Use of raw data for diffraction space visualization: What are we missing in an integrated HKL file?

Jim Britten

McMaster University

Canada

Outline

Visualization of area detector scans
Supercells
Incommensurate scattering
Diffuse scattering
Twinning
Thin films
Teaching Crystallography
Other diffraction patterns worth saving

Visualization of area detector scans

Supercells

Incommensurate scattering

Diffuse scattering

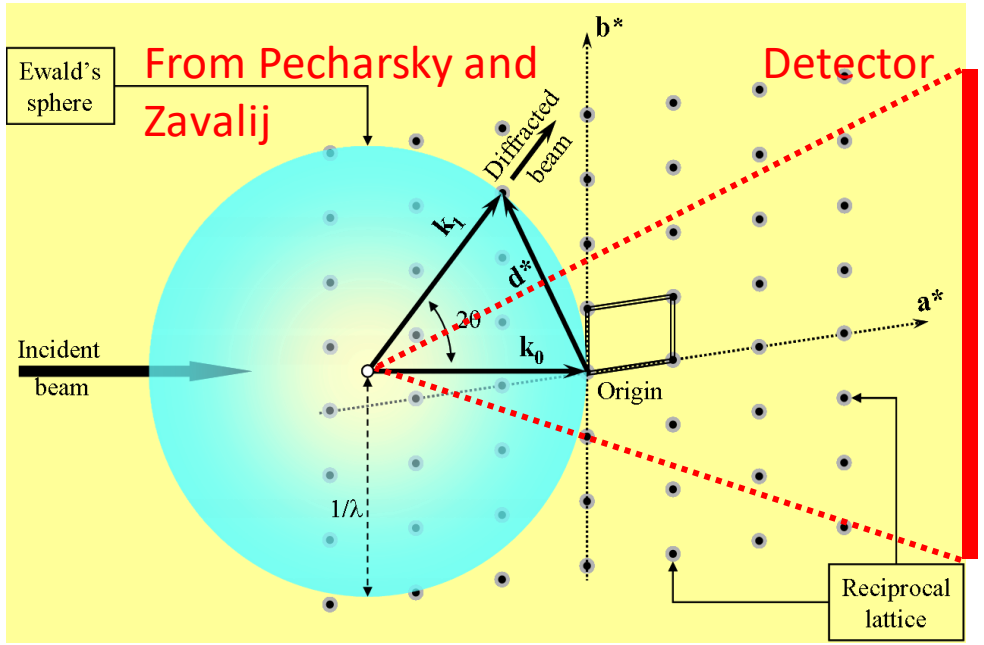
Twinning

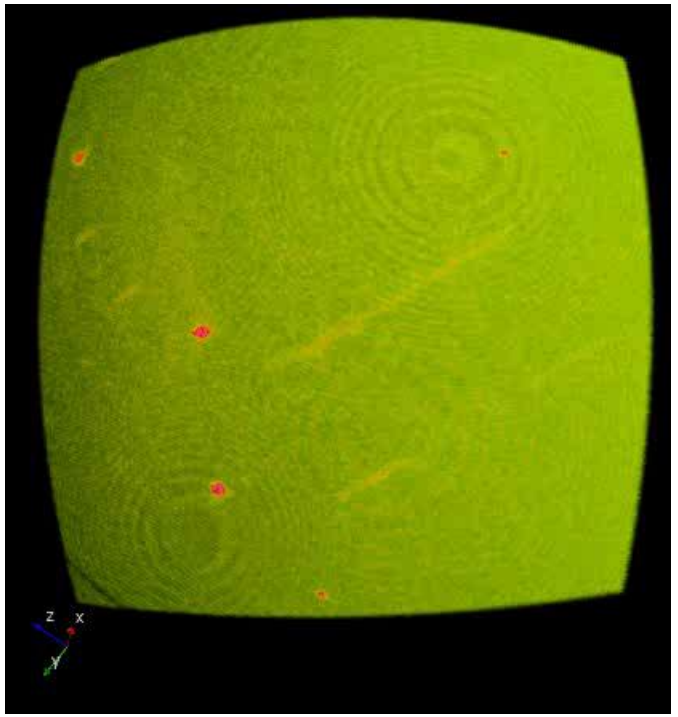
Thin films

Teaching Crystallography

Other diffraction patterns worth saving

Rotate the sample in the beam and collect 2D frames.

