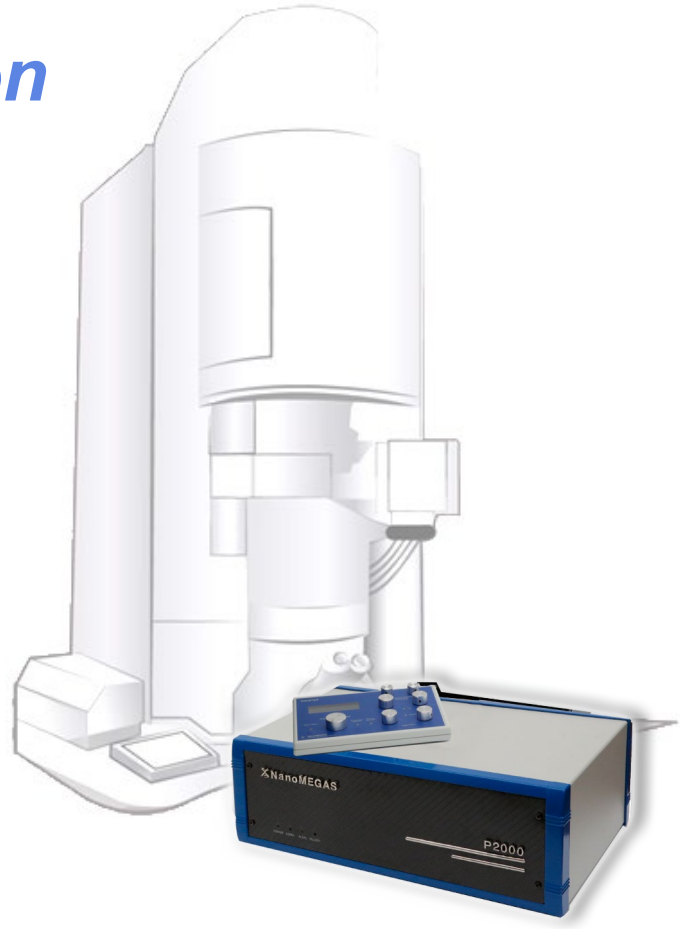
Precession enhanced Electron Diffraction applications in TEM for nano crystals.



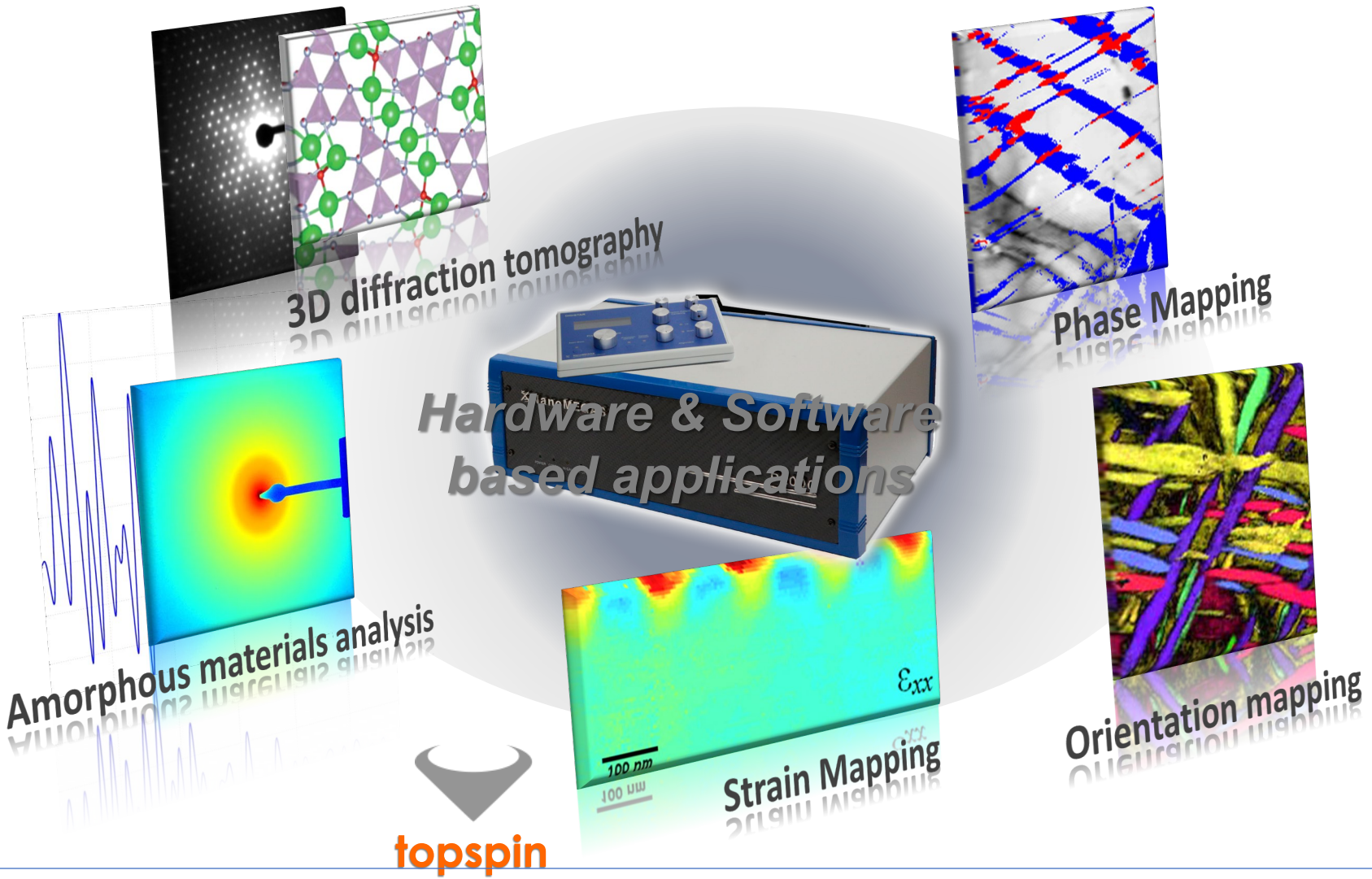
Thanos Galanis *Application Specialist*

NanoMEGAS
Advanced Tools for electron diffraction

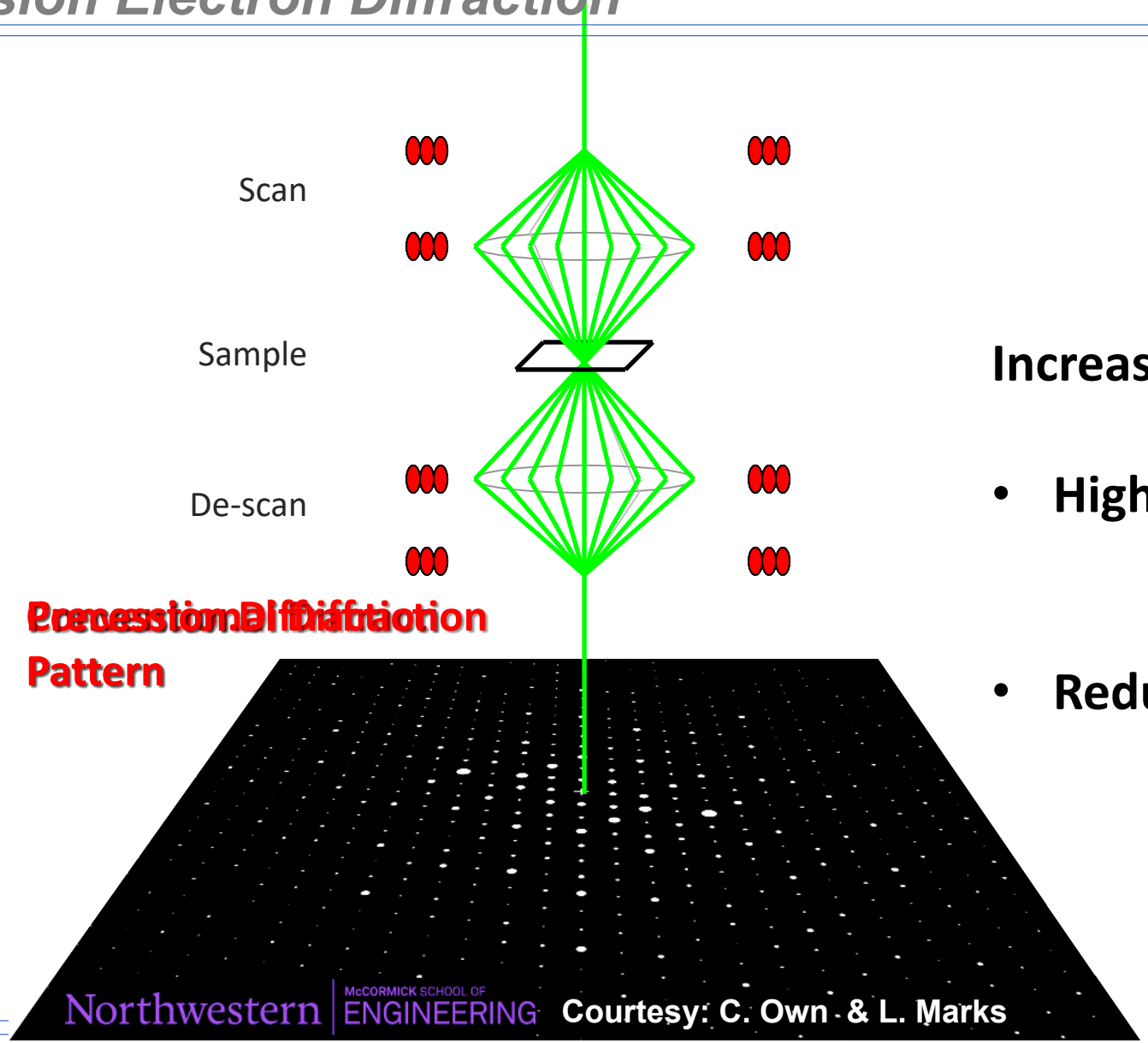
Brussels, Belgium
nanomegas.com

16th Multinational Congress on Microscopy, *September 4 – 9, 2022*

Precession Electron Diffraction Solutions



Precession Electron Diffraction



Increasing Precession angle:

- **Higher resolution**

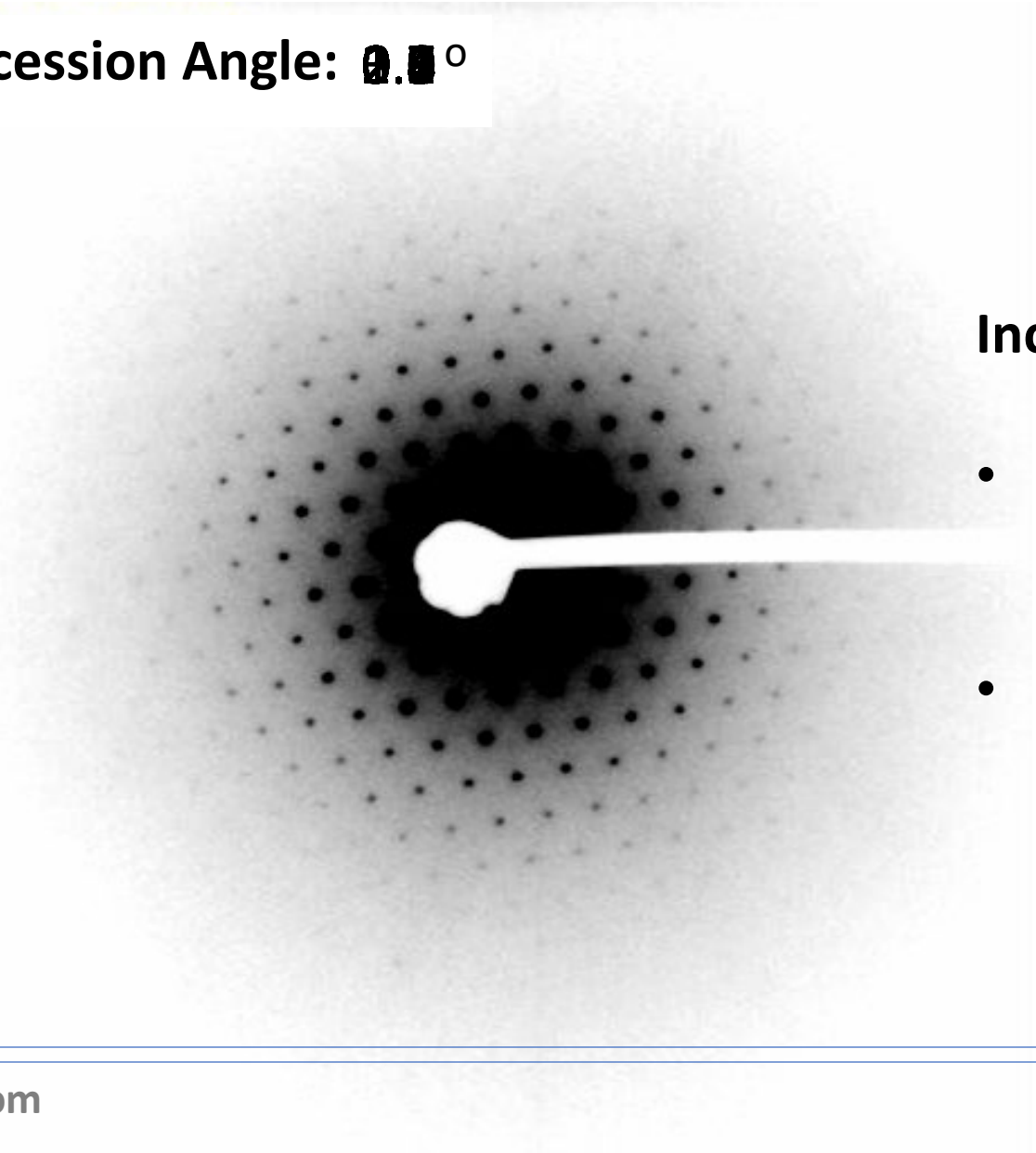
  higher order reflections

- **Reduce of dynamical effect**

  Close to kinematical / real reflections

Precession Electron Diffraction

Precession Angle: [illegible]°



Increasing Precession angle:

