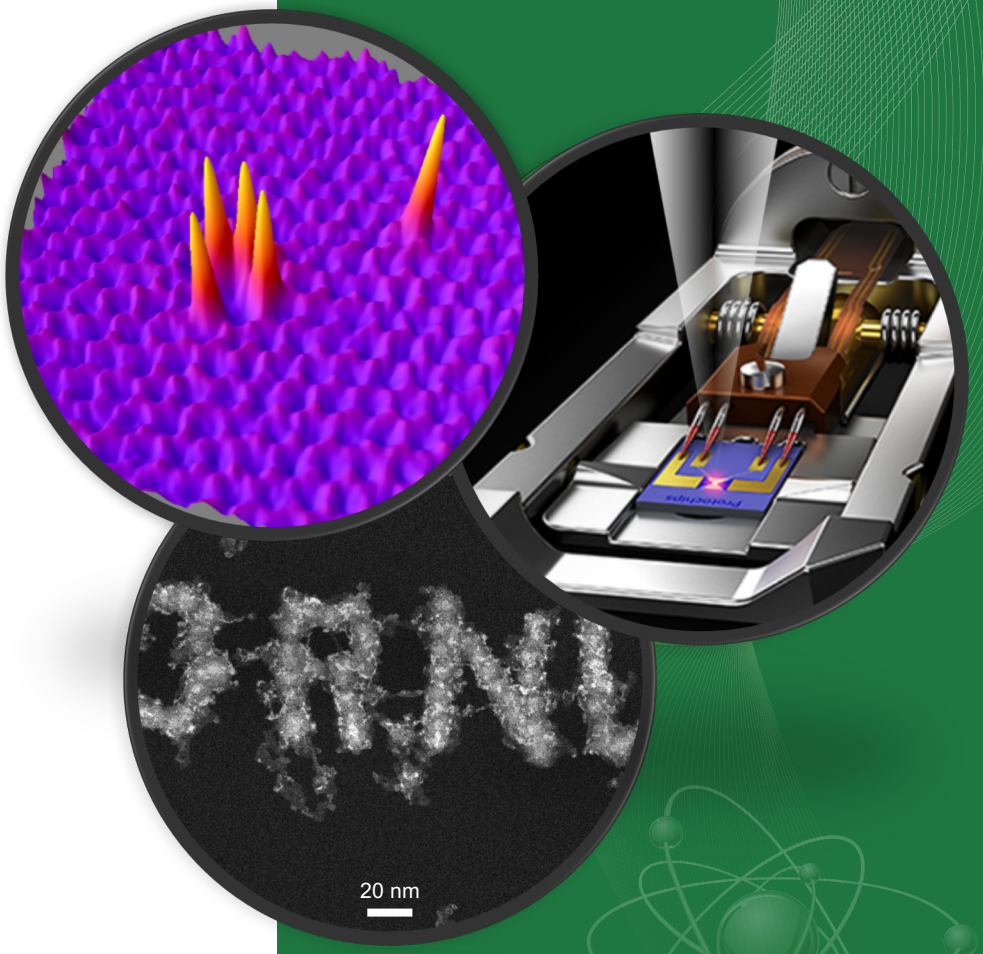
AI and ML in Microscopy: A Material Opportunity



Sergei V. Kalinin

The Center for Nanophase Materials Sciences (CNMS),
Oak Ridge National Laboratory

October 17, 2019

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SCANNING PROBE MICROSCOPY
Electrical and Electromechanical Phenomena at the Nanoscale
Sergei Kalinin
Alexei Gruverman
EDITORS
VOLUME 1
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Sergei V. Kalinin
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Scanning Probe Microscopy of Functional Materials
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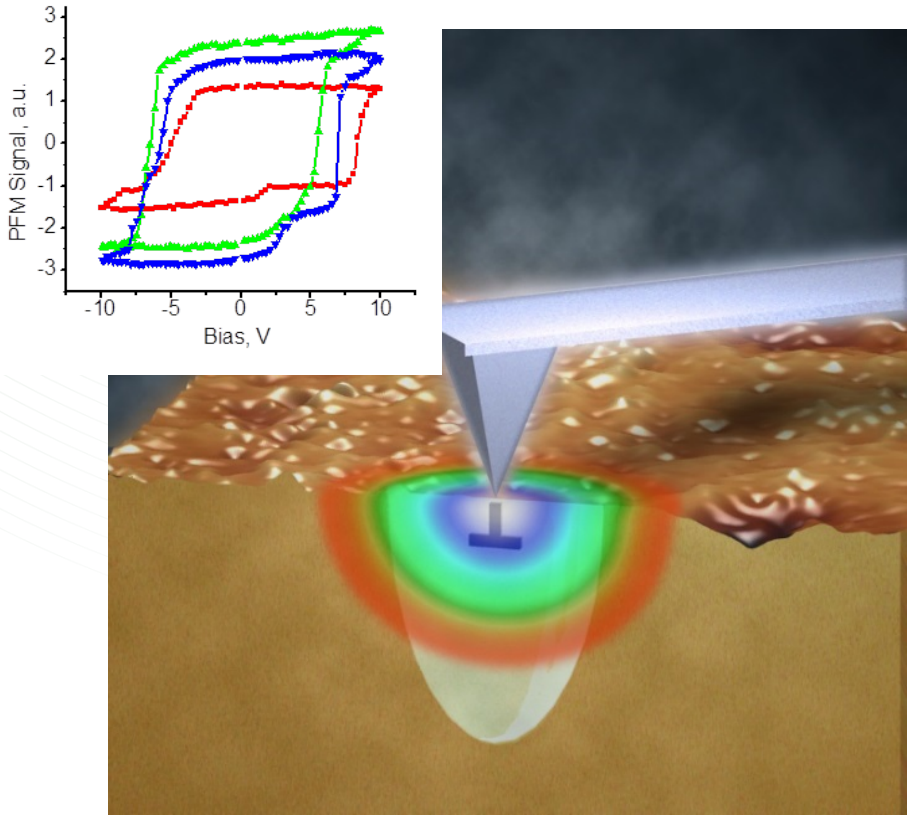
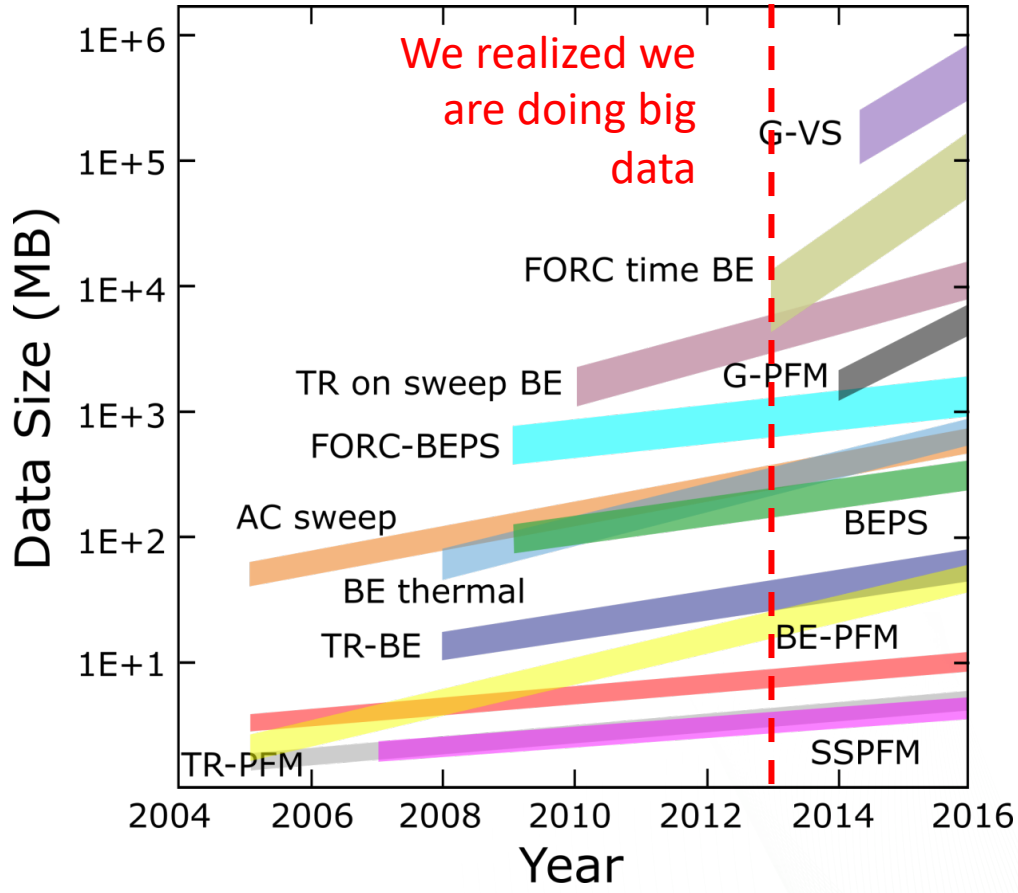
Multidimensional SPM modes





Physics perspective:

1. Local stimulus: spectroscopic (3D) SPM modes
2. Phase transitions are hysteretic: First Order Reversal Curves
3. Phase transitions can be rate-controlled

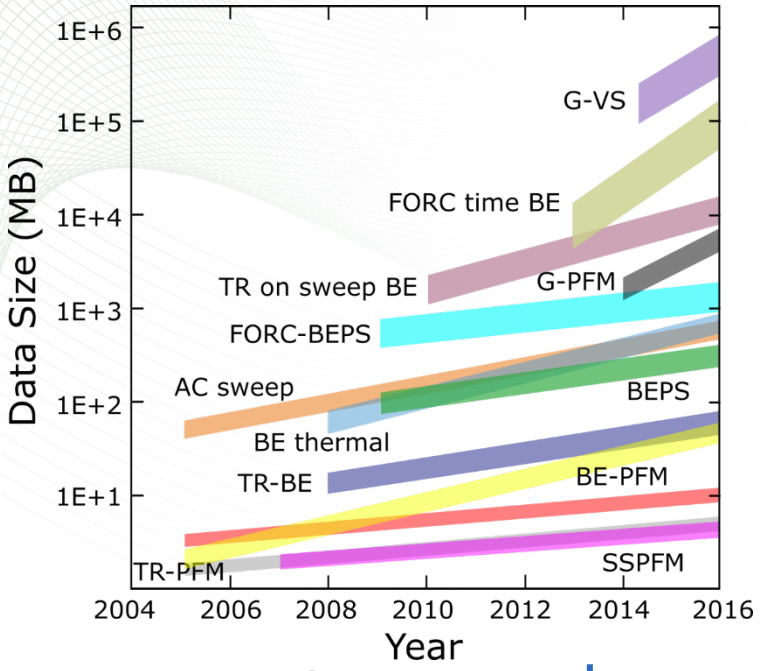
SPM Perspective:

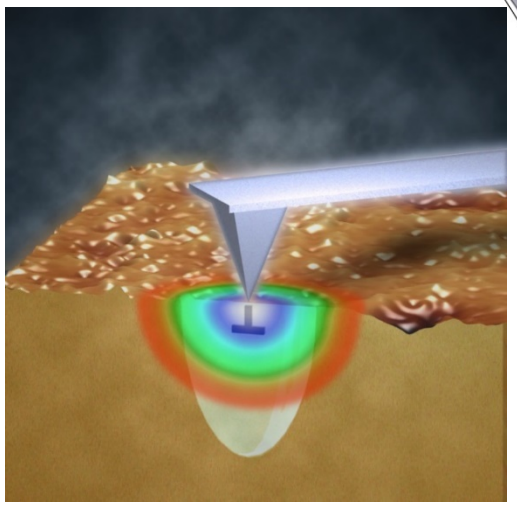
4. SPM requires resonance enhancement (frequency domain)

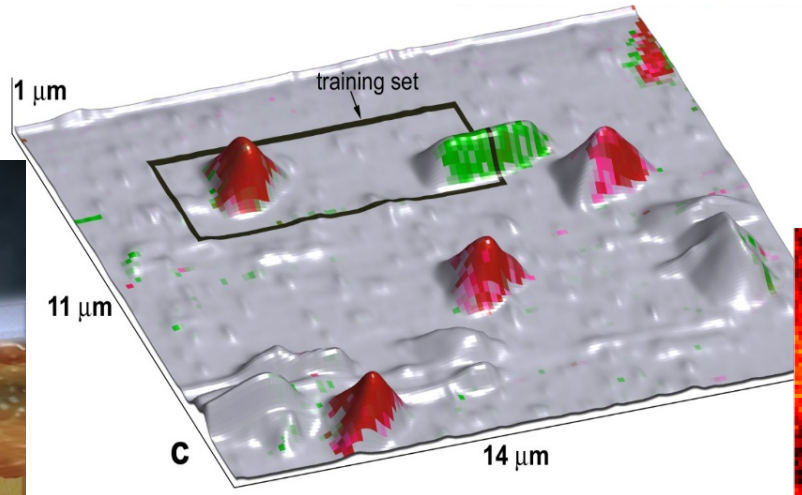
Instrumental limit: photodetector bandwidth (~10 MHz) x DAQ performance (32 Bit)

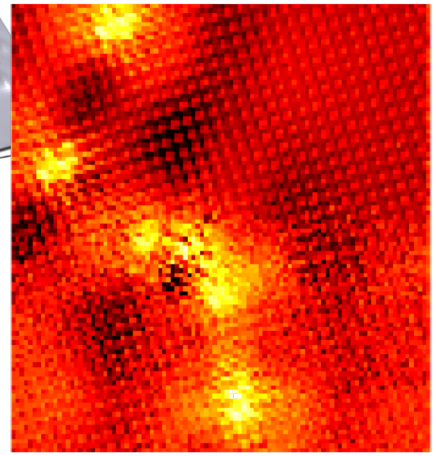
- **Single frequency/heterodyne:** lock-in compression to ~ 1 kHz
- **Band excitation:** 10^2 bins at ~ 1 kHz = 100 kHz
- **G-mode:** full streaming at ~10 MHz

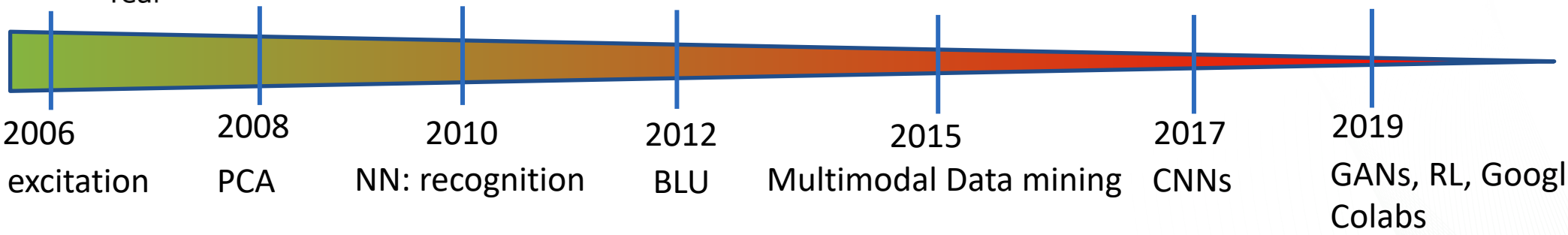
We have the data… We need to do something with it!





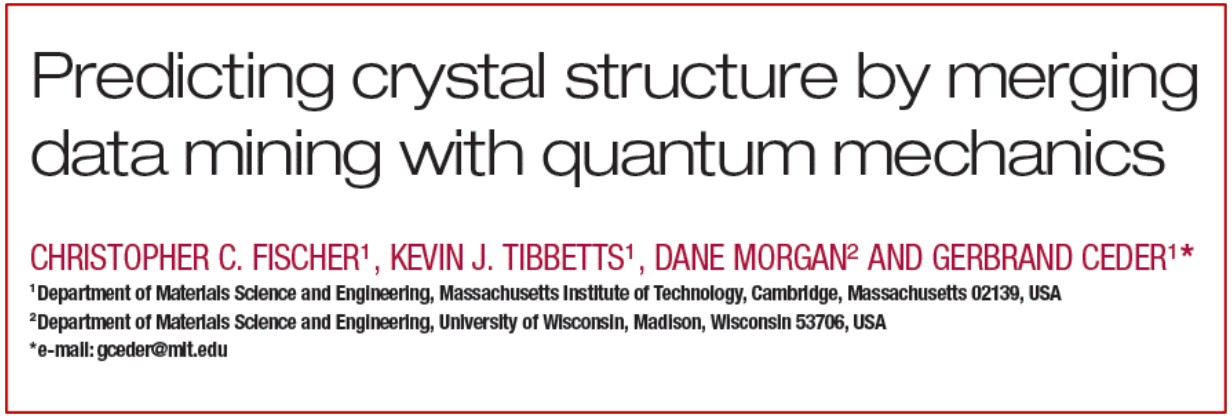






M.P. NIKIFOROV, A.A. VERTEGEL, V.V. REUKOV, G.L. THOMPSON, S.V. KALININ, and S. JESSE, *Functional recognition imaging using artificial neural networks: Applications to rapid cellular identification by broadband electromechanical response*, Nanotechnology **20**, 405708 (2009).

Opportunities in Materials Science



Predicting crystal structure by merging data mining with quantum mechanics

CHRISTOPHER C. FISCHER[1], KEVIN J. TIBBETTS[1], DANE MORGAN[2] AND GERBRAND CEDER[1]*
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[2]Department of Materials Science and Engineering, University of Wisconsin, Madison, Wisconsin 53706, USA
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- **"Improve":** Renewable energy, self-driving cars, transparent displays, new memory technologies
- **"Discover":** Room temperature superconductivity, high mechanical stress materials
- **"Engineer":** Quantum computing, single-atom catalysts, biomolecules

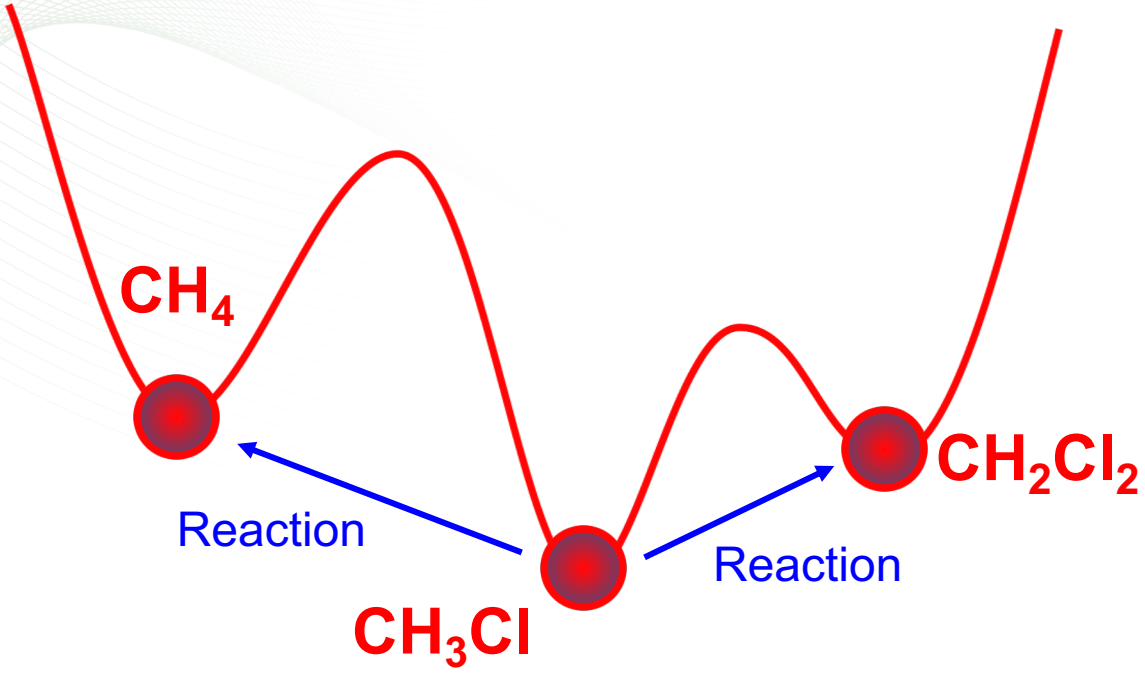
Functionality, manufacturability, cost

“New directions in science are launched by new tools much more often than by new concepts. The effect of a concept-driven revolution is to explain old things in new ways. The effect of a tool-driven revolution is to discover new things that have to be explained.”

Freeman Dyson

Materials in the Chemical Space

- Full atomic coordinate space: un-tractable
- Chemical space: minima corresponding to (meta)stable compounds



- Materials and molecules are points in chemical space
- Finding "the right" material is then a search/optimization problem.
- Machine learning is great at this. Right?

- Chemical space is non-differentiable
- The pathways between different regions are non-obvious
- "Useful" functionalities can be very complex and poorly understood
  - We can calculate bandgaps and ideal Young moduli
  - Biological activities and superconductivity, not so much

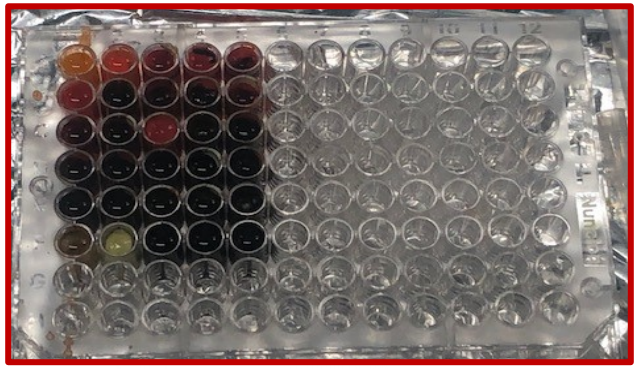
We typically need outliers!

But:

- There are underlying physical laws that determine what is possible

Molecular Systems:

Chematica (B. Grzbowski)



M. Ahmadi

M. Ceriotti

Opentrons

- Chemical space is a graph: 10^{63} (only less then 30 atoms)
- Edges (reactions) can be optimized individually
- Amenable to the literature mining (*Chematica*)
- Area of known compounds can be expanded via retrosynthesis
- Amenable to laboratory robotics

Biology took 10^{9} years for search: MCMC with ~10^{20} steps and ~$10^{35\text{-}40}$ chains

But: functionalities at the nodes are defined within the context (CO_2 adsorption, water desalination, biological functionality)

Solid Materials

S. Curtarolo

Let's think about it as a search problem:

- **Alloying:** need maintain composition ~1%
- **Doping:** need maintain composition ~ 10^{-6}
- Grid search is out for D > 3 (experiment)
- MCMC type problem: how do we make it work?

