H2020-MSCA ITN
Grant n. 956099

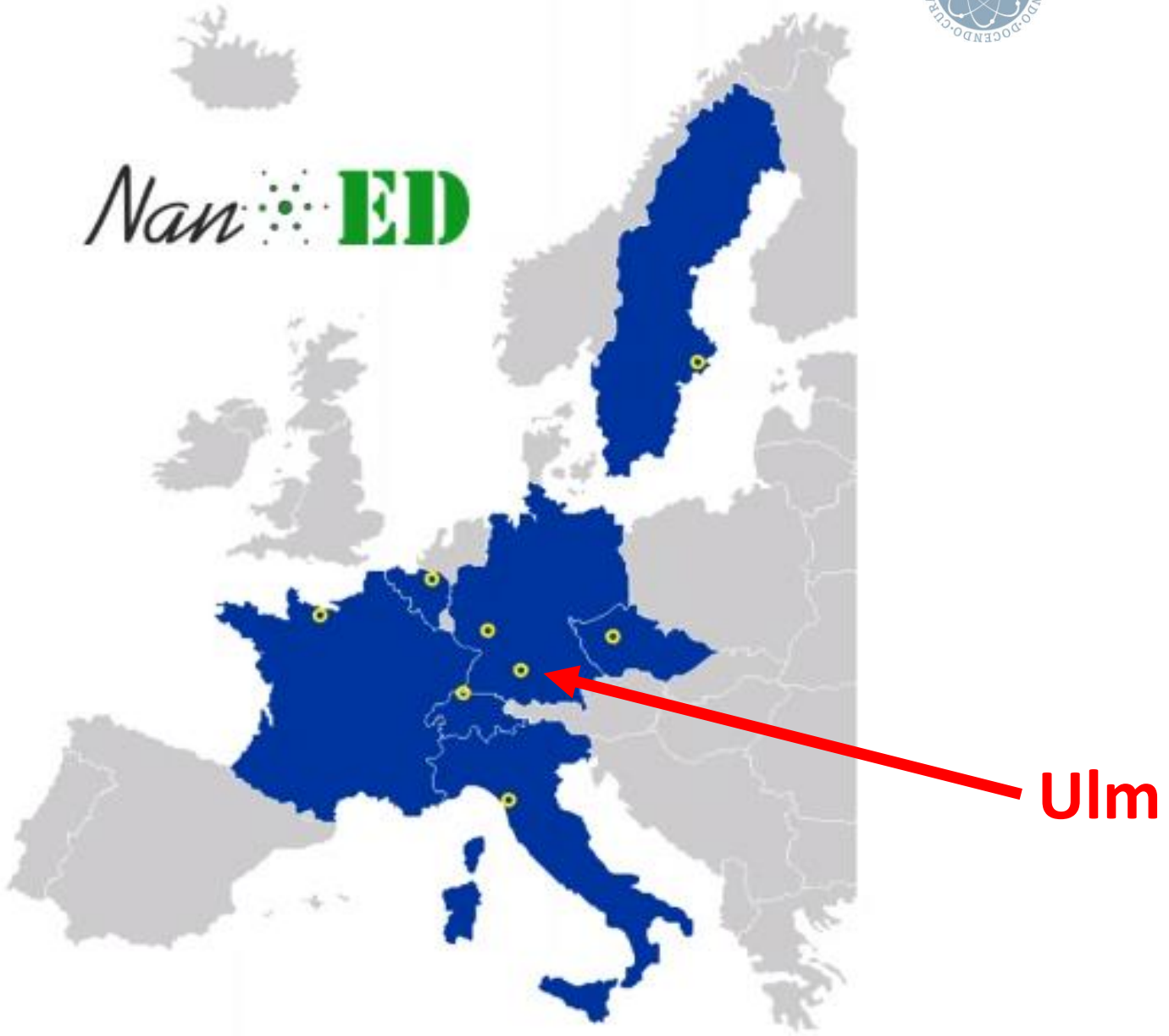
WP4 Crystallography beyond nanocrystals

Research theme ESR6

Tatiana Gorelik, Ute Kaiser (Ulm University)

Ute Kaiser
2D materials, AC-HRTEM

Tatiana Gorelik
3D electron diffraciton


NanoED
Ulm

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UUlm group – People

Materials Science Electron Microscopy (U. Kaiser)

2 Professors

1 Scientific staff position

3 Technical assistants

9 PostDocs

7 PhD students

3 Master students

2009-2018

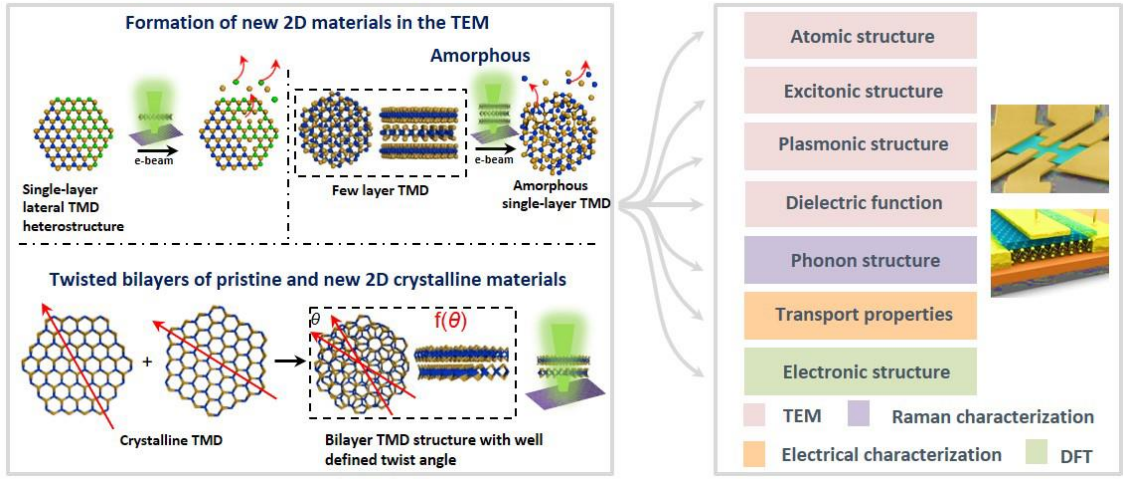
- The Instrument
  CEOS Cc/Cs quadrupole/octupole corrector
  FEI Titan Themis³ column
  Gatan Quantum GIF
  20, 30, 40, 60, and 80 kV
- Imaging theory at low voltages

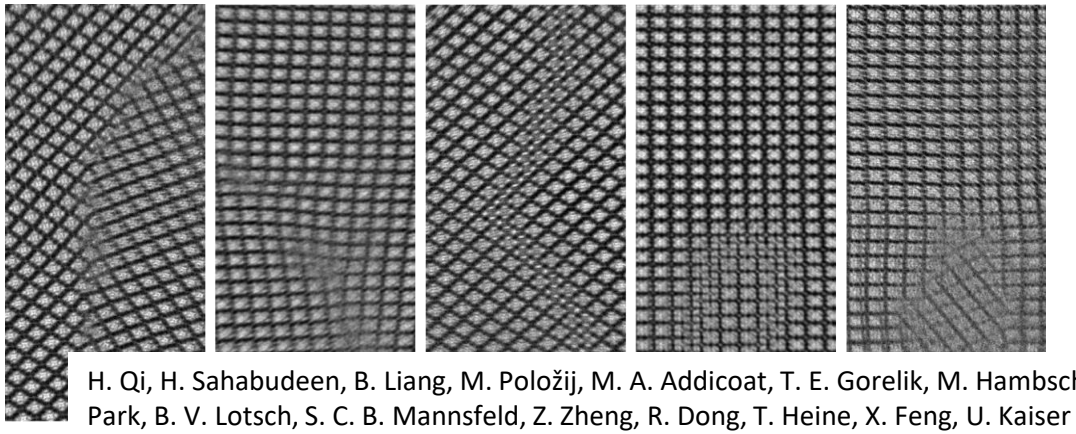
HV kV	wavelength pm	measured resol. pm	resolution wavelengths
SALVE			
80	4.2	76	18
60	4.9	83	17
40	6.0	90	15
30	7.0	115	16.5
20	8.6	139	16
FEI Titan			
80	4.2	180	43

https://www.uni-ulm.de/en/einrichtungen/emms/ and www.salve-project.de

Research Topics

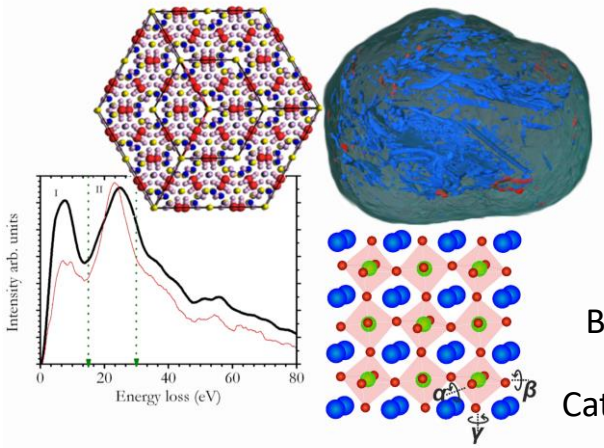
Inorganic low-dimensional materials





H. Qi, H. Sahabudeen, B. Liang, M. Položij, M. A. Addicoat, T. E. Gorelik, M. Hambsch, M. Mundszinger, S. Park, B. V. Lotsch, S. C. B. Mannsfeld, Z. Zheng, R. Dong, T. Heine, X. Feng, U. Kaiser
Near–atomic-scale observation of grain boundaries in a layer-stacked two-dimensional polymer
Science Advances 6 (2020), eabb5976

3D materials



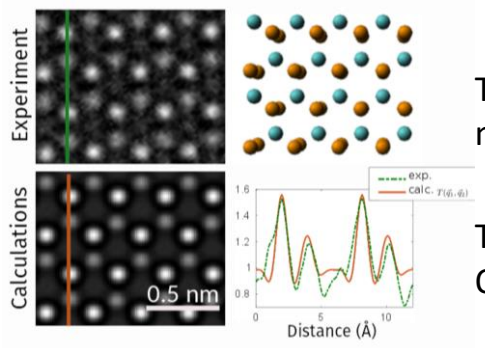
Rare-earth nickelate superlattices

LED materials

Battery materials

Catalysis

Low-voltage Imaging Theory



Transmission electron microscopy instrumentation

Theory of image formation
Quantitative Fit

Research Methods

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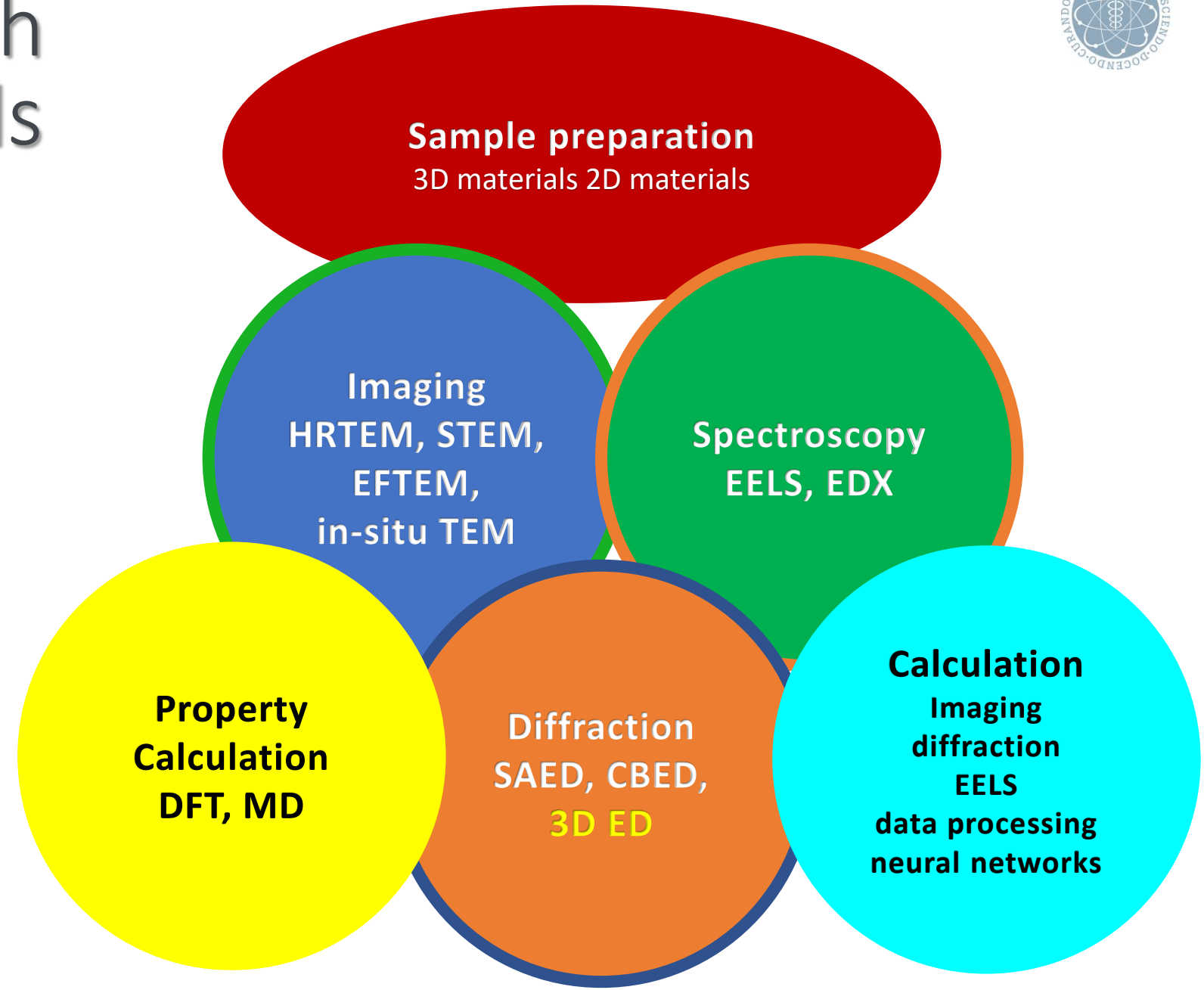


Instrumentation TEMs

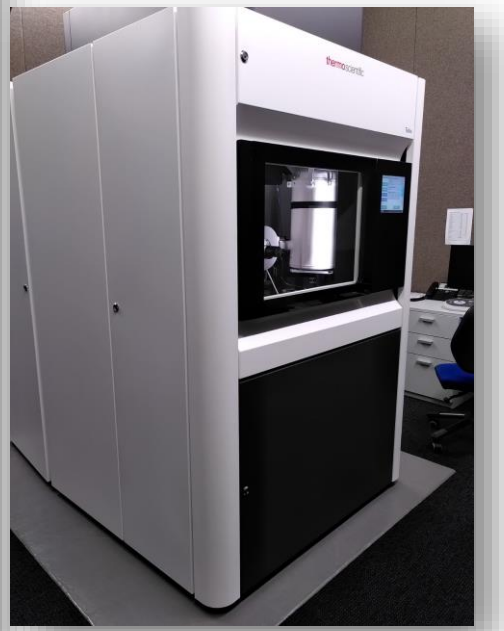
- SEM/FIB Nvision
  Ga ion beam (2 - 30 kV)
  Electron beam (0.5 - 30 kV)
- SALVE 20 – 80
  Monochromator and low voltage energy filter for spectroscopy and energy filtered TEM.
  Dedicated low voltage corrector for geometric and chromatic aberrations
- FEI Titan 80-300
  HRTEM, STEM, EELS, EnergyFiltered TEM, Tomography, Electron Holography, Lorentz microscopy
- Thermofisher TALOS 200x
  (TEM, HRTEM, STEM with SuperX EDX detector)