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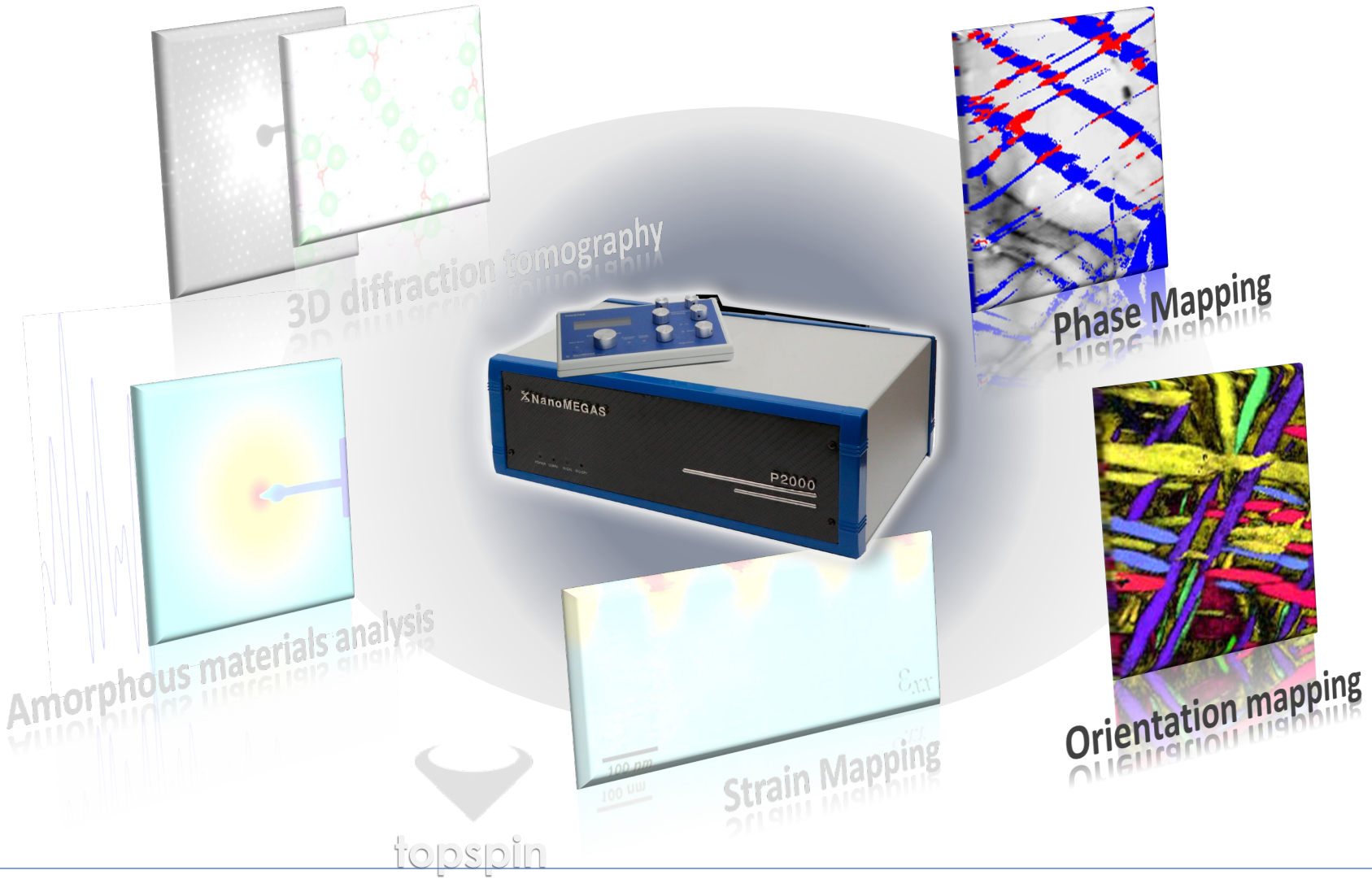
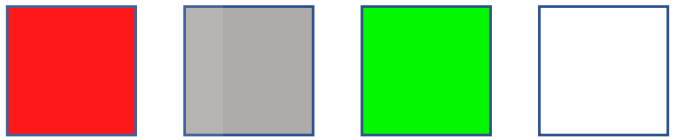
Precession Electron Diffraction Solutions



ASTAR: Orientation & Phase mapping in nm scale

ASTAR

Orientation & Phase Mapping in TEM



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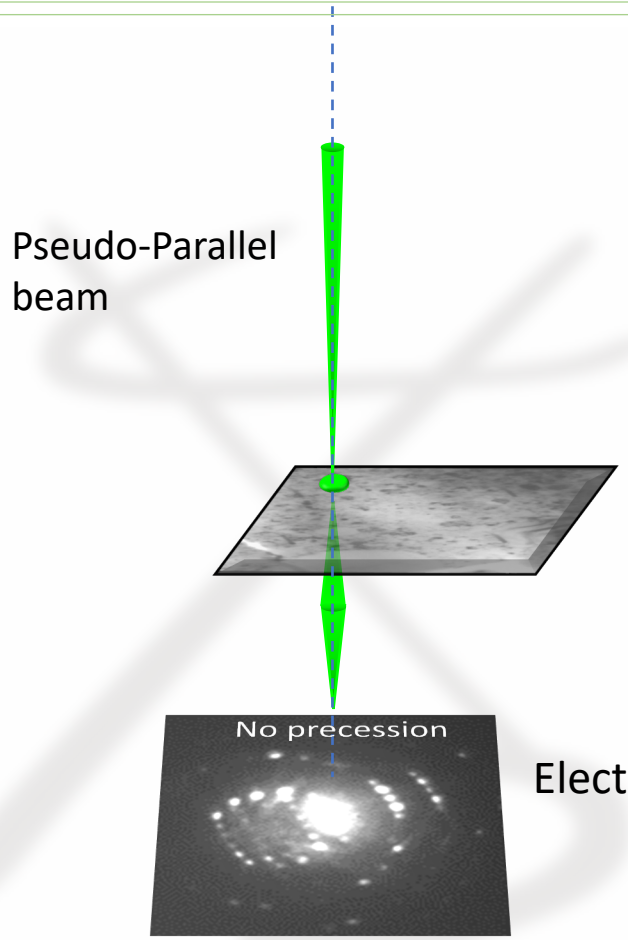
ASTAR: Orientation & Phase mapping in nm scale

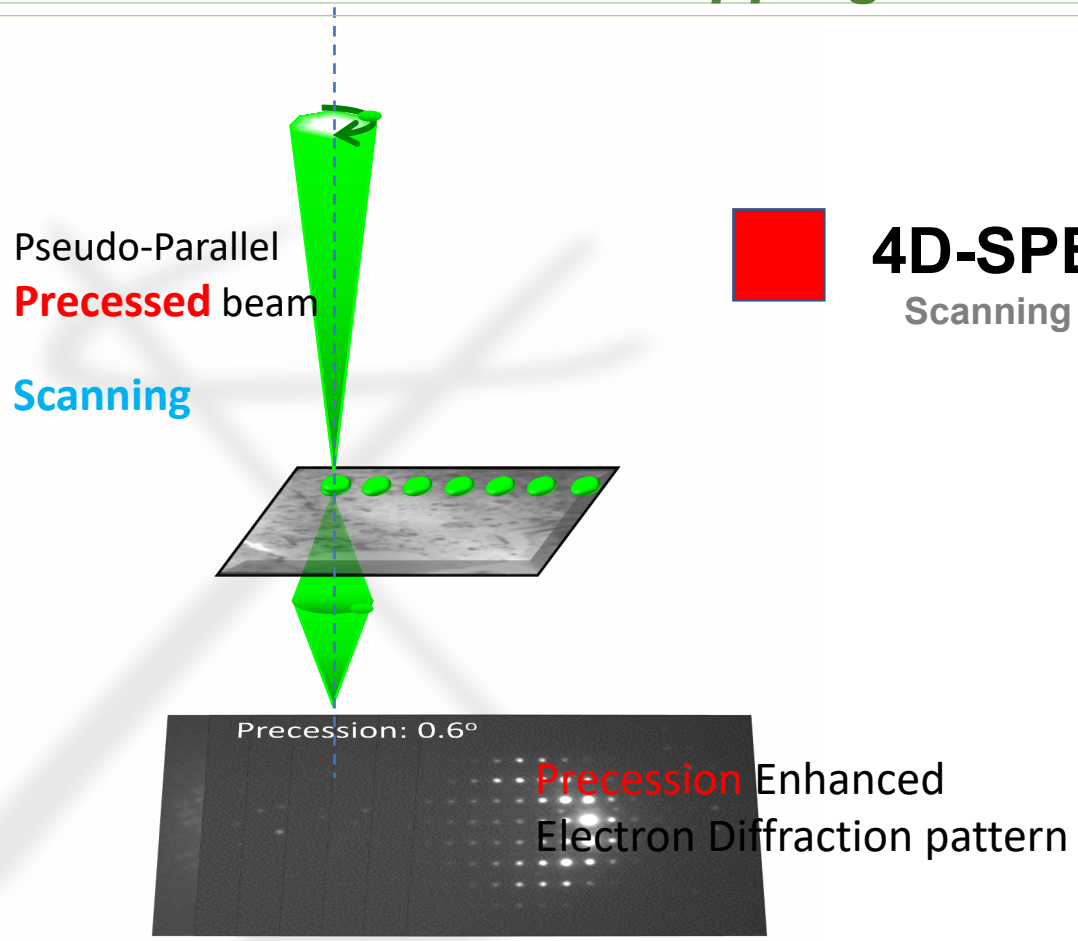


Electron Diffraction pattern

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

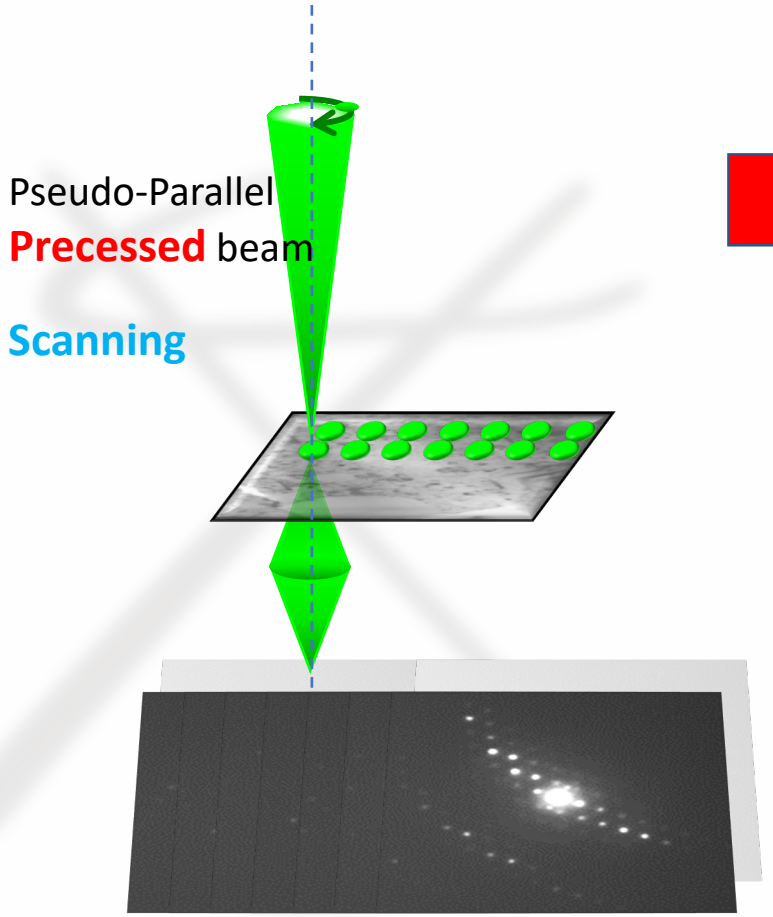
ASTAR: Orientation & Phase mapping in nm scale



4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

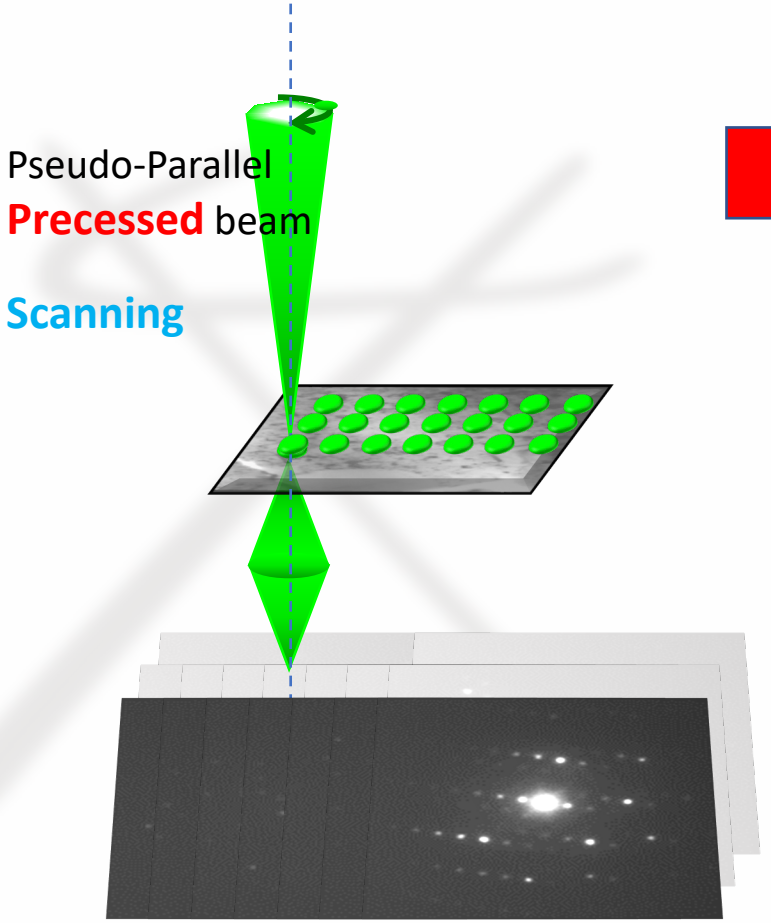
ASTAR: Orientation & Phase mapping in nm scale



4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

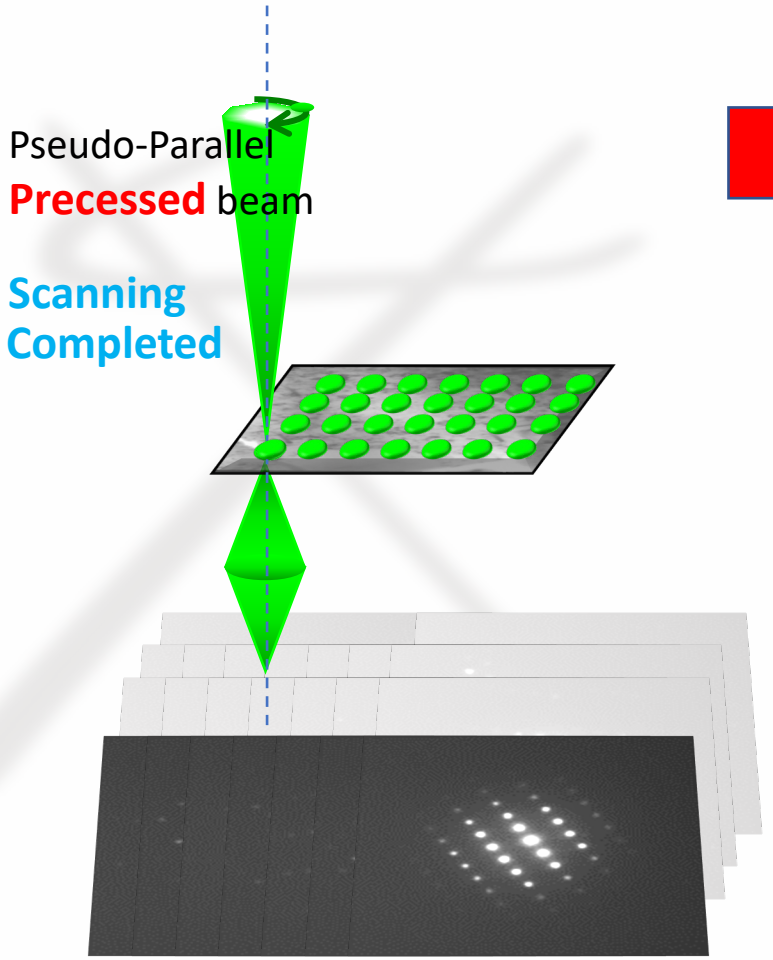
ASTAR: Orientation & Phase mapping in nm scale