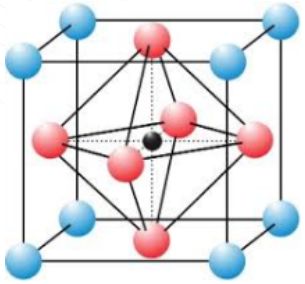
Discovery of copper HTSC, MgB_2, iron pnictides. Serendipitous discovery followed by exploring large families. Could not predict – no theory

The Solid Problem

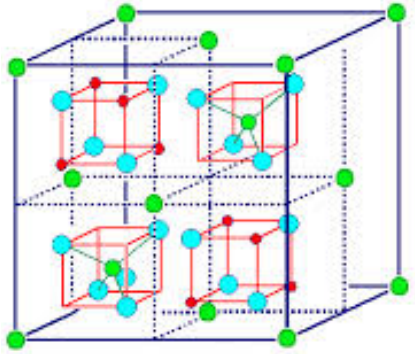
Chemical space is heavily degenerate: can use mean field descriptors

- *Symmetry,*
- *Concentrations,*
- *Order parameters*
- *Thermodynamic potentials*

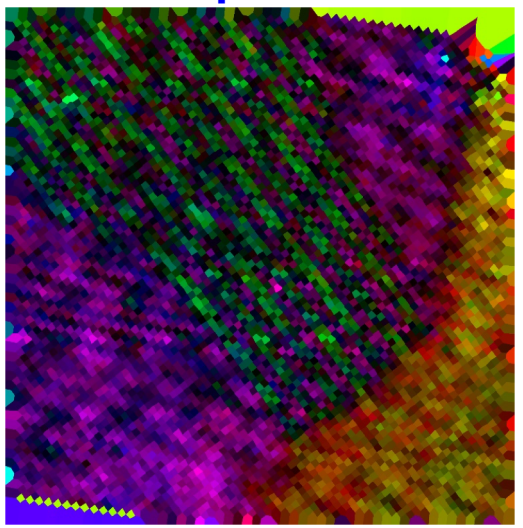
Simple unit cell



Complex unit cell: partial occupancies

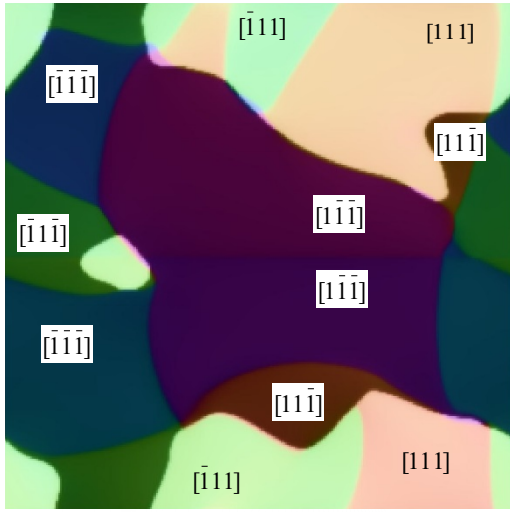


Nanoscale phase separation



C. Nelson

Mesoscale: microstructures



L.Q. Chen

- Really interesting materials are those when it is not the case (relaxors, Kitaev materials) – some correlations and disorder in ground state.
- Many physical properties cannot be predicted. Bandgaps ok, superconductivity not so much
- Large scale organization: defects, microstructures, etc.

How do we start describing structures of solids beyond symmetry-based methods, establish structure-property relationships, and use this information for prediction of materials and discovery of synthesis pathways?

More then atoms

Hasten high resolution

Build precision microscopes to map atoms, say **Stephen J. Pennycook** and **Sergei V. Kalinin**.

Nature 515, 487 (2014)

J.J. Guo et al.,
Nat. Comm. 5, 5389 (2014)

Atomic positions can be determined to <10-pm precision	Bond length: Chemical reactivity, catalytic activity	Bond angles: Magnetism and transport	Configurations and repeating elements?

The Lab on a Beam

Image atomic columns

Spectra from single atoms

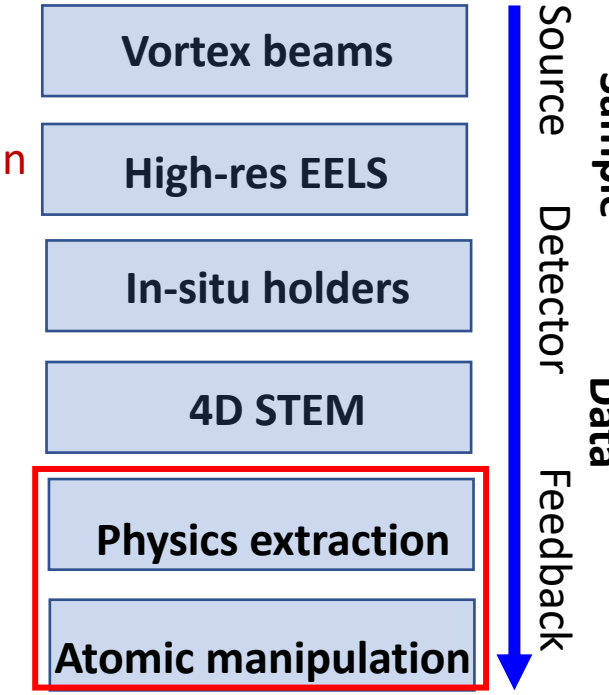
Image light atoms and sensitive materials

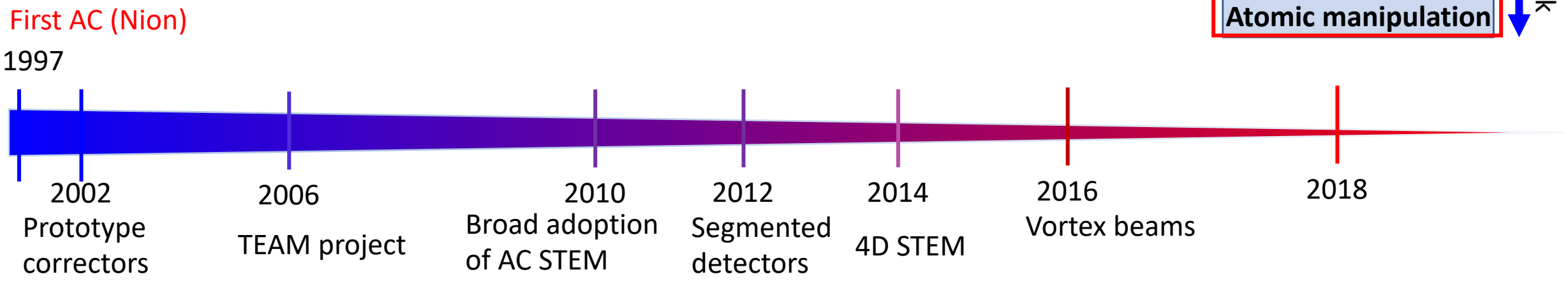
Diffraction from subatomic volumes

Beams with orbital momentum

Beam manipulation

0.61 A resolution





Challenge 1:
Data Infrastructure

Standard image: ~10 MB
Dynamic data: ~ 100 MB/S
4D STEM: ~TB data (limited by the availability of data infrastructure)
Potentially: Large Hadron Collider level data flows (from single microscope)

Physics from STEM and SPM data

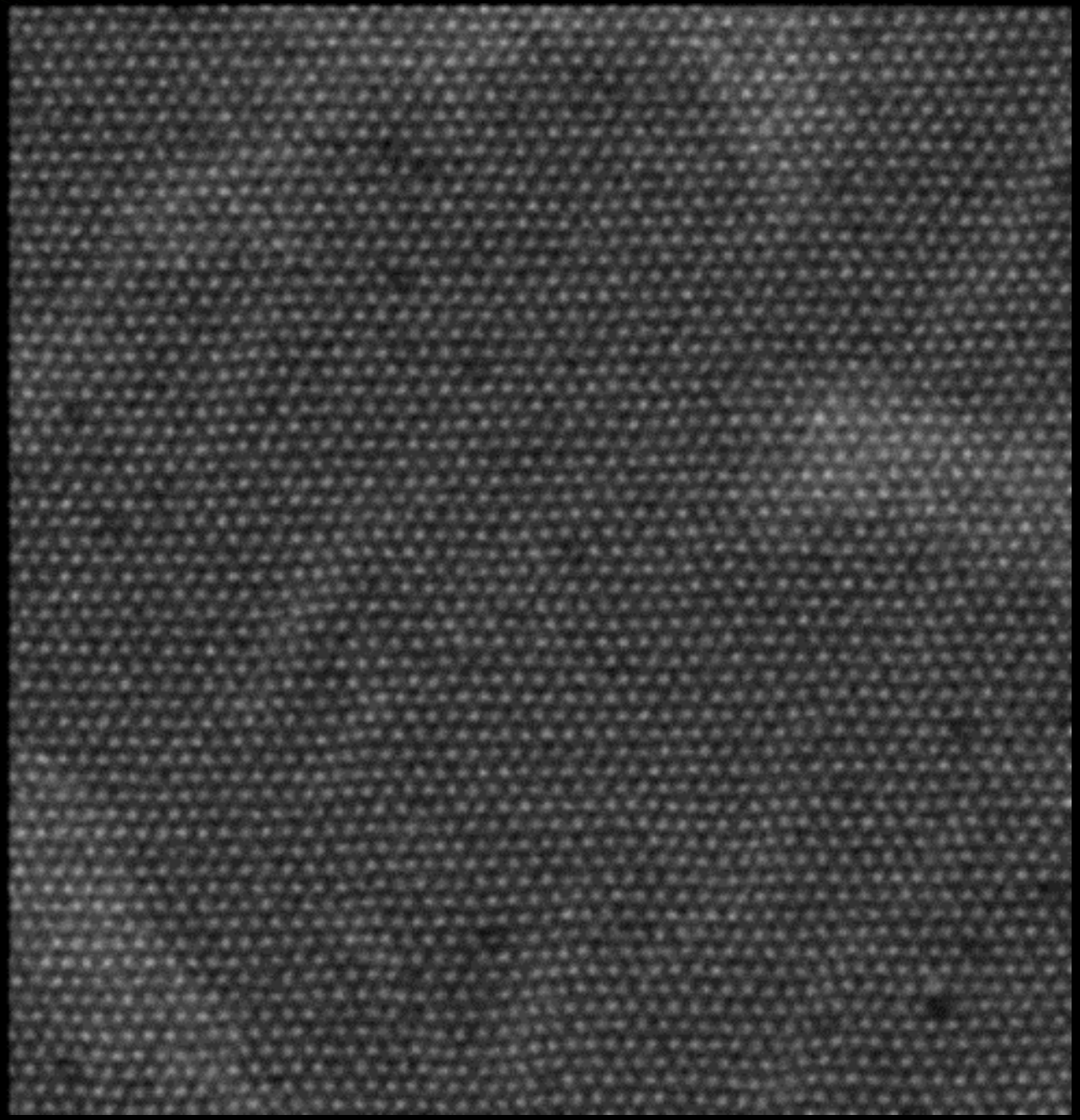
- **Can we get materials specific information** (e.g. atomic coordinates from STEM, scattering potentials from 4D STEM, etc.) **from microscopy data**, at which level of confidence, and how this knowledge is affected and can be improved from knowledge of imaging system (e.g. classical beam parameters, resolution function, all the way to full imaging system modelling), and knowledge of material.

- **Can we use the materials-specific information** with uncertainties determined by incomplete knowledge of imaging system or intrinsic limitations **to infer physics and chemistry, either via correlative models or recovery of generative physics** (force fields, exchange integrals, etc.)

- **Can we use thus determined materials information**, either correlative or causative, **to reconstruct materials behaviors** (phase diagrams, etc) in the broader parameter space (e.g. for temperatures and concentrations different for specific sample studied), and determine how reliability of such prediction depends on position in parameter space.

- Can we harness the data stream from the microscope to engender real time feedback, e.g. for autonomous experimentation and atomic manipulation

Dynamic Atomic Changes



Observations of atomic dynamics induced by beam (or temperature, field, etc.) gives information on multiple atomic configurations as they form and evolve. Can we learn:

- Effective interaction parameters (e-ph coupling)?
- Force fields?
- Phase transition dynamics

Deep learning for atomically resolved images

Top 3 predictions

Staffordshire bullterrier 43 %

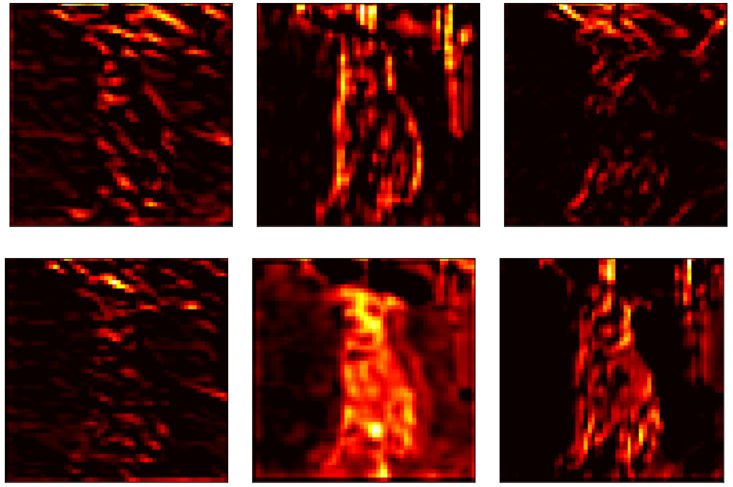
Very close

American pit bull terrier 23 %

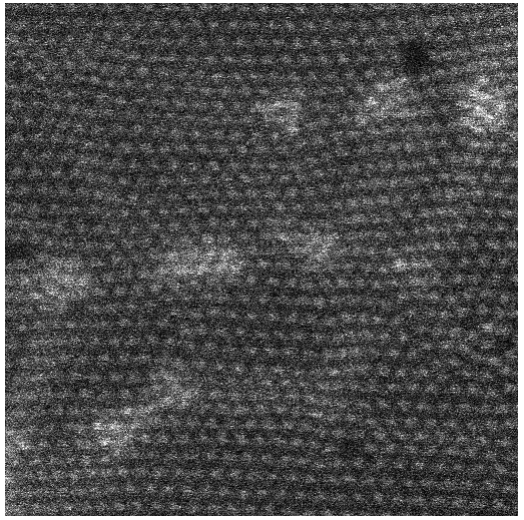
Basenji 11 %

Meet Duffy: Pitbull-Shepard-Collie mix

Randomly selected feature maps



STEM of WS2



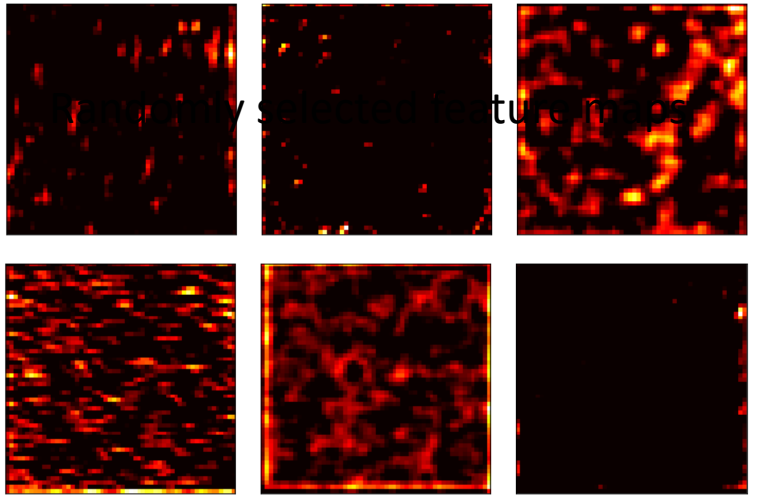
Top 3 predictions

Wool 6.3 %

Velvet 5.5 %

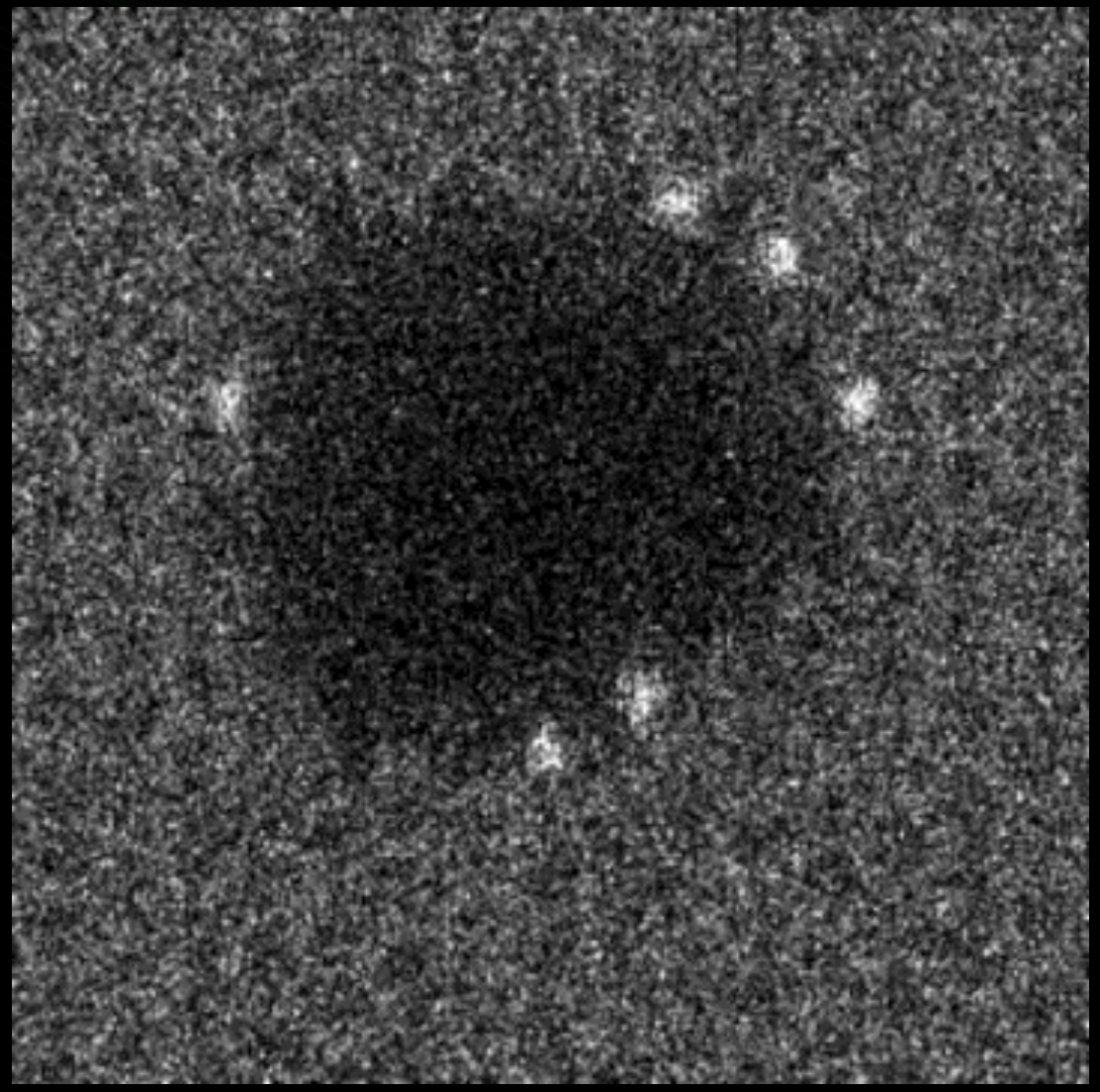
Window screen 3.8 %

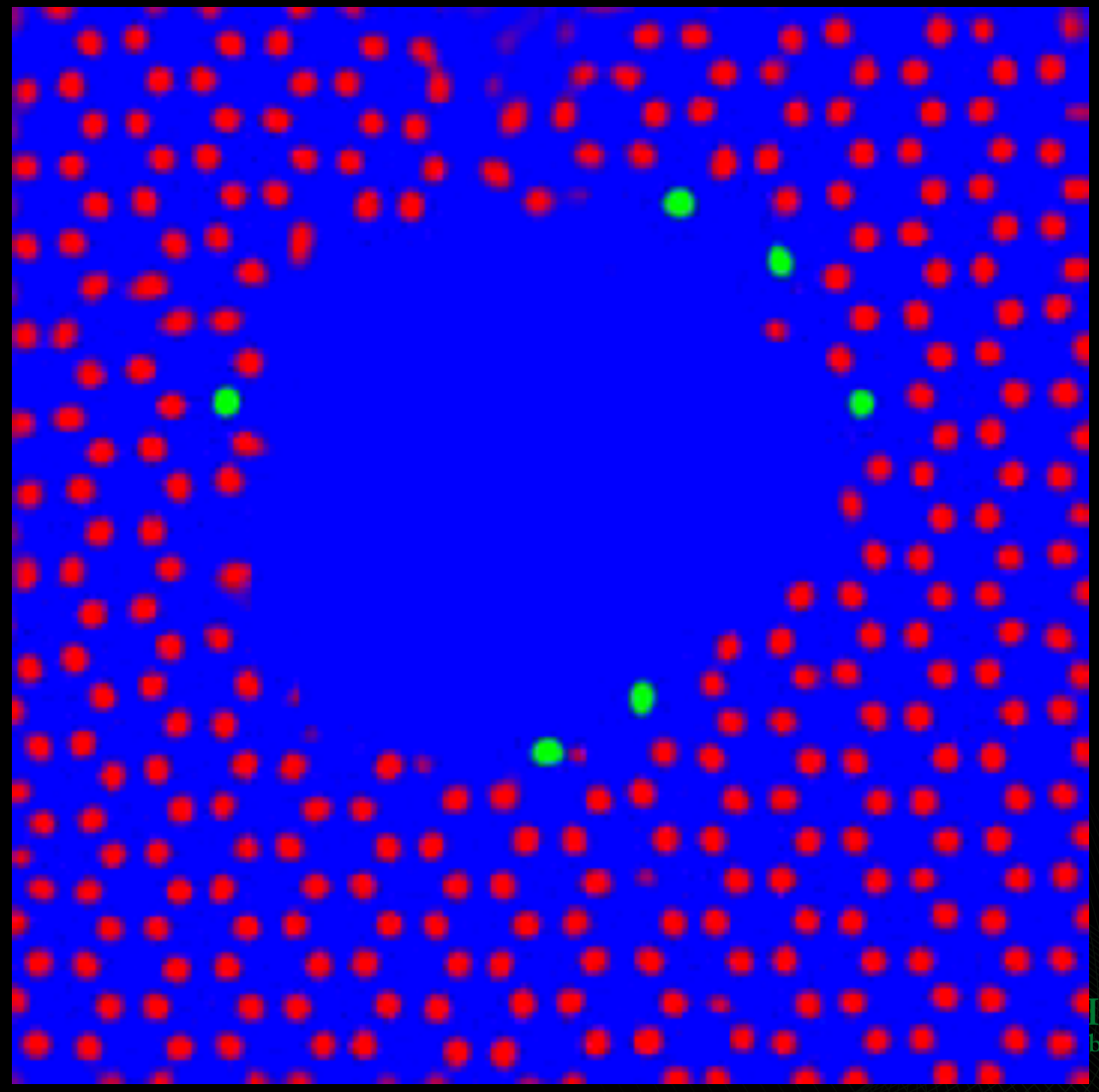
Randomly selected feature maps



Deep learning works like a charm for:

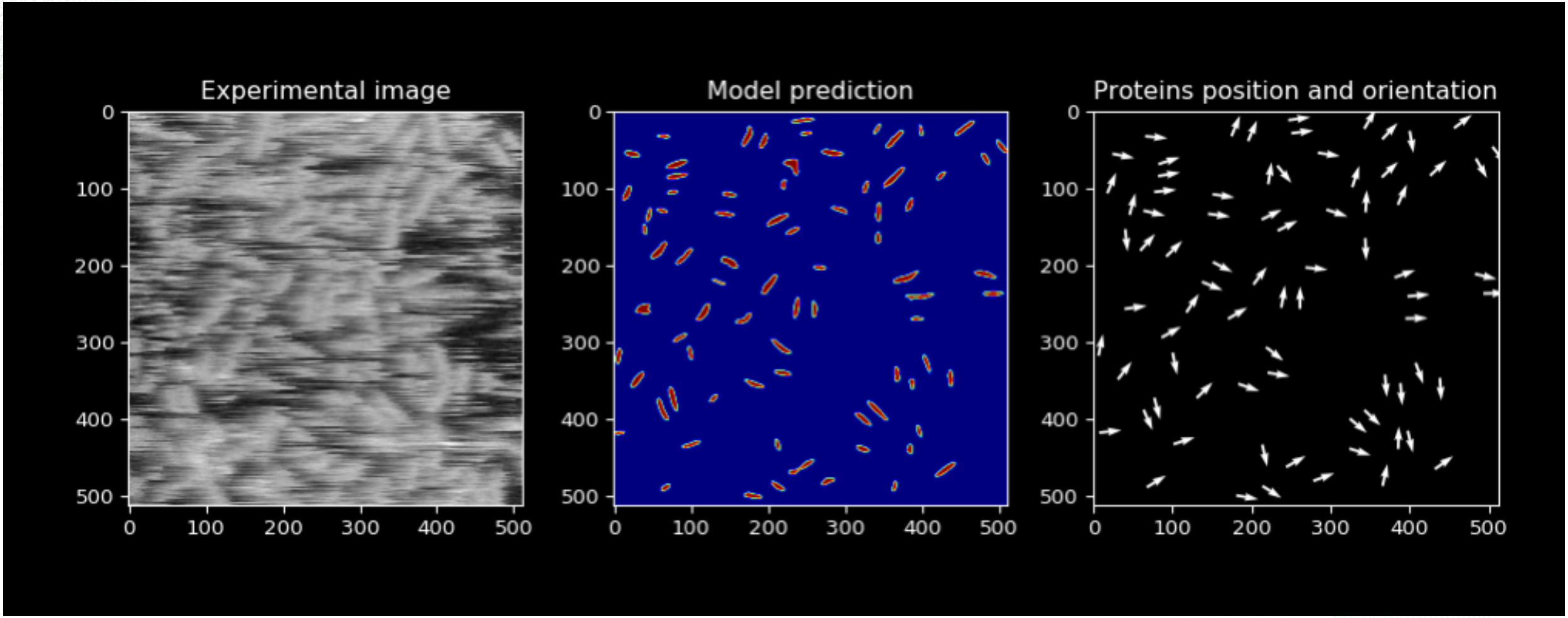
- Drift correction
- Denoising
- Data processing/dimensionality reduction
- Feature finding (physics is in the training set)





Deep learning in AFM

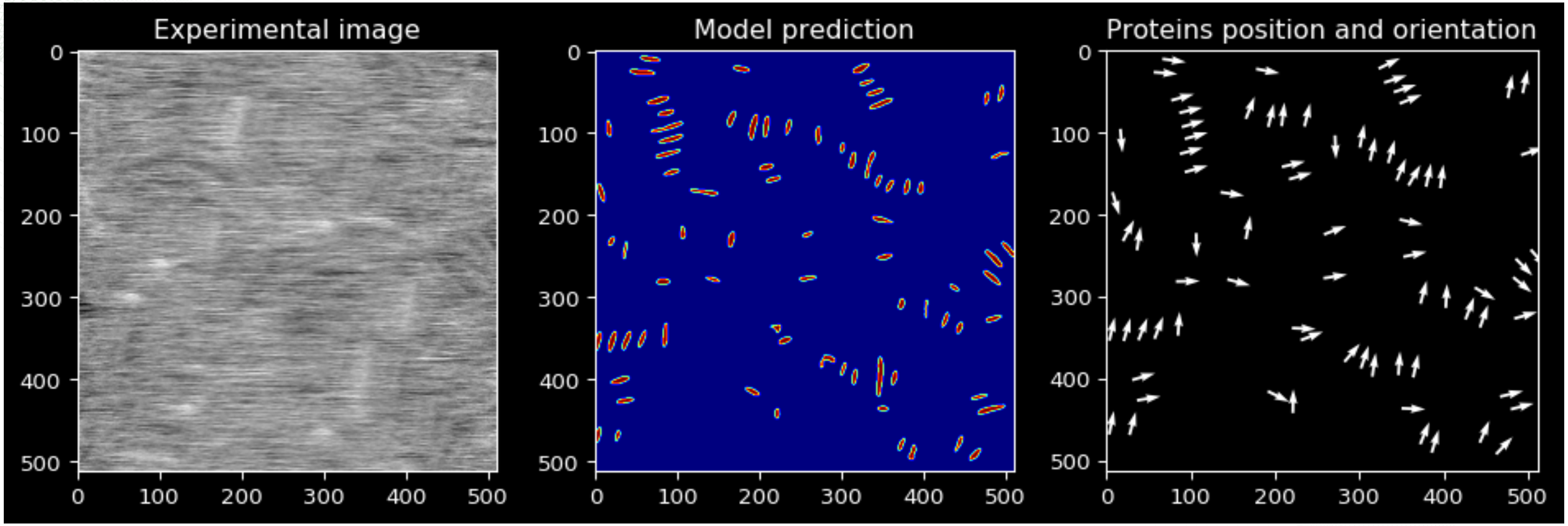
Model trained on a single movie frame from the well-ordered phase and applied to the entire movie



Maxim Ziatdinov, Xin Li, Shuai Zhang, Harley Pyles, David Baker, James J. De Yoreo, Sergei V. Kalinin

Deep learning in AFM

Model trained on a single movie frame from the well-ordered phase and applied to the entire movie



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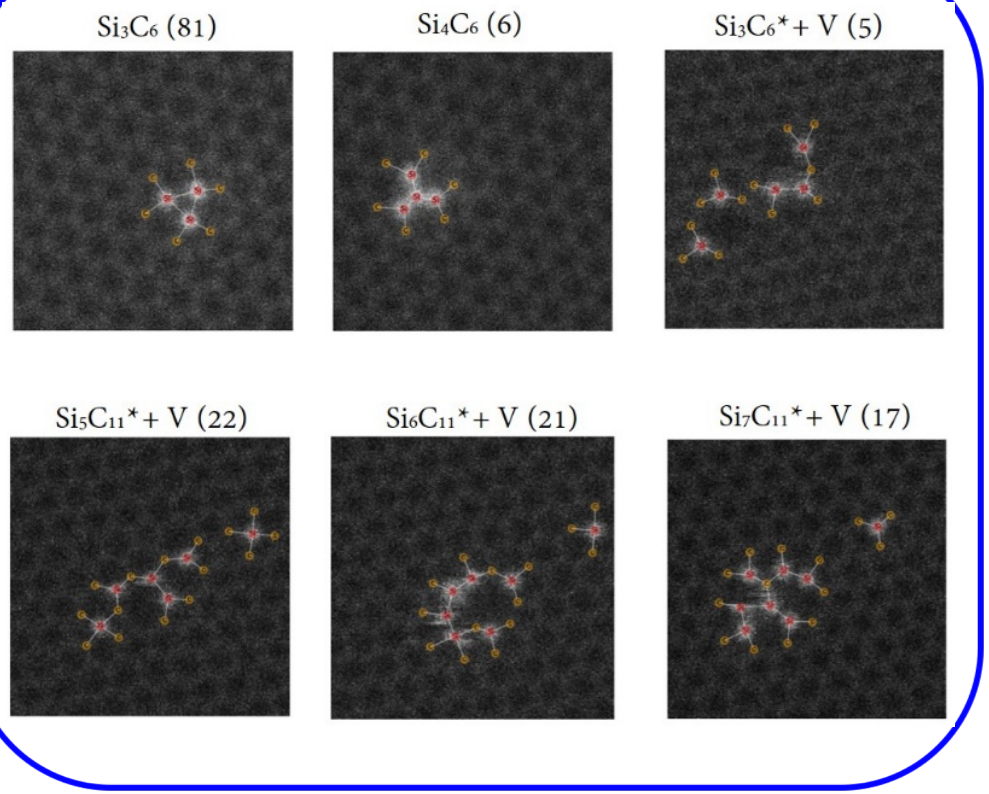
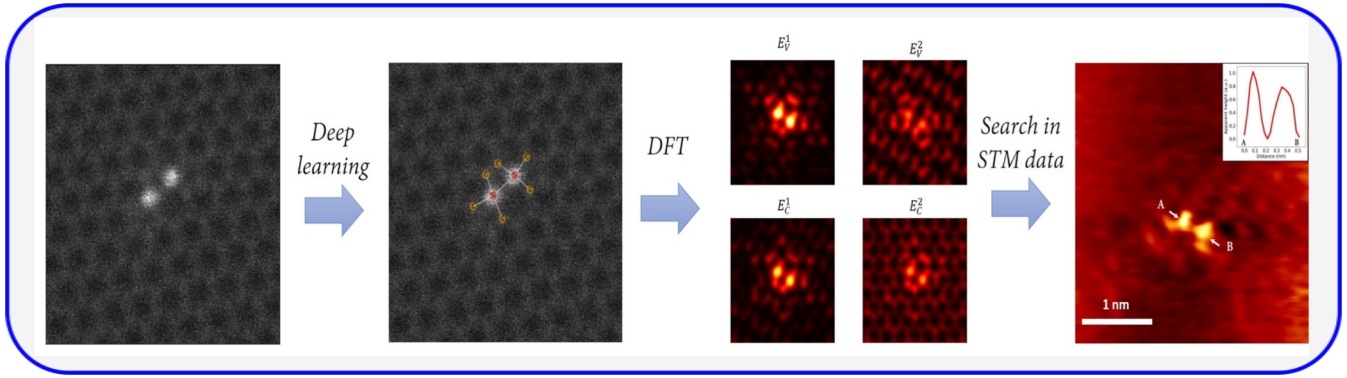
Defect Libraries

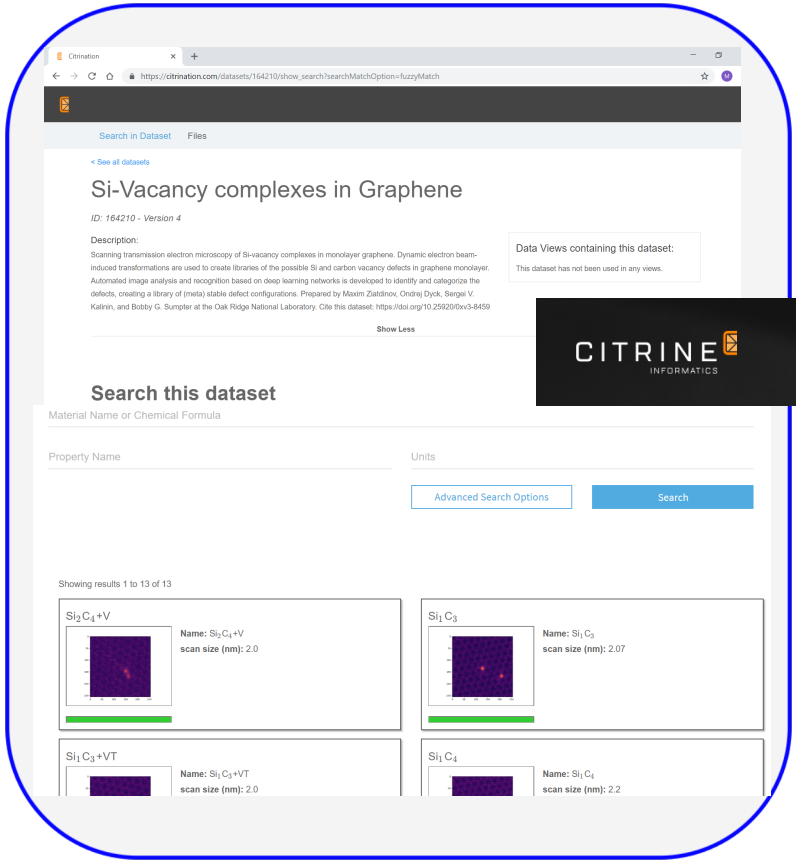


- Identify the “real” defects
- Theory to get energies, strains, etc

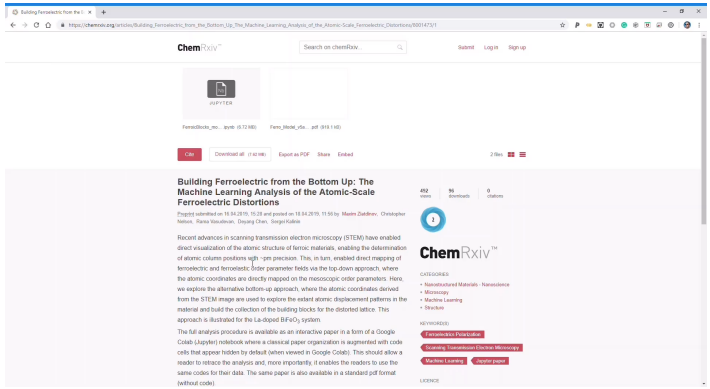
Analysis workflows



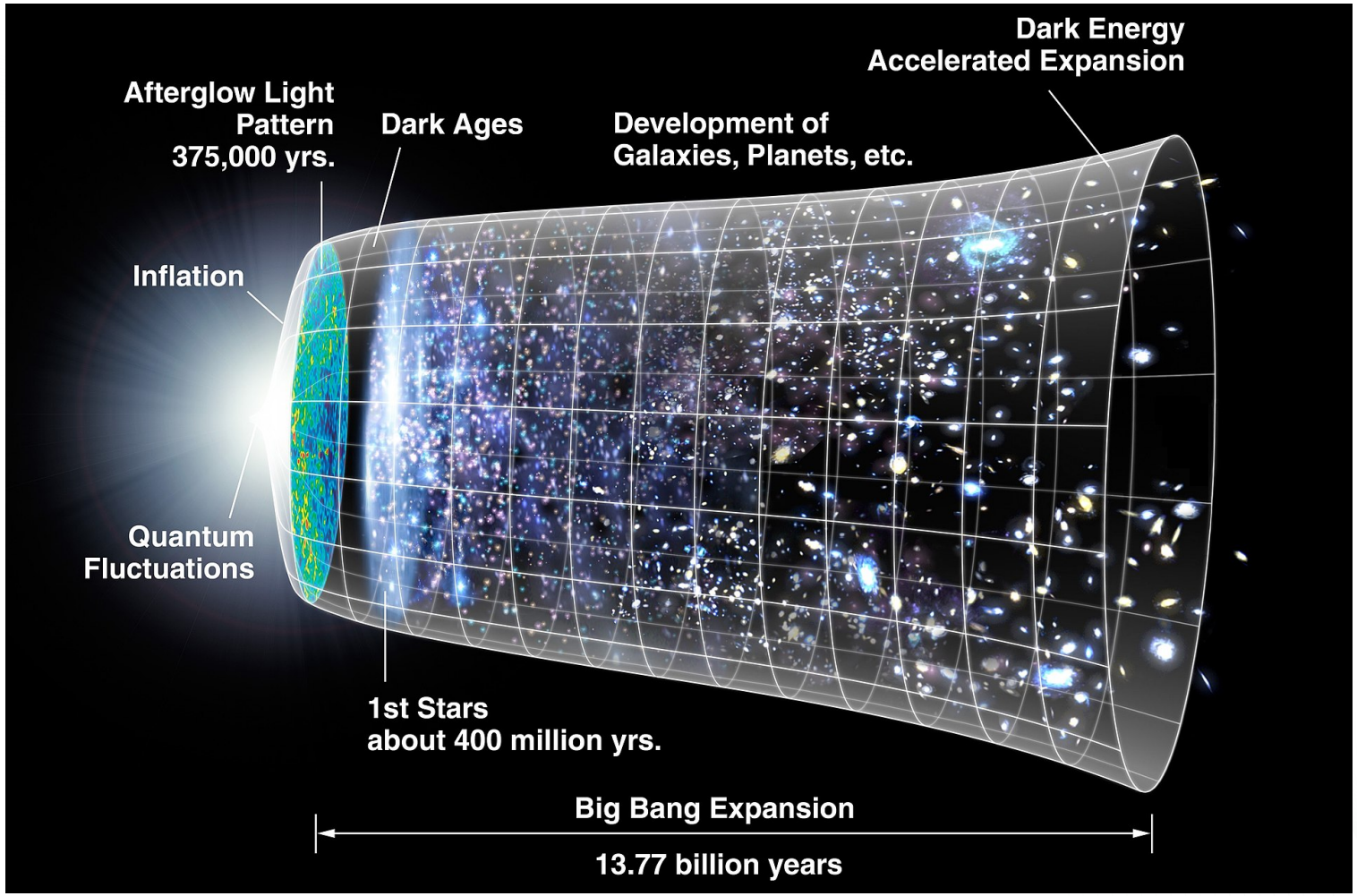
Open data



Open code: Jupyter papers

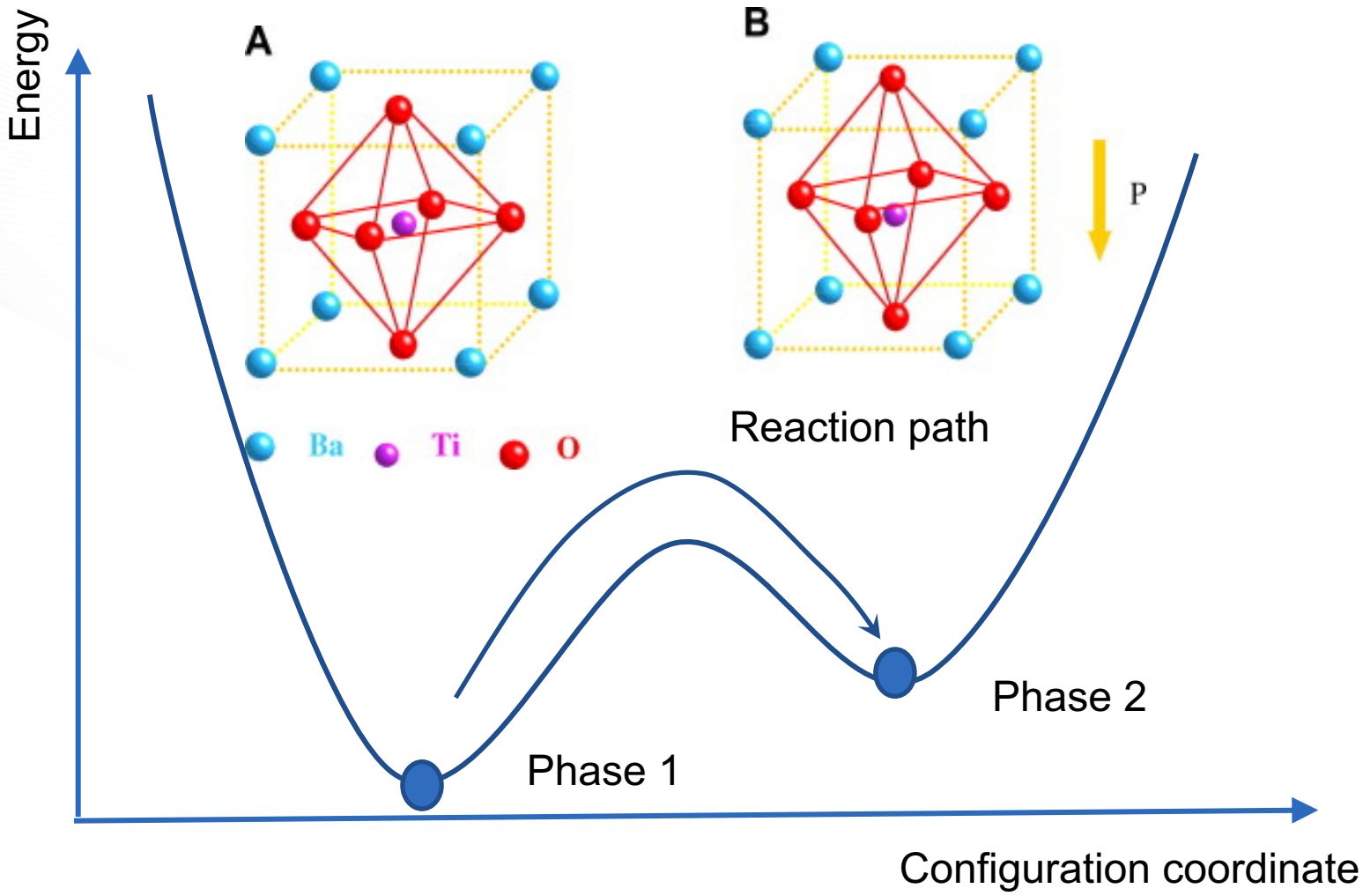


From Correlation to Causation:



Wikipedia, NASA

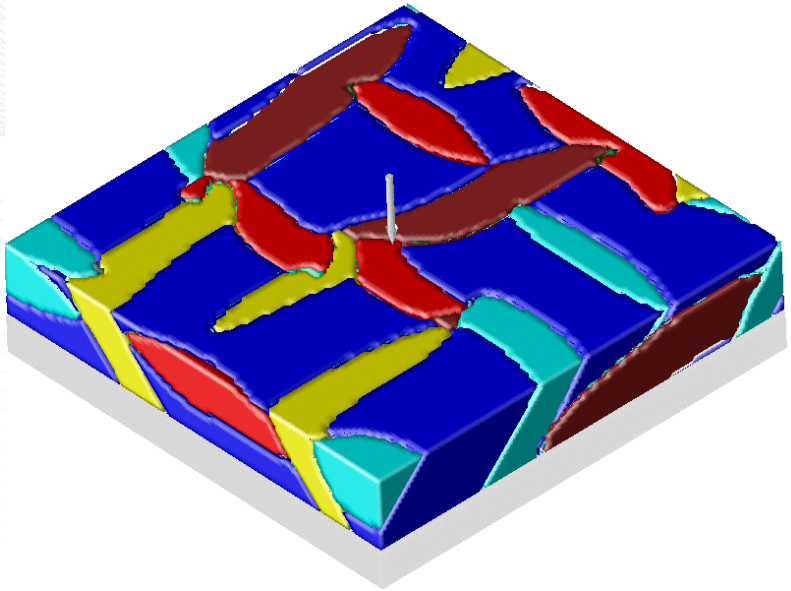
- Problems can be intractable combinatorically, but have simple constitutive laws
- Low dimensional non-linear manifold in the very high dimensional linear spaces
- Observational data: astronomy



Problems:

- There are $10^{(10 - 23)}$ degrees of freedom
- Which (in most materials) correspond to very small number of collective variables (order parameters)
- Naturally, materials where this is likely not the case are really interesting

Physics and chemistry from structural STEM data?