SEM/FIB NVision

Cs-corrected TEM TITAN

TEM SALVE
Cs/Cc corrected
20 - 80 kV

Thermofisher
TALOS 200x



Instrumentation Sample Prep

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Lab for 3D Materials

- 3 Plasma cleaners
  TEM sample cleaning
  2 Ion Beam Thinning machine
  Fishione Ion Mill 1010
  Fishione Nanomill 1040

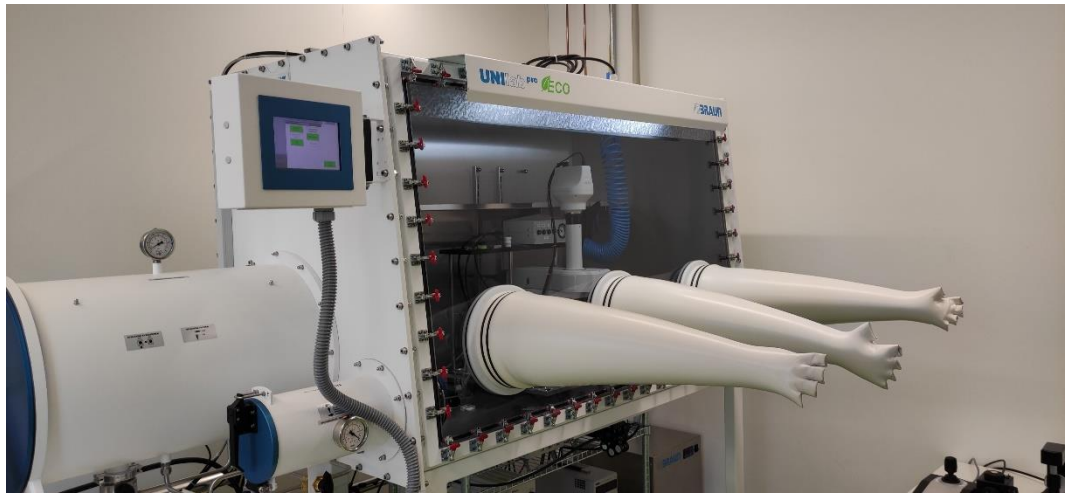
Lab for 2D Materials

- Plunch Freezer
- Glove Boxes
- Light microscopes

2D Sample preparation laboratory

Preparation Laboratory 1: Spincoater and preparation for battery materials

Preparation Laboratory 2: Liquid exfoliation and CVD transfer



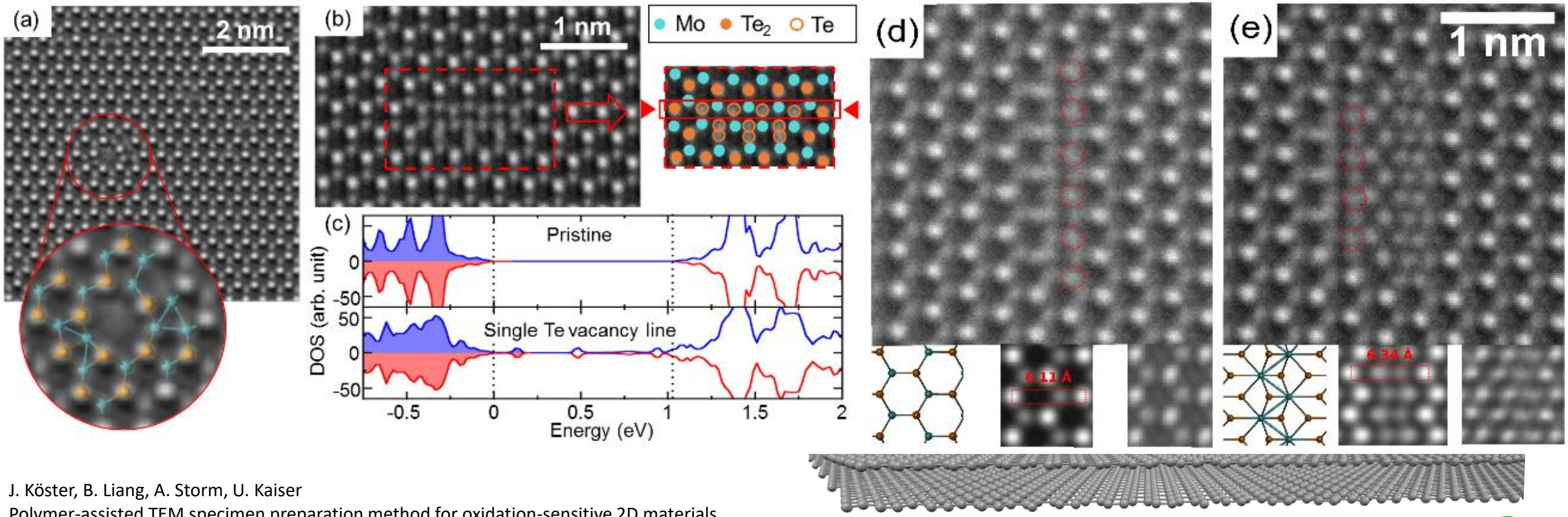
Preparation Laboratory 3: Glovebox for preparation for oxygen sensitive materials (viscoelastic stamping method)

Preparation Laboratory 3: Light microscope for classical 2D preparation (wet etching method)

2D Sample preparation laboratory

From 2D direct and spectroscopic space to 3D reciprocal space of 2D materials

40kV SALVE $MoTe_2$



J. Köster, B. Liang, A. Storm, U. Kaiser
Polymer-assisted TEM specimen preparation method for oxidation-sensitive 2D materials
Nanotechnology 32 (2021) 075704
T. Lehnert, M. Ghorbani-Asl, J. Köster, Z. Lee, A. V. Krasheninnikov, and U. Kaiser
Electron-Beam-Driven Structure Evolution of Single-Layer MoTe2 for Quantum Devices
ACS Appl. Nano Mater. 2 (2019) 3262-3270

WP4 Crystallography beyond nanocrystals

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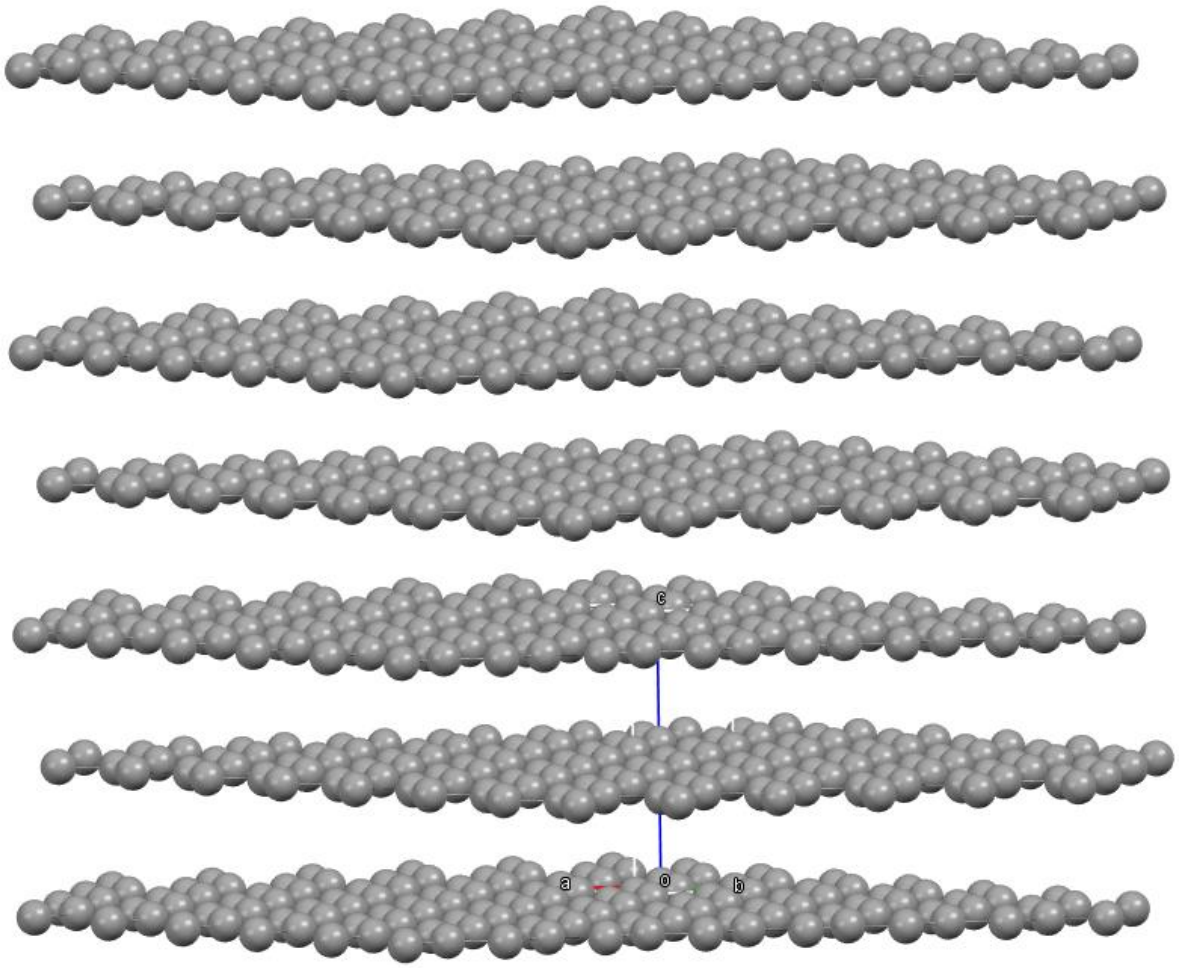
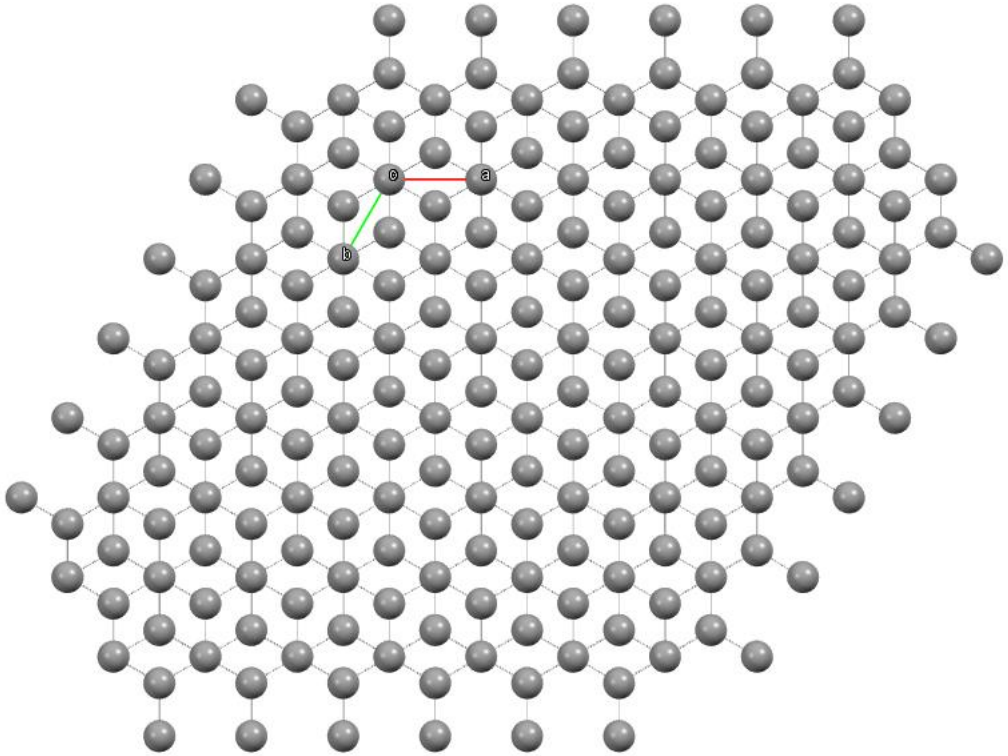
- **ESR6 Exploring 3D reciprocal space of 2D crystals**
- ***ESR7*** *Structure of poorly crystalline materials from their electron total scattering data (ePDF)*