4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

ASTAR: Orientation & Phase mapping in nm scale



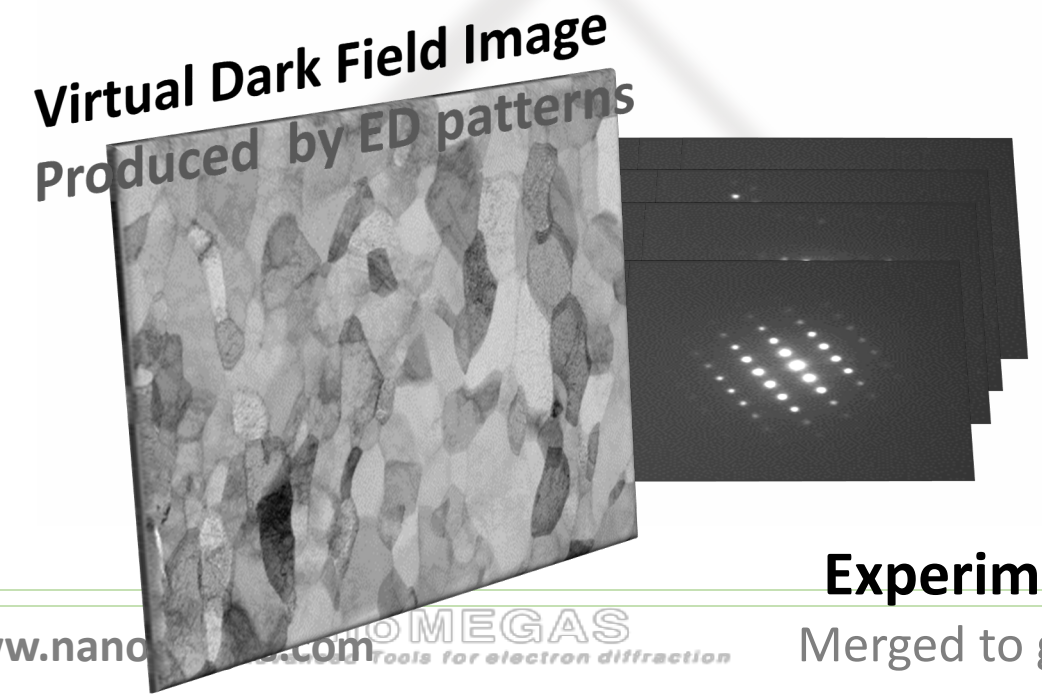
4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

ASTAR: Orientation & Phase mapping in nm scale

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

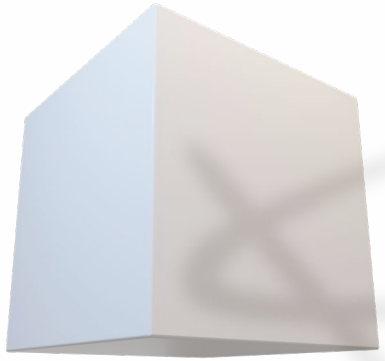


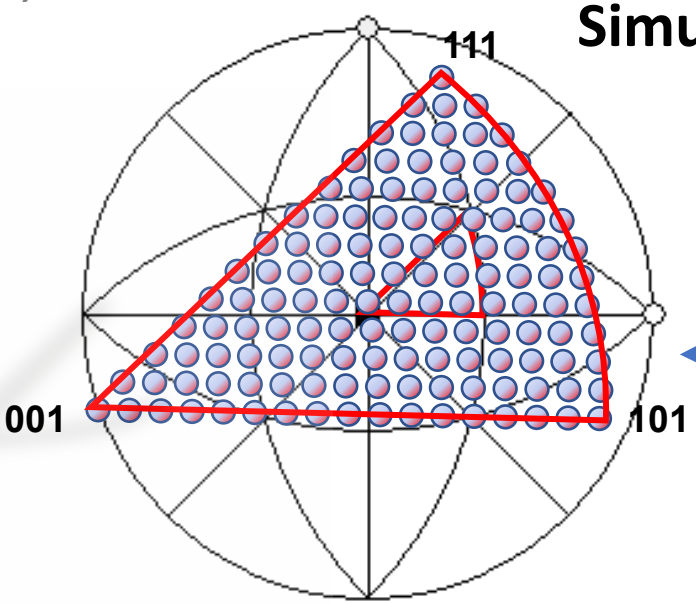
Experimental ED data

Merged to generate block file

**Application example: cubic; all crystal systems can be used*

Crystal Cubic System



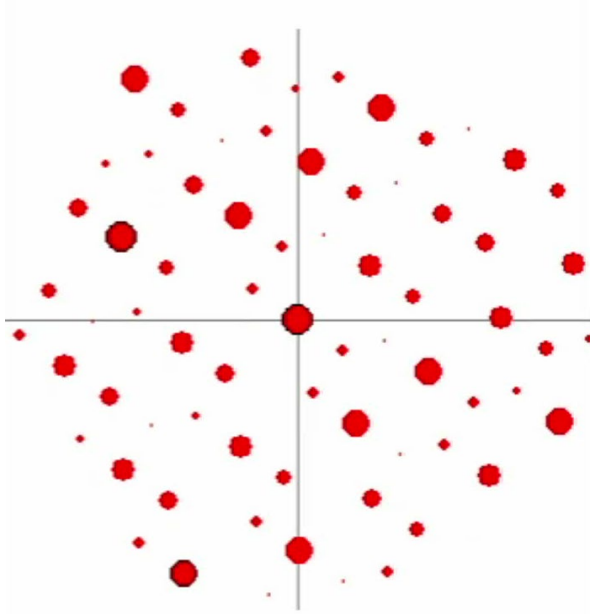


Stereographic Projection of all possible Crystal Orientations in Cubic Crystal System

Sector with all possible Crystal Orientations

Simulated ED patterns for each orientation

generated by crystallographic file



ASTAR Templates

Simulated ED patterns generation

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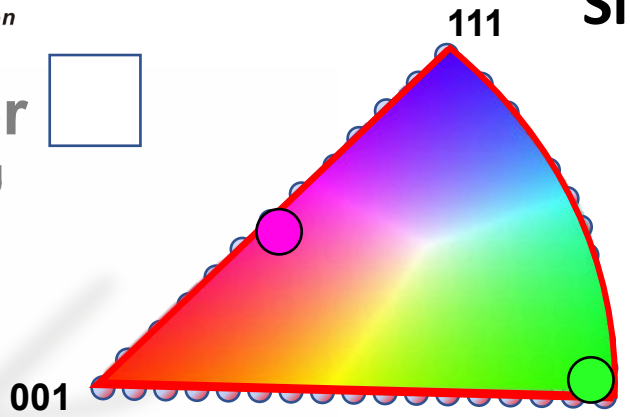
ASTAR MapViewer

Index Experimental dps with templates

Orientation & Phase Mapping



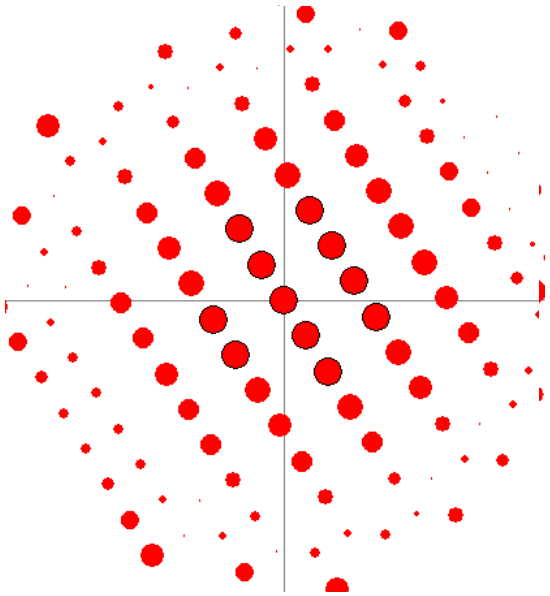
Sector with all Possible Orientations

Color coded Inverse Pole figure

in Cubic Crystal system

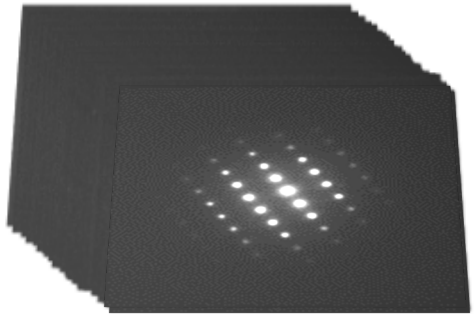
Simulated ED patterns for each orientation

generated by crystallographic file



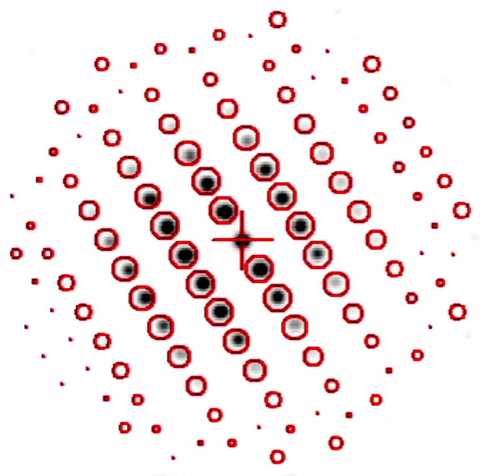
Virtual Bright Field Image

Orientation Data



Experimental ED data

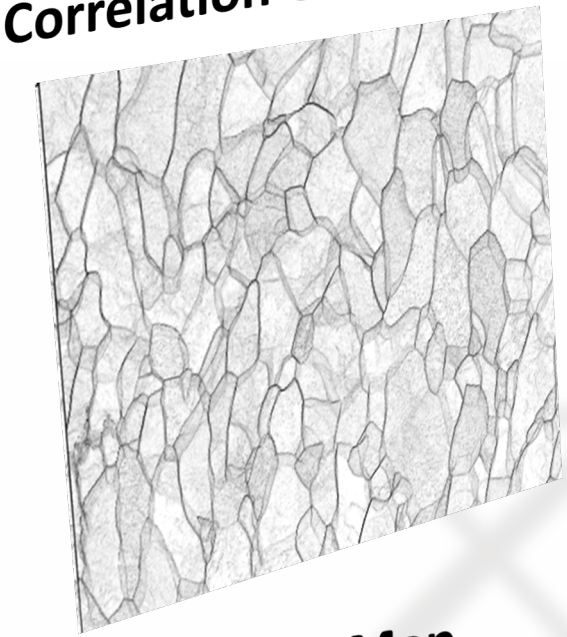
Merged to generate block file



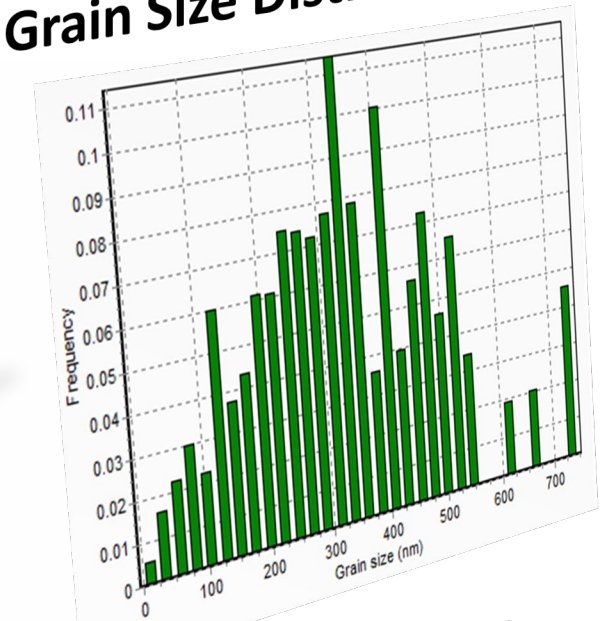
Experimental & simulated patterns

Matching procedure

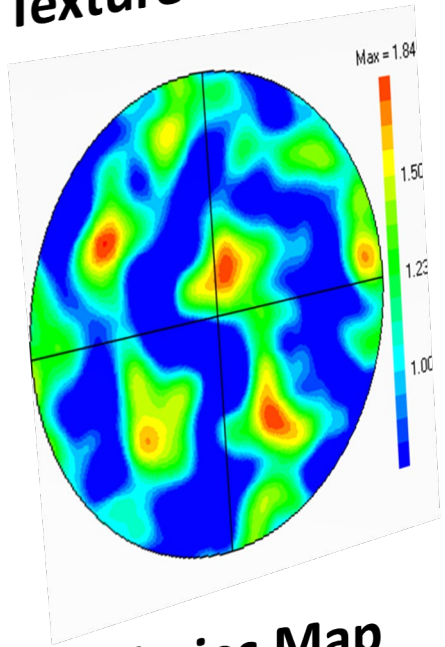
Correlation Coefficient Map



Grain Size Distribution



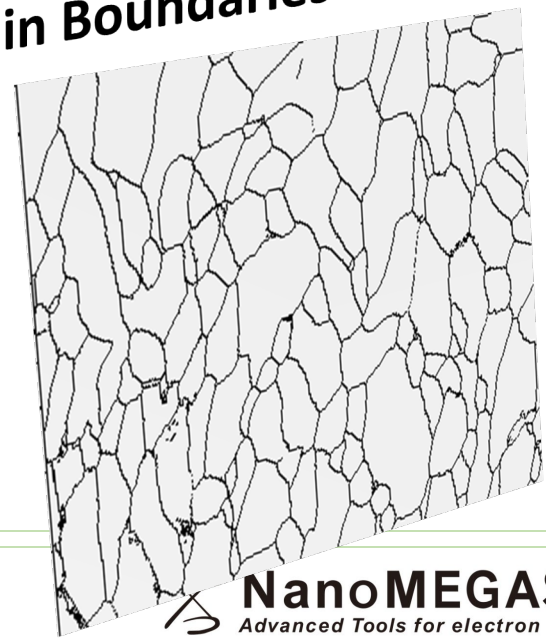
Texture Analysis



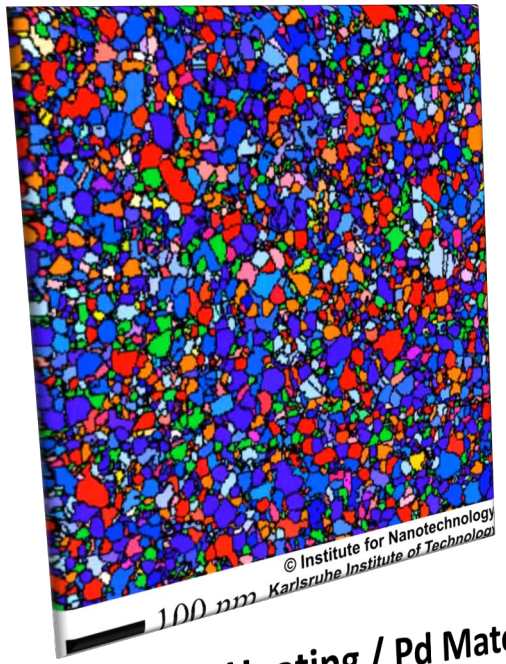
Orientation Map

Virtual Dark Field Image

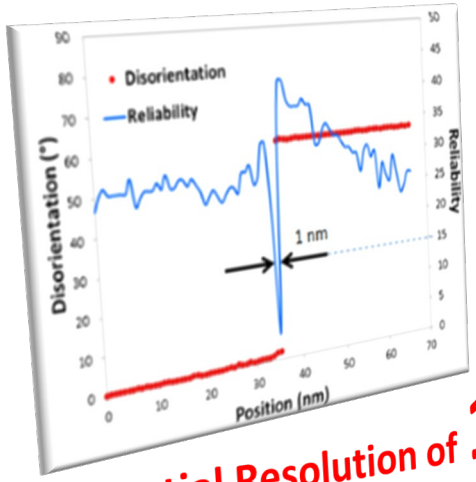
Grain Boundaries Map



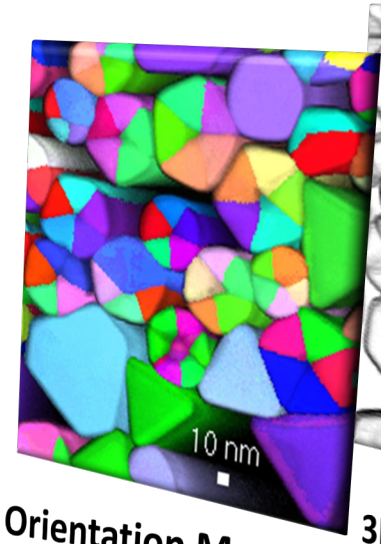
ASTAR: Orientation & Phase mapping in nm scale