LGD equation:

$$F = \int_V \left[f_{bulk}(P_i) + f_{grad}(P_{i,j}) + f_{elas}(P_i, \varepsilon_{kl}) + f_{elec}(P_i, E_i) \right] dV$$

LGD equation:

$$\frac{dP_i}{dt} = -L \frac{\delta F}{\delta P_i}$$

Conventional fixed φ b.c.

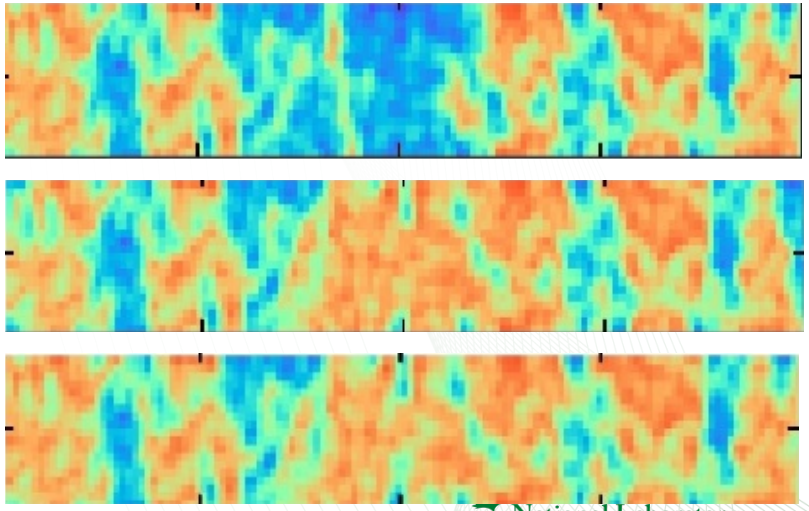
$$\phi\big|_{z=L+\lambda} = V_{\text{planar}}$$

$$\left.\frac{\partial P_z}{\partial z}\right|_{z=0,L+\lambda} = 0$$

On mesoscale, materials functionalities can often be described via order parameter fields:

- Mesoscopic order parameter is often known from macroscopic measurements
- What are the boundary conditions at surfaces and interfaces
- What are the roles of defects?
- Can we describe spatially inhomogeneous states (relaxors, charge ordered materials, MPB systems)?

Disordered states

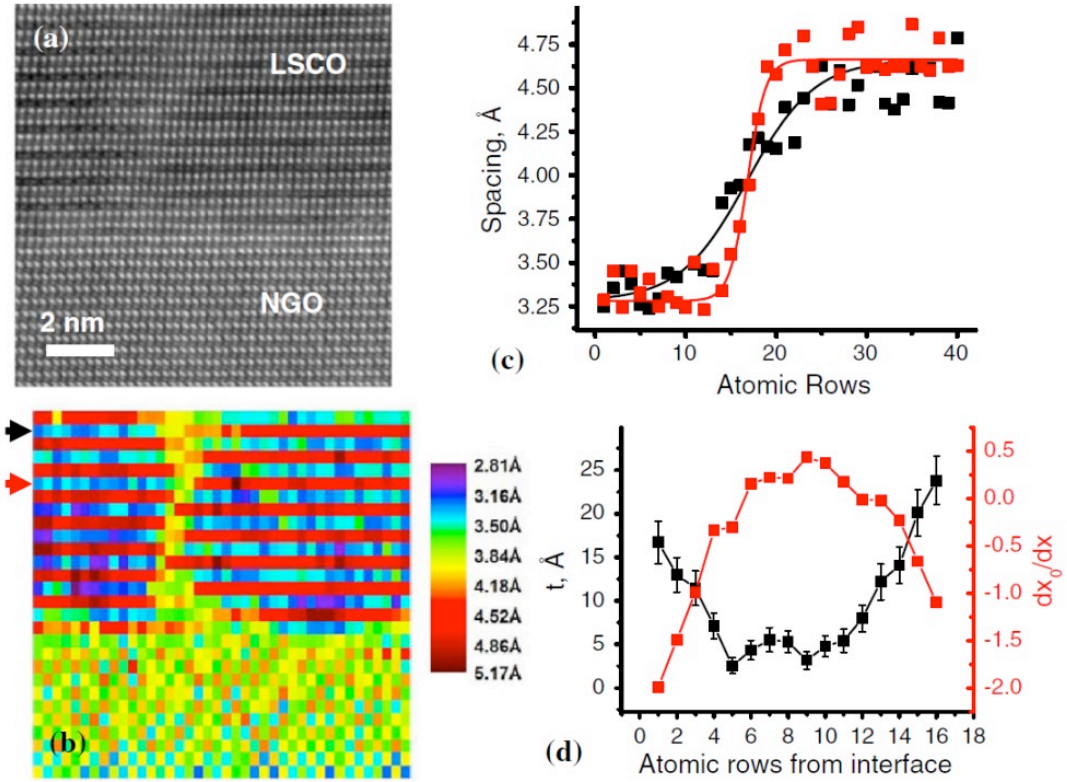


Building the mesoscopic picture top down

Model System: Lanthanum-strontium cobaltite with topological defects and interfaces

Antiphase domain boundary

$$\eta(x, z) \approx \eta_{SD}(z) \tanh\left(\frac{x - x_0}{L_C(z)}\right)$$

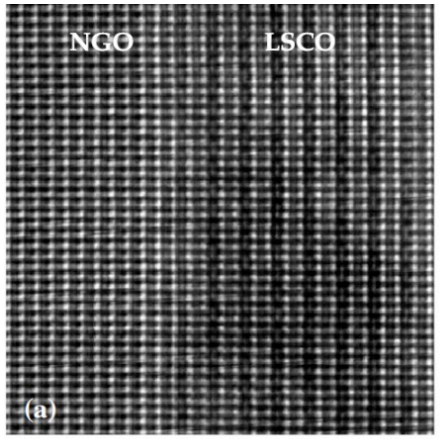


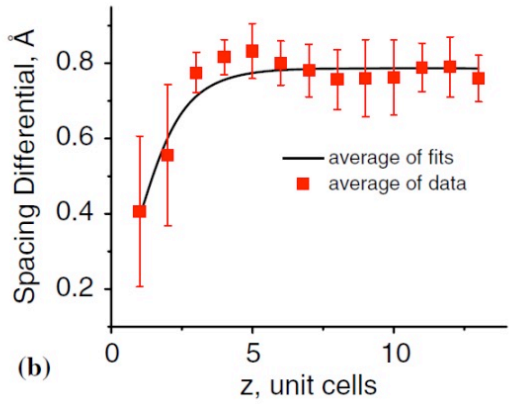
Fitting the experimentally observed atomic profiles to the functional form of order parameter

$$\eta_{SD}(z) \approx \eta_b\left(1 - \frac{1}{1 + \sqrt{2}\lambda / L_C(0)} \exp\left(-\frac{\sqrt{2}z}{L_C(0)}\right)\right)$$

Ordering behavior at the interfaces

$$\eta(z) = 0.787(1 - 0.5\exp(-\tfrac{z-1}{1.1}))$$





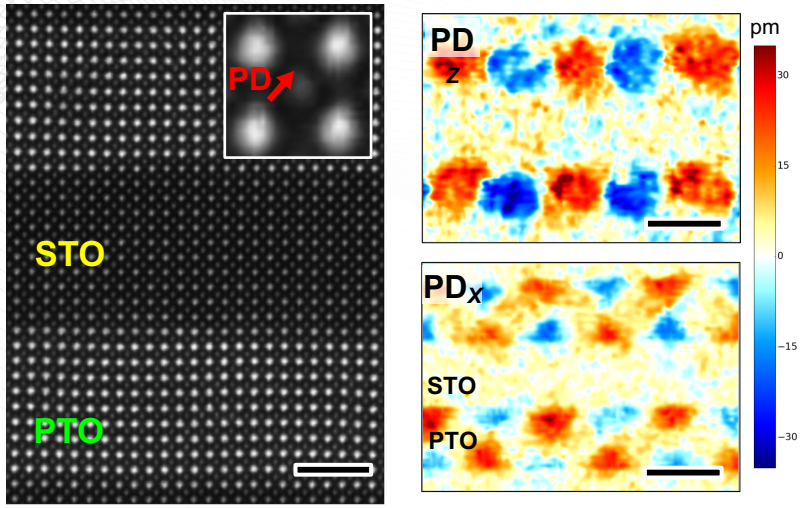
Allows to analyze the interplay between ordering, chemical composition, and mechanical effects at domain walls, interfaces and structural defects

A.Y. Borisevich *et al.*, *PRL* **109**, 065702 (2012)

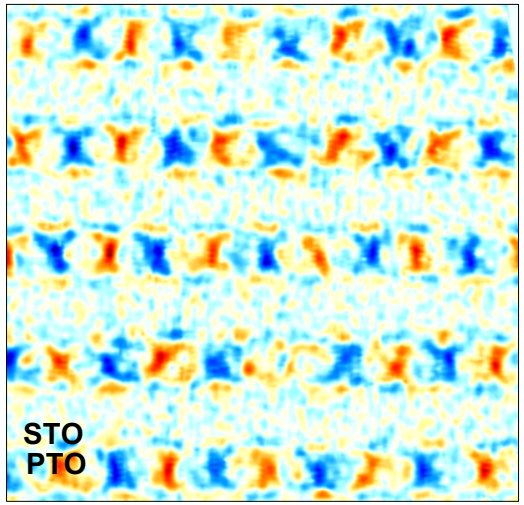
OAK RIDGE National Laboratory

Flexoelectricity by Computer vision

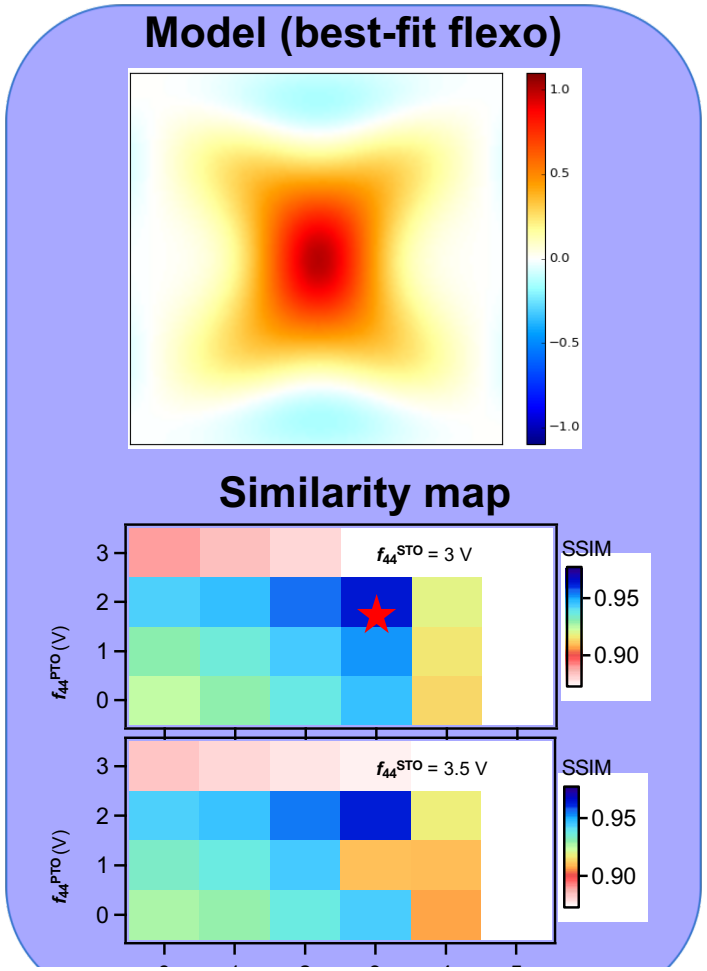
HAADF-STEM images



Vorticity/polar gradient



Theory-experiment matching

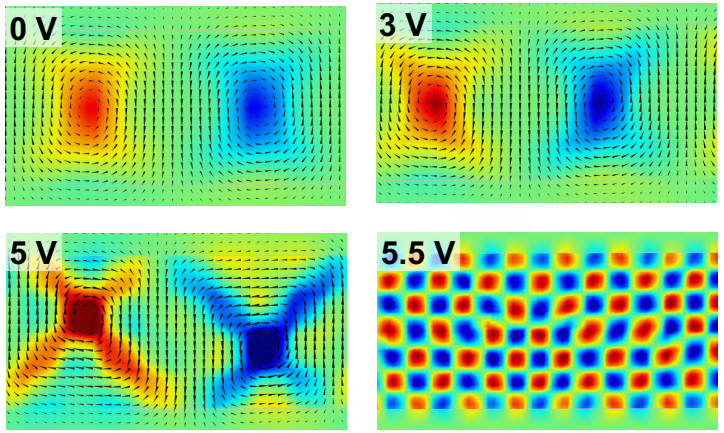


Effect of flexoelectricity

$$F = \alpha_{ij}P_iP_j + \alpha_{ijkl}P_iP_jP_kP_l + \alpha_{ijklmn}P_iP_jP_kP_lP_mP_n + \frac{1}{2}g_{ijkl}P_{i,j}P_{k,l} +$$

$$\frac{1}{2}c_{ijkl}\varepsilon_{ij}\varepsilon_{kl} - q_{ijkl}\varepsilon_{ij}P_kP_l - \frac{1}{2}\kappa_0 E_iE_i - E_iP_i + f_{ijkl}(P_{k,l}\varepsilon_{ij} - \varepsilon_{ij,l}P_k)$$

Longitudinal/transverse flexocoupling f_{11}/f_{12} ($f_{11} \sim -f_{12}$)



The flexocoupling in both PTO and STO layers is considered, revealing different modulation effects.

Li et al., Nat. Comm. 2017

- Polarization and tilt behavior at interfaces and topological defects: gradient terms and physical BCs
- Coupling with (electro)chemical boundary conditions
- Defect effects: transition from localized perturbation to collective responses

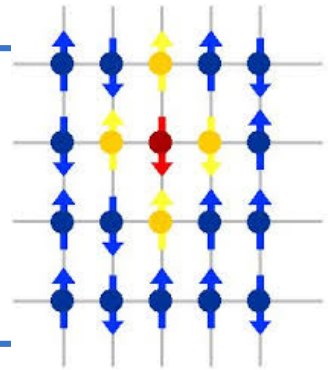
Physics from Microscopic Degrees of Freedom

Macroscopic observables:
- Property
- Average structure
- fluctuations

Macroscopic measurements
Scattering data

Can ML do it?
(Melko 2017)

Microscopic models
- Ising model
- Heisenberg model
- Kitaev model
- ….



Can ML do it?

Real material

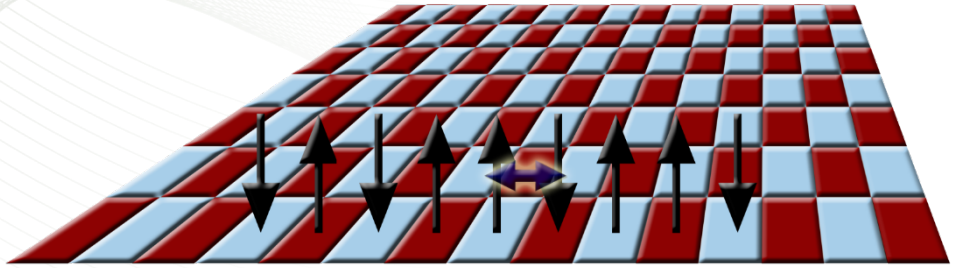
A. Sefat

On atomistic scale, we often use lattice Hamiltonian models:

- Can we determine local interactions from STEM or SPM data
- What if some information is lost (1 << observables << degrees of freedom)

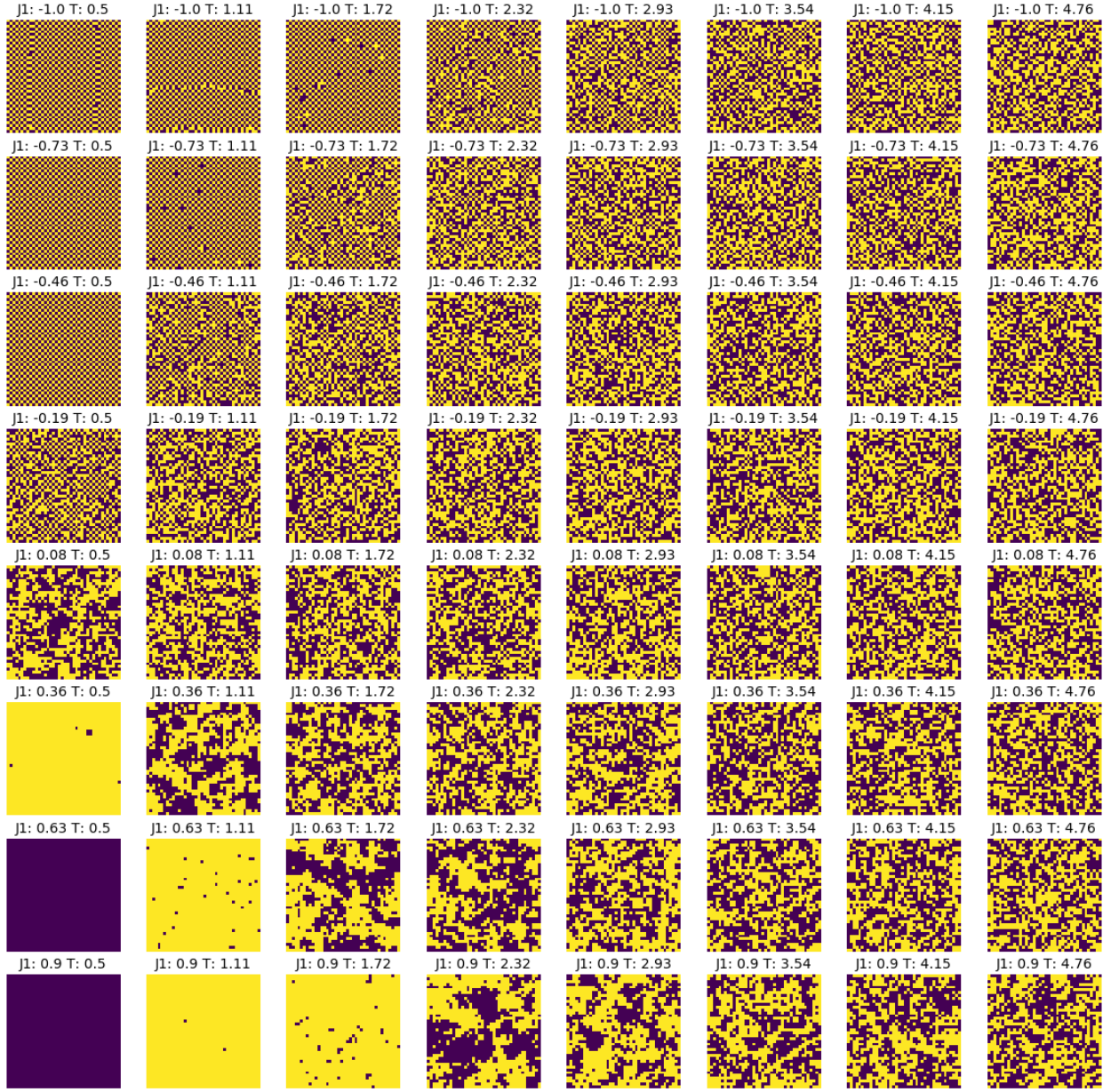
Can we study physics and chemistry locally?

Ising model



$$\mathrm{H}(H) = \sum_{i.j} J_{ij} S_i S_j + \sum_i (h_i + H) S_i$$

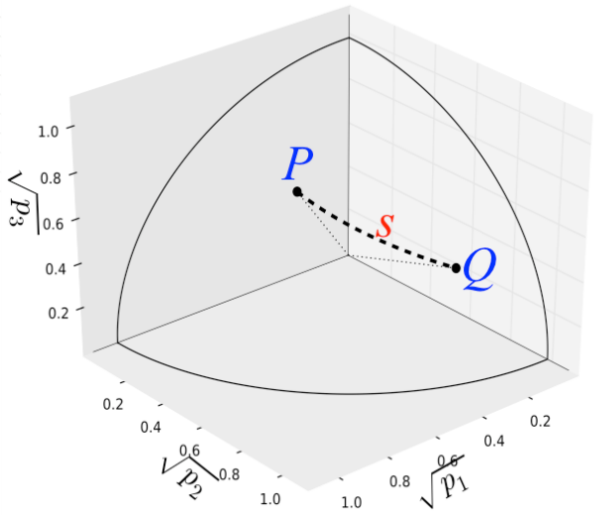
- Collection of two-state “spins” on a geometric lattice
- Interactions with nearest neighbors
- No long range depolarization fields
- Universal model that can represent physical (magnetism), chemical (alloys, surface adsorbates) systems



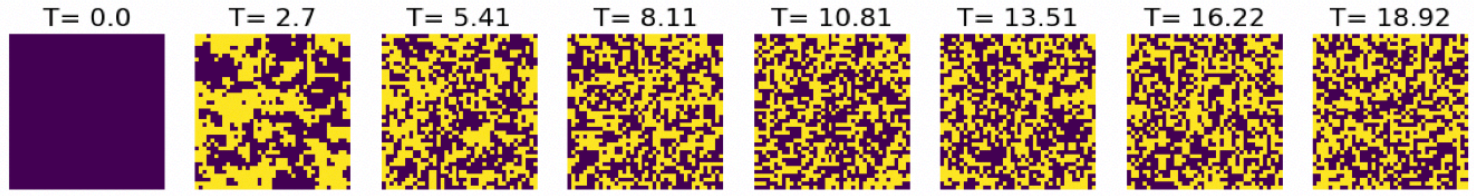
Statistical distance minimization

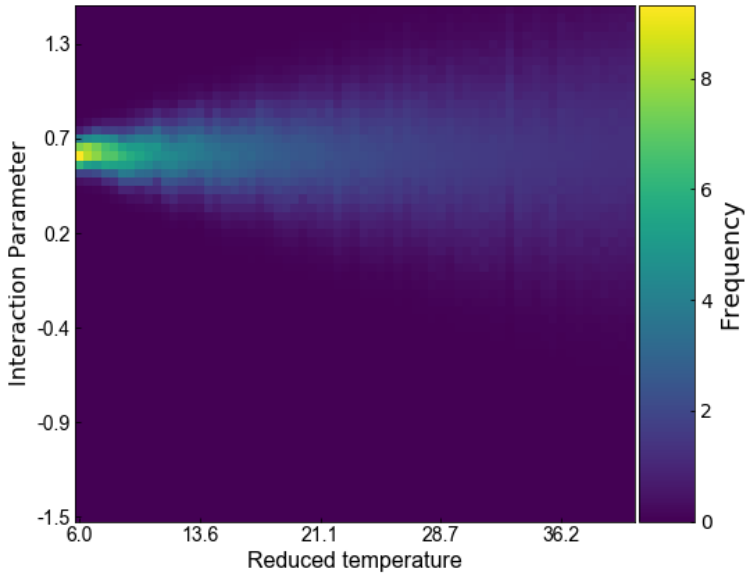
Minimize statistical distance between histograms