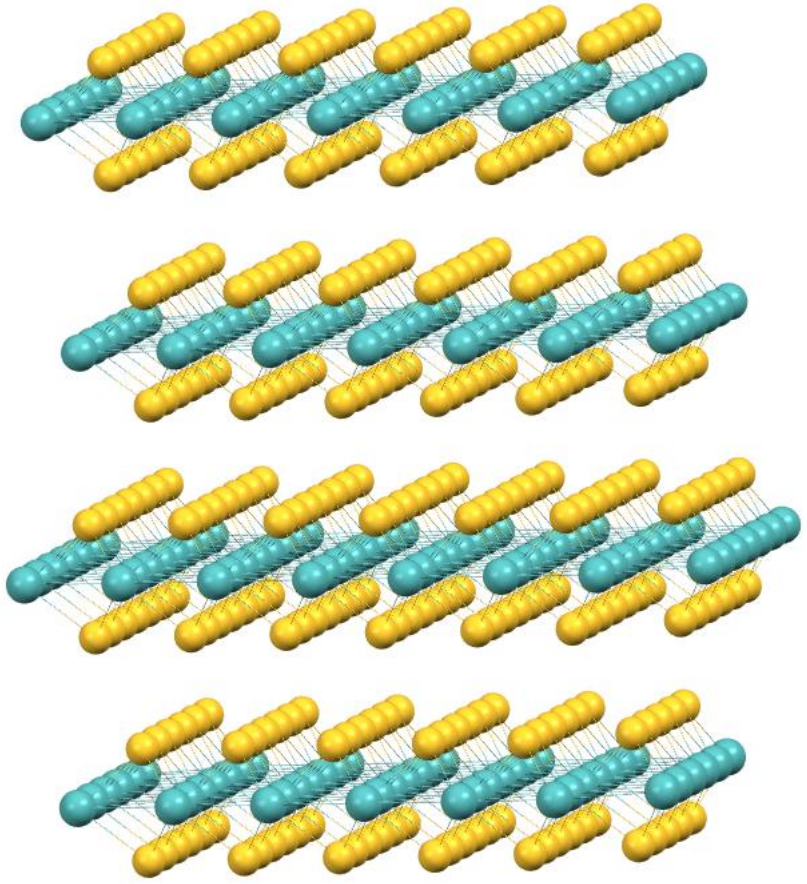
-> Stockholm

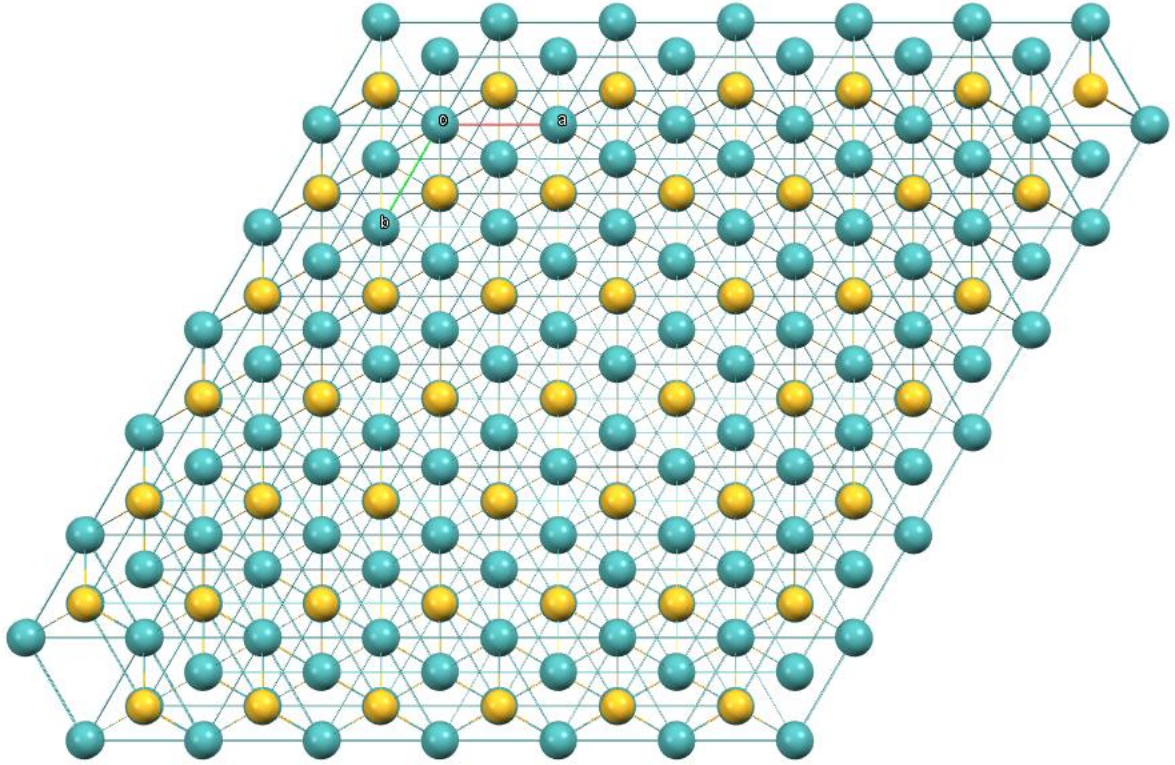
Layered materials: graphite



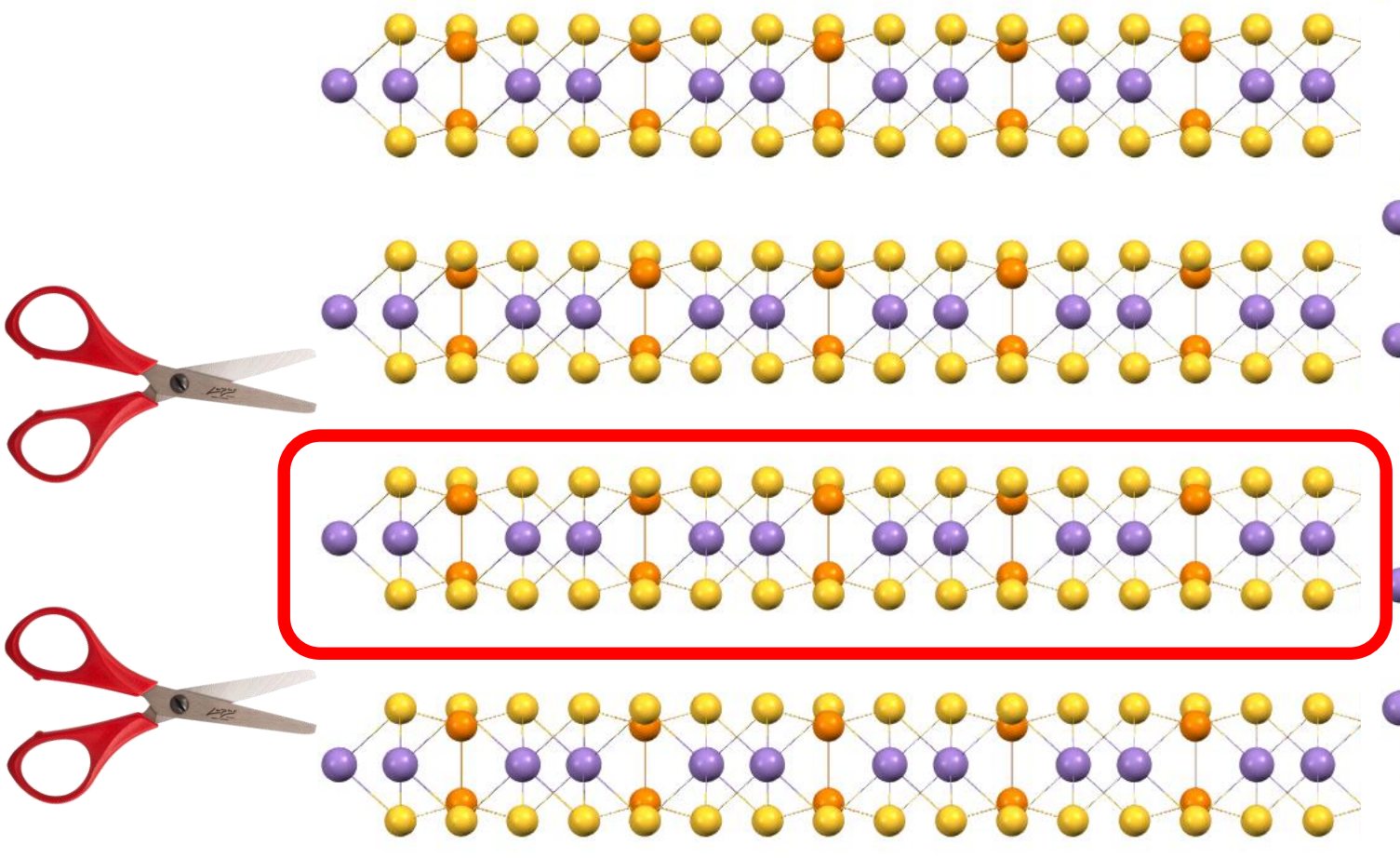


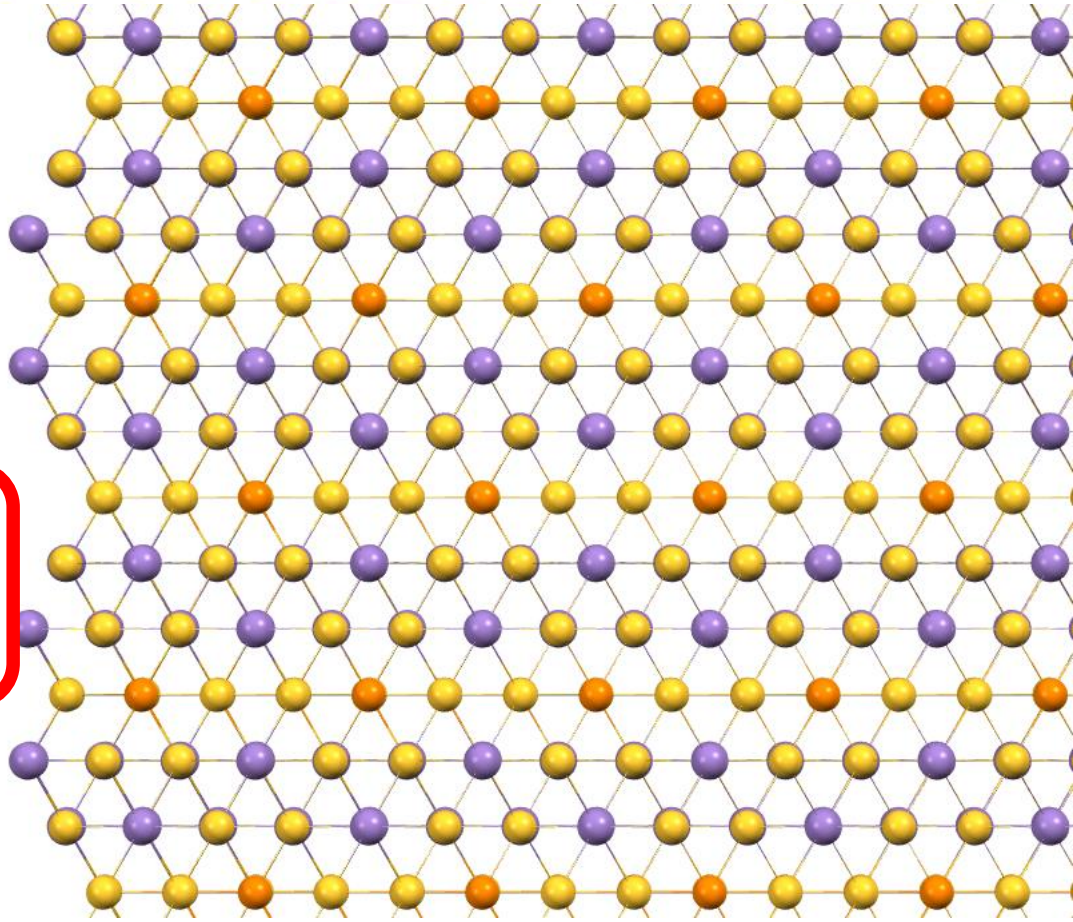
Layered materials: MoS_2 (TMDs)



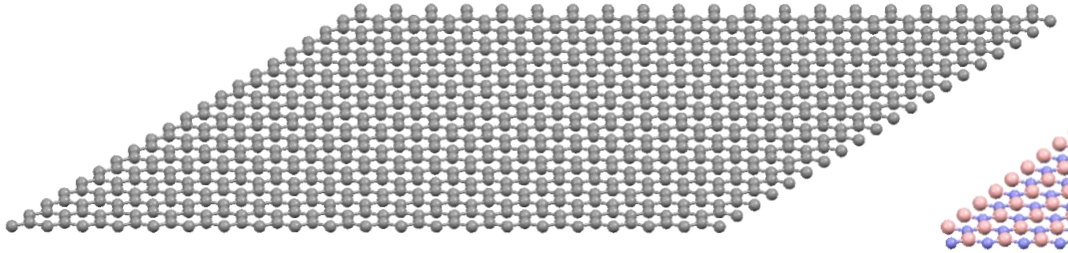


Layered materials: $MnPS_3$ (TMPTs)

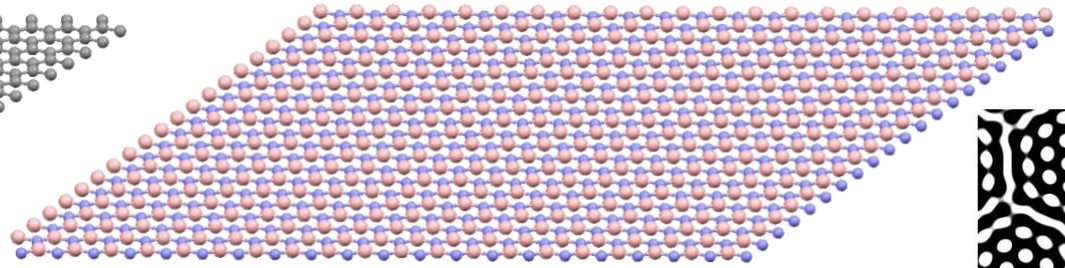




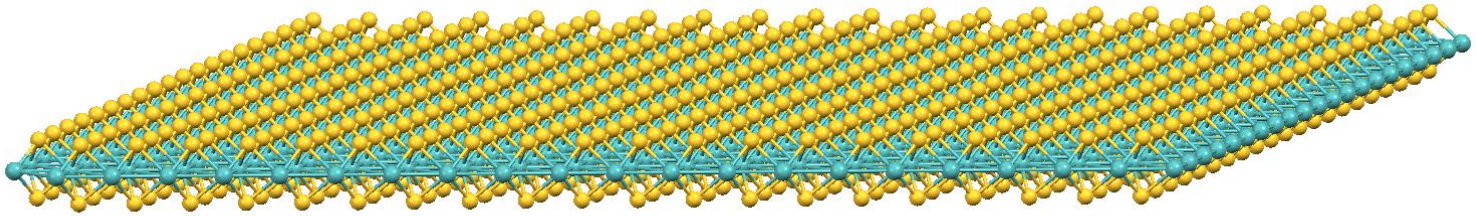
2D crystals



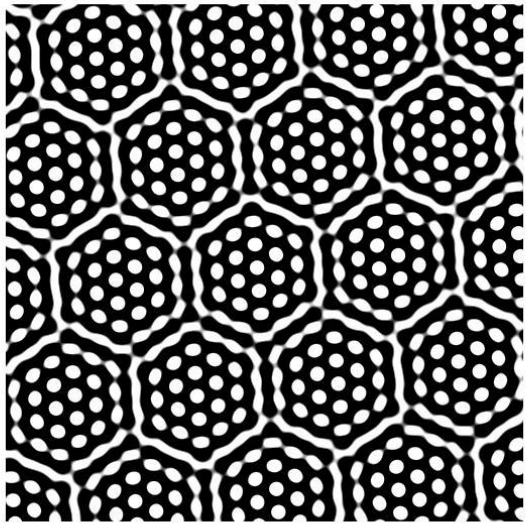
graphene



BN



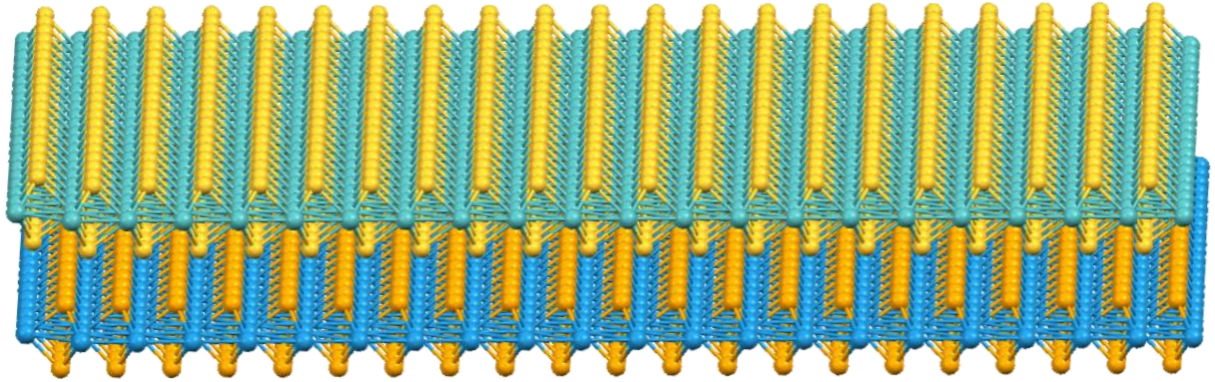
MoS_2



Two graphene sheets 10° twist

Diverse 2D materials and their heterostructures:

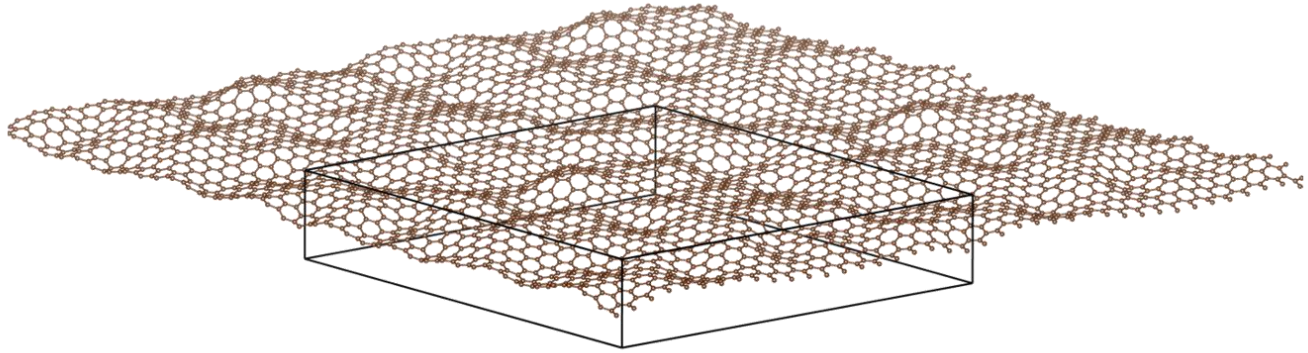
- The same materials twisted
- Different materials (different lattice costants) stacked
- Different materials twisted, …