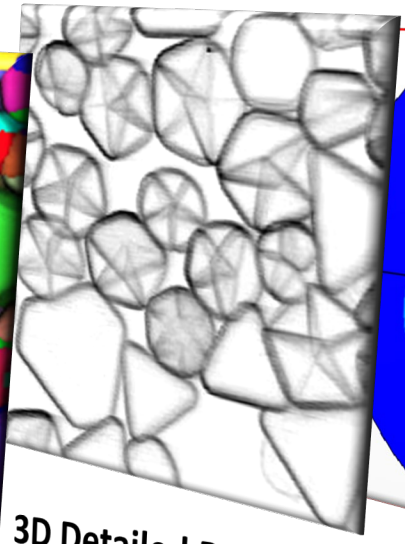
In Situ Heating / Pd Material



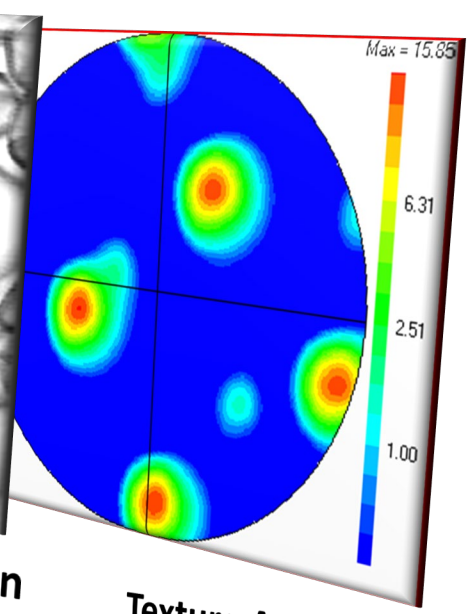
Spatial Resolution of 1nm



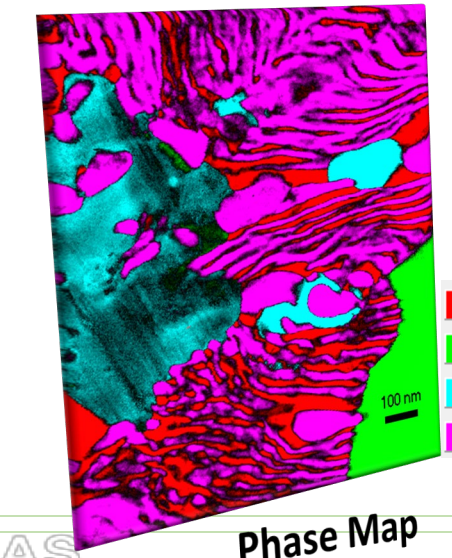
Orientation Map



3D Detailed Projection

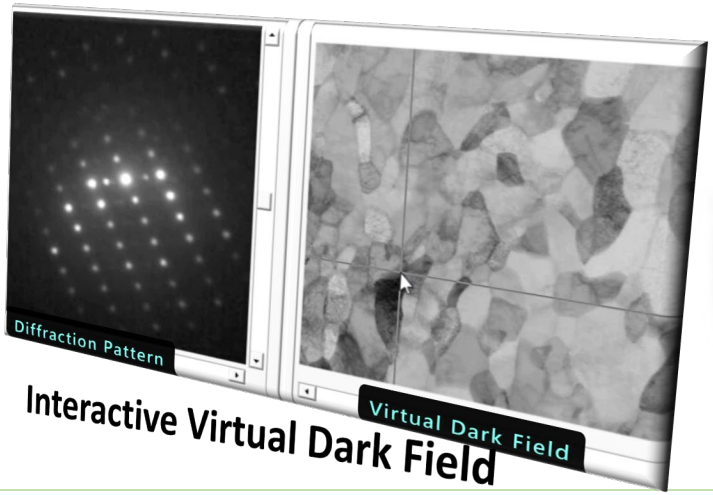


Texture Analysis



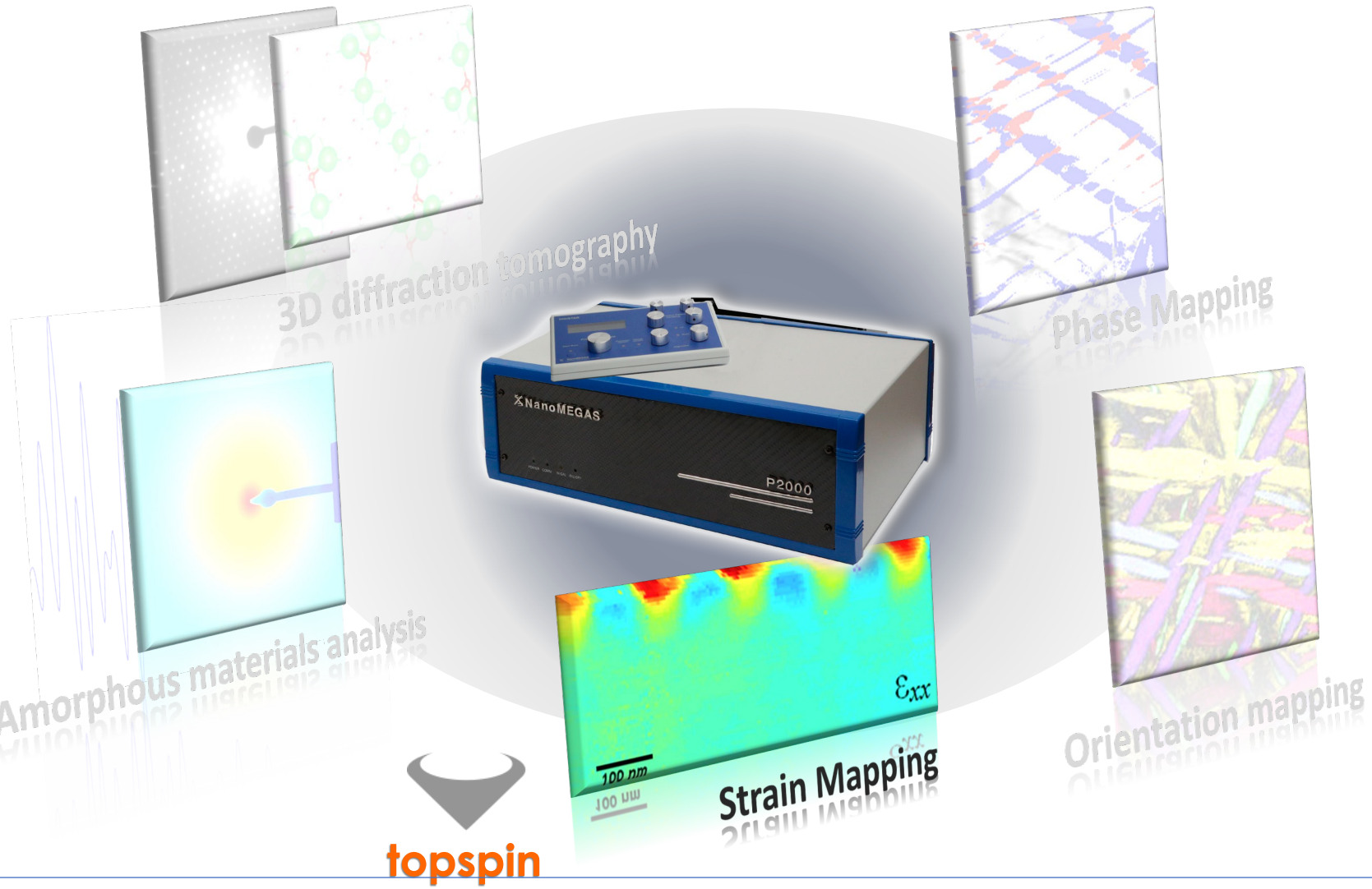
- Fe
- $Fe_{14}Nd_2B$
- Fe_2B
- NdH_2

Phase Map



Interactive Virtual Dark Field

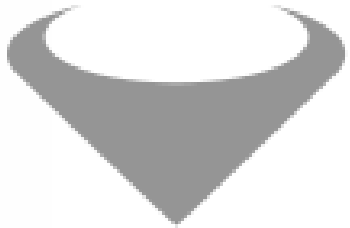
Precession Electron Diffraction Solutions



TopSPIN: Strain mapping in nm scale and high sensitivity

TopSPIN

PED Strain Analysis in TEM



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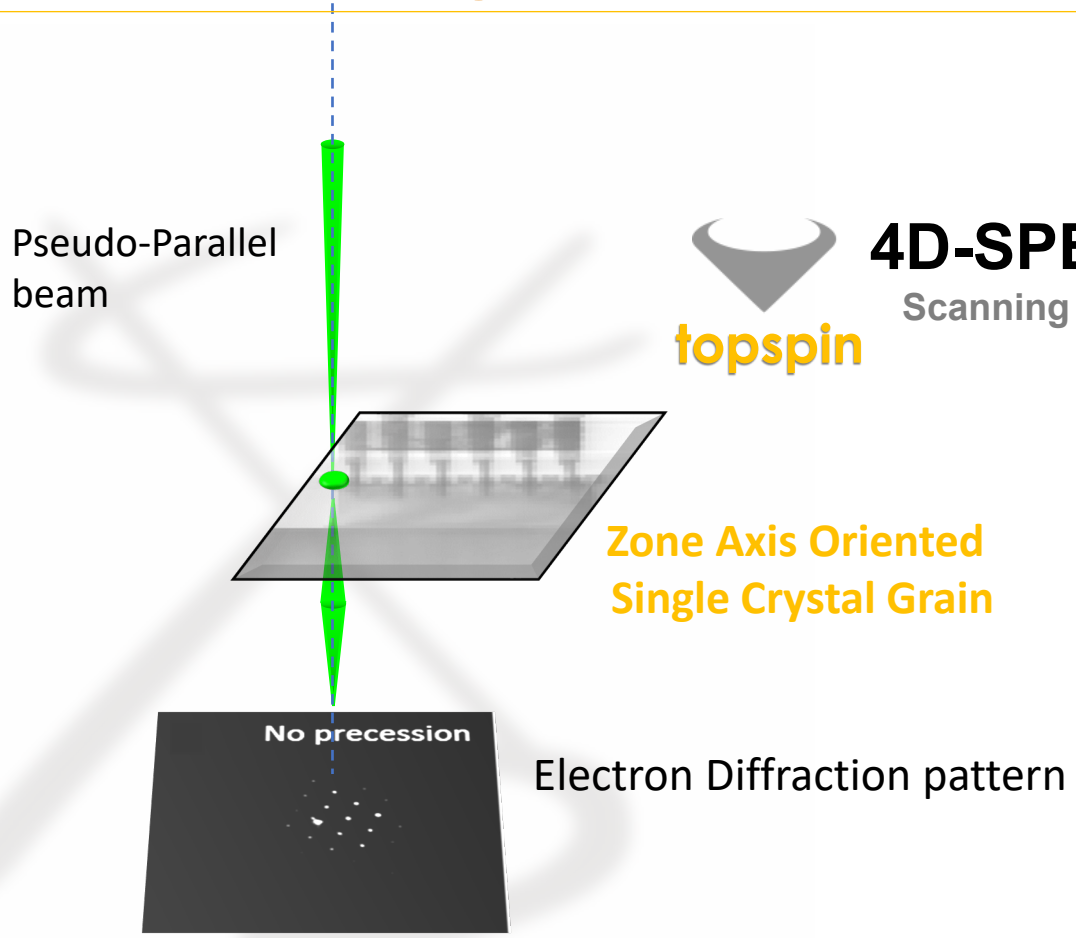
TopSPIN: Strain mapping in nm scale and high sensitivity