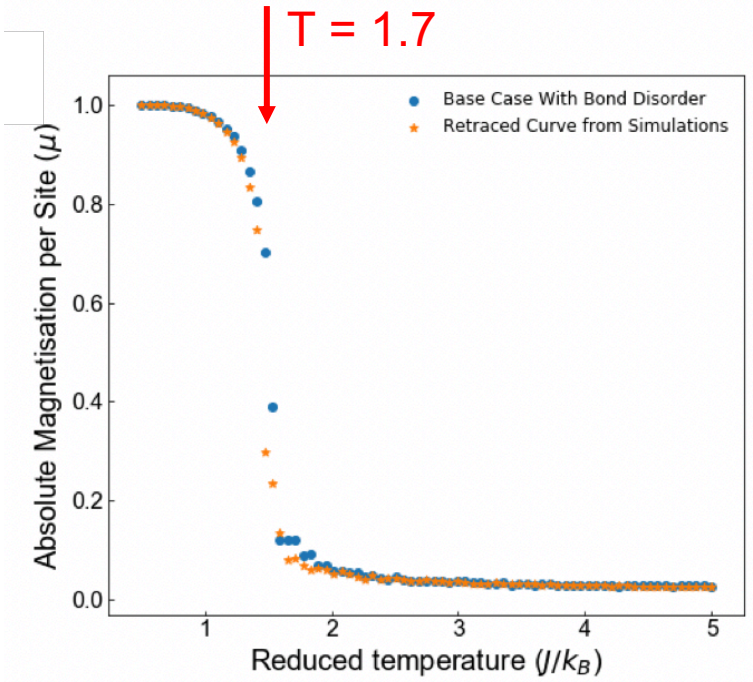
$$s = \arccos\left(\sum_{i=1}^{k}\sqrt{p_i}\sqrt{q_i}\right)$$



Vlcek et al., ACS Nano (2017)



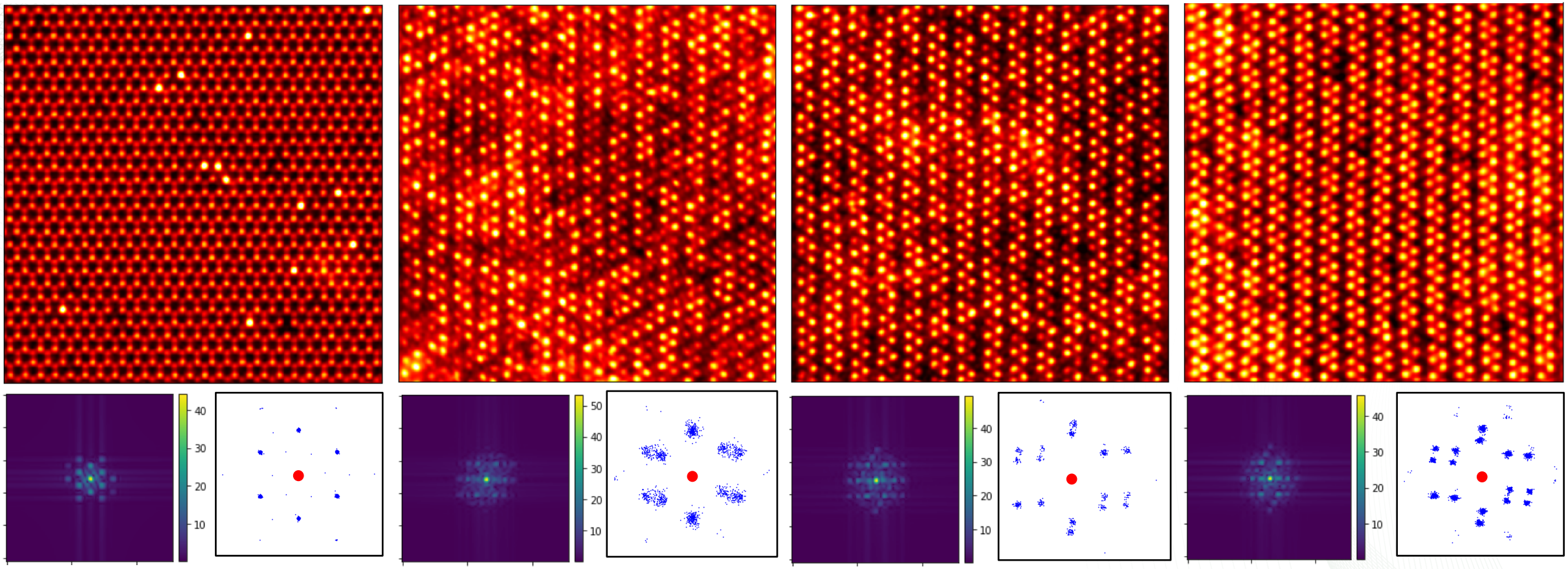




- Utilize all available statistical information in the image
- Using generative model, infer parameters from the experiment.

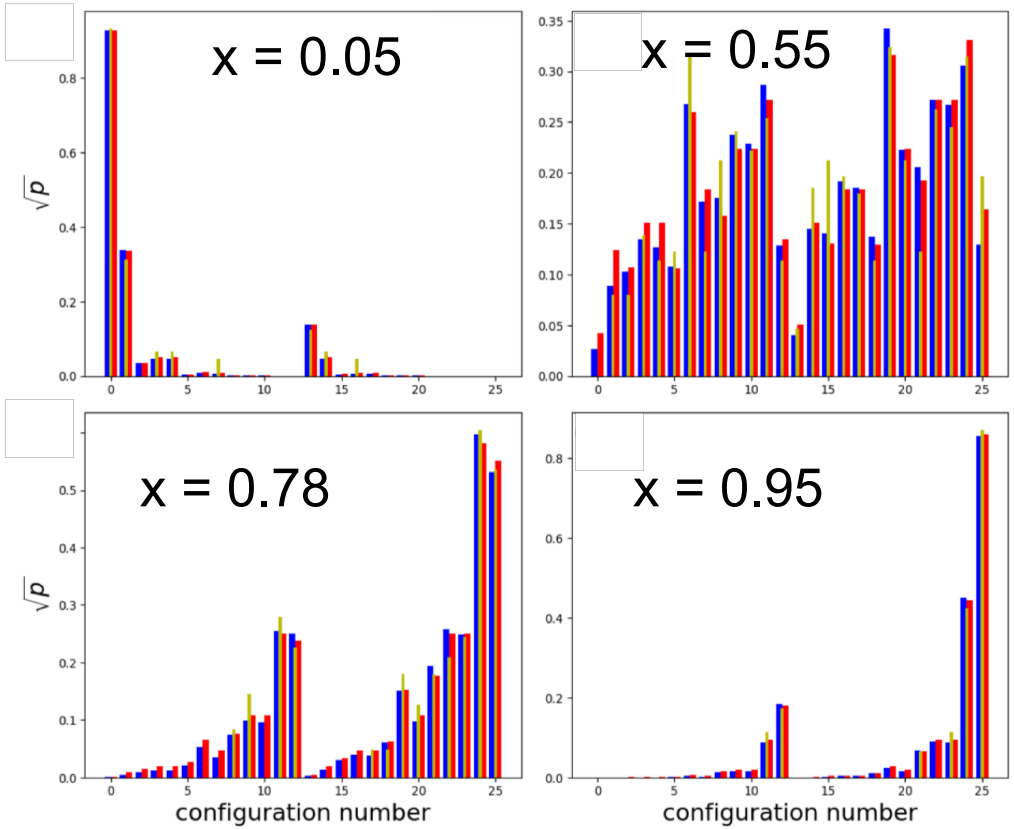
MoS_2 – ReS_2 Solid solutions by STEM

5% ReS_2 | 55% ReS_2 | 78% ReS_2 | 95% ReS_2

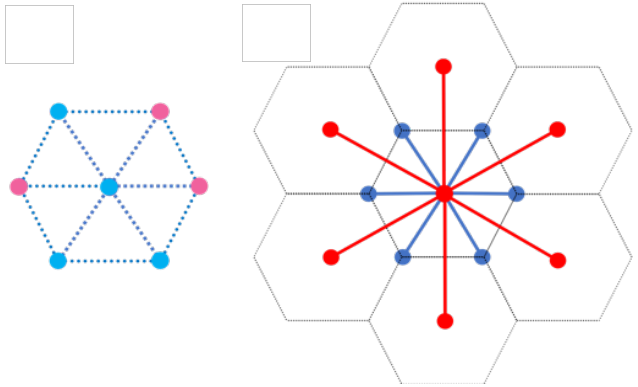


Data by Shize Yang and Matt Chisholm

Thermodynamics of solid solution



Nearest and next-nearest interactions



1. Pair-additive Model

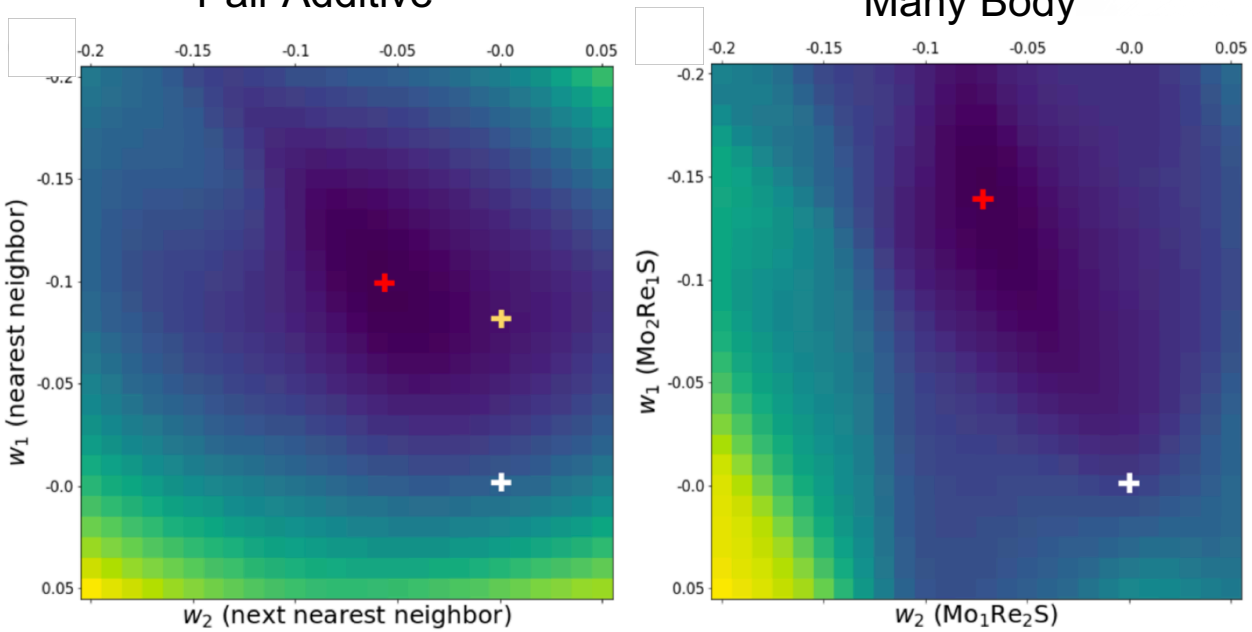
$$u_i = w_1 \sum_{\{NN\}} \delta_{MoRe} + w_2 \sum_{\{NNN\}} \delta_{MoRe}$$

2. Many-body Model

$$u_i = w_1 \sum_{\{S\}} \delta_{MoMoRe} + w_2 \sum_{\{S\}} \delta_{MoReRe}$$

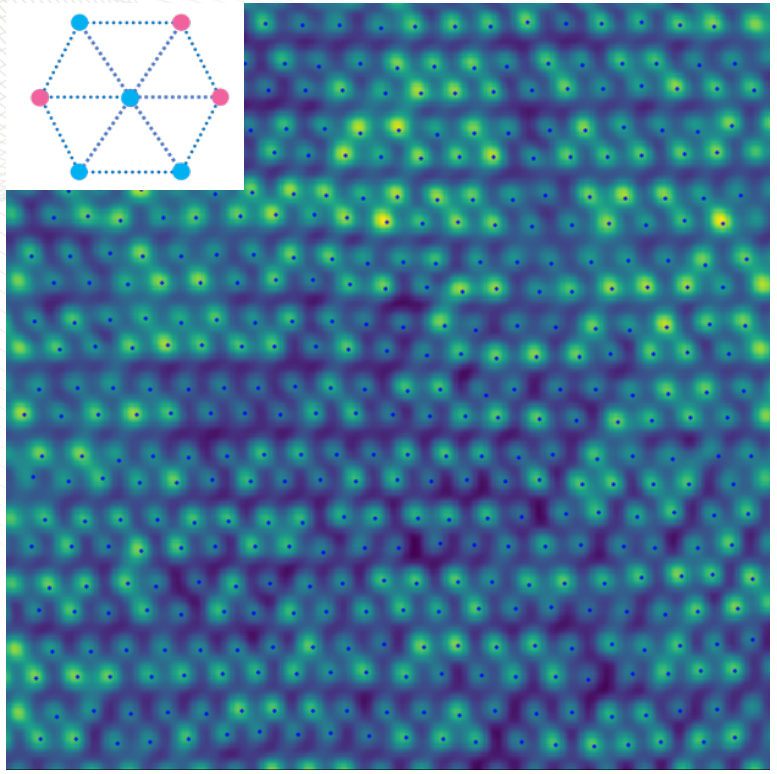
Pair Additive

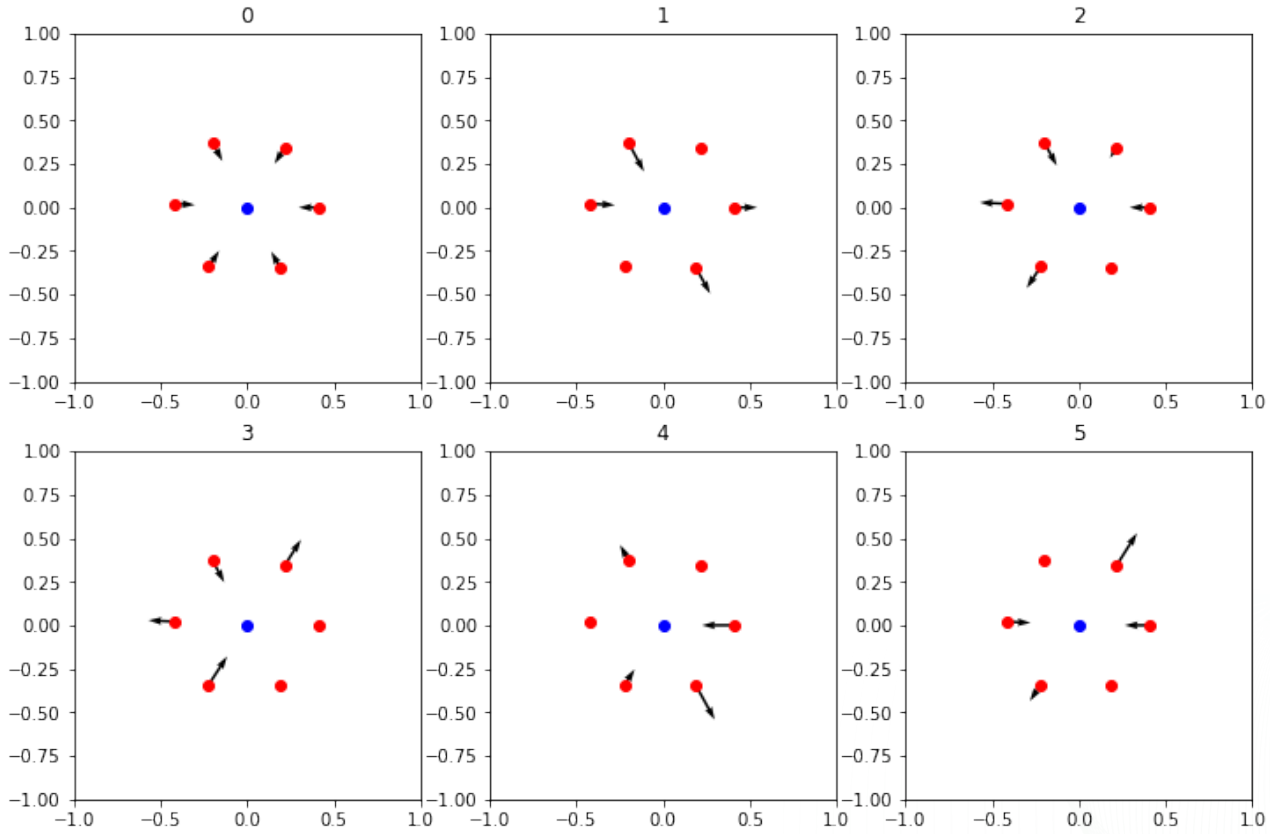
Many Body

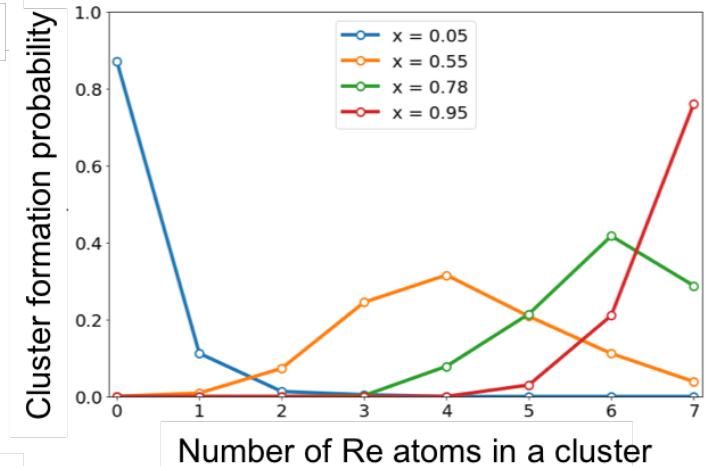


L. Vlcek, S. Yang, Y. Gong, P. Ajayan, W. Zhou, M. F. Chisholm, M. Ziatdinov, R. K. Vasudevan, S. V. Kalinin, [submitted], 2019.

Exploring physics: statistical normal modes



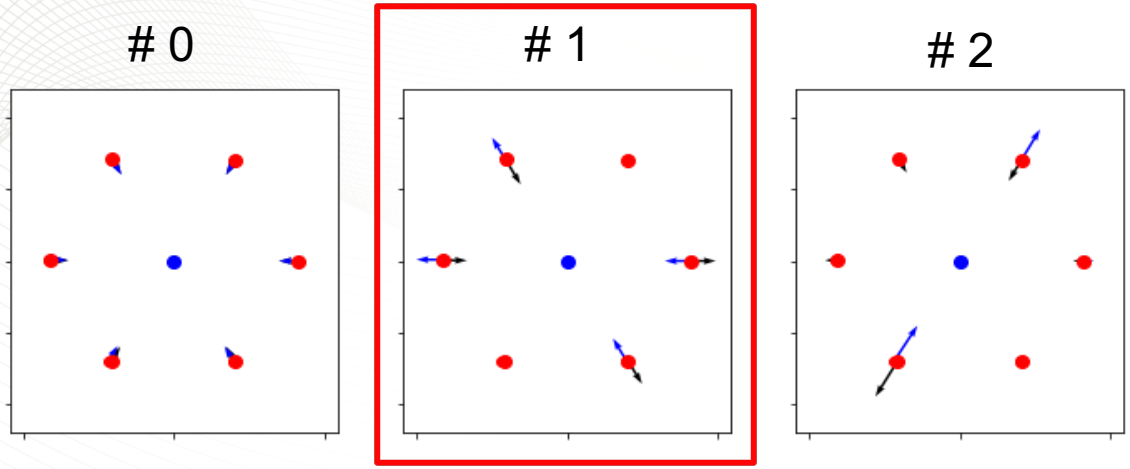




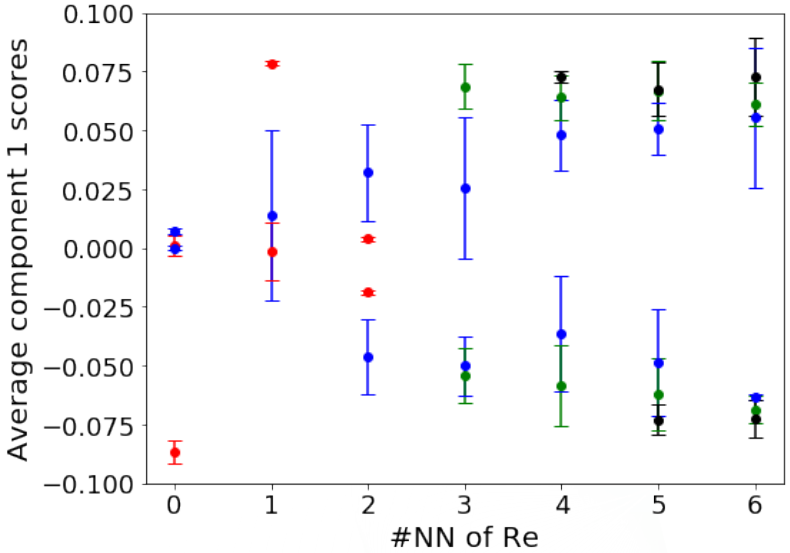
- Traditioonally, the order parameter is defined based on symmetry and atomistic representation is established in the *ad hoc* manner
- But what if we define order parameter from the bottom up – based on the statistics of atomic distortions?
- And further correlate it to local chemical composition?

Phase transition via statistical normal modes

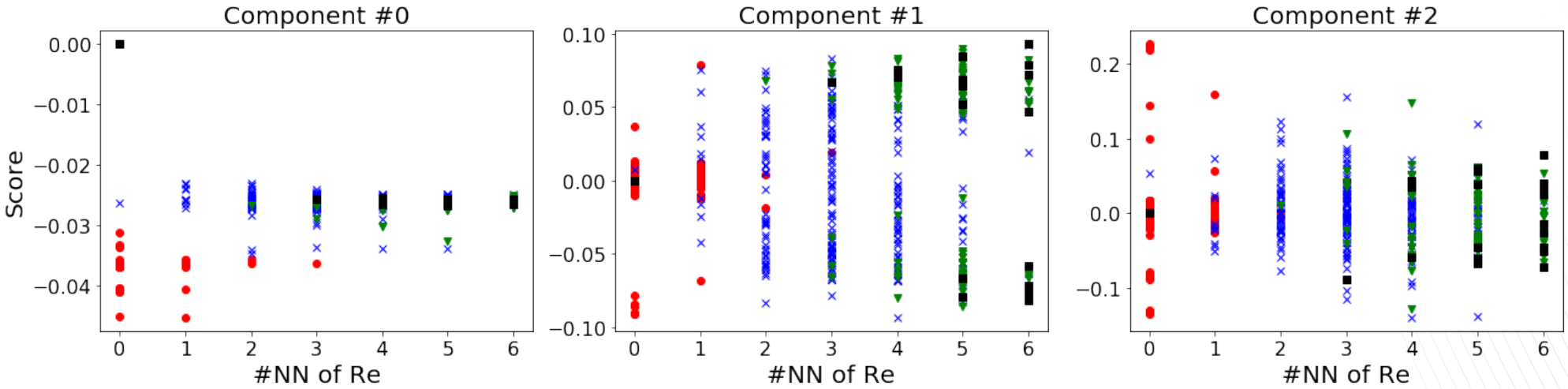
Three dominant distortion modes



Local symmetry breaking!