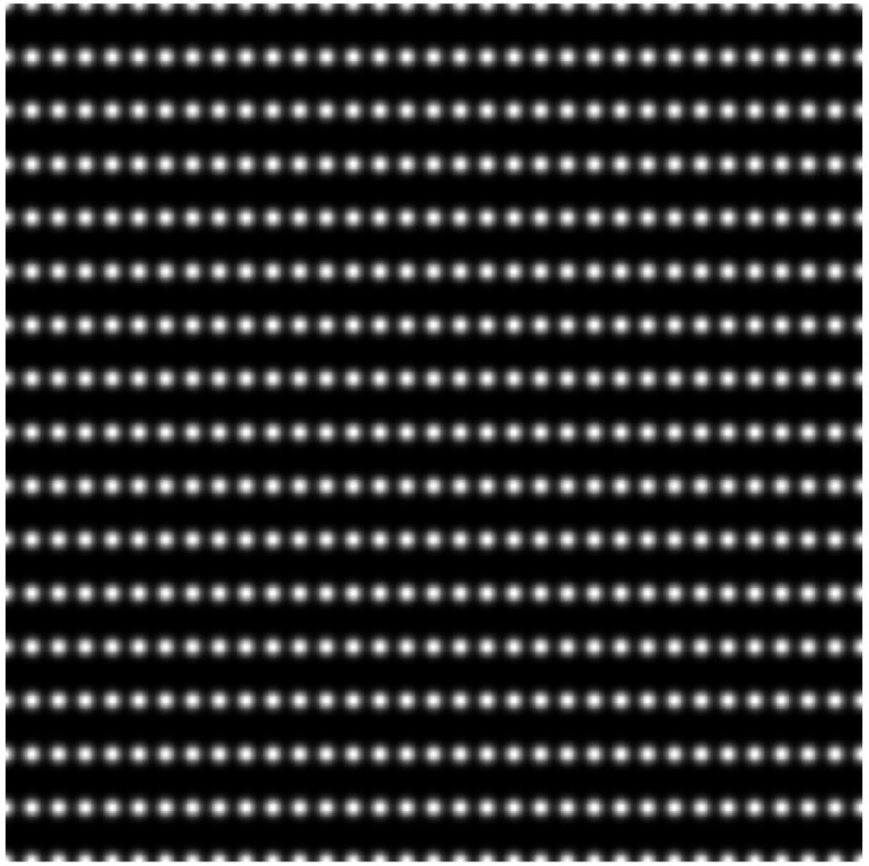
What happens to a crystal structure when it is