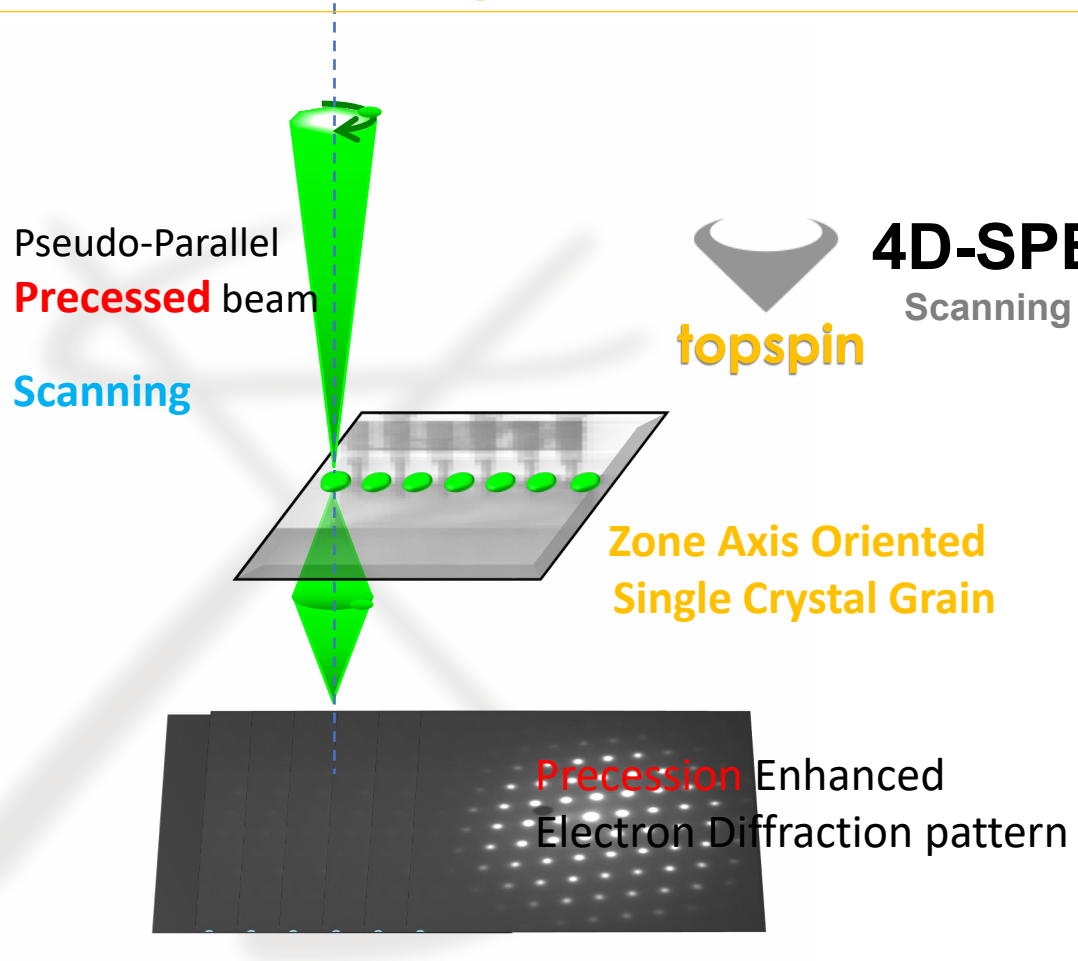


topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

TopSPIN: Strain mapping in nm scale and high sensitivity

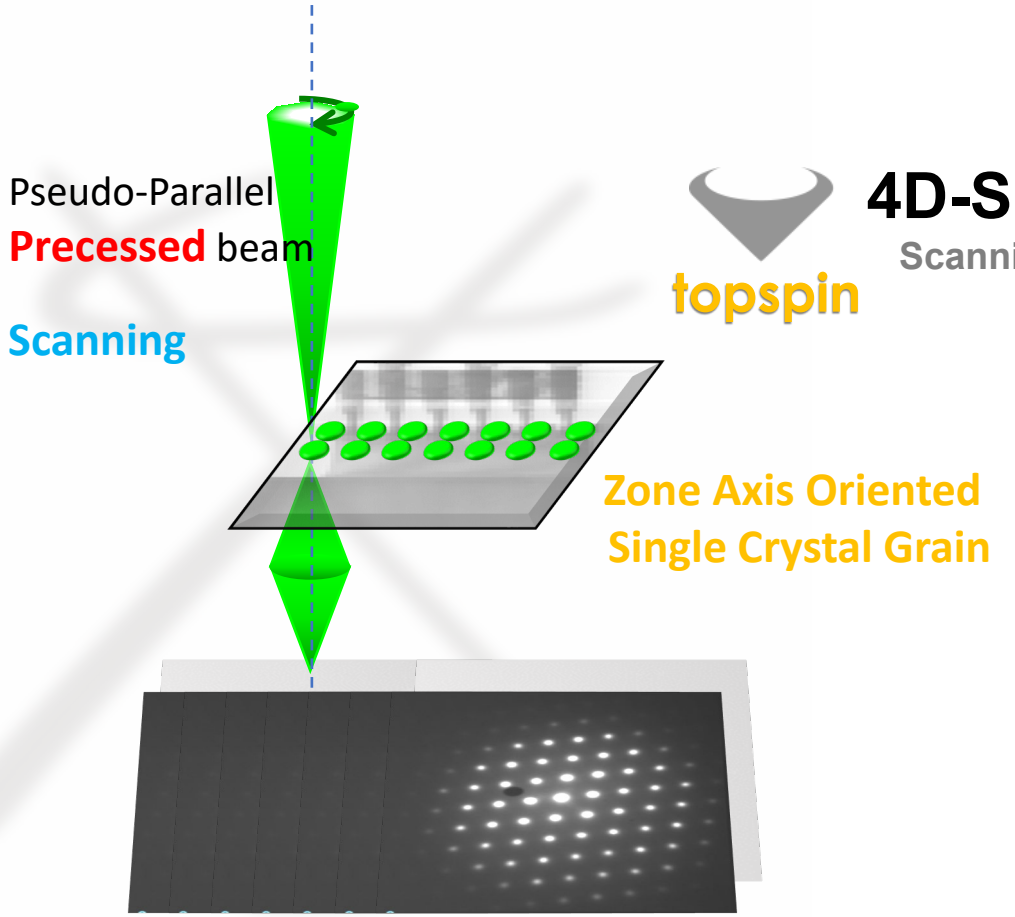


topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

TopSPIN: Strain mapping in nm scale and high sensitivity

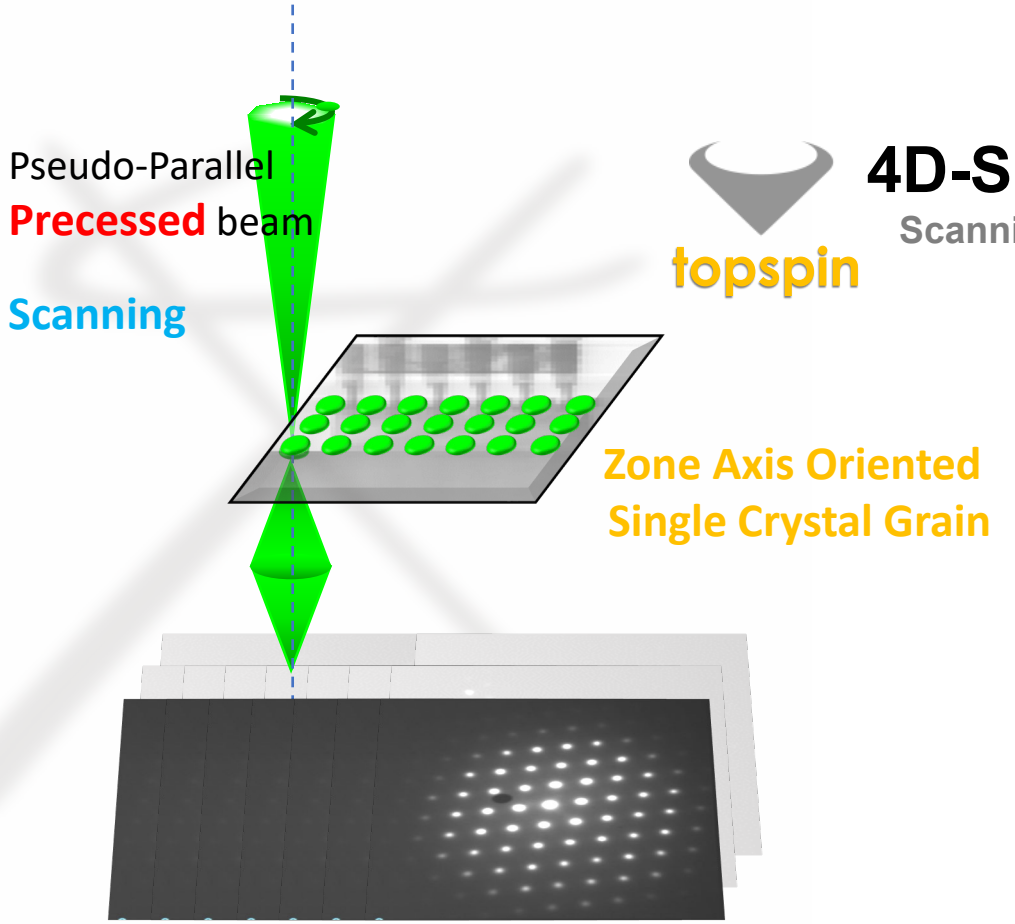


topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

TopSPIN: Strain mapping in nm scale and high sensitivity

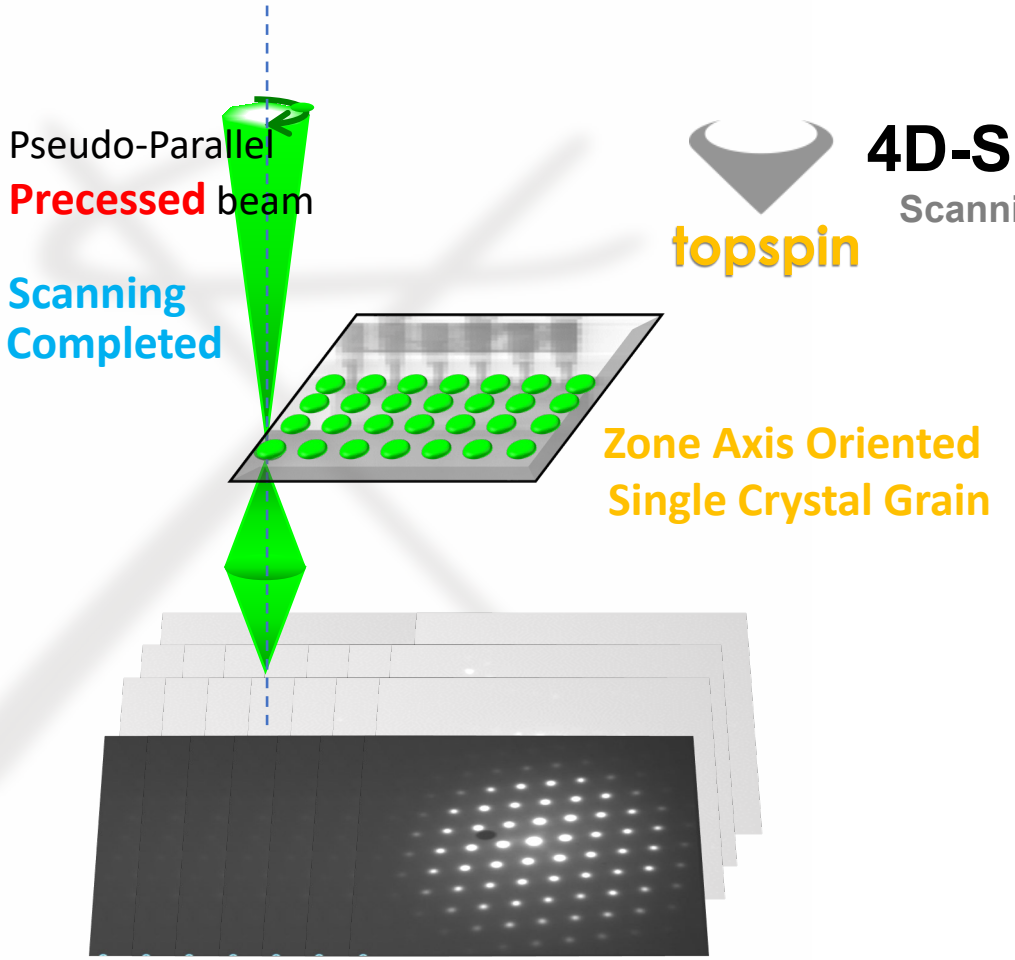


topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

TopSPIN: Strain mapping in nm scale and high sensitivity

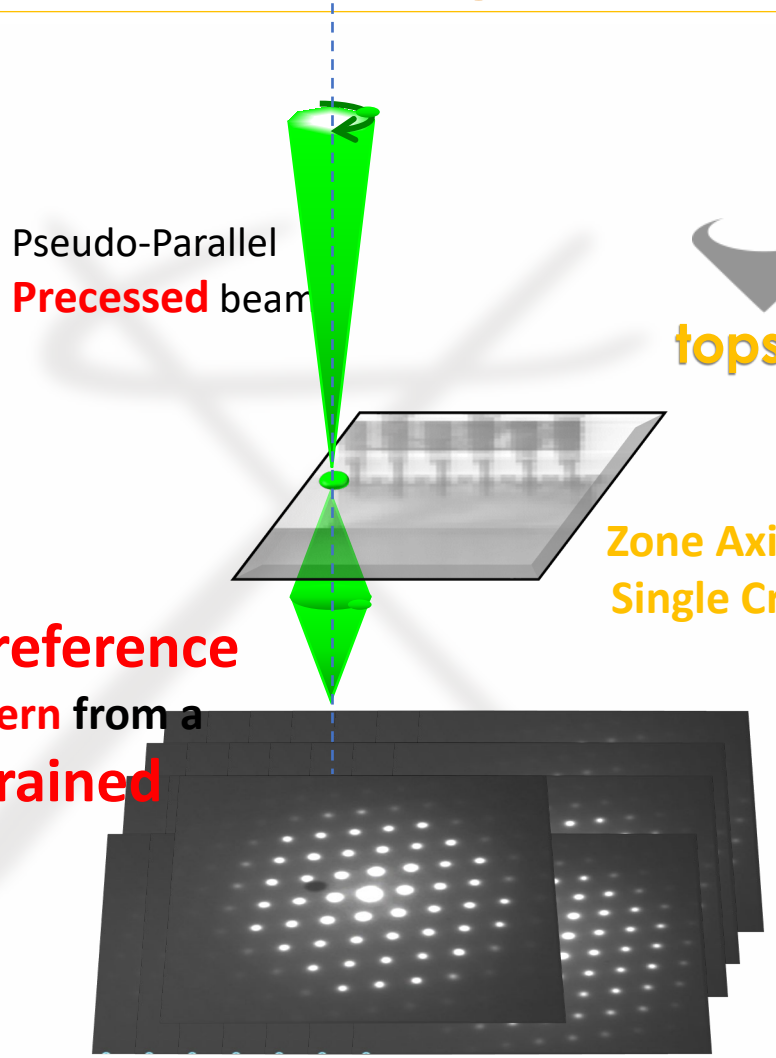


topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

TopSPIN: Strain mapping in nm scale and high sensitivity



topspin

4D-SPED Data Acquisition

Scanning Precession Electron Diffraction

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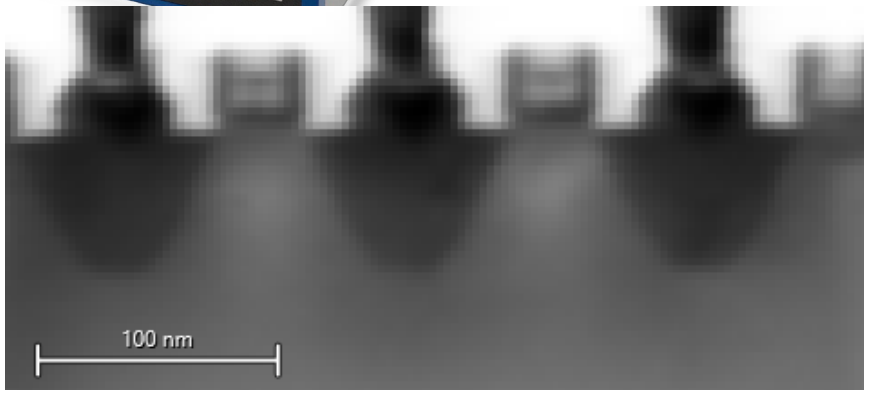
Zone Axis Oriented
Single Crystal Grain



Series of PED patterns

Scanned area

STEM / VDF image

Scanned area

TopSPIN: Strain mapping in nm scale and high sensitivity

topspin

TopSPIN strain mapping

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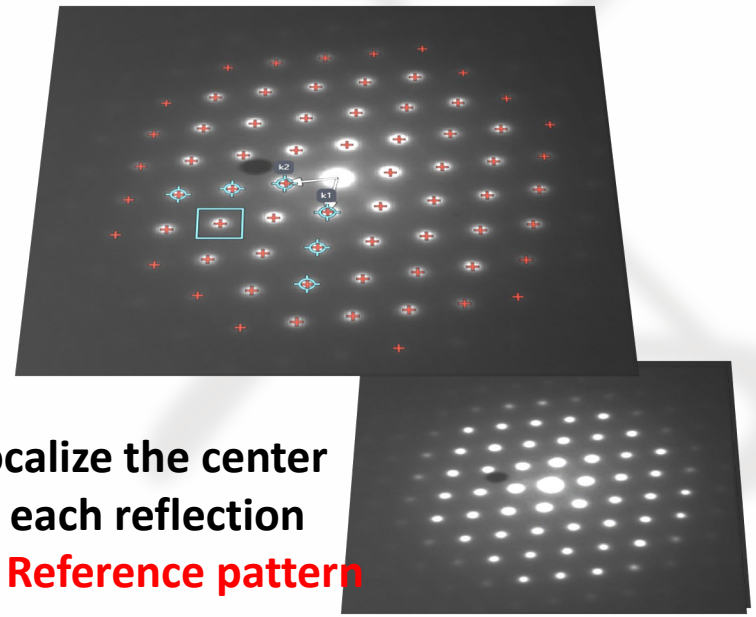
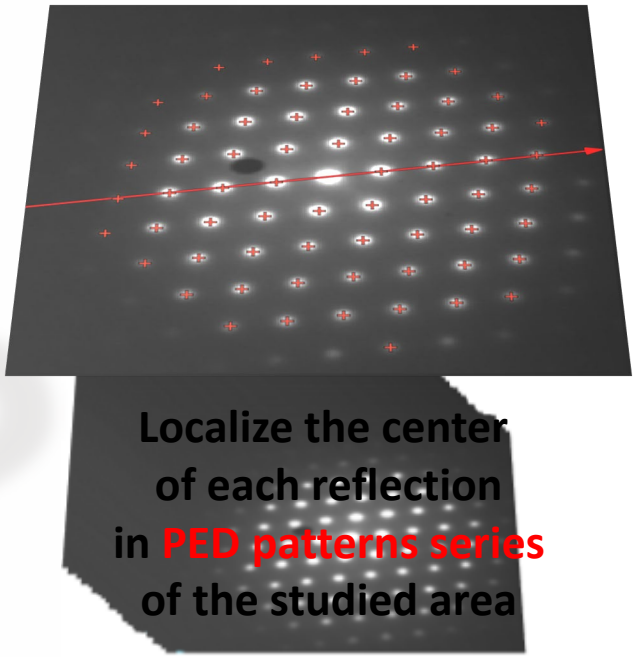
Advanced Tools for electron diffraction