Mode distributions vs. global and local composition



5% ReS_2

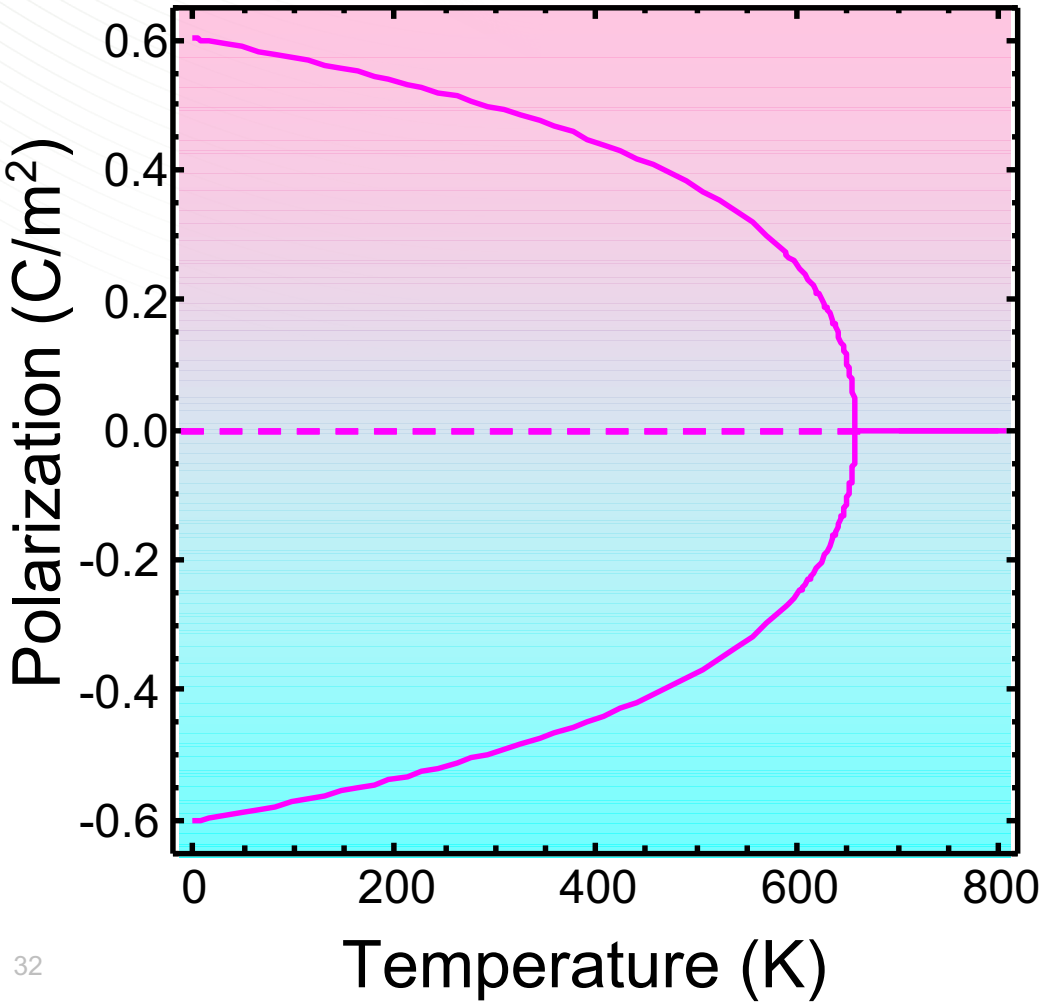
55% ReS_2

78% ReS_2

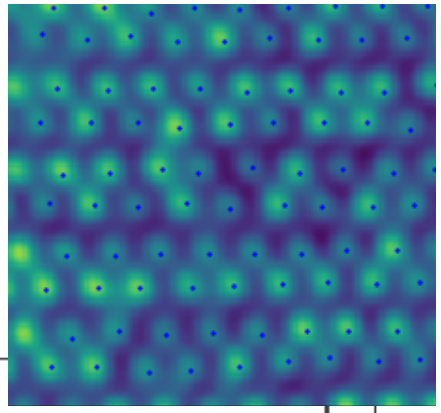
95% ReS_2

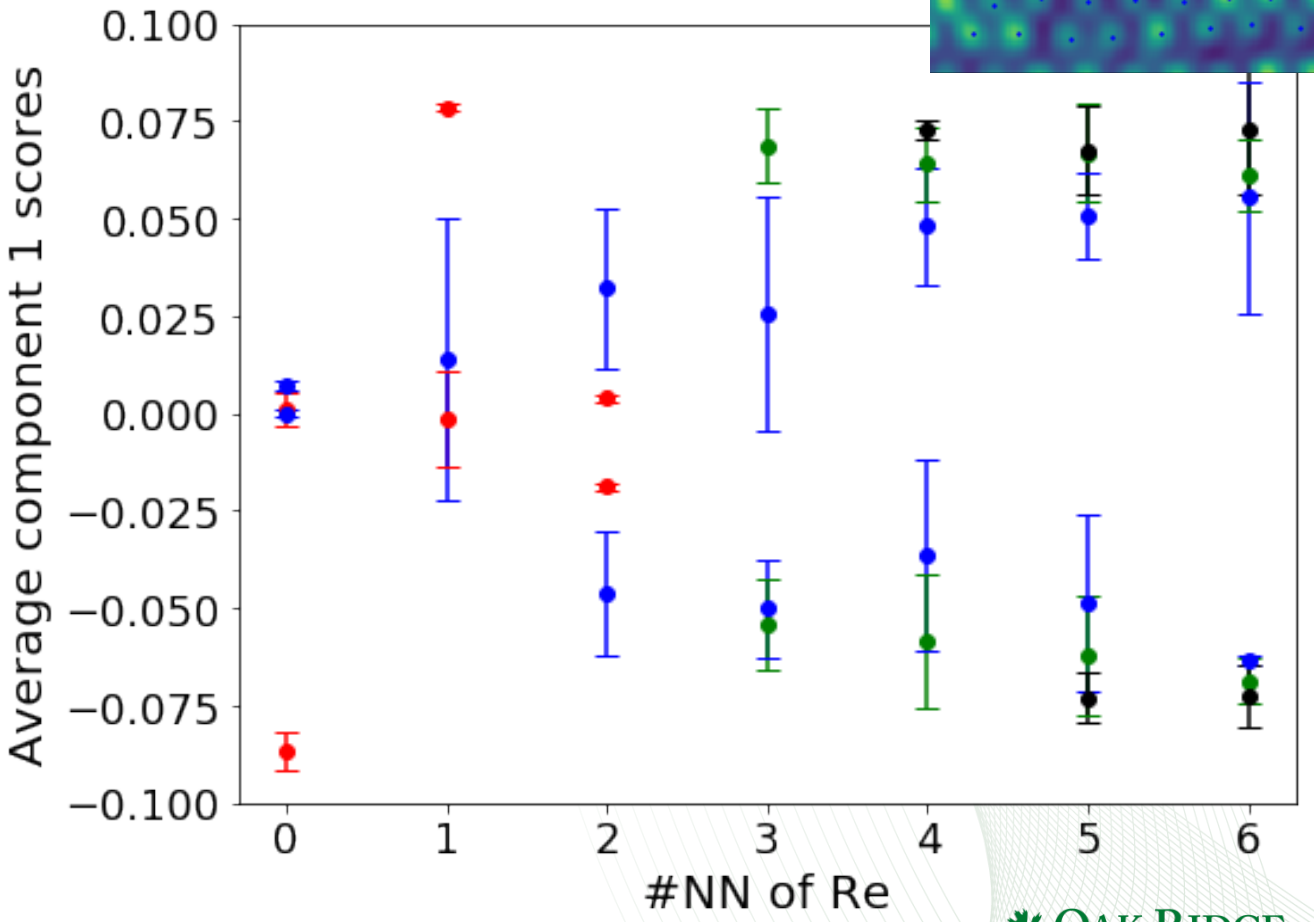
Phase transition from the bottom up

Global symmetry breaking

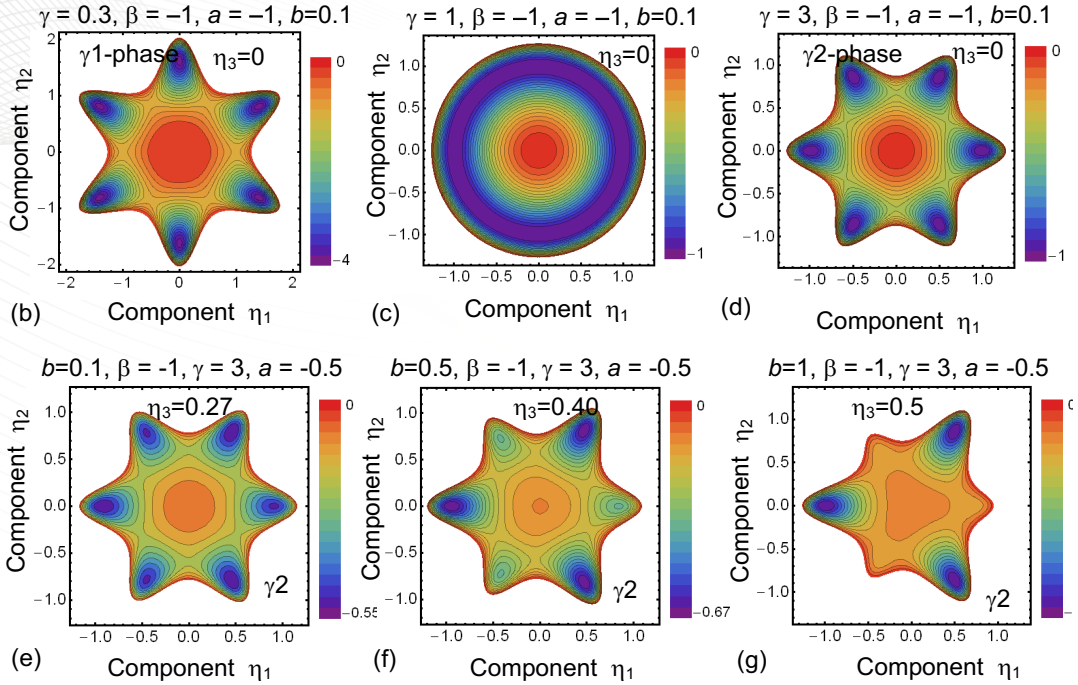


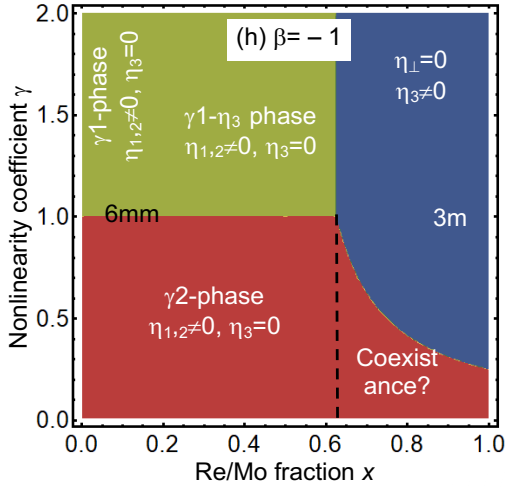
Local symmetry breaking!

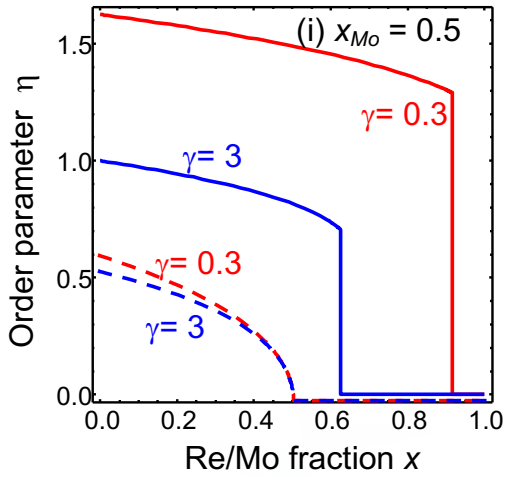




Transition to mesoscopic free energy







Free energy functional for 2D materials

$$F[\boldsymbol{\eta}] = \int_S d^2 r \left(f_L[\boldsymbol{\eta}(\vec{r})] + f_{EL}[\boldsymbol{\eta}(\vec{r})] + \frac{g_{ijkl}}{2} \frac{\partial \eta_i}{\partial x_j} \frac{\partial \eta_k}{\partial x_l} + \frac{f_{ijkl}}{2} \left(\frac{\partial \eta_i}{\partial x_j} u_{kl} - \eta_i \frac{\partial u_{kl}}{\partial x_j} \right) \right)$$

Local free energy term:

$$f_L[\boldsymbol{\eta}(\vec{r})] = \alpha_{11}\eta_\perp^2 + \alpha_{33}\eta_3^2 + \beta_{11}\eta_\perp^4 + \beta_{13}\eta_\perp^2\eta_3^2 + \beta_{33}\eta_3^4 + \beta_{15}\eta_1\eta_3\left(\eta_1^2 - 3\eta_2^2\right) + \gamma_{111}\eta_1^2\left(\eta_1^2 - 3\eta_2^2\right)^2 + \gamma_{222}\eta_2^2\left(3\eta_1^2 - \eta_2^2\right)^2 + \gamma_{113}\eta_\perp^4\eta_3^2 + \gamma_{133}\eta_\perp^2\eta_3^4 + \gamma_{333}\eta_3^6$$

Opportunity:

- Phase diagrams
- Electronic phenomena induced by curvature via flexoelectric coupling
- Field- and doping induced transitions

A Bit of (Ancient and Modern) Wisdom

…there are known knowns; there are things we know we know. We also know there are known unknowns; that is to say we know there are some things we do not know. But there are also unknown unknowns—the ones we don't know we don't know.

D. Rumsfeld

Har kas ke bedanad va bedanad ke bedanad
Asb-e kherad az gombad-e gardun bejahanad
Har kas ke nadanad va bedanad ke nadanad
Langan kharak-e khish be manzel beresanad
Har kas ke nadanad va nadanad ke nadanad
Dar jahl-e morakkab'abad od-dar bemanad

Anyone who knows, and knows that he knows
Makes the steed of intelligence leap over the vault of heaven
Anyone who does not know, but knows that he does not know
Can bring his lame little donkey to the destination nonetheless
Anyone who does not know, and does not know that he does not know
Is stuck forever in the double ignorance

Naser od-Din Tusi (1201-1274)

The World is Bayesian: Physics from Observations

…there are known knowns; there are things we know we know. We also know there are known unknowns; that is to say we know there are some things we do not know. But there are also unknown unknowns—the ones we don't know we don't know.

D. Rumsfeld

Hypothesis driven science:
What we want to learn

Forward model:
Theory

Domain expertise:

$$P(Theory|Data) = \frac{P(Data|Theory)\,P(Theory)}{P(Data)}$$

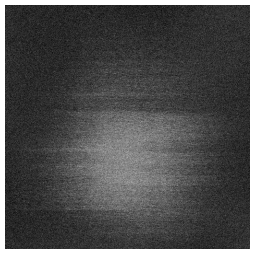
High Performance Computing

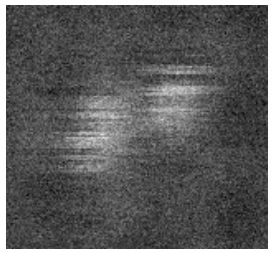
- Experimentalists know the priors. Albeit they do not know that they know it, or how to convert them to algorithmic form
- **How can we add Bayesian priors to:**
  - Reinforcement learning (functionality optimization in experiment)
  - GANs (inverse problems, image reconstruction)
  - VAEs (physical constraints on latent variables)

The chemistry challenge

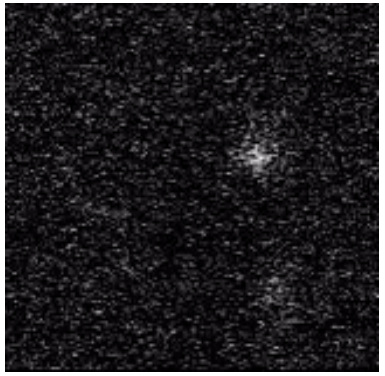
R. Ishikawa

O. Dyck

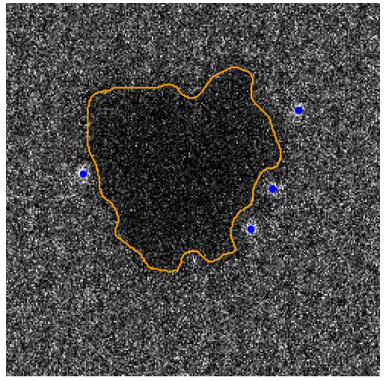




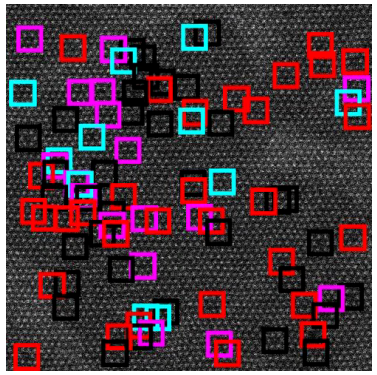
O. Dyck



O. Dyck

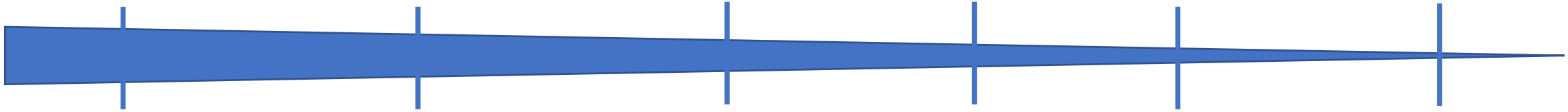


O. Dyck



A. Lupini

M. Chi



Only one atom moves in well defined potential

One or few atoms move in defined potential

One or few atoms move in weakly changing potential

Point defect dynamics

Extended defect dynamics

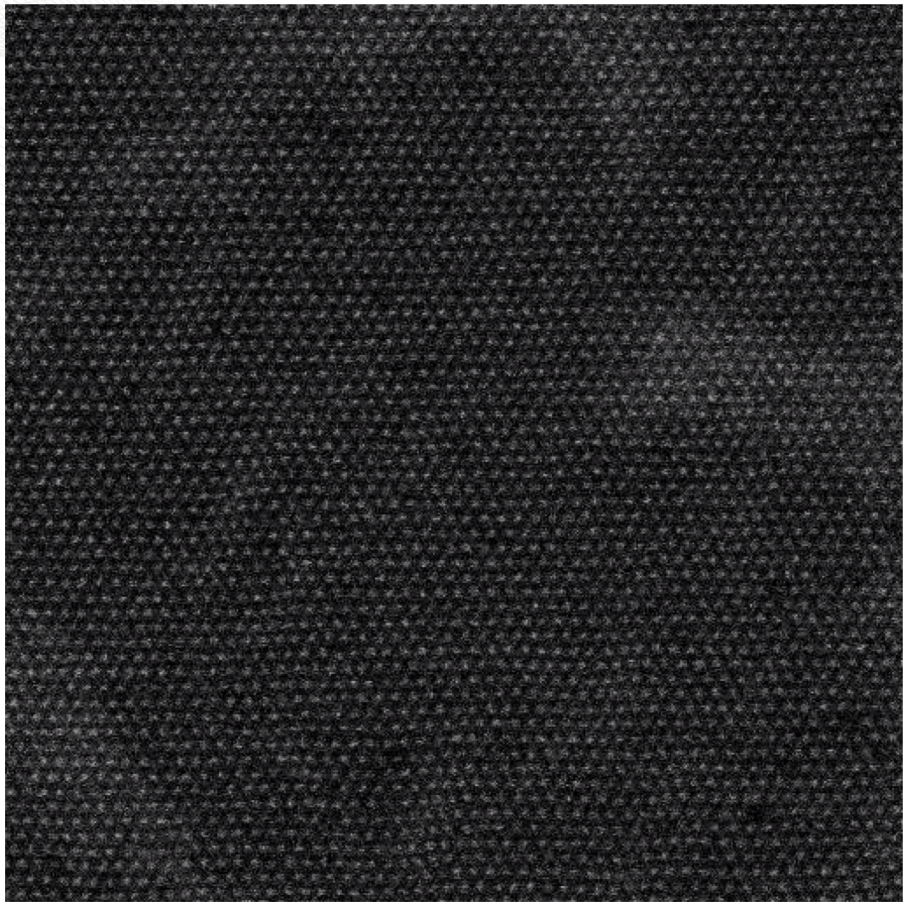
Everything changes: defect formation, nucleation, mechanical deformation

What about chemistry?