absolutely free

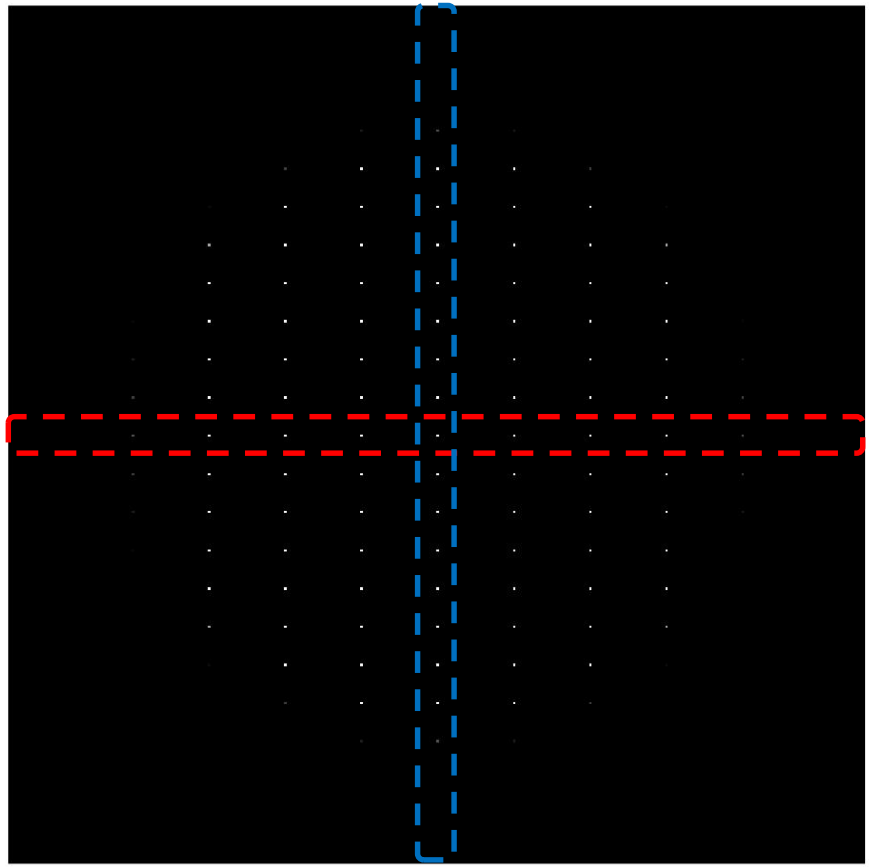
in one direction



2D crystals – diffraction space

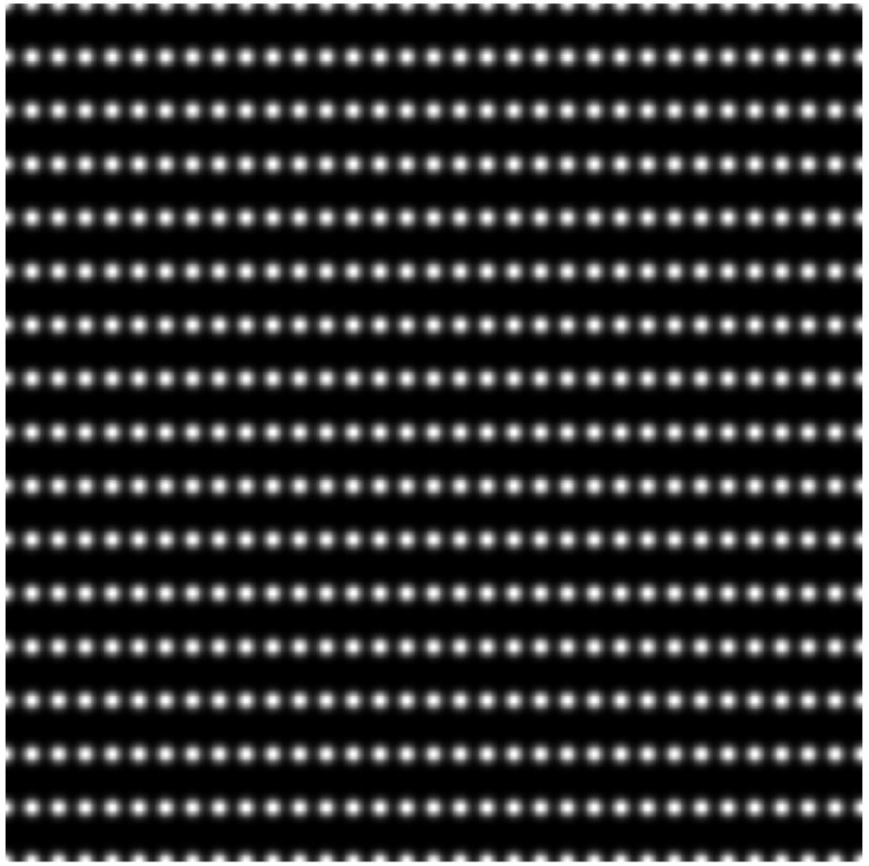


Direct space - crystal

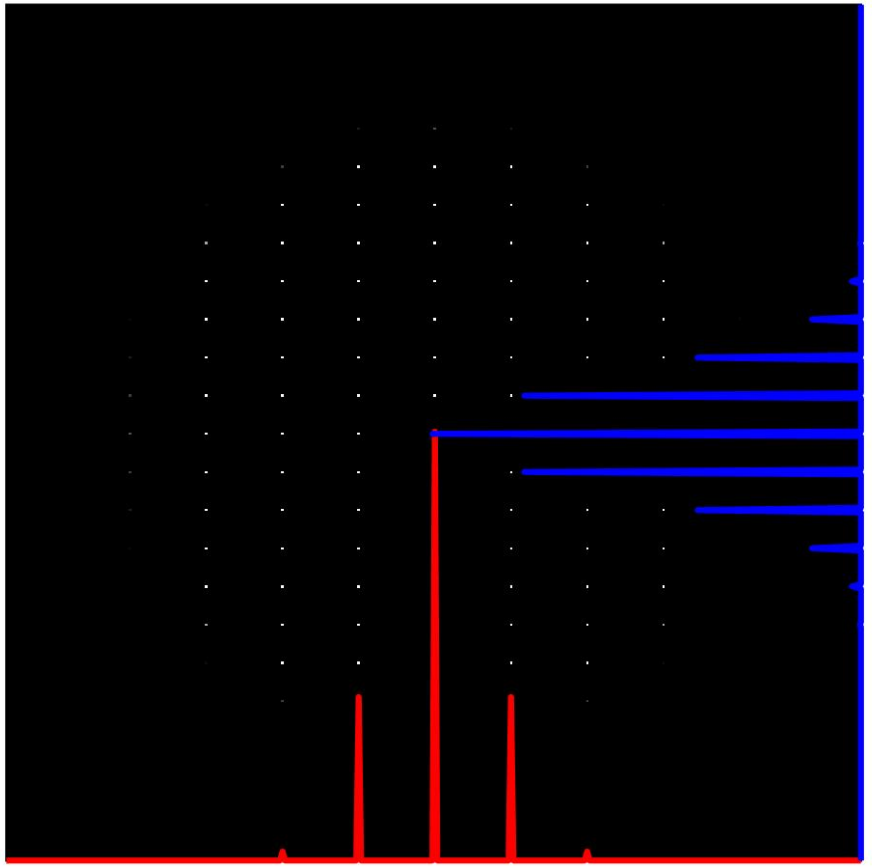


Reciprocal space - diffraction

2D crystals – diffraction space

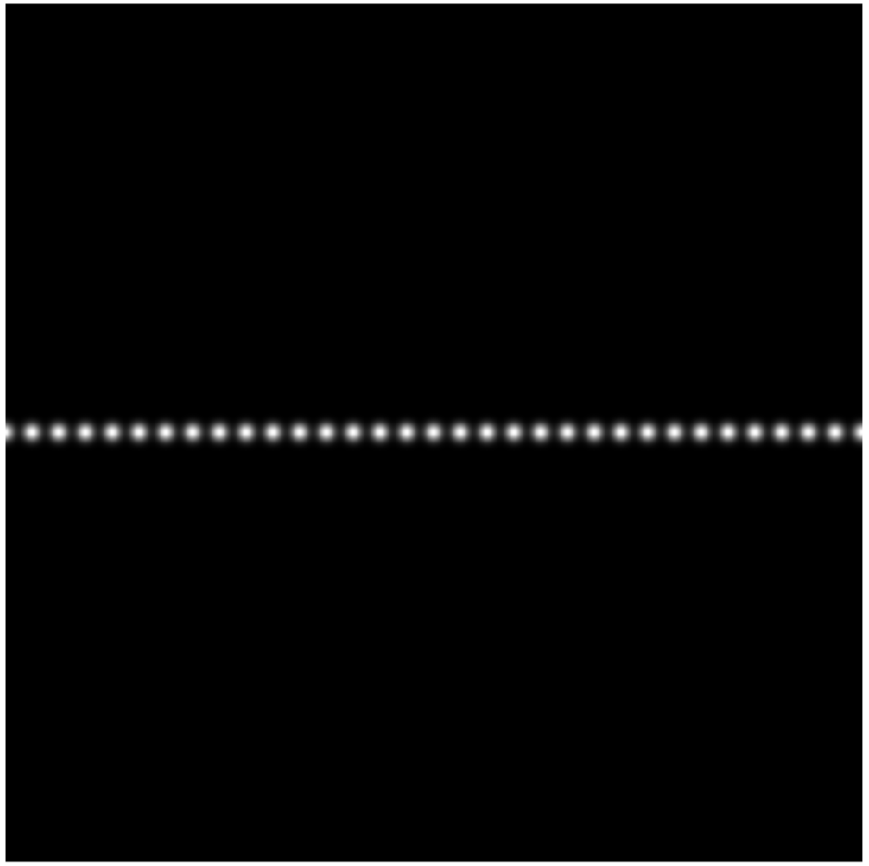


Direct space - crystal

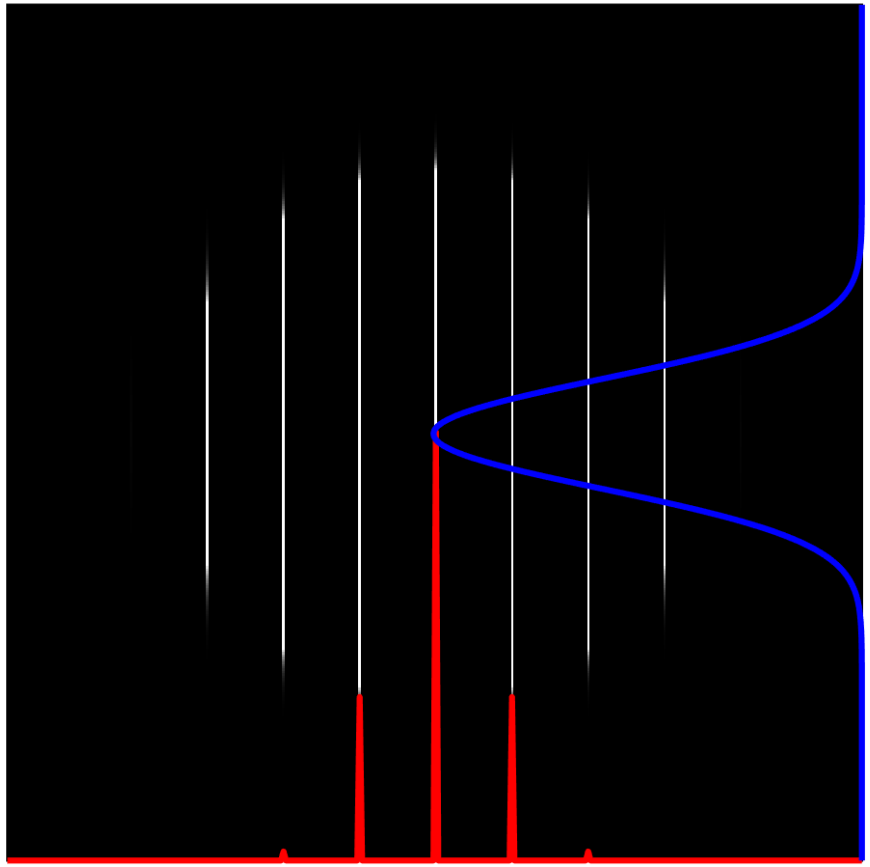


Reciprocal space - diffraction

2D crystals – diffraction space

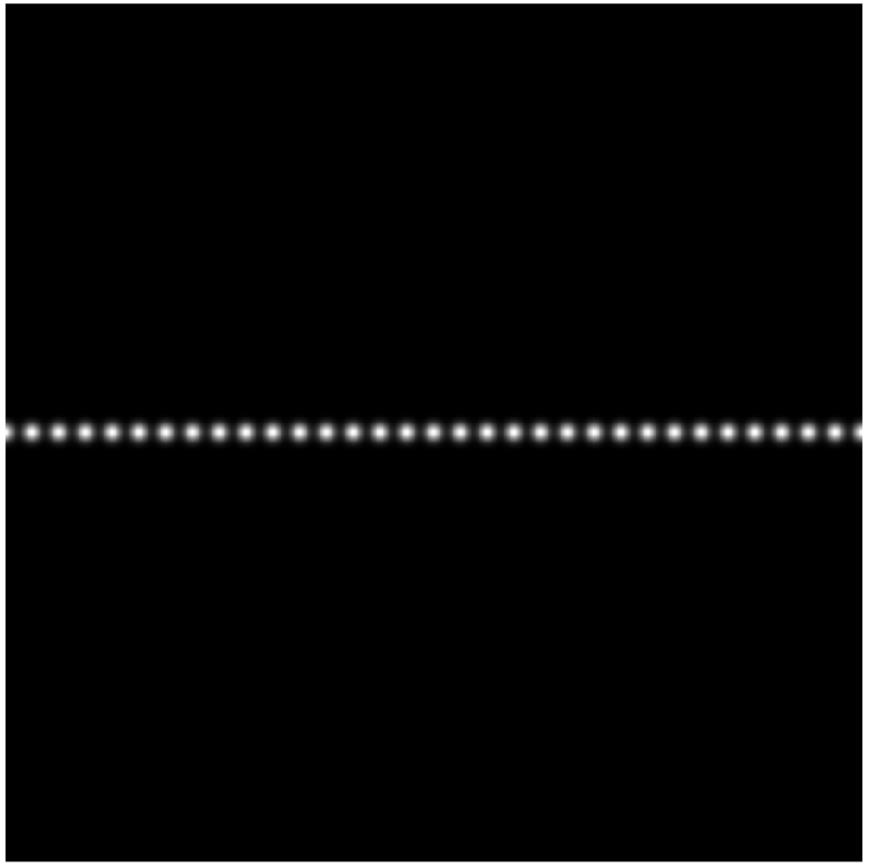


Direct space - crystal

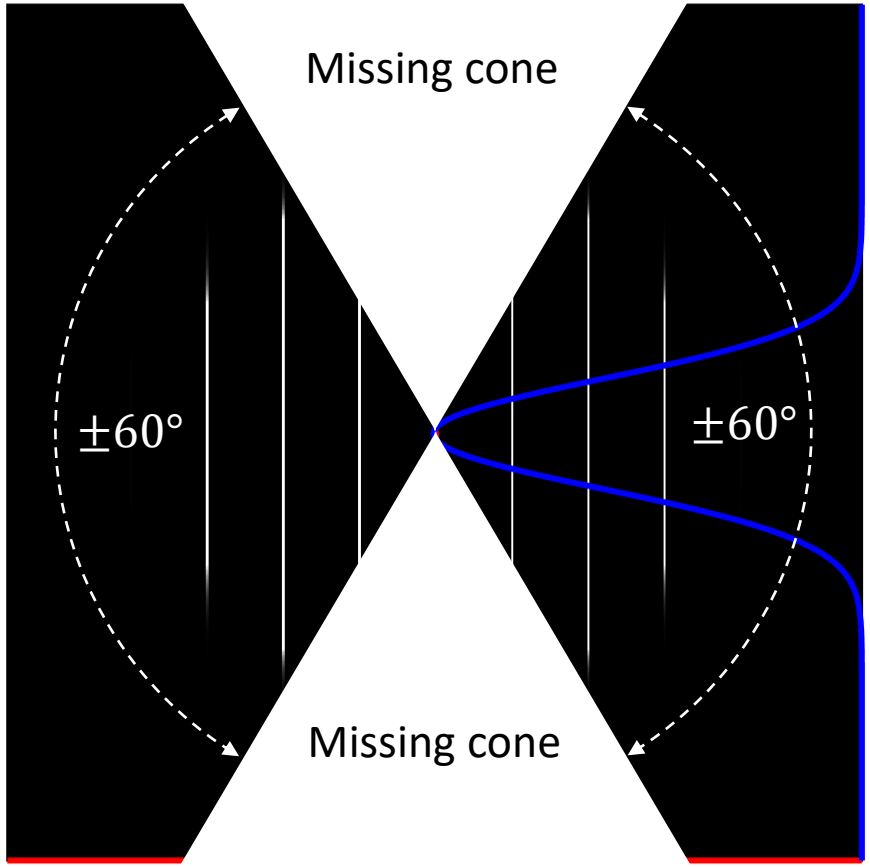


Reciprocal space - diffraction

2D crystals – diffraction space

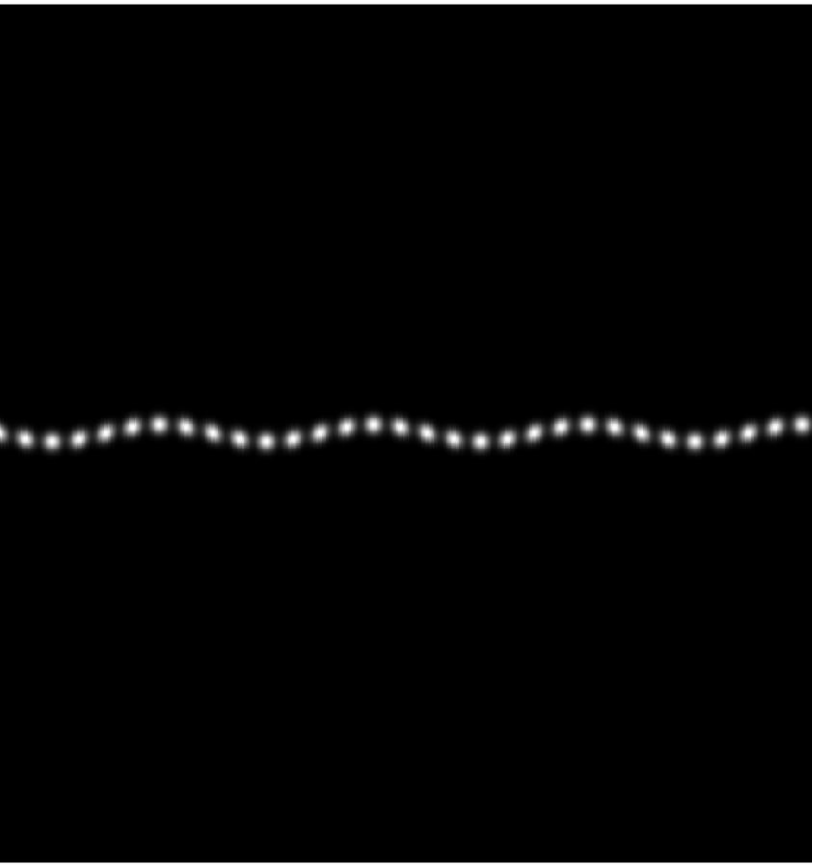


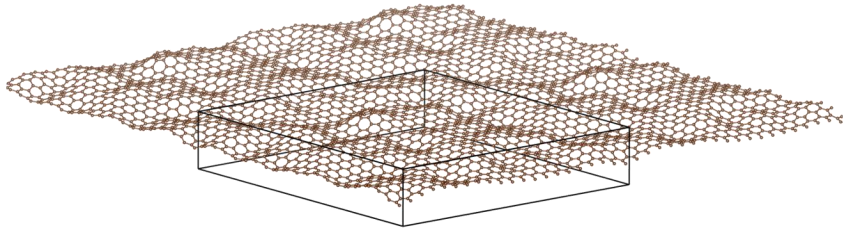
Direct space - crystal

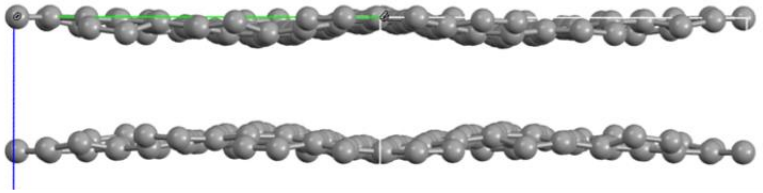


Reciprocal space - diffraction

2D crystals – diffraction space



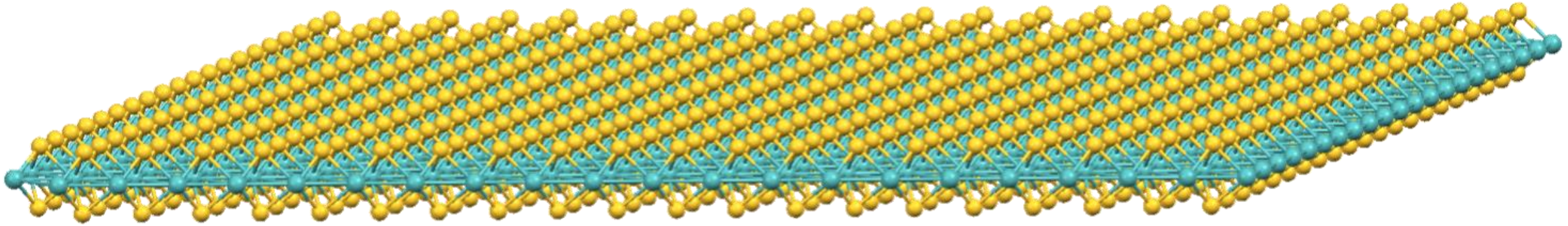




2D crystals – diffraction space

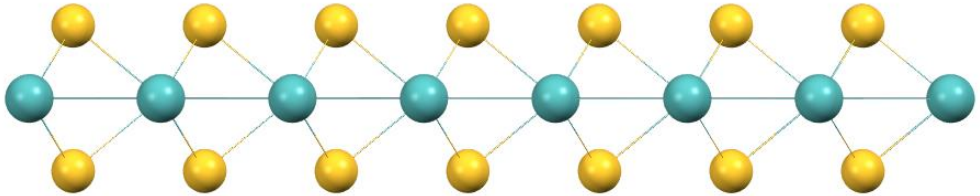
Real data

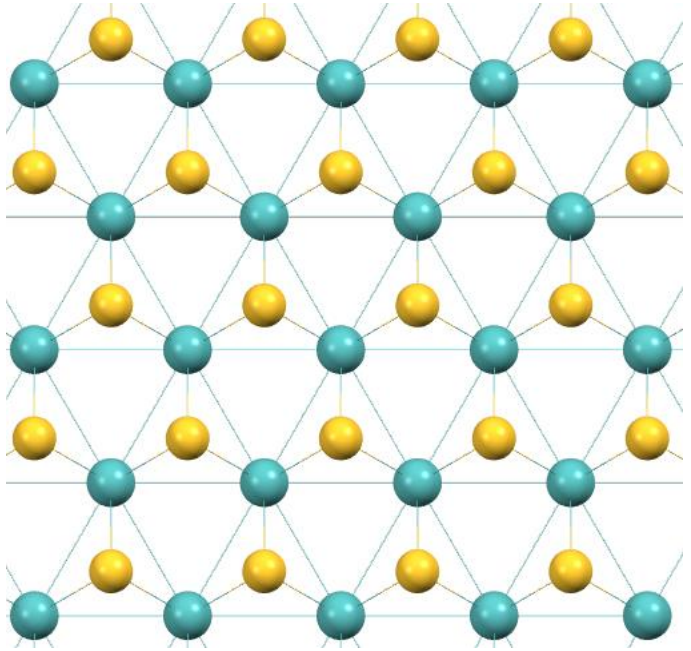
2D crystals – diffraction space



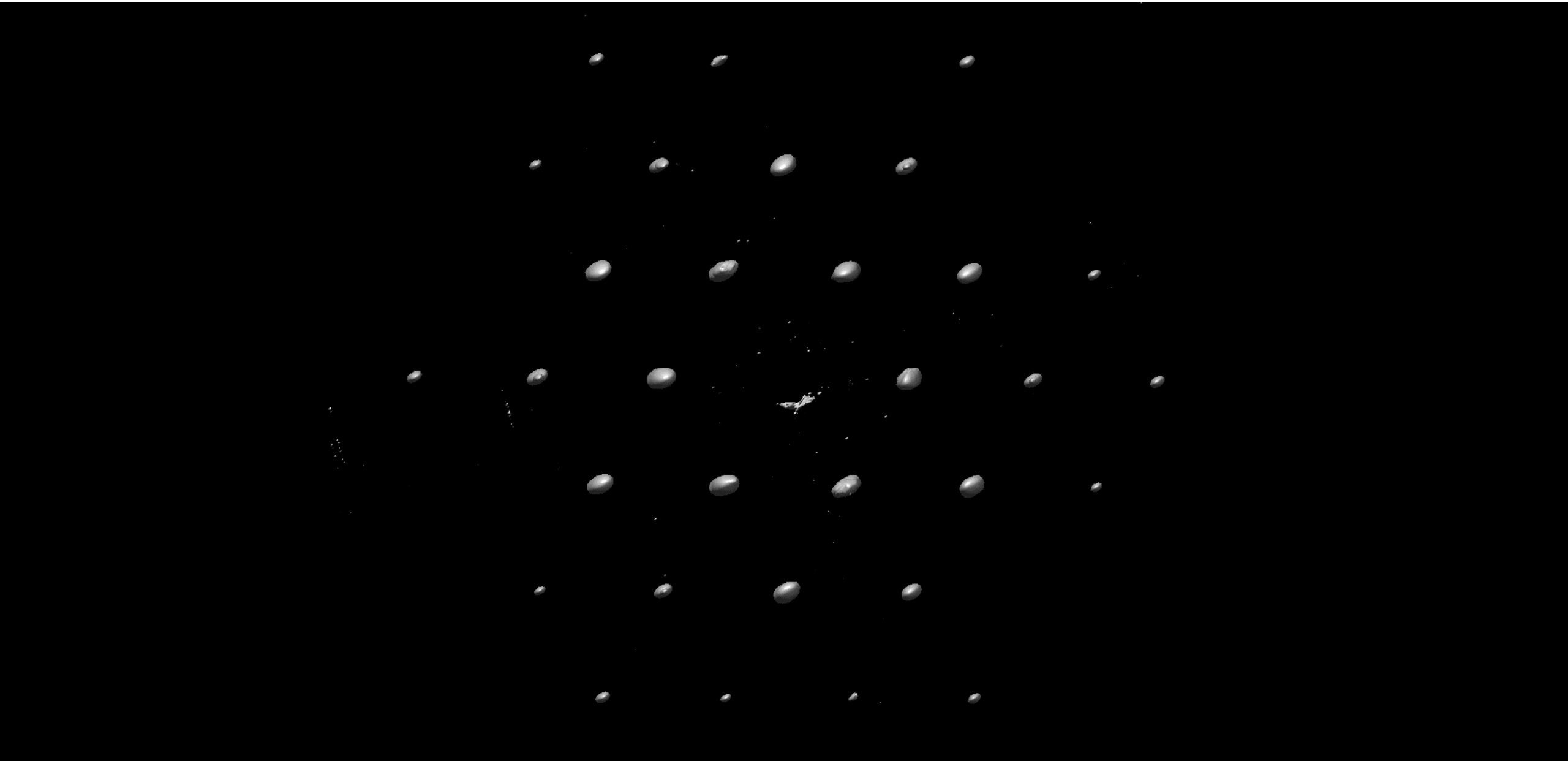
Transition metal di-chalcogenides – TMDs: MoS_2, $MoTe_2$