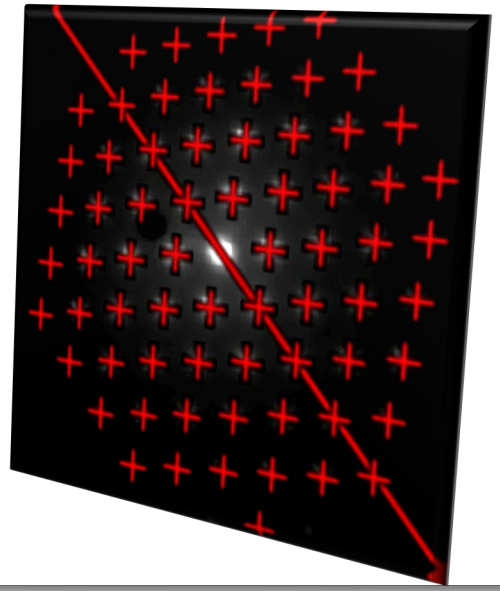
Strain Calculation:
PED series patterns are matched against the reference pattern.

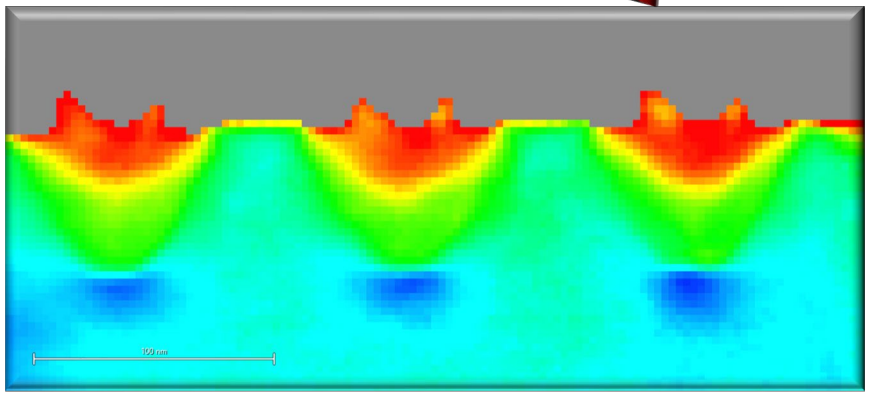


Localize the center of each reflection in Reference pattern



Localize the center of each reflection in PED patterns series of the studied area





Reference PED pattern
non-strained area

Series of PED patterns
Scanned area

STEM / VDF image
Single crystal grain area marked

TopSPIN: Strain mapping in nm scale and high sensitivity

topspin PED Strain Mapping

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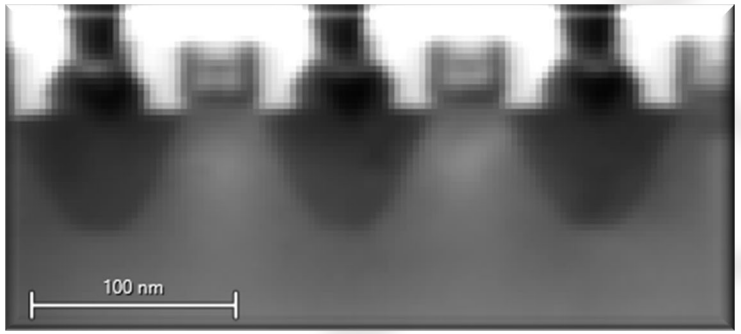
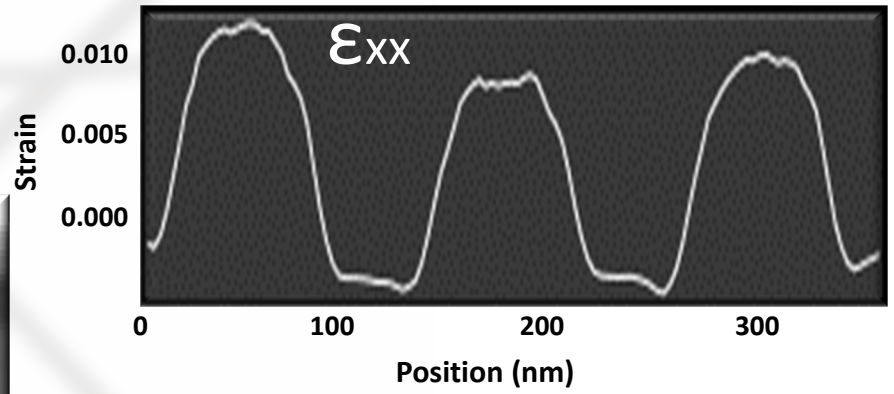
pMOS device