- Markov state descriptions
- Potential energy landscape reconstructions
- Force fields (and excitation) from observed dynamics

Learning the defect evolution

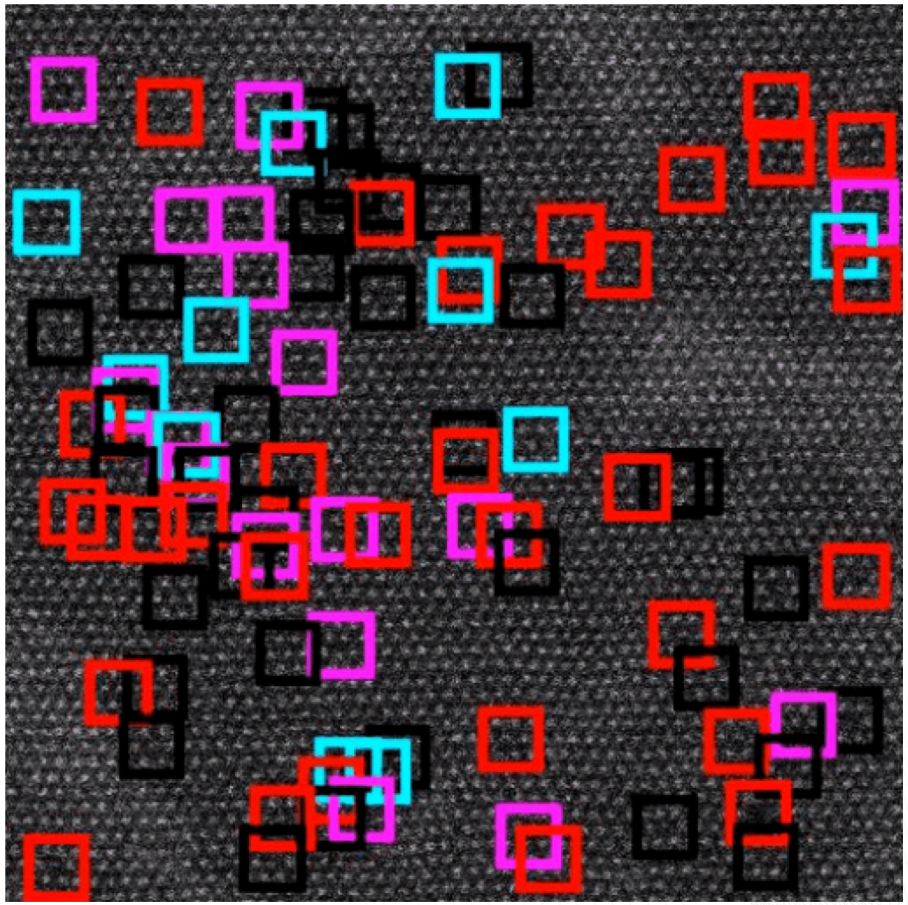
Experimental

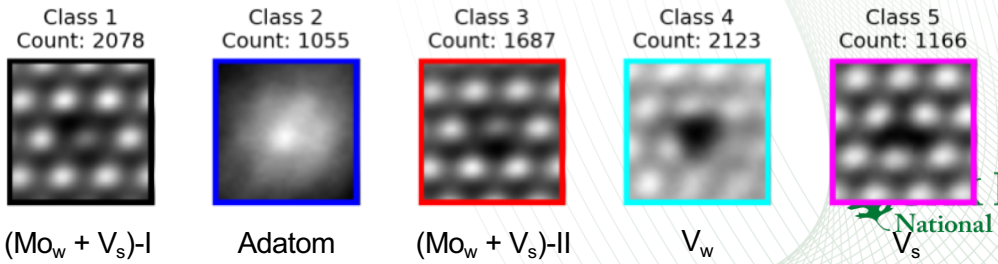


Sample: WS2
E-beam energy: 60 kV

Data collected by Ondrej Dyck (CNMS/ORNL)

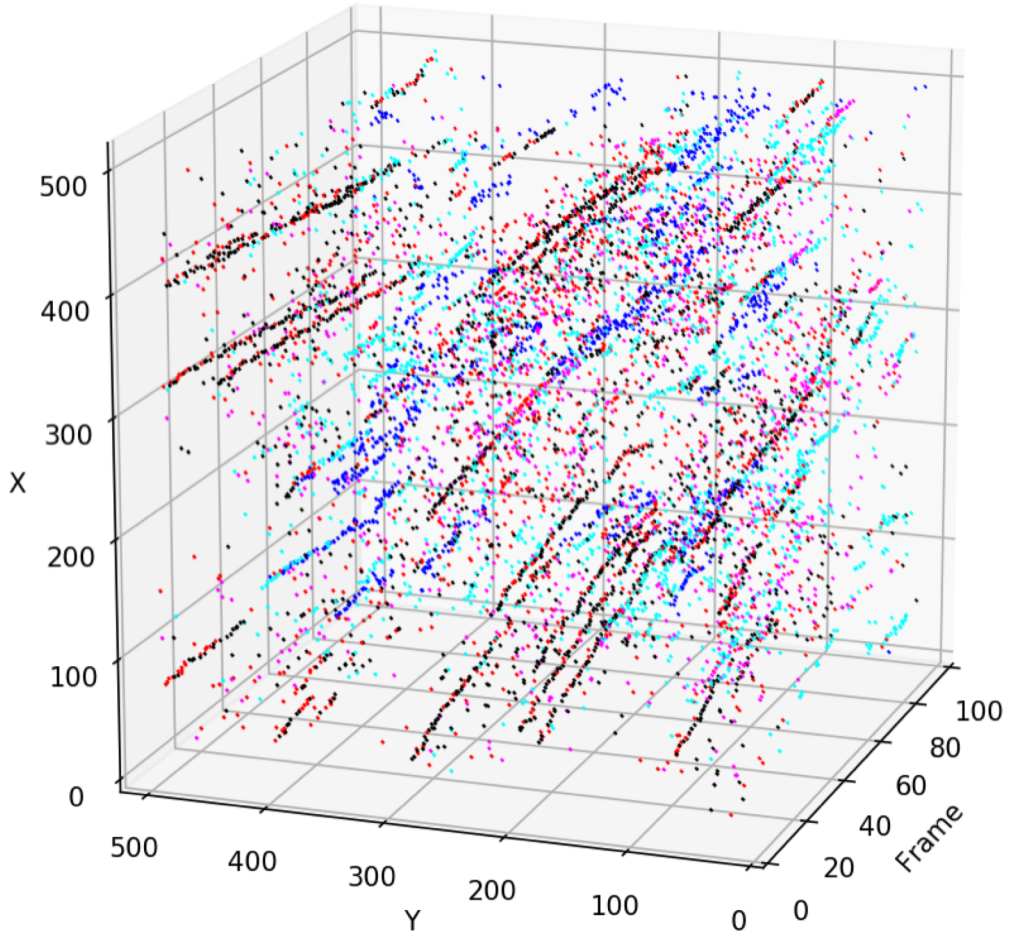
Decoded





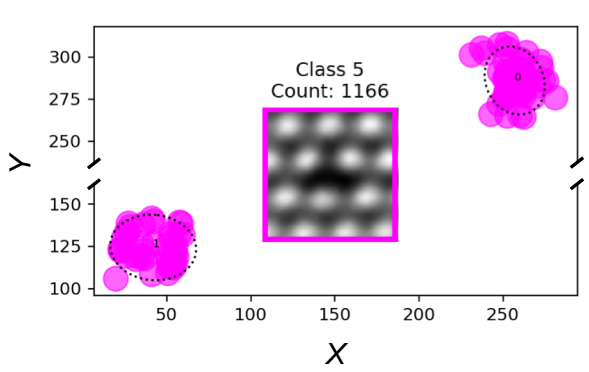
Physics extraction

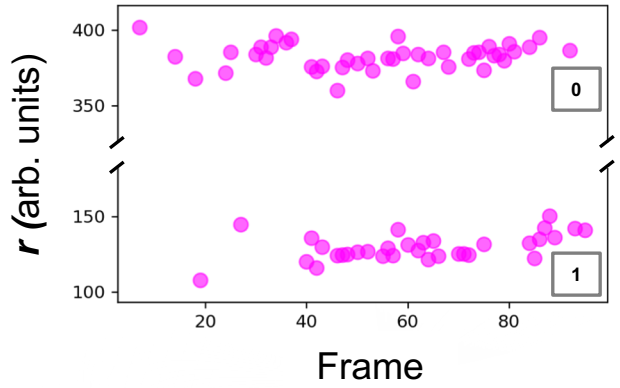
Spatio-temporal trajectories



Maksov *et al.*, npj Computational Materials 5, 12 (2019)

Diffusion parameters for selected defect types



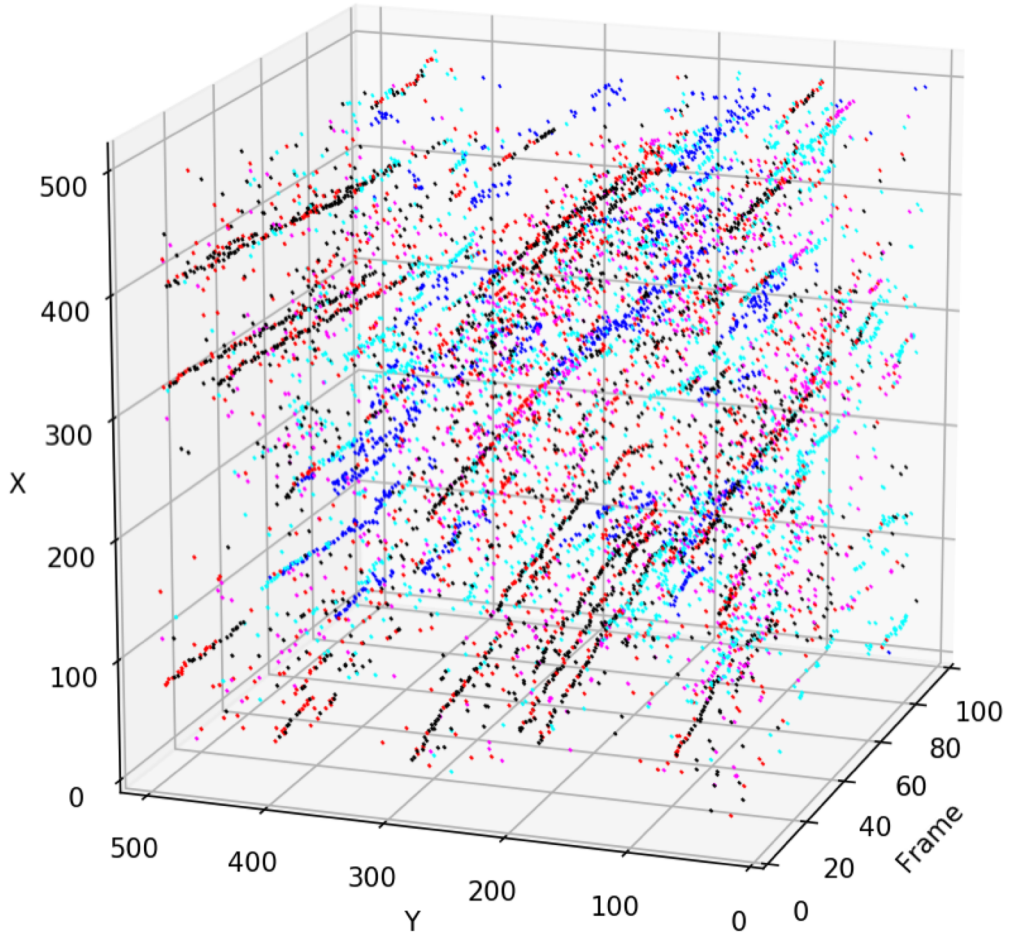


Diffusion coefficient: 3×10^{-4} nm^2/s - 6×10^{-4} nm^2/s (within 2D random walk approximation)

- Identification of dominant point defects and their characteristic statistical behaviors
- Analysis of diffusion parameters for the selected defect species
- Study of transformation pathways and transition probabilities for composite defects

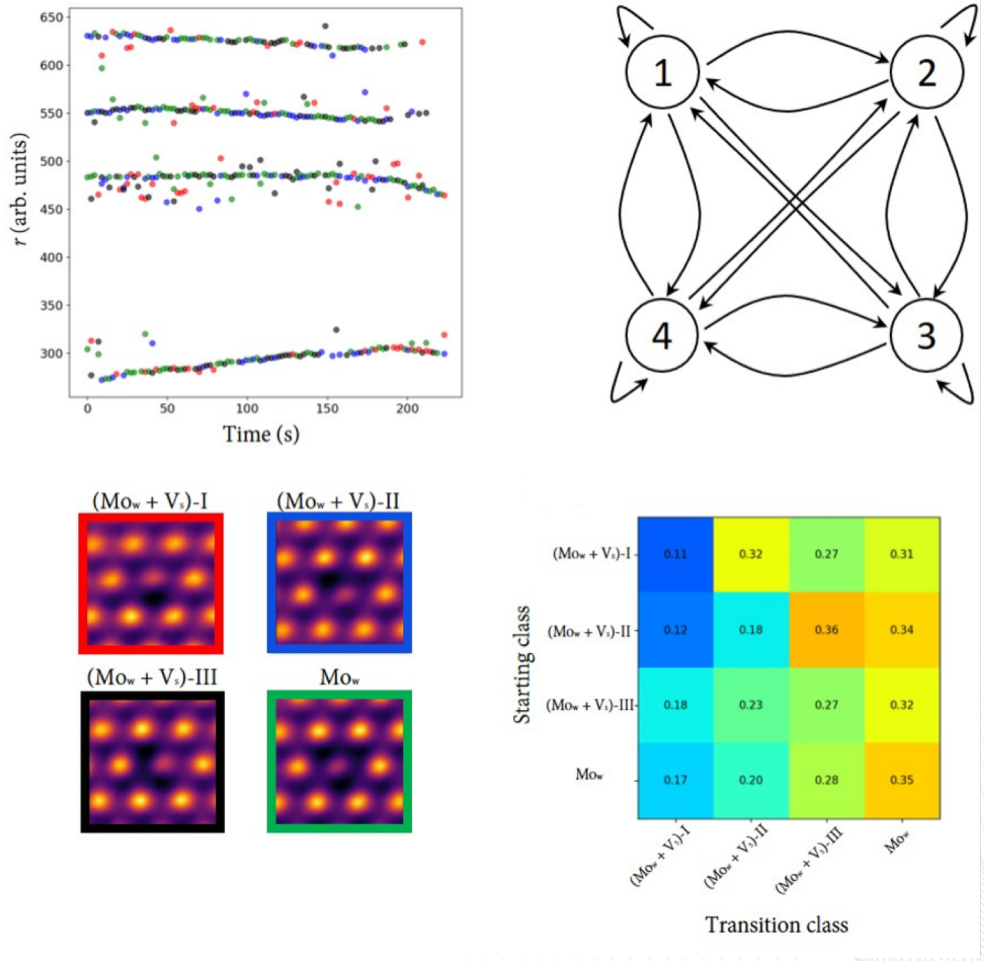
Physics extraction

Spatio-temporal trajectories



Maksov *et al*., npj Computational Materials 5, 12 (2019)

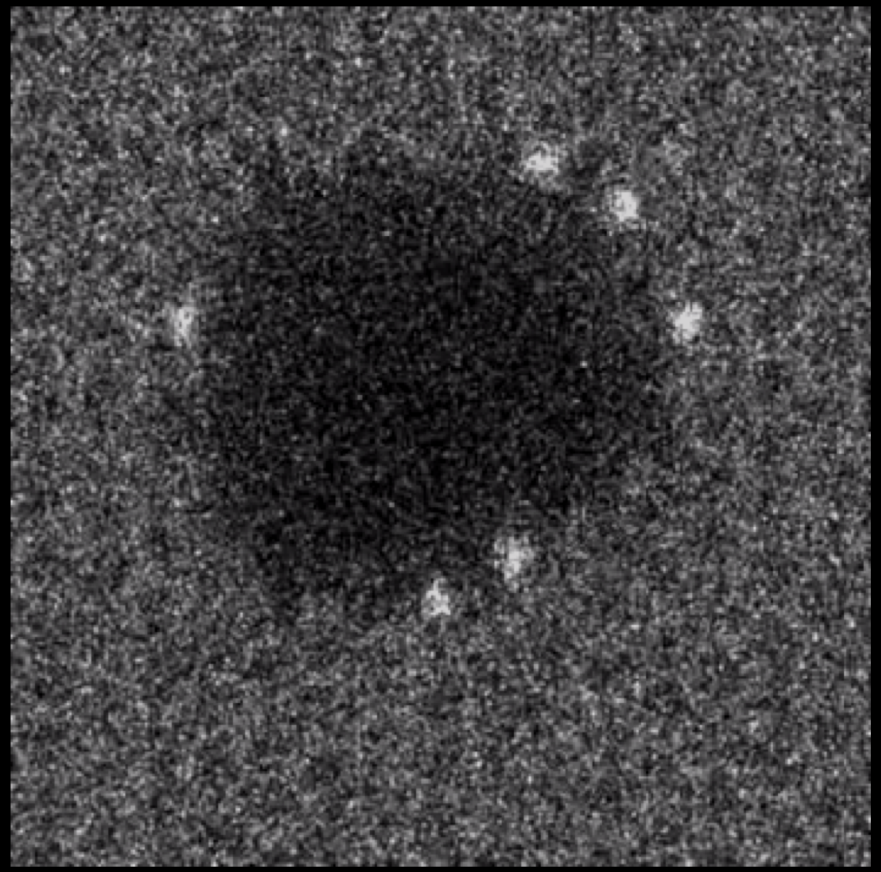
Evolution of defects as Markov process



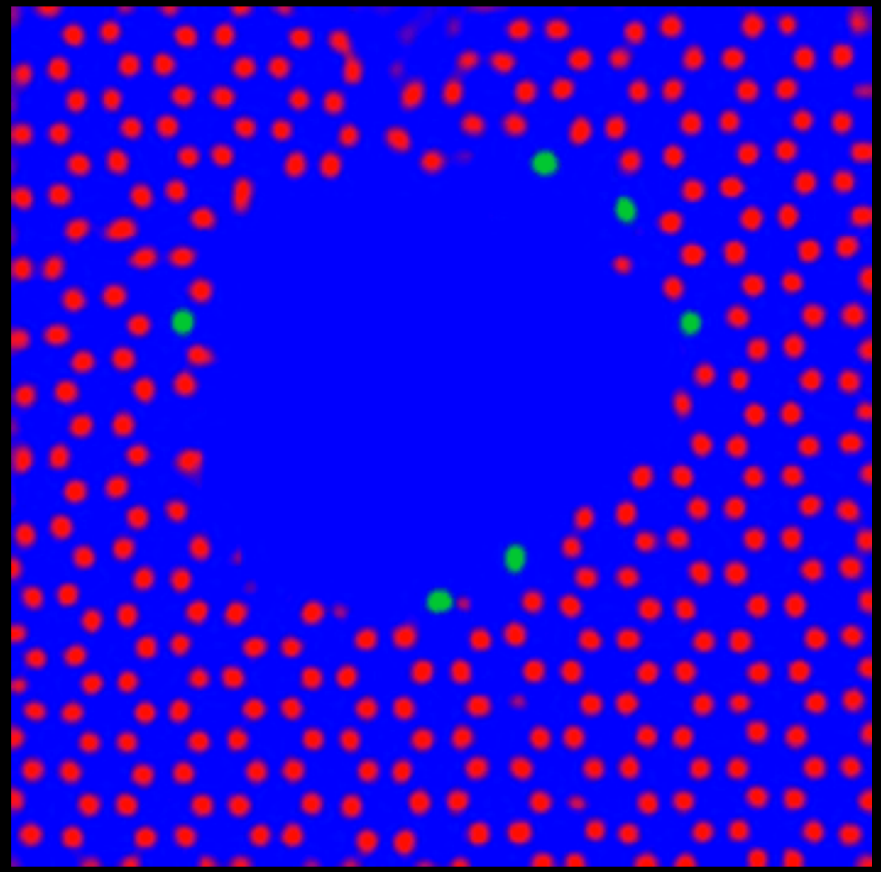
Beam induced reactions of Si atoms on the edge of graphene

Reconstructing Si impurity configurations at graphene edge

Experimental data



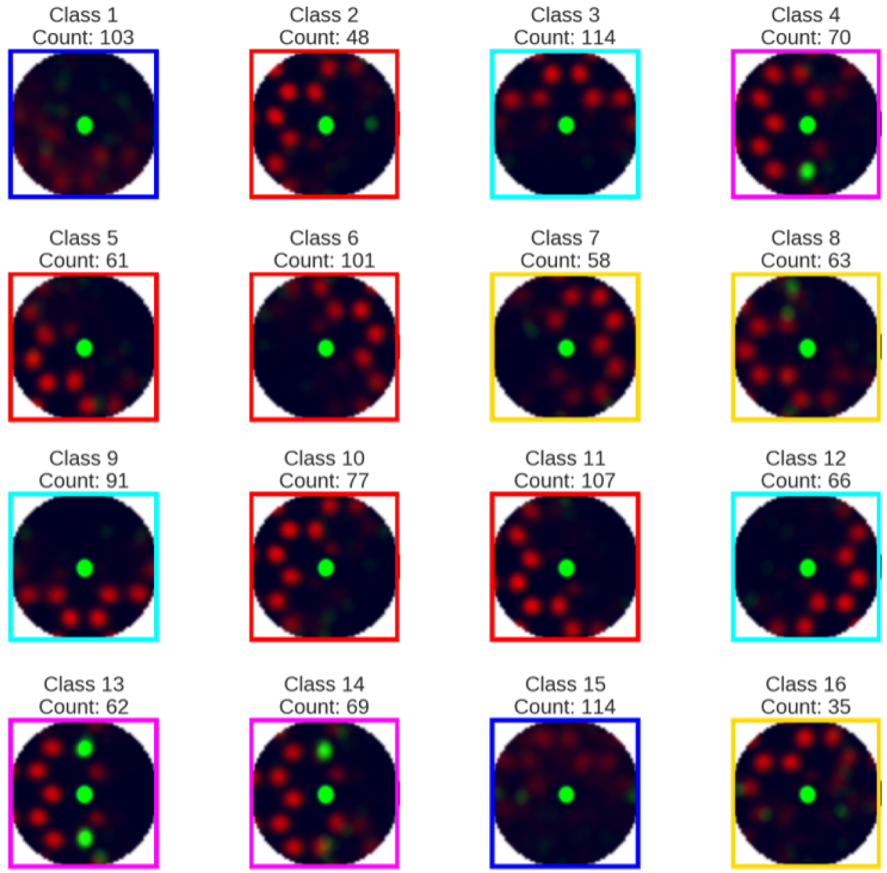
Network's output



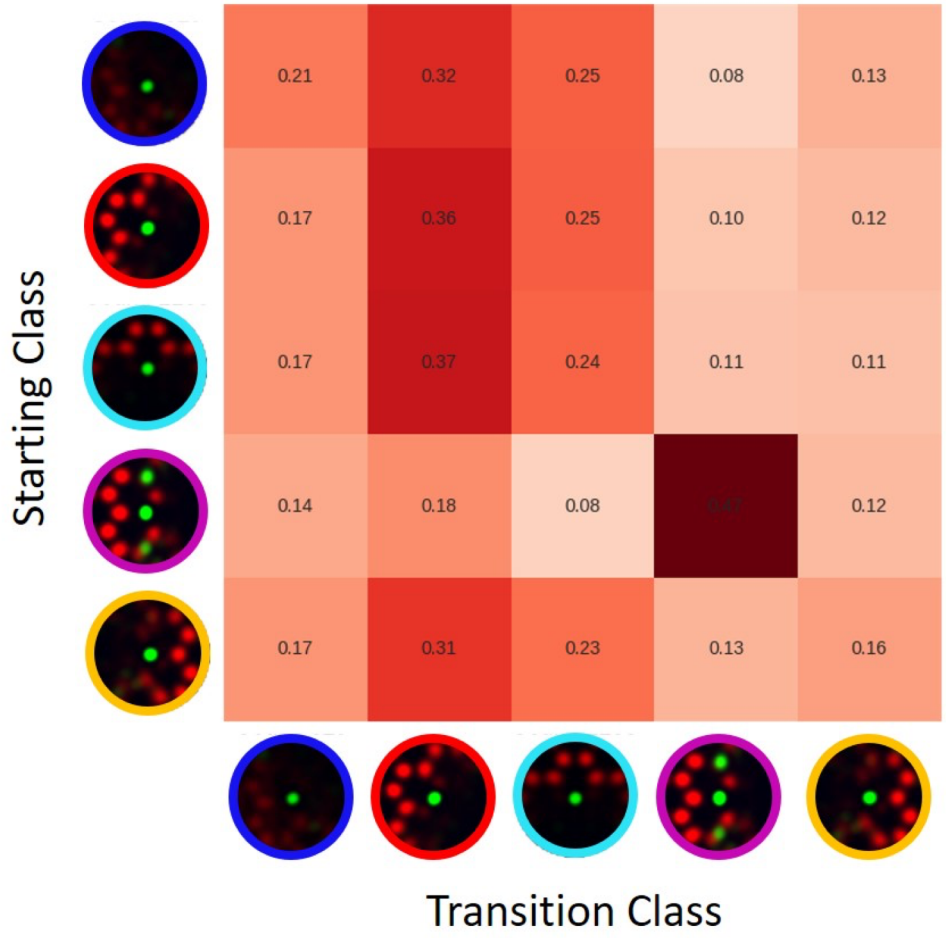
Ziatdinov *et al*., ArXiv:1901.09322 (2019)

Chemical transformations on the edge

Derived classes of Si-C edge configurations



Transition probabilities matrix



- Gaussian mixture model

- Discrete rotation symmetry + structural similarity algorithm

- Markov state analysis

Ziatdinov *et al.*, ArXiv:1901.09322 (2019)

“What I cannot create, I do not understand.”