MoS_2 monolayer,
3 atoms in z

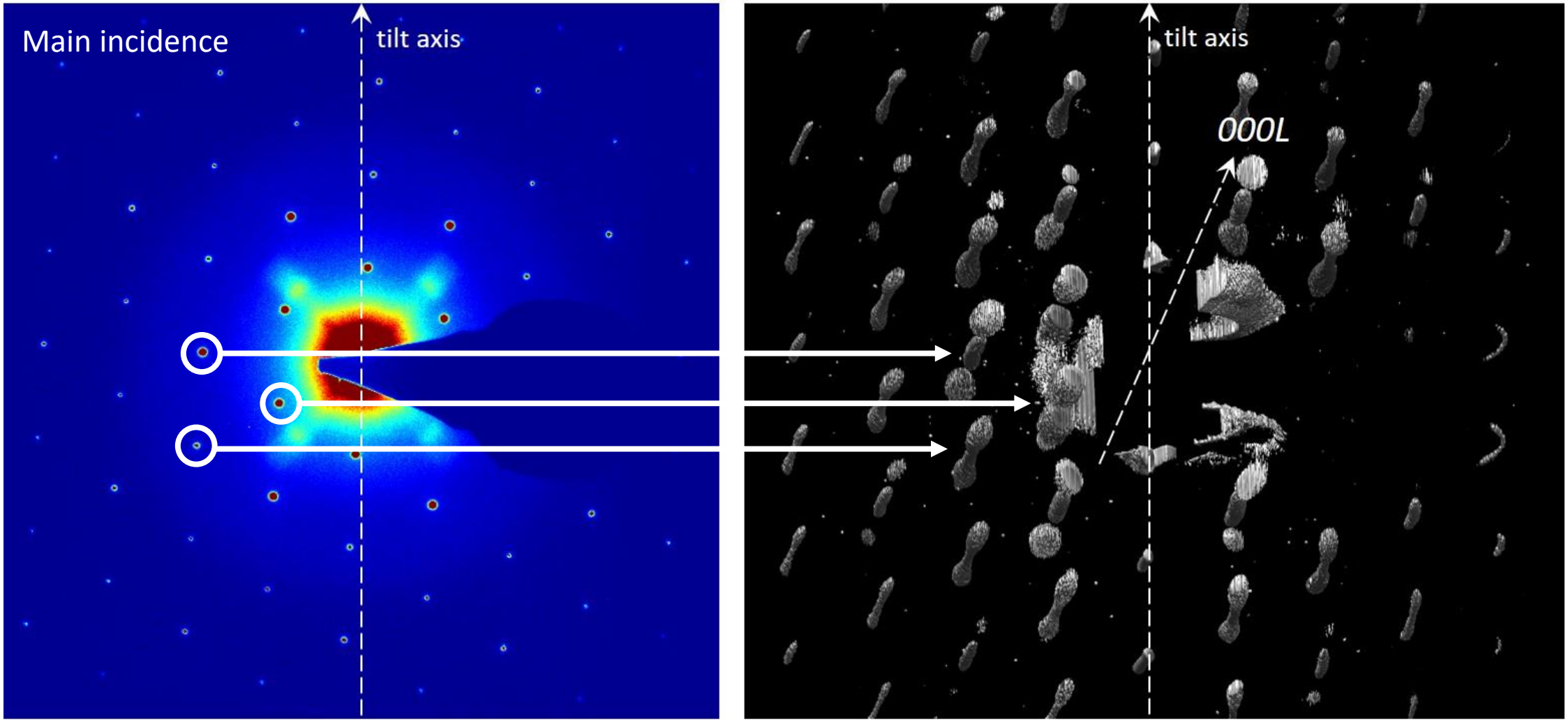




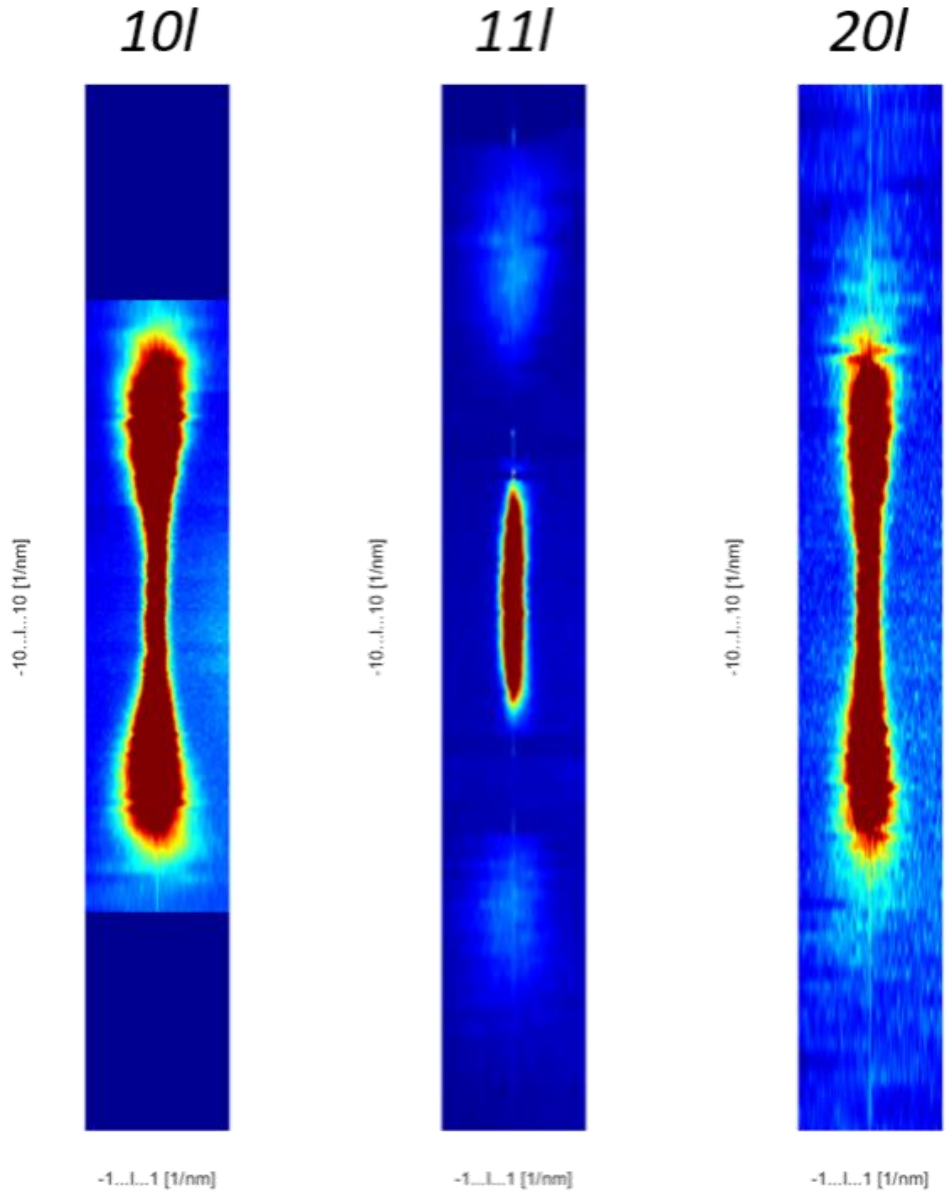
Electron diffuse scattering – crystal size confinement – 3D ED on 2D crystal