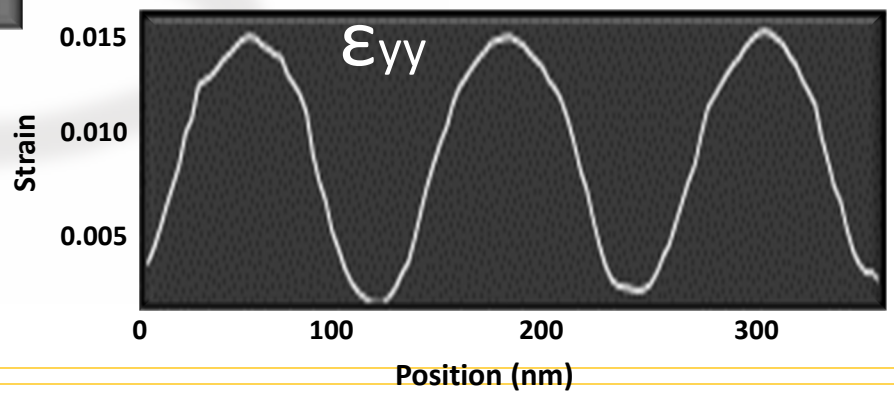


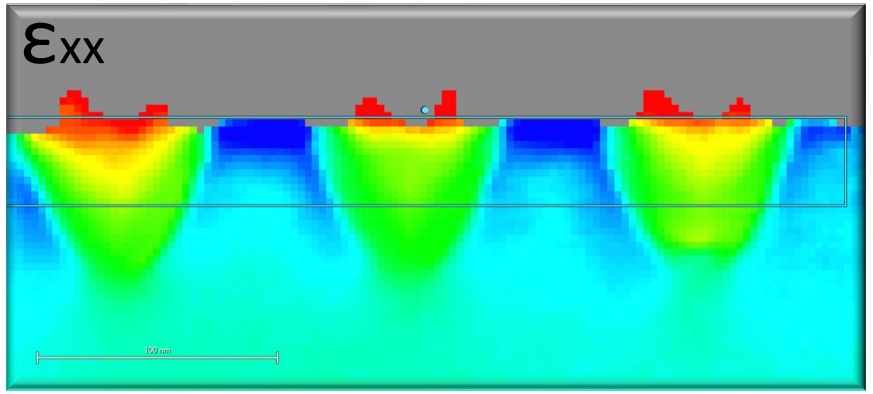
Spatial resolution: 1-5 nm

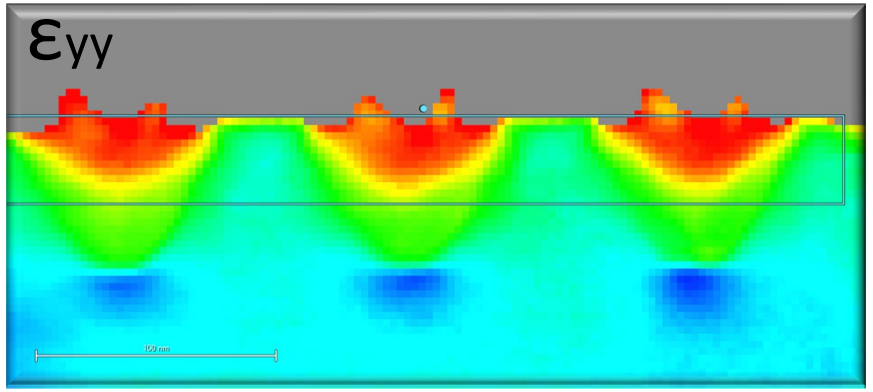
Strain Sensitivity: >0.02%

Strain Profile





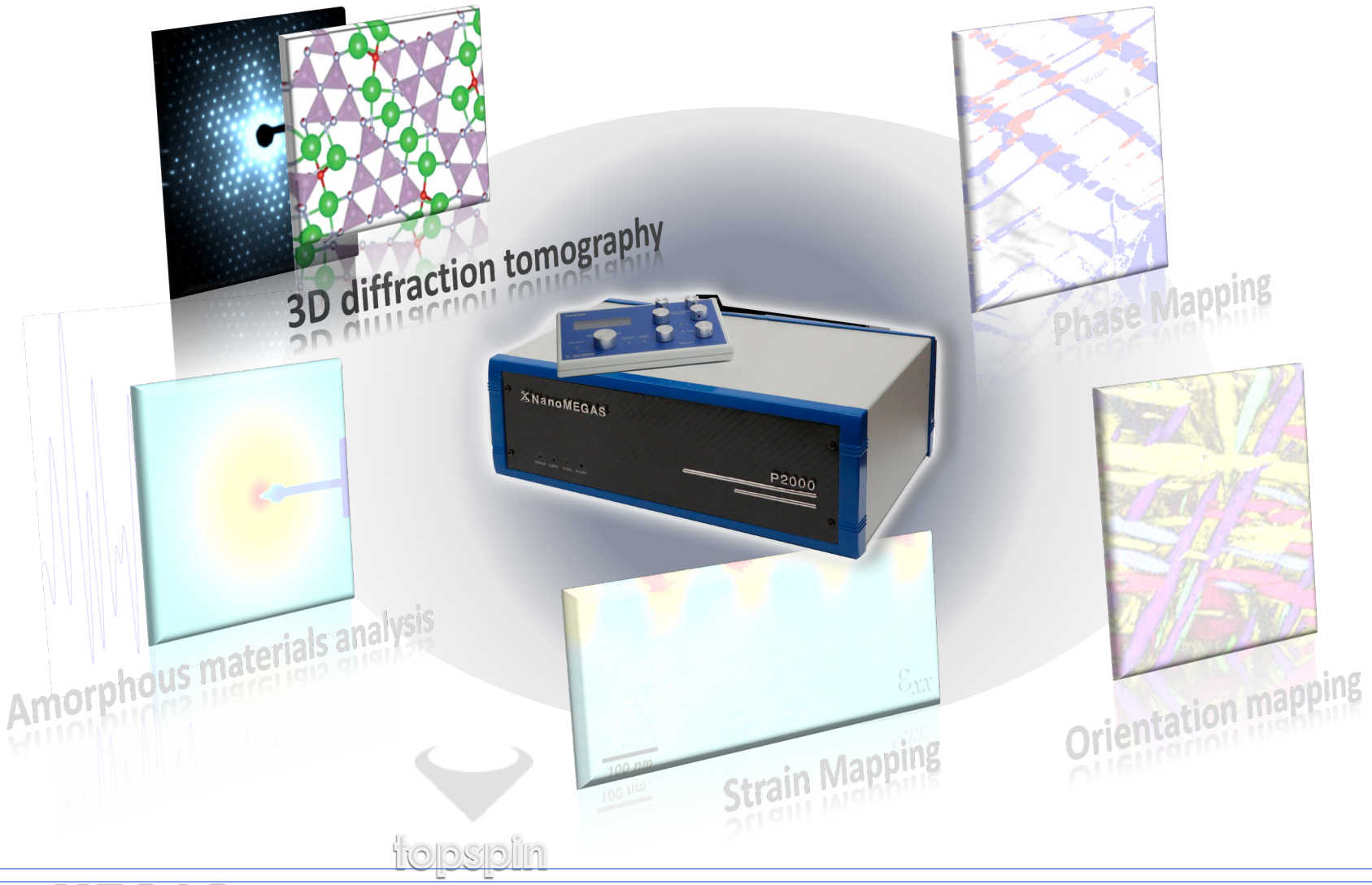




Strain Mapping

Single crystal grain area calculated

Precession Electron Diffraction Solutions



Precession Electron Diffraction Tomography (PEDt) – Solve crystal structures

3D - Electron Diffraction Tomography

for crystal structure determination

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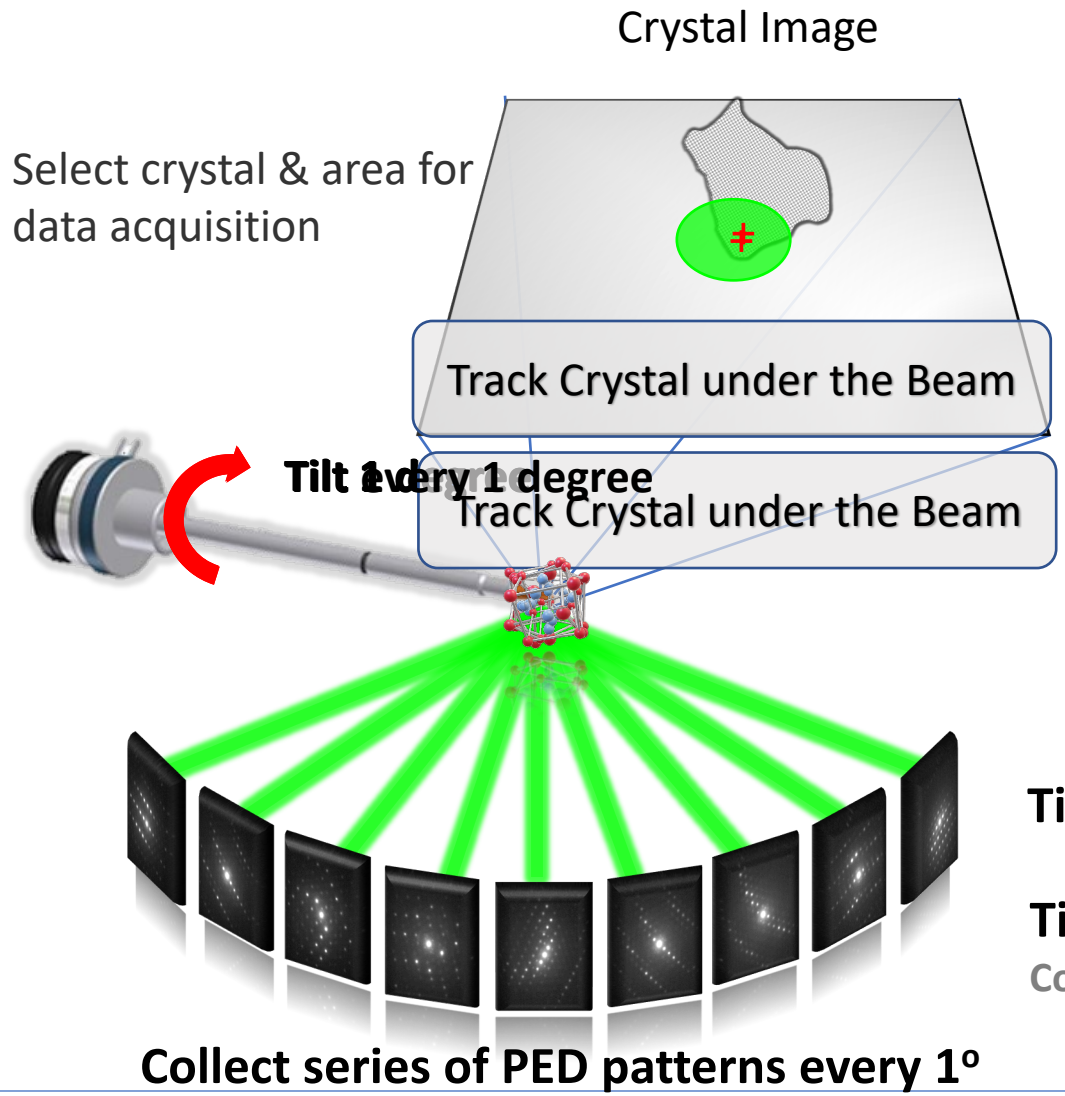
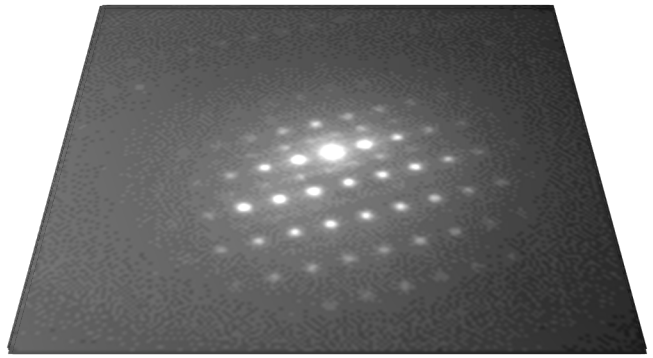
Precession Electron Diffraction Tomography (PEDt) – Solve crystal structures



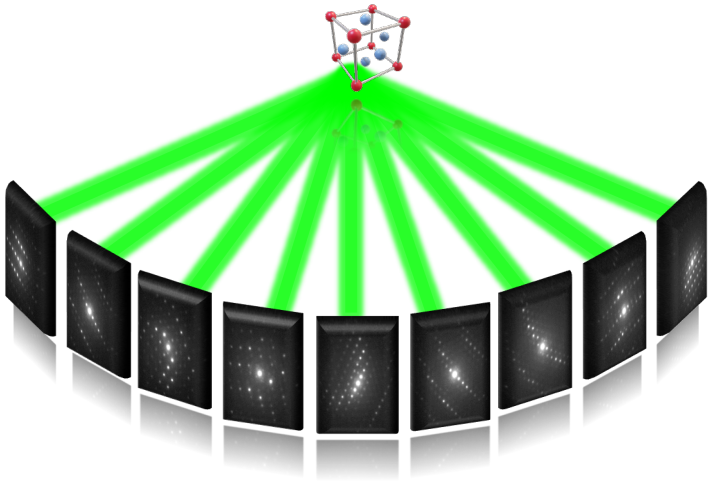


Tilt range for cell parameters determination: -30º to +30º

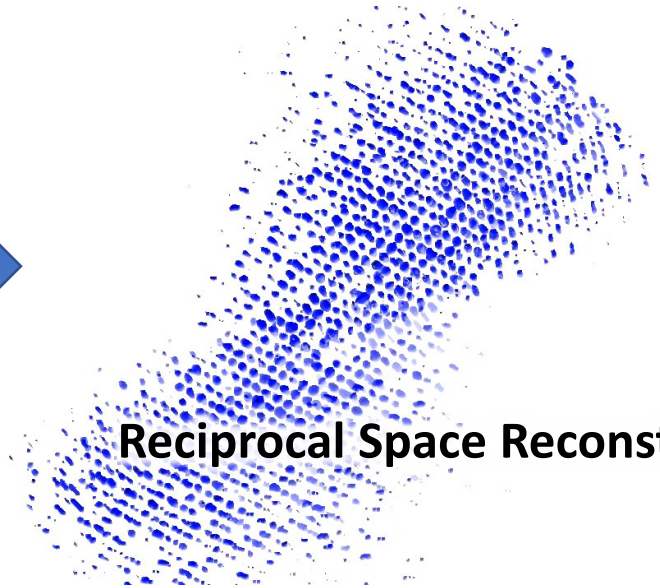
Tilt range for structure determination: -60º to +60º

Completeness of reciprocal space

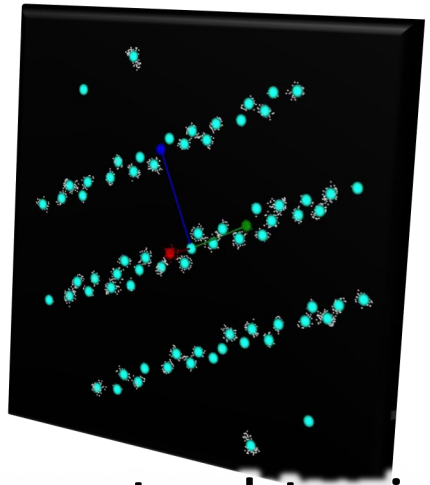
Precession Electron Diffraction Tomography (PEDt) – Solve crystal structures