— Richard Feynman

Molecular machines

2016 Nobel Prize

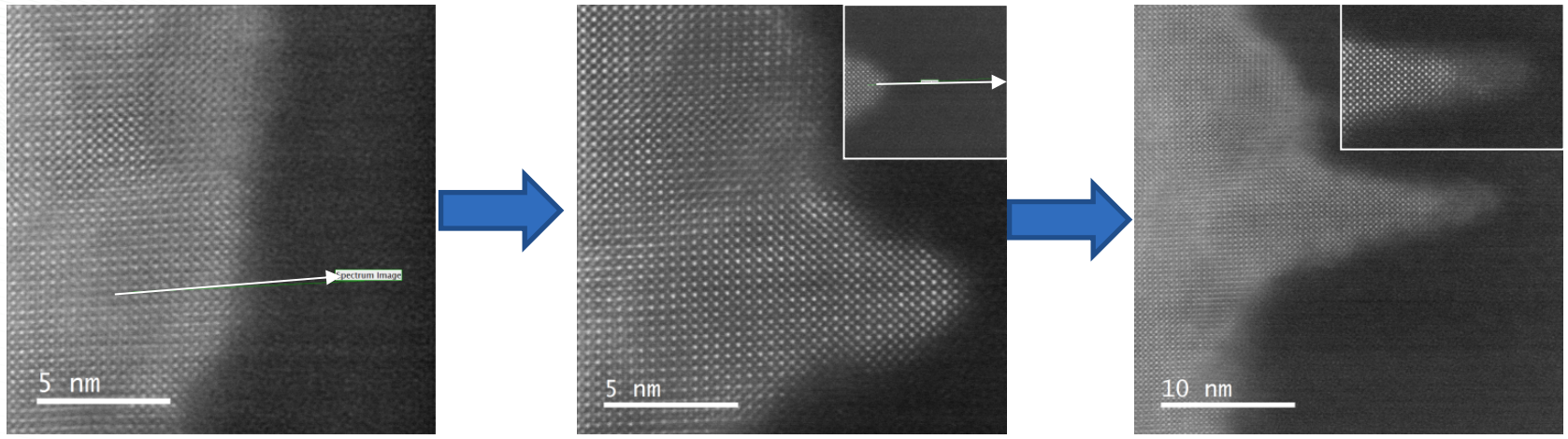
J.P. Sauvage, F. Stoddard, and B. Feringa

“What I cannot create, I do not understand.”

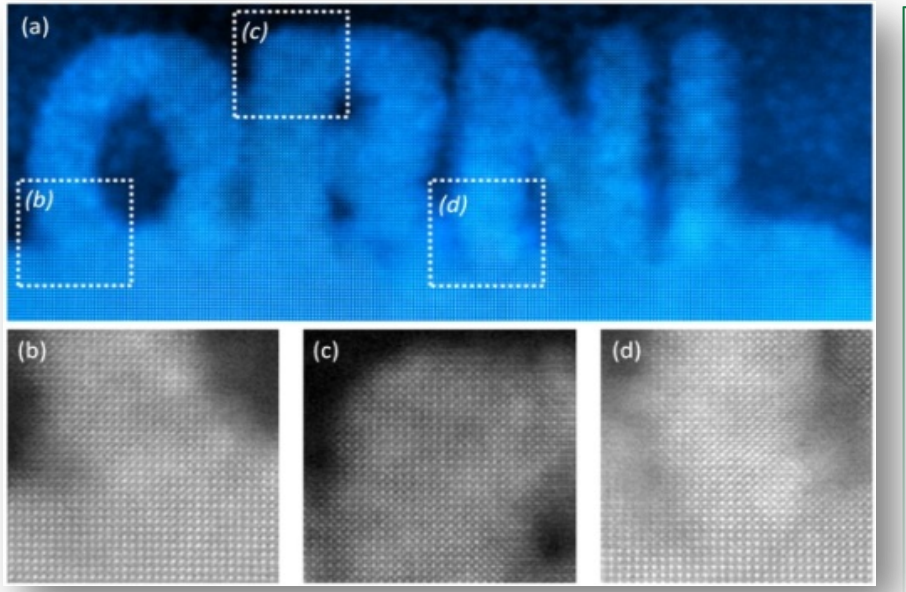
— Richard Feynman

Is There a Third Way?

Synergy of STEM and SPM: Sculpting

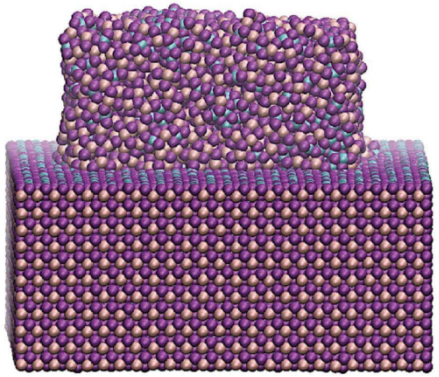


We induce local amorphous/crystalline transition in a defined area

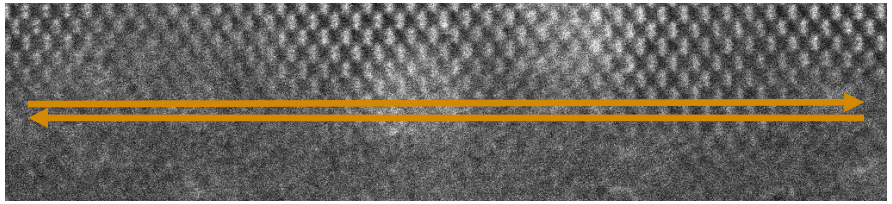


- No crystallization observed away from interface
- Electron-beam induced solid phase epitaxy
- Control of beam position and speed
- We chose to explore, exploit, and understand this behavior

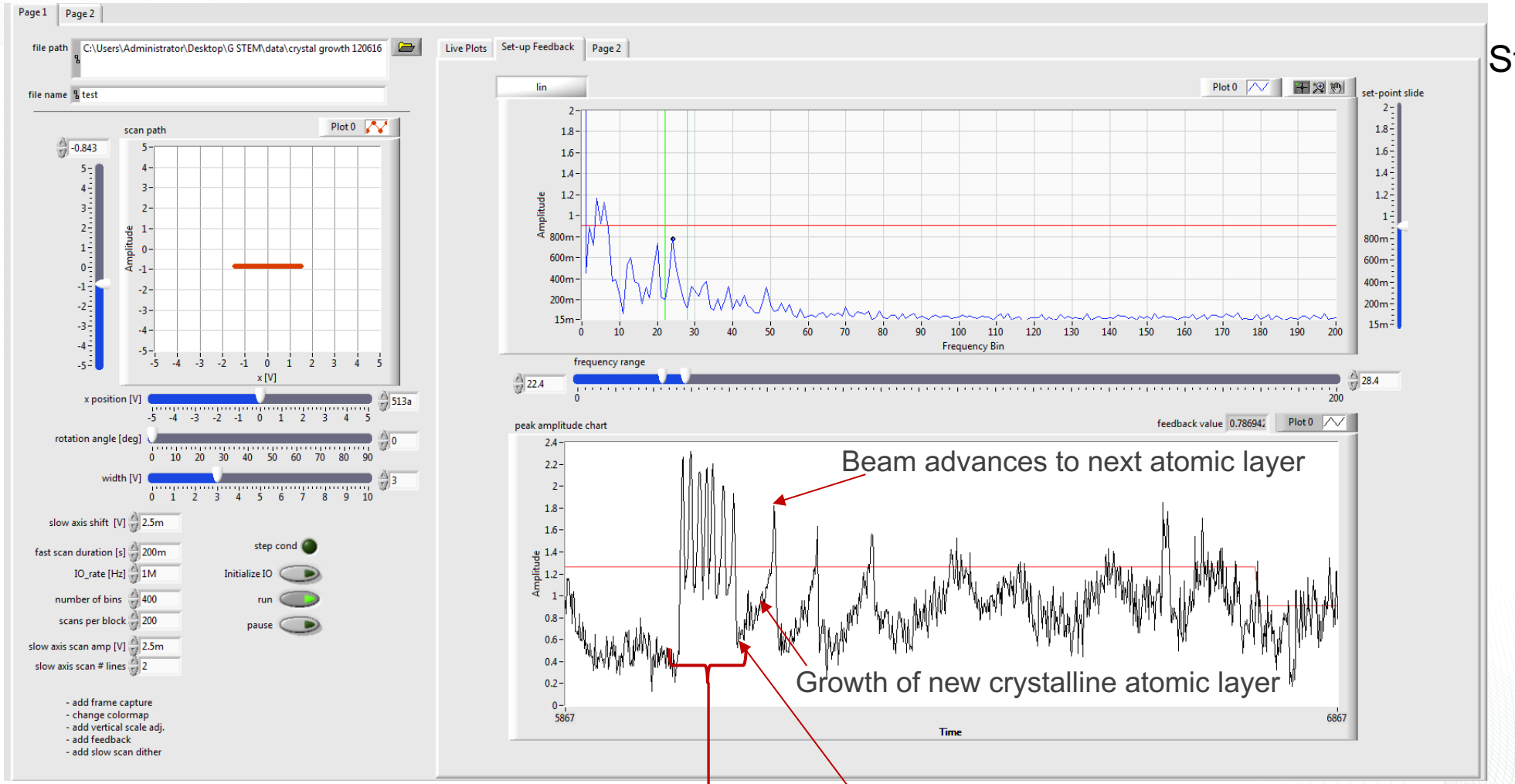
- Formation of 3D structures inside solid
- Epitaxial registration
- MD simulations have confirmed experimental observations
- Foundation for making atomically precise, multi-component, multi-layer systems



Beam Induced Transformations in Solids: *Feedback*



Stephen Jesse



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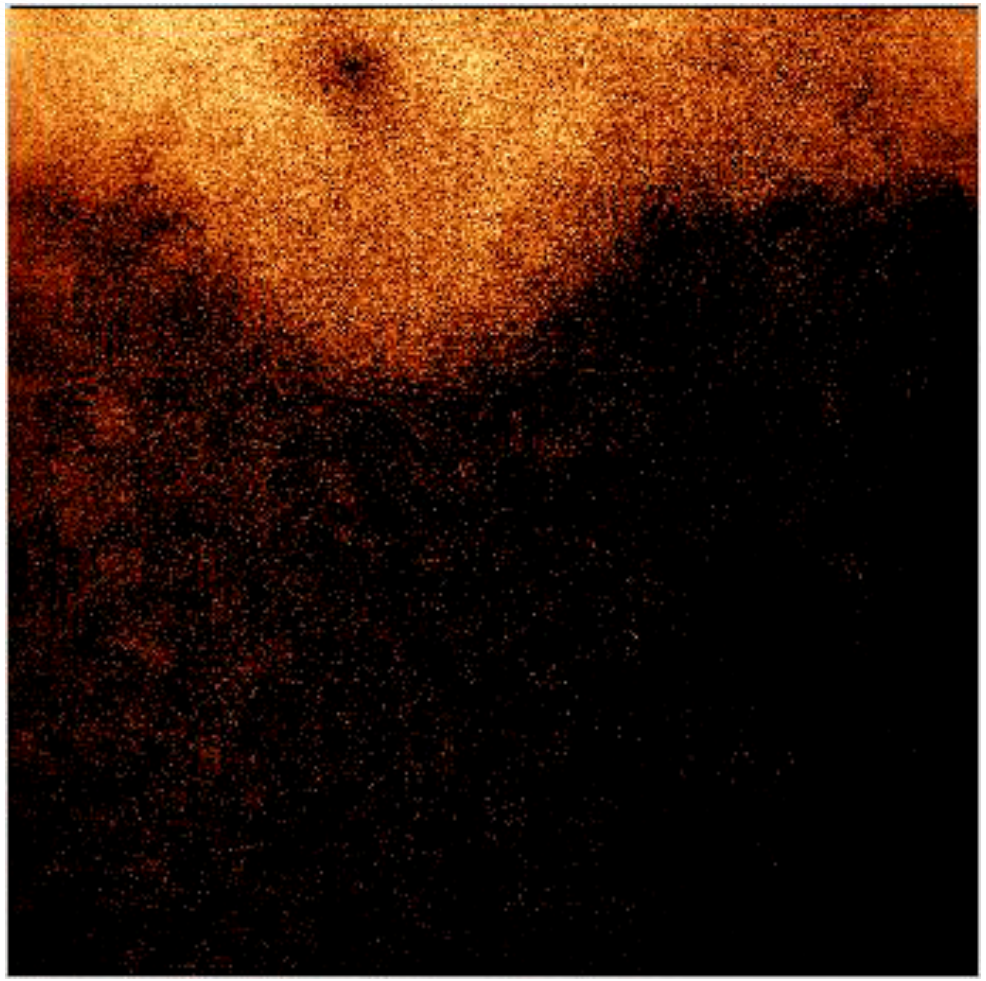
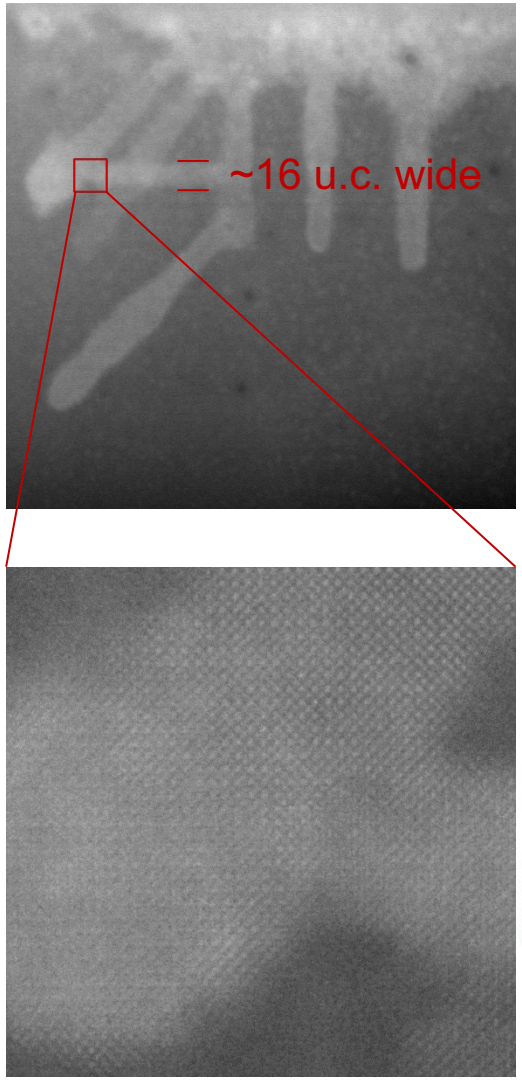
Manufacturing at the Atomic Scale with Beams

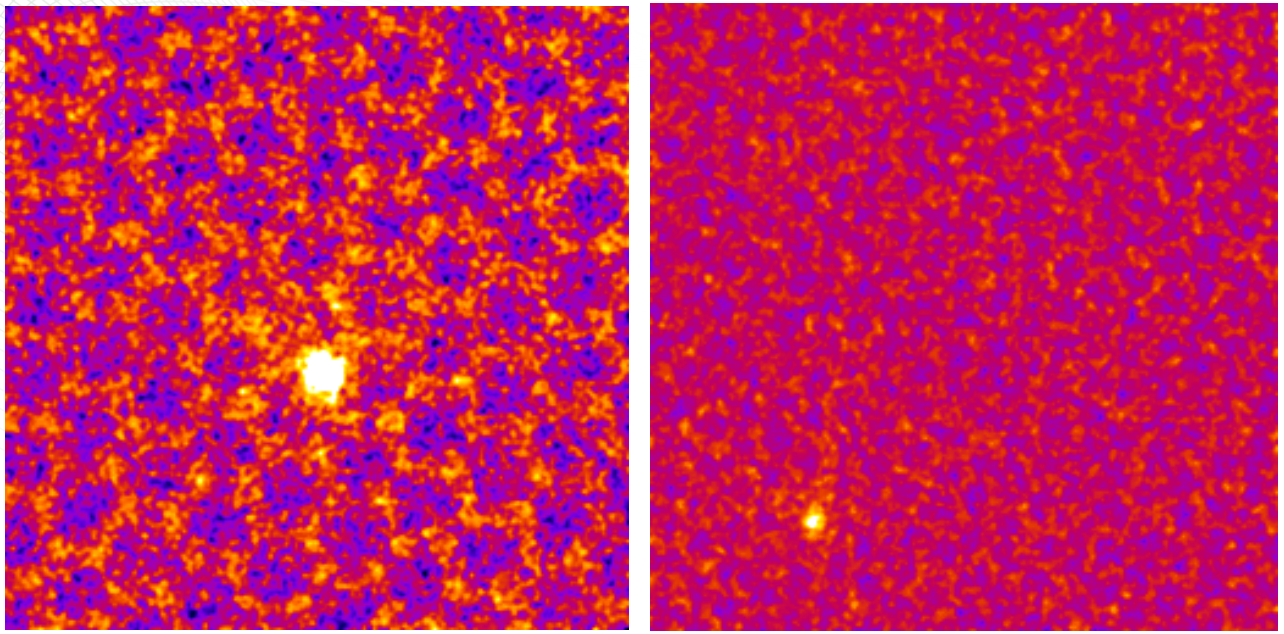
Directed Crystal Growth in STO



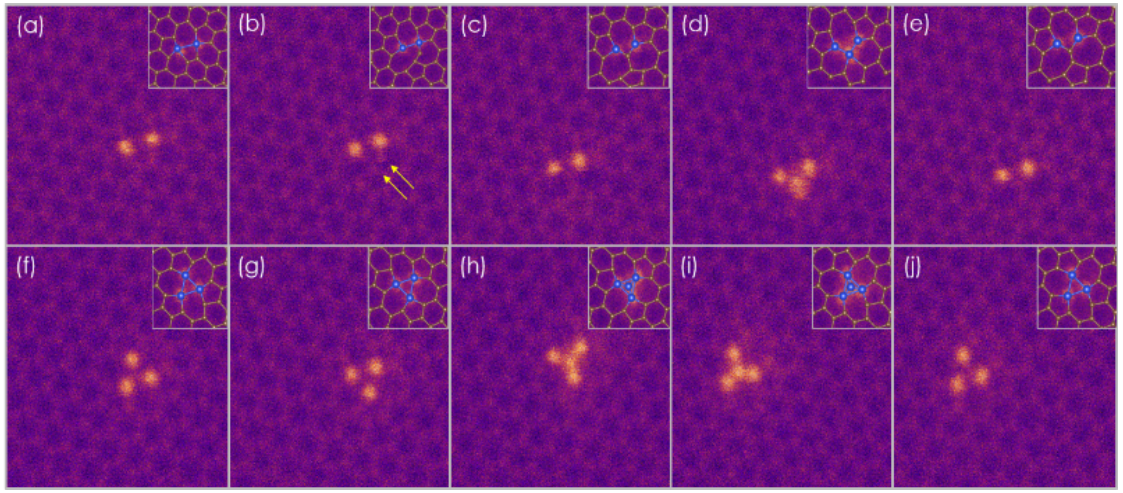


Atomic manipulations

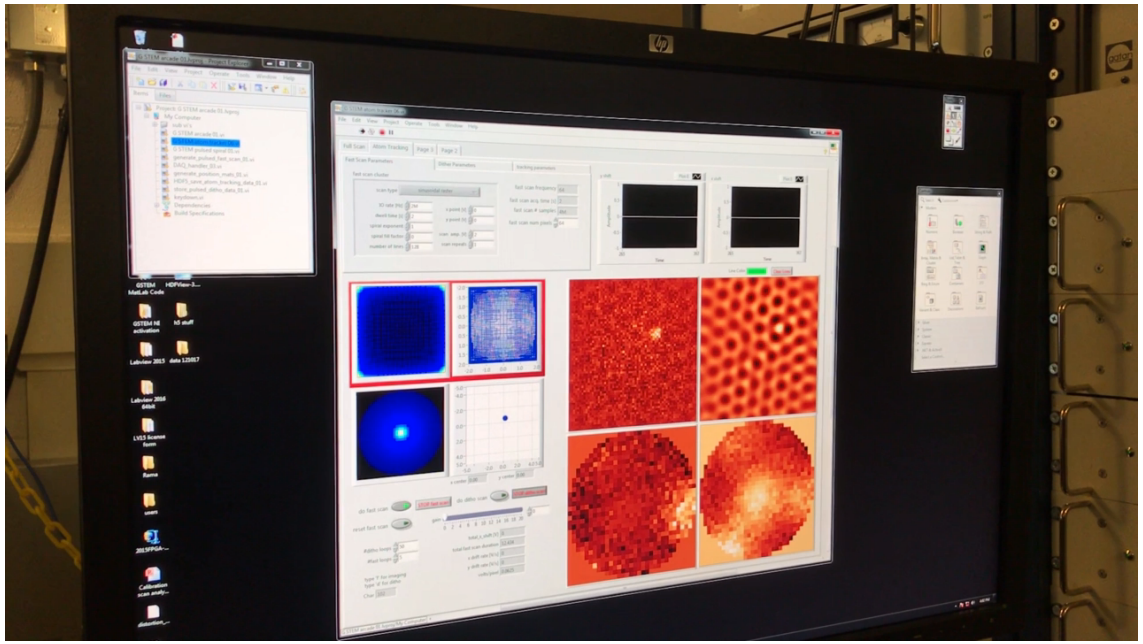
Moving Si dopants



Assembling primitive structures



Custom platform for dopant atom movement with real-time image updates during manipulation

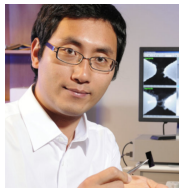


Ondrej Dyck

Stephen Jesse

Elisa Jimenez-Izal

Anastasia N. Alexandrova



Songkil Kim

Dyck, O., Kim, S., Jimenez-Izal, E., Alexandrova, A. N., Kalinin, S. V. & Jesse, S. Building Structures Atom by Atom via Electron Beam Manipulation. *Small* **14**, 1801771, doi:doi:10.1002/smll.201801771 (2018).

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STEM: The Lab on the Beam

What I cannot create, I do not understand
R. Feynman

X-ray and neutron scattering: where the atoms are on average

Electron and probe microscopy: where exactly are the atoms

Dynamic microscopies: what the atoms do

- Nanotechnology
- Beyond Moore
- Molecular machines
- Materials design

Need to find out :

- What are local atomic functionalities
- Why do atoms do it ?

- How we direct them to do what we want ?

Time

Present

Concluding:

- Data matters (but only if physics and chemistry is analyzed!)
- Building physics and chemistry from atomic level up
- E-beam atomic fabrication

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See cnms.ornl.gov for more information.

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Connect!

YouTube

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