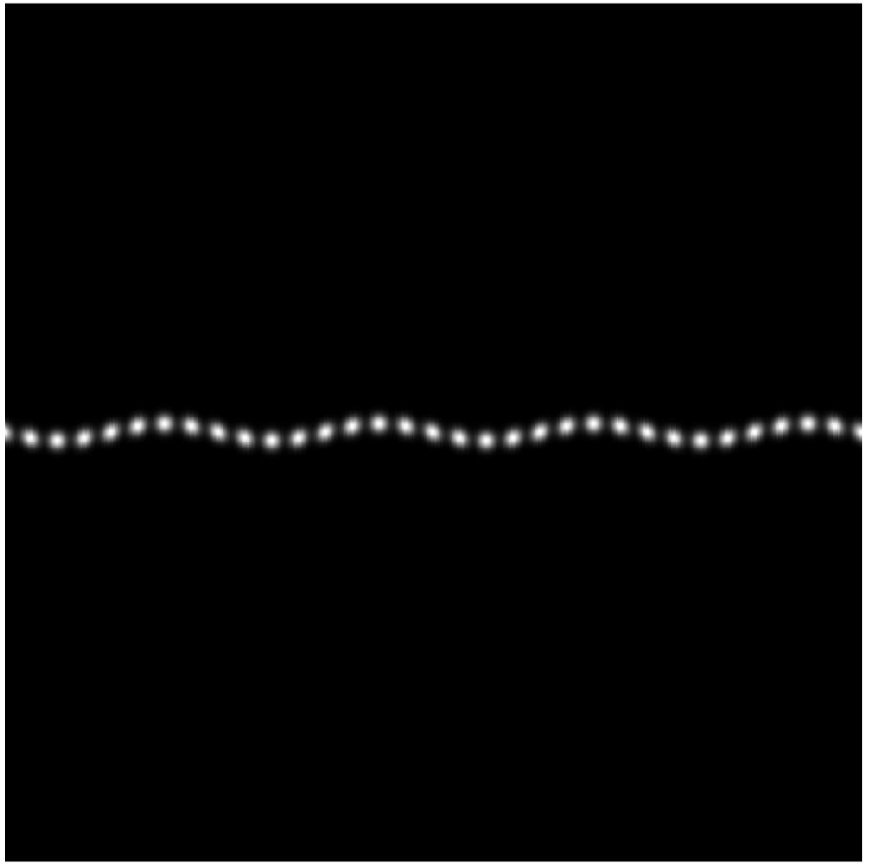


2D crystals – diffraction space

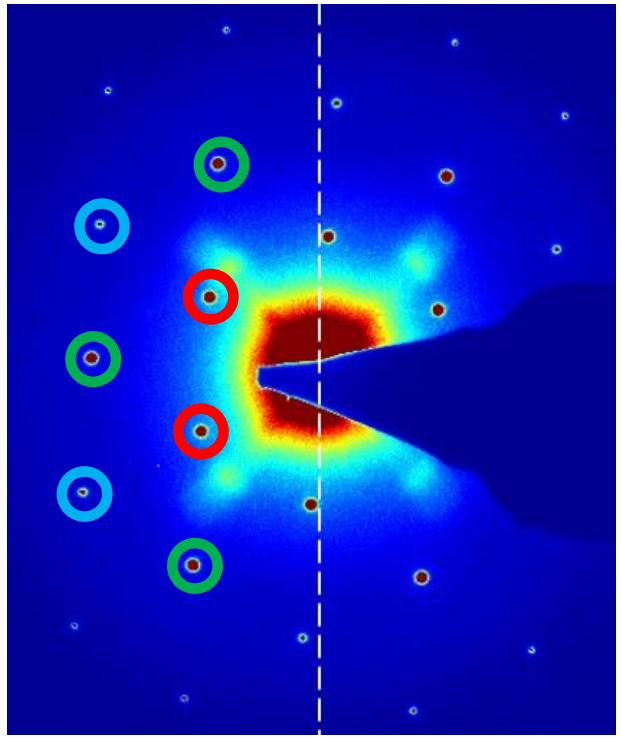


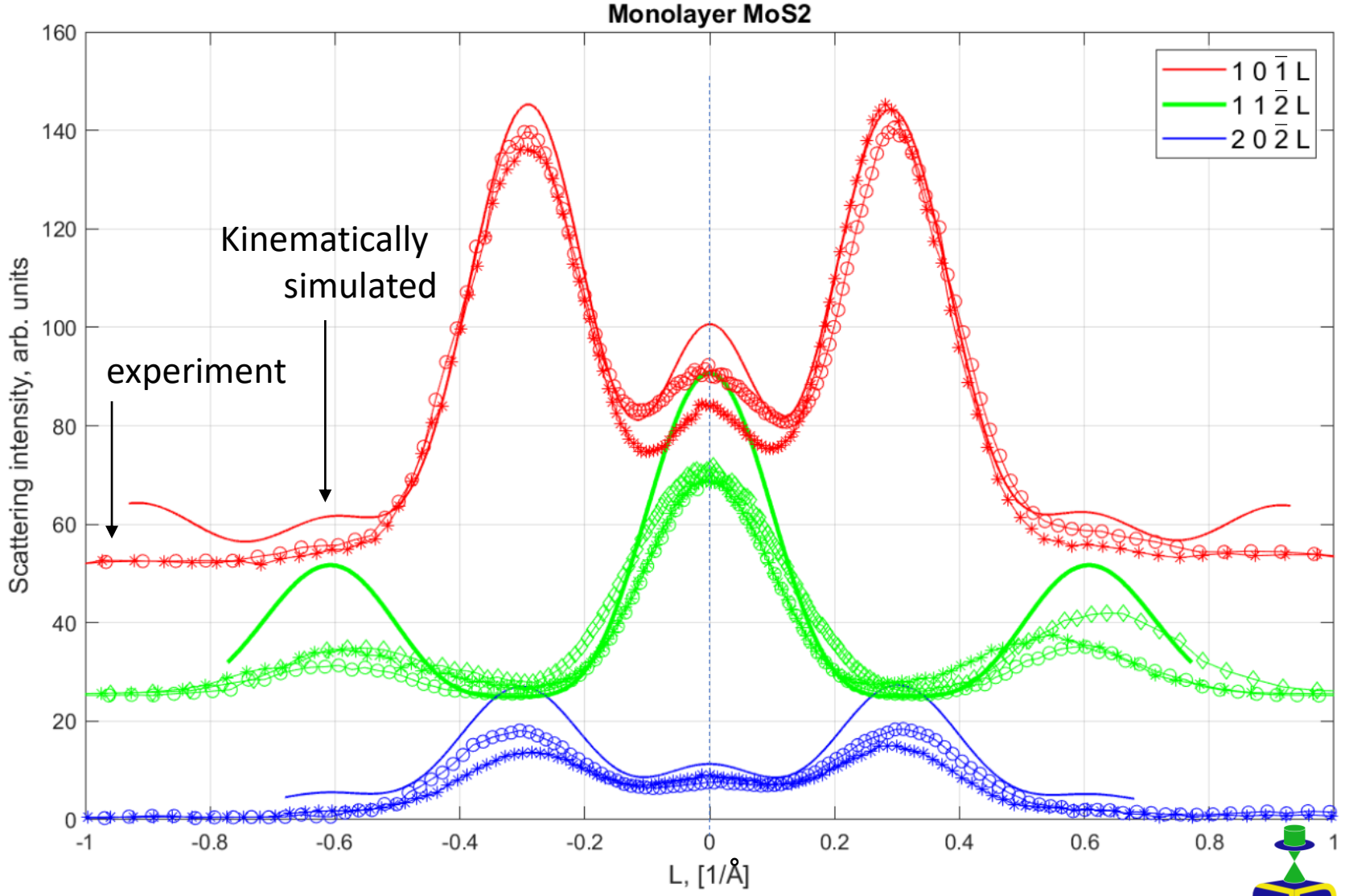
2D crystals – diffraction space





3D ED on 2D MoS_2 crystal





Peak width, symmetry

Gorelik et al., Micron 146 (2021) 103071

3D ED on 2D MoS_2 crystal

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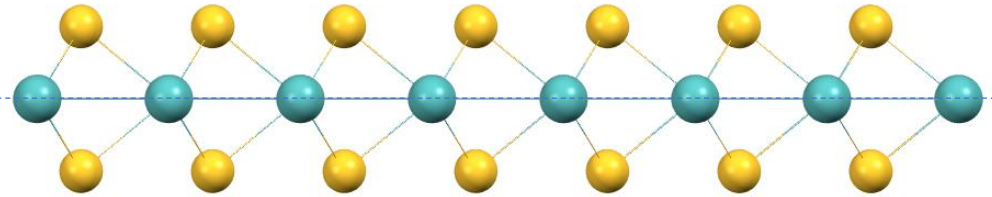
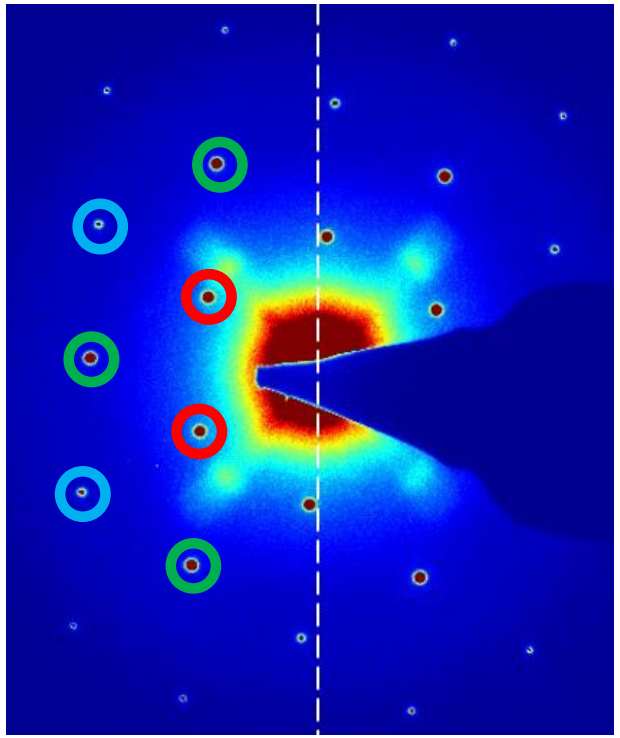
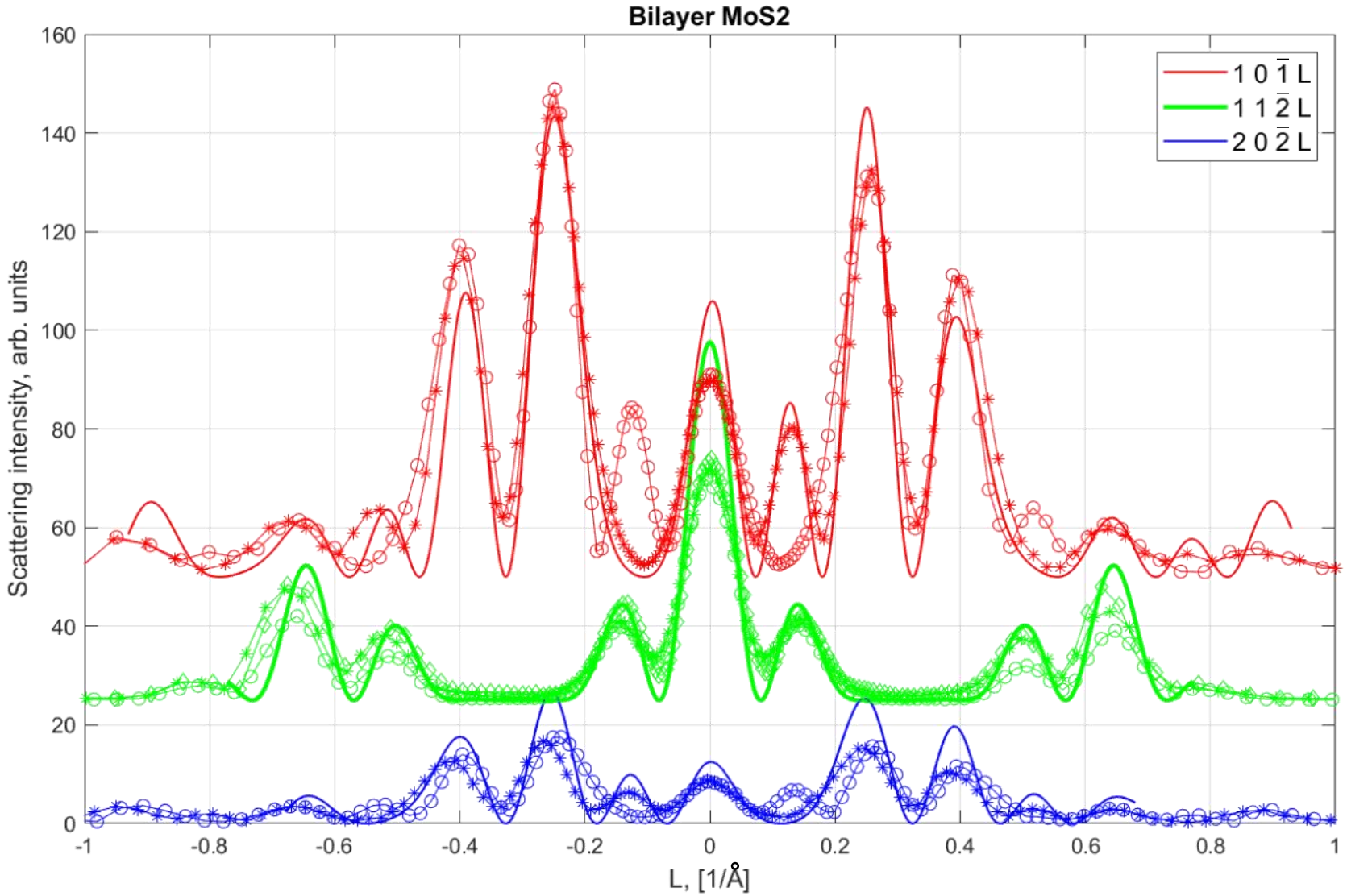


3D ED on 2D MoS_2 crystal





Gorelik et al., Micron 146 (2021) 103071

3D ED on 2D MoS_2 crystal

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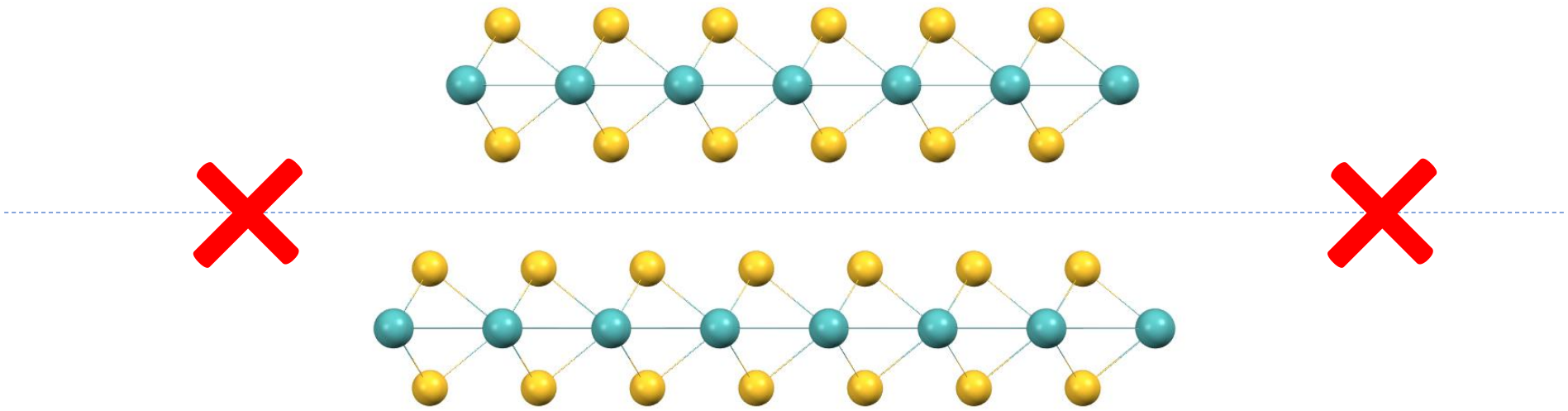
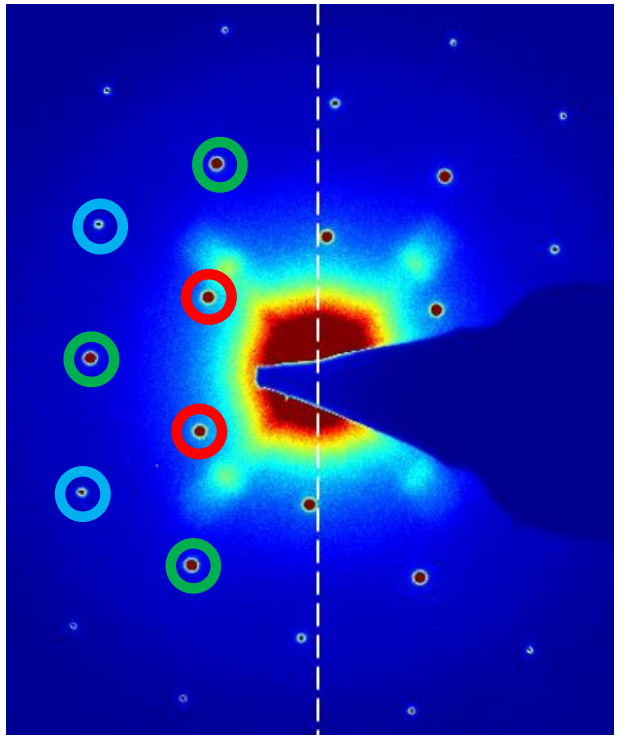
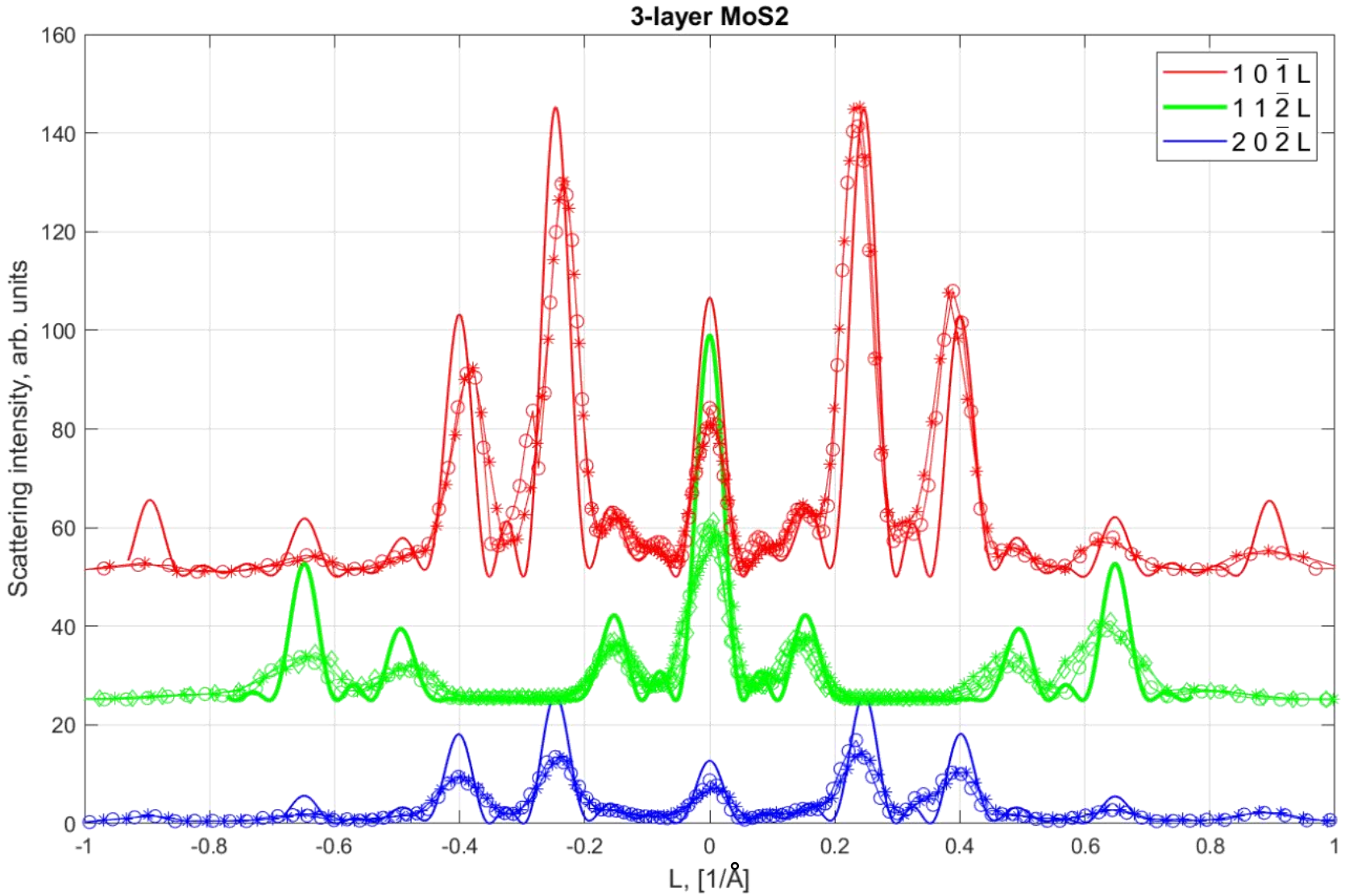