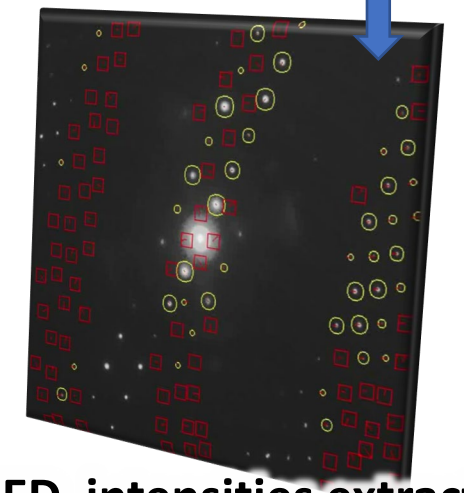
PED patterns every 1º



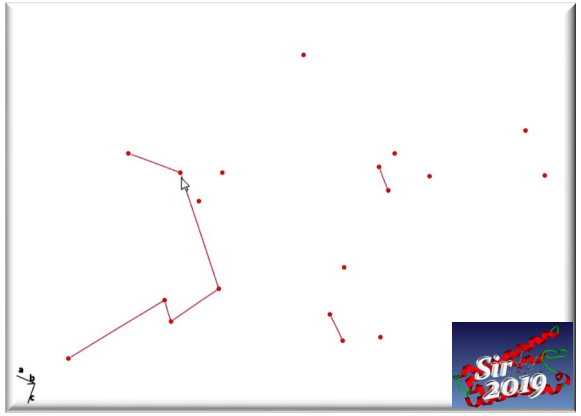
Reciprocal Space Reconstruction



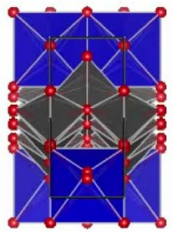
Cell parameters determination



ED intensities extraction



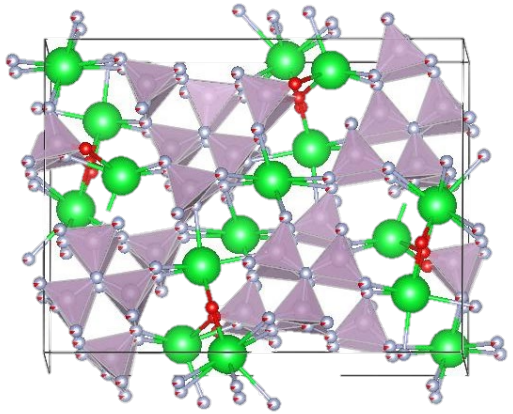
Solving Crystal Structure



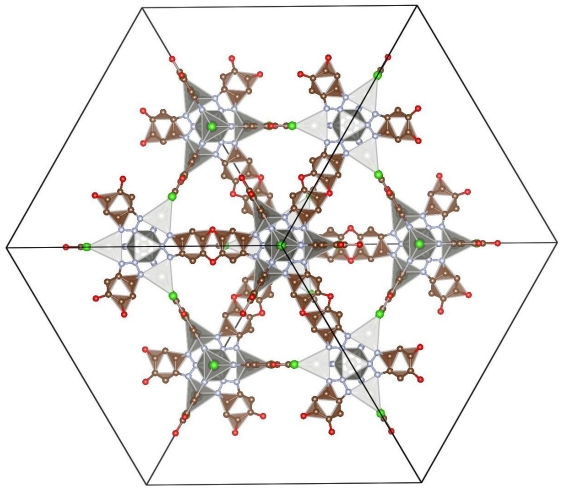
Final Structural Model

Precession Electron Diffraction Tomography (PEDt) – Solve crystal structures

Light-Emitting Diodes

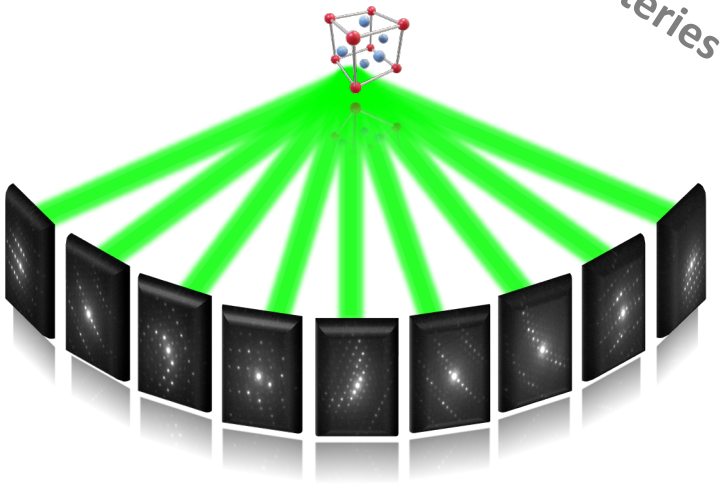


catalysts



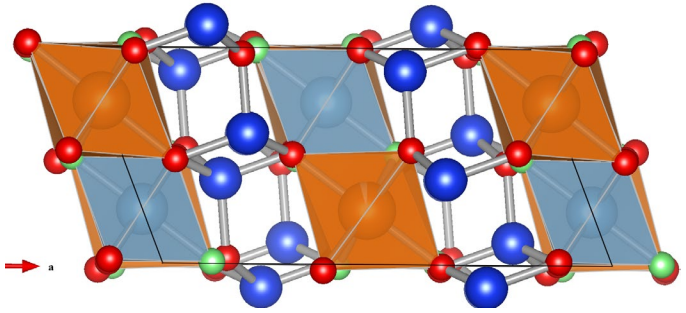
MFU4L_MOF

batteries



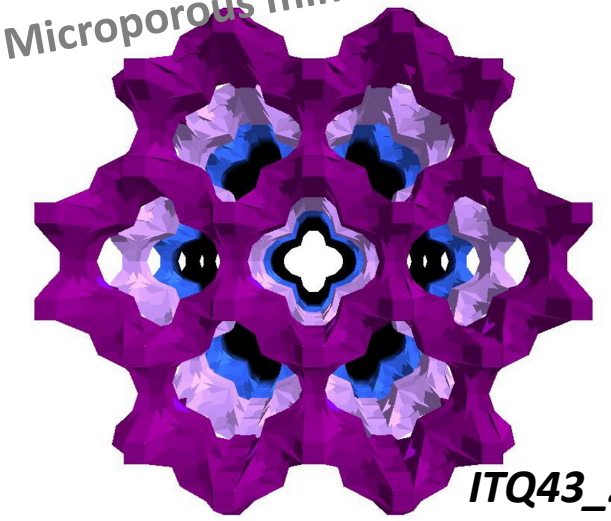
PEDt tomography

minerals



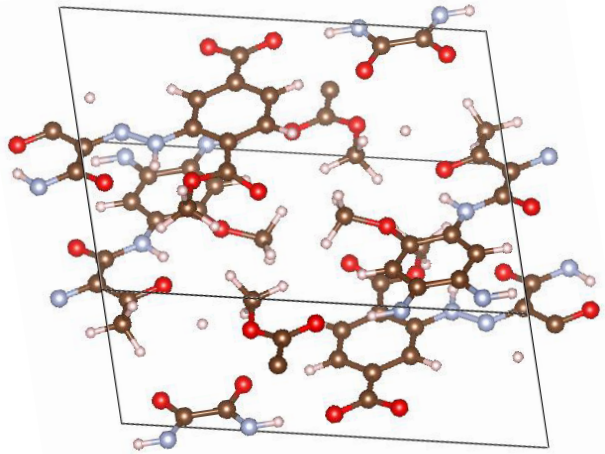
HAPY_$Mg_2Al(OH)_2AlSiO_6$

Microporous minerals



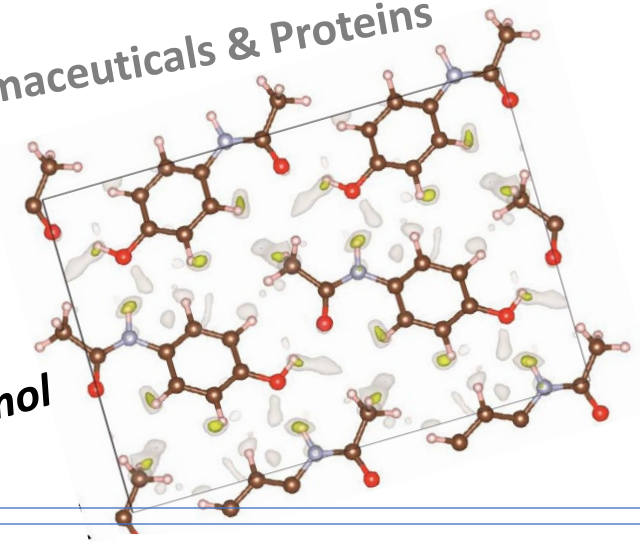
ITQ43_Zeolite

pigments



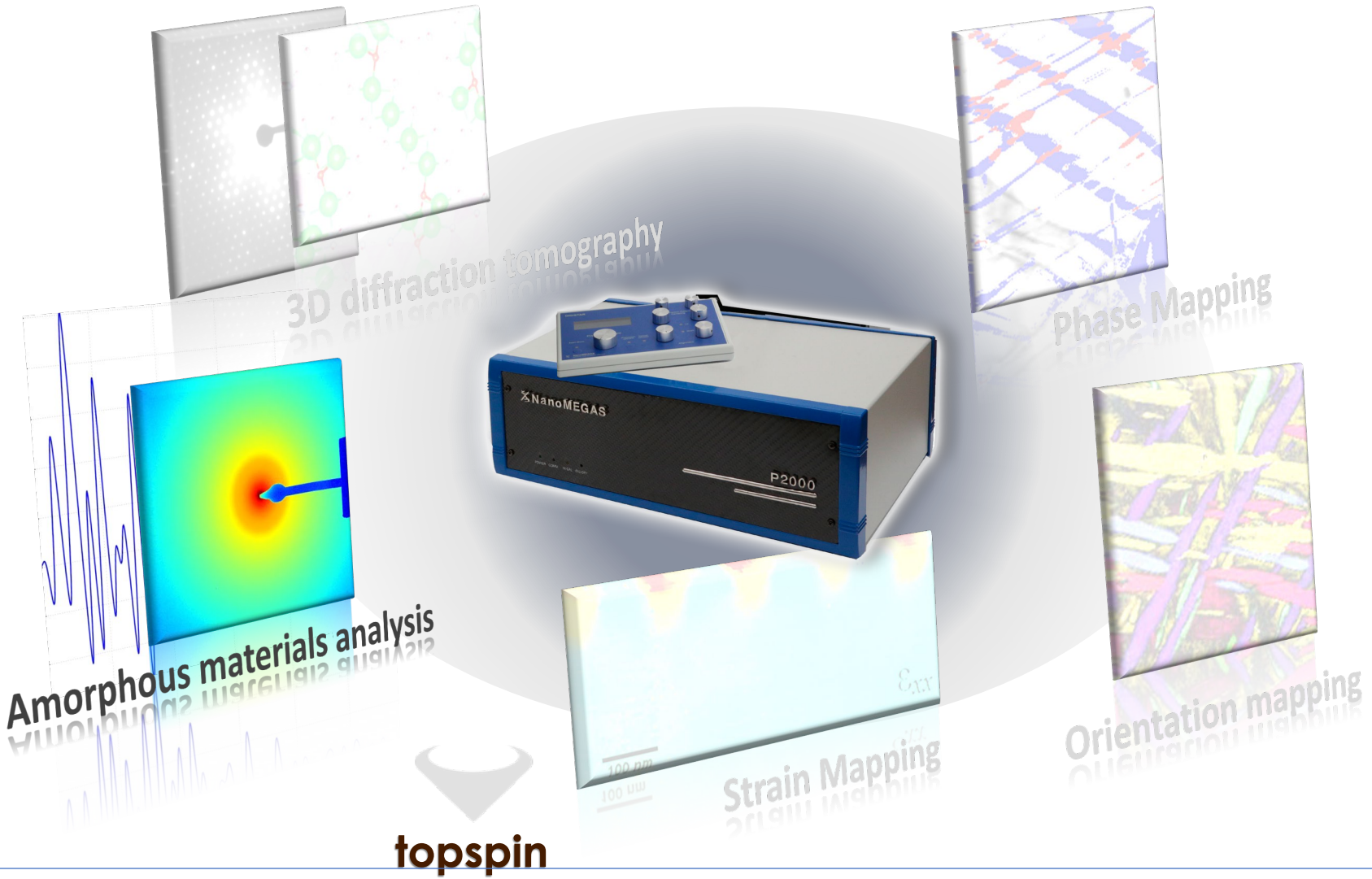
Pigment Yellow

Pharmaceuticals & Proteins



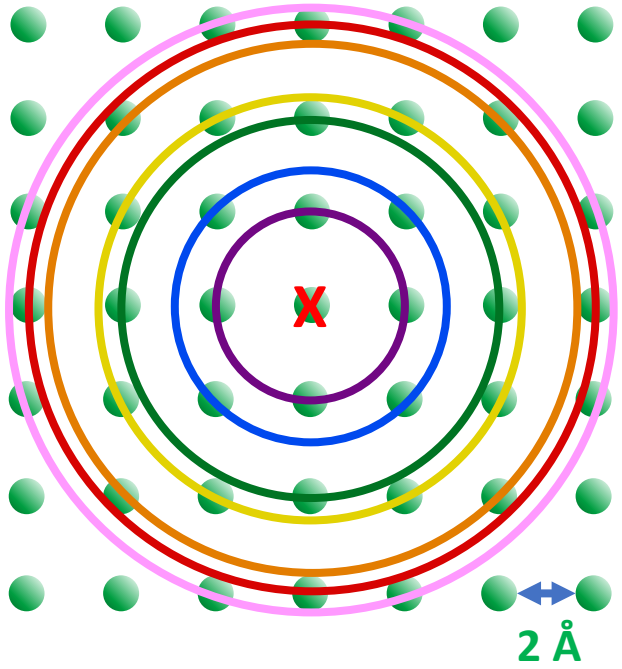
Paracetamol

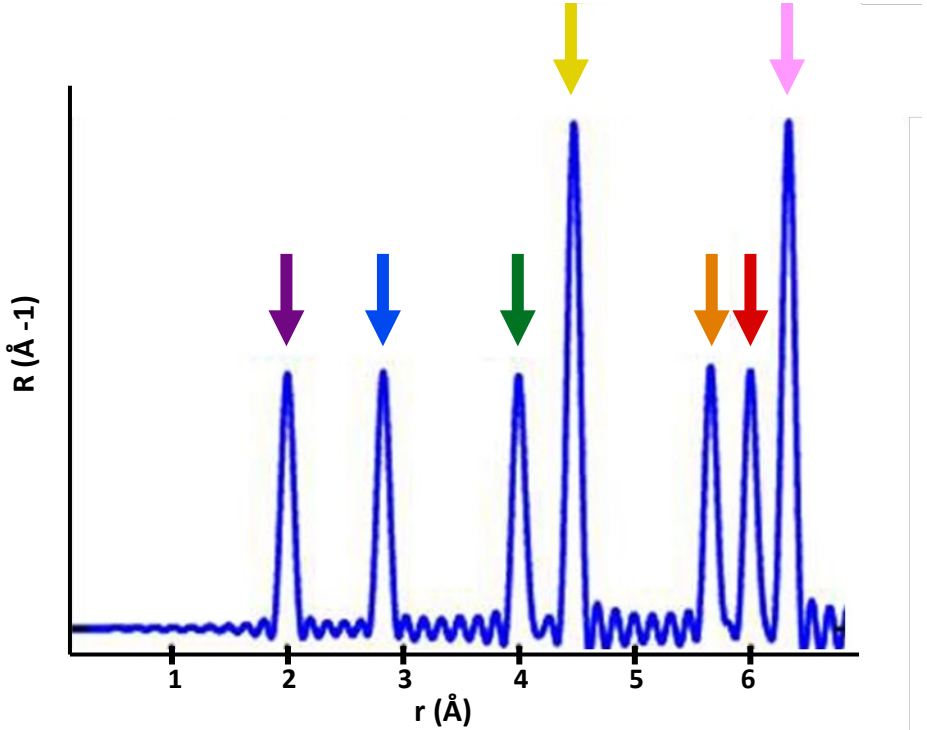
Precession Electron Diffraction Solutions



ePDFsuite: Pair Distribution Function– Characterization of Amorphous materials

Pair Distribution Function G(r) is probability of finding an atom in a distance r

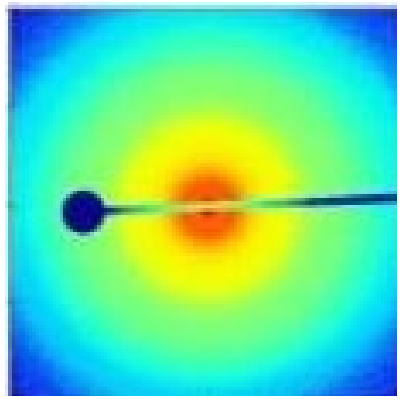


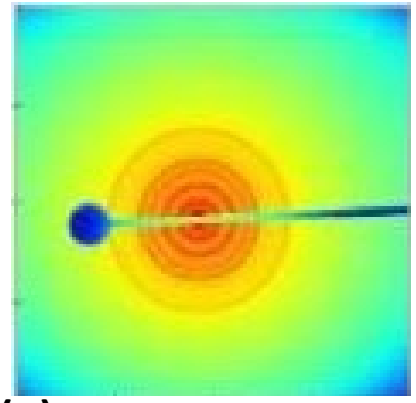


A total pair-distribution function (PDF) is obtained by repeating this process systematically by placing each atom in the origin.

ePDFsuite: Pair Distribution Function– Characterization of Amorphous materials

Amorphous material

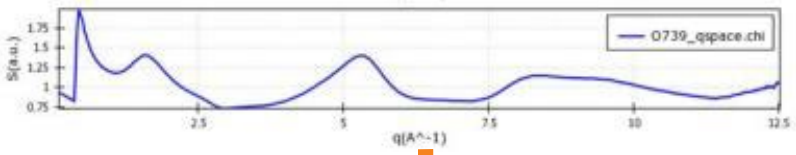


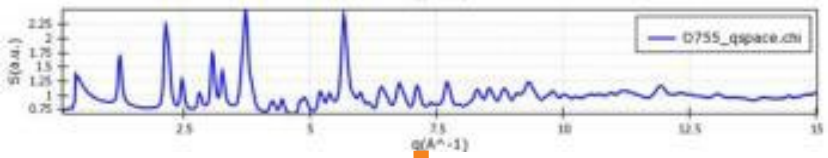


Nanocrystalline

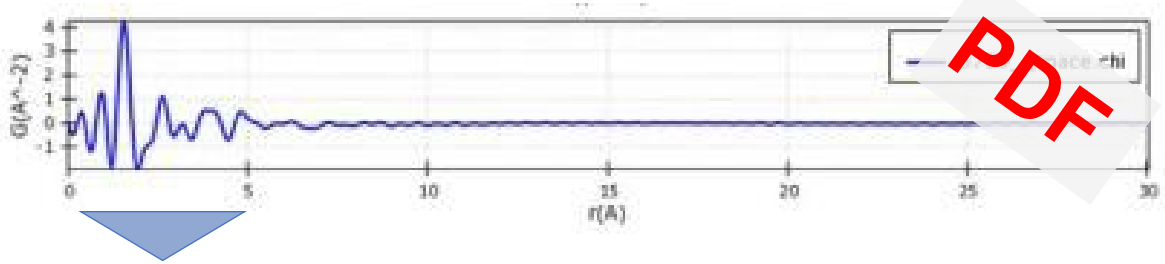
Corrected and Integrated data I(q)

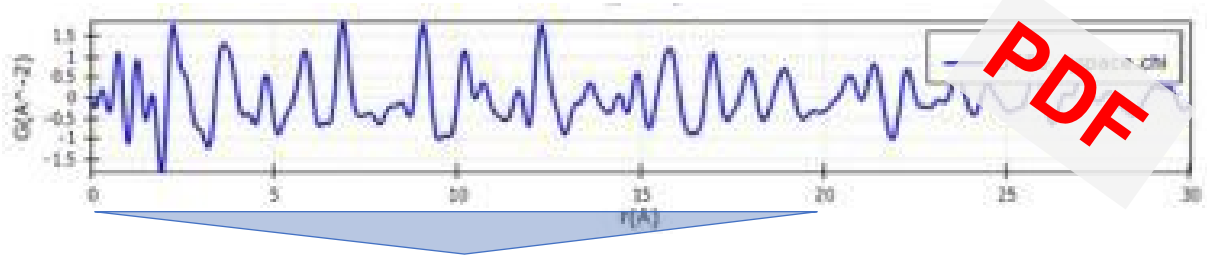
Normalization to calculate S(q)





Fourier transformation

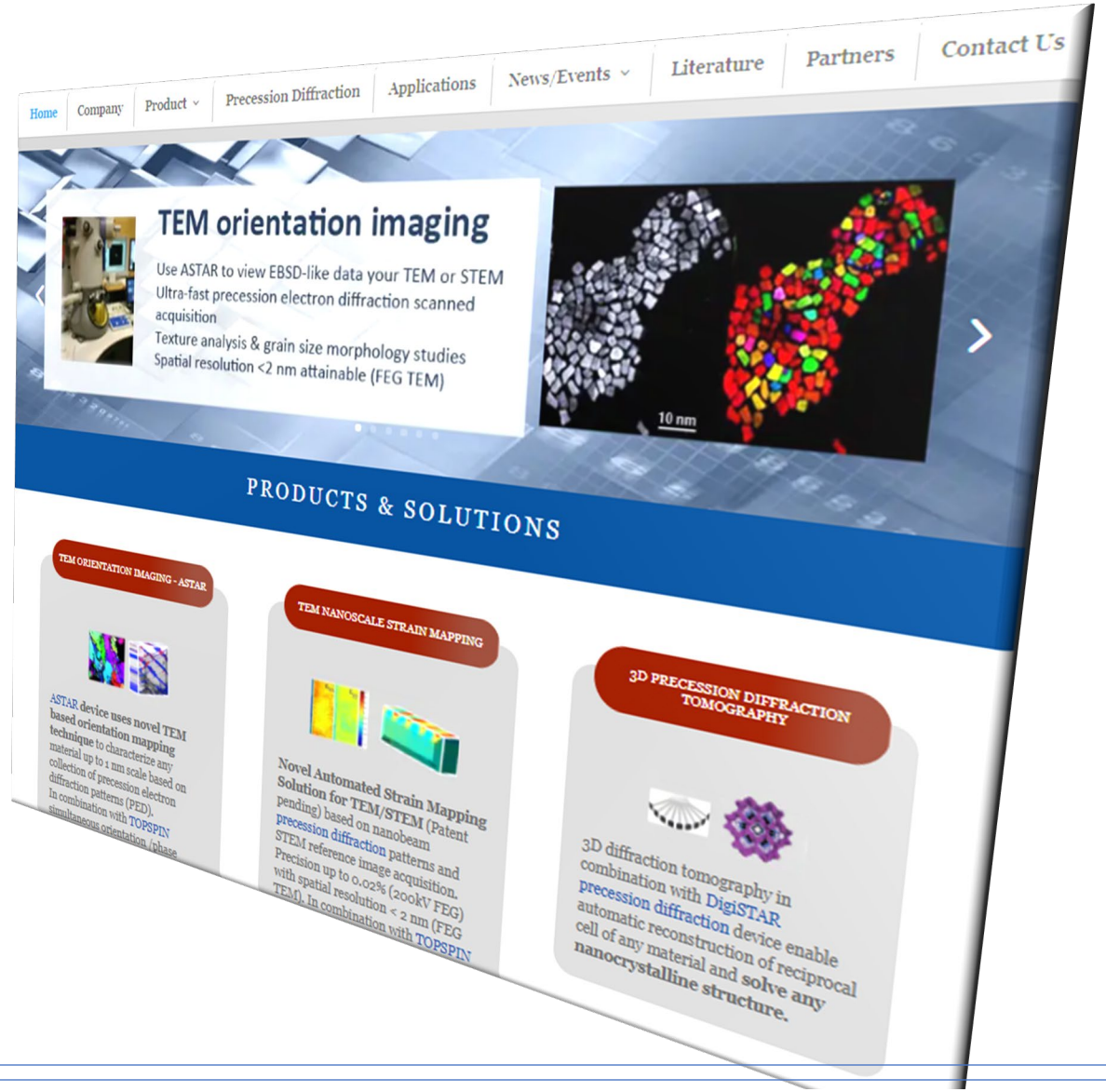




Short range order

Long range order

- Applications details
- Literature on PED techniques
- Past & Forthcoming Webinars



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Thank you!