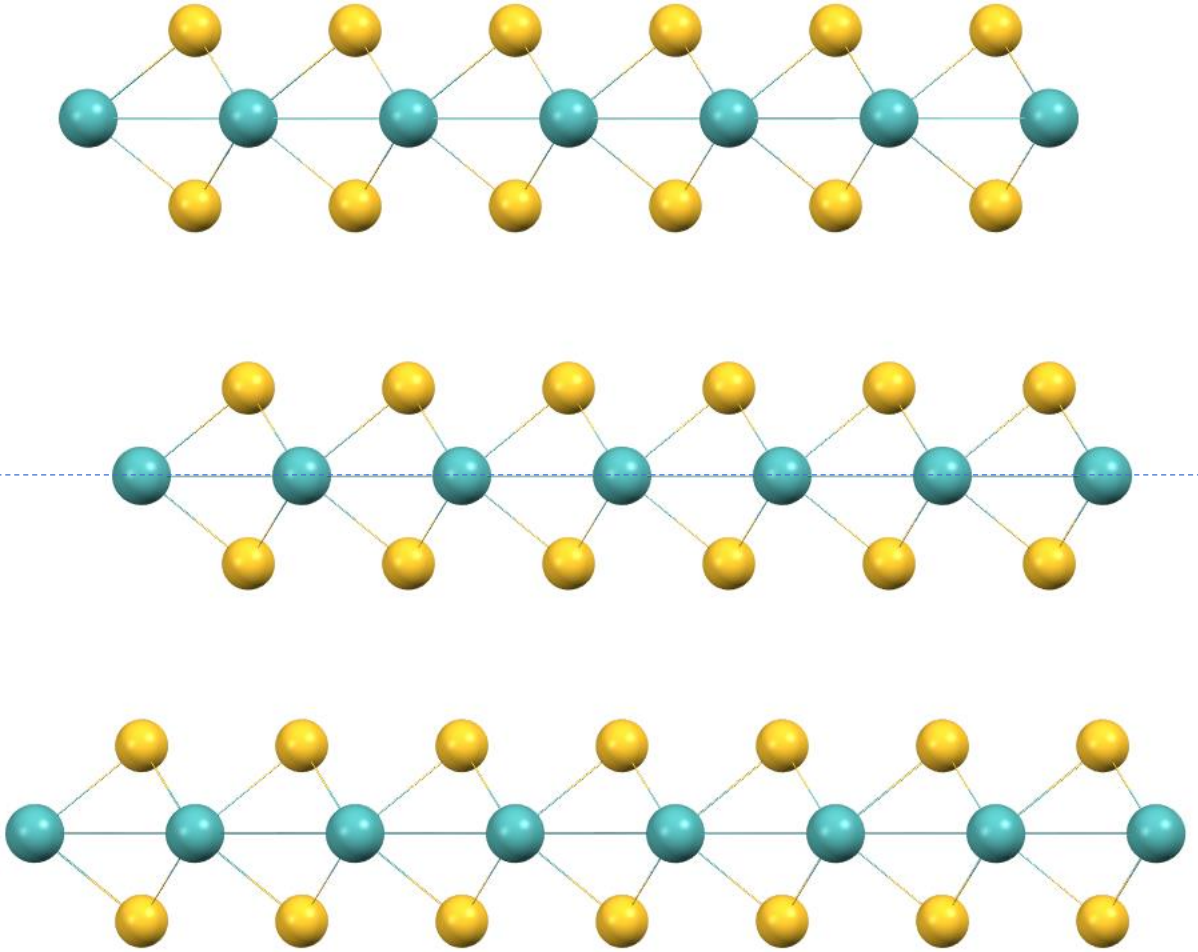
3D ED on 2D MoS_2 crystal





Gorelik et al., Micron 146 (2021) 103071

3D ED on 2D MoS_2 crystal



3D ED on 2D MoS_2 crystal

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For MoS_2 thin crystals
we can count
layers (atoms)

Gorelik et al., Micron 146 (2021) 103071

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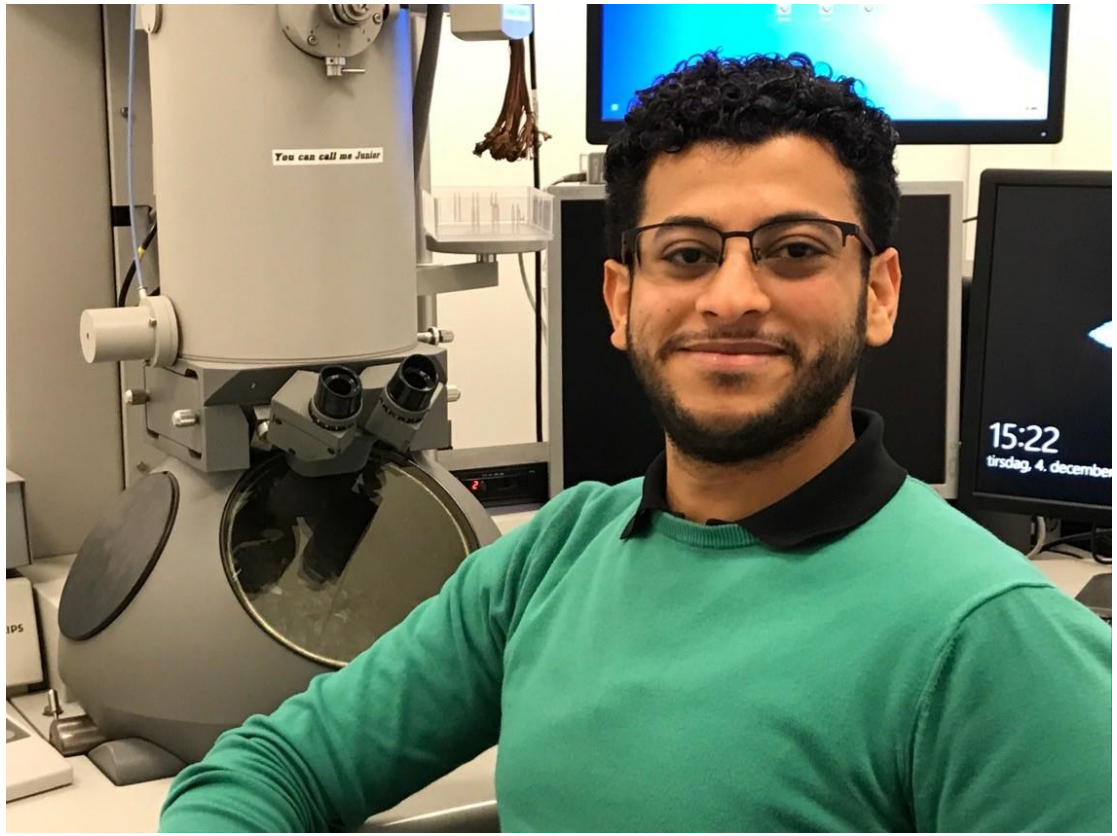
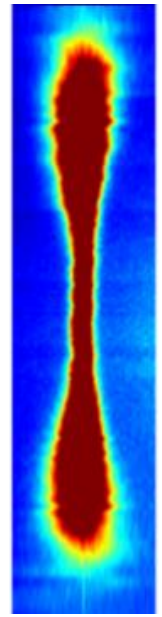
ESR6 Exploring 3D reciprocal space of 2D crystals

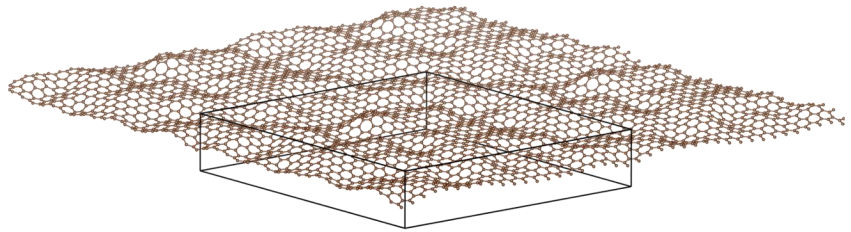


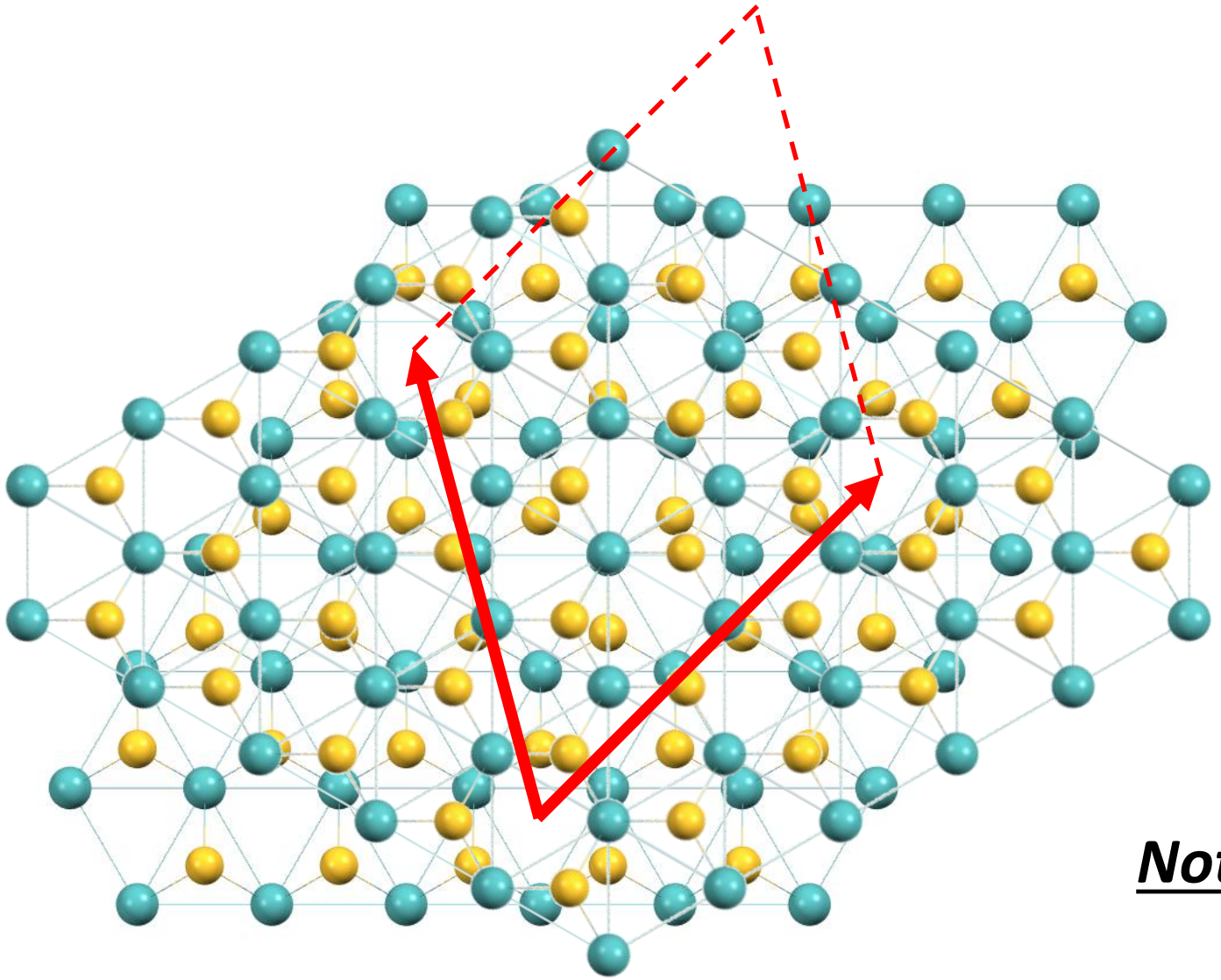
Sameh Okasha <samehokasha@gmail.com>



Sameh is going to

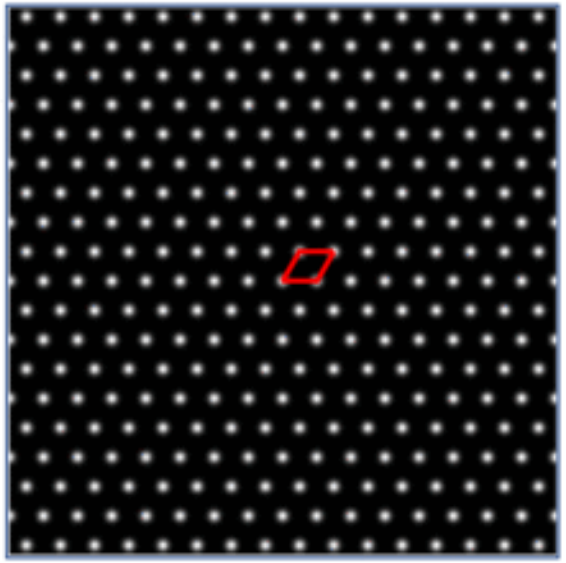
- Learn how to collect and analyse 3D ED data
- Learn how to prepare 2D crystals
- Quantify crystal waviness
- Prepare heterostructures (twisted)…



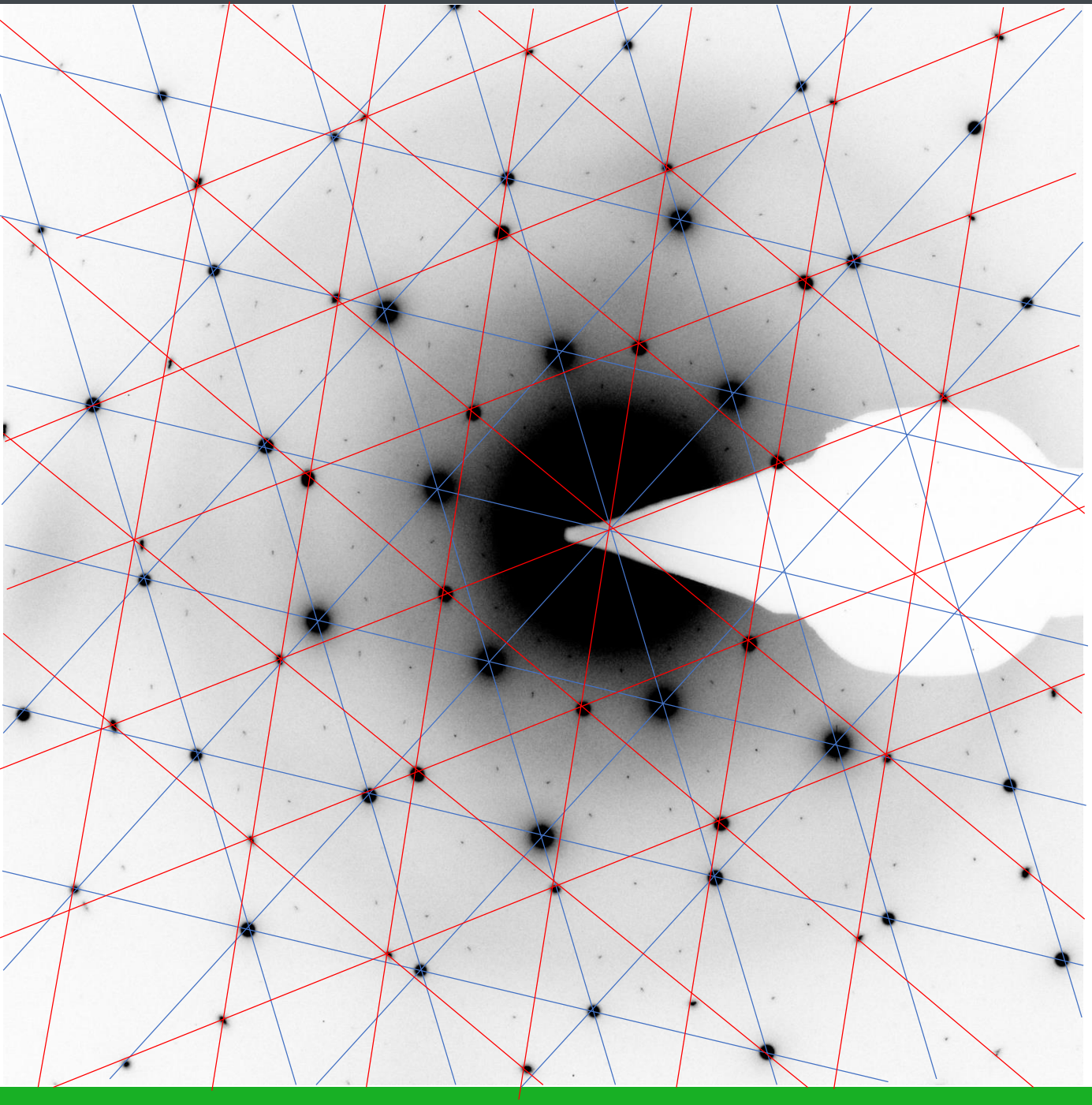


Superstructure

Not nessesarily comensurate



0 degrees



MoS_2

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Sameh is going to

- Understand and analyse 3D ED data of twisted heterostructures
- Study the 3D appearance of superstructure reflections
- Relate *everything* to structural parameters

THANK YOU
FOR YOUR ATTENTION