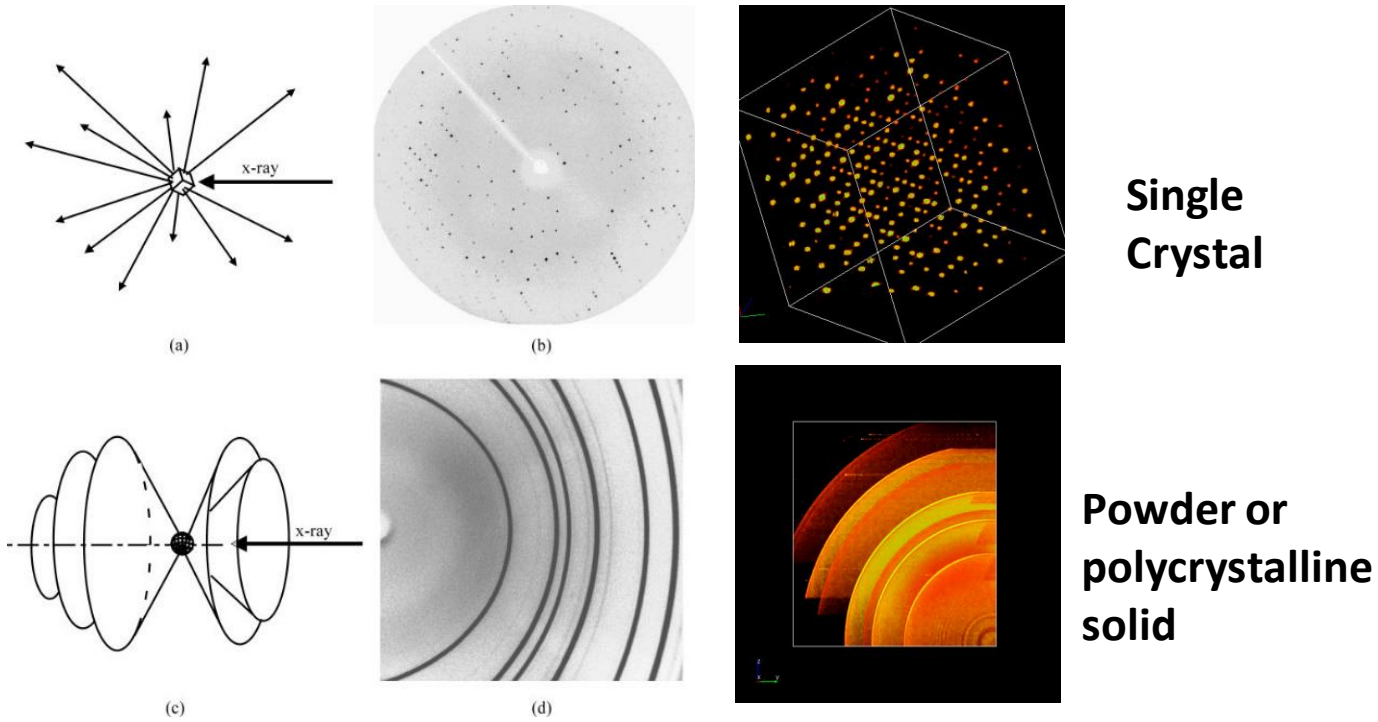




The 2D images can be mapped into reciprocal space – onto the surface of Ewald's Sphere

SCD - 2D image + scan –> 3D Int vs 2θ

XRD³ - 2D image + scan –> 3D Int vs 2θ



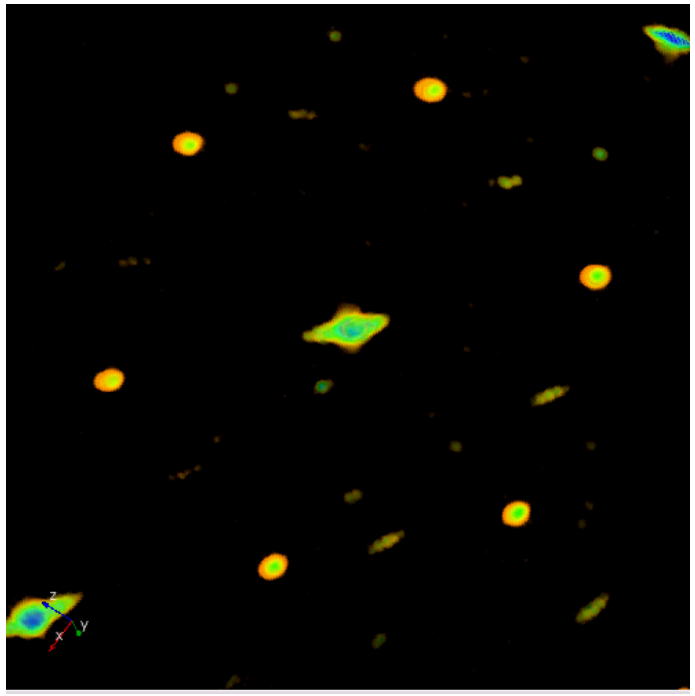
Single Crystal

Powder or polycrystalline solid

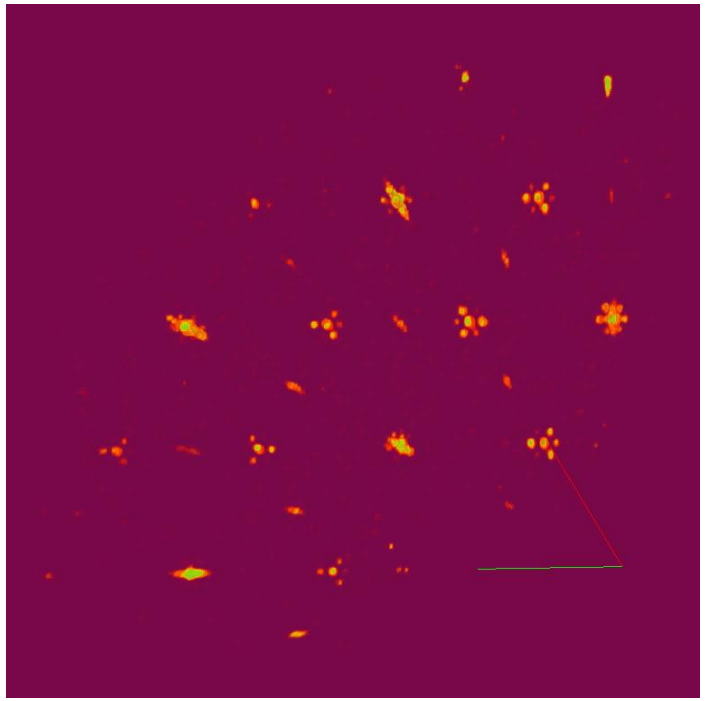
From Bob He's book: Two-Dimensional X-Ray Diffraction

Single Crystal With Long *and* Short Range Ordering ($LuFe_2O_4$)

Y.J. Kim, Toronto



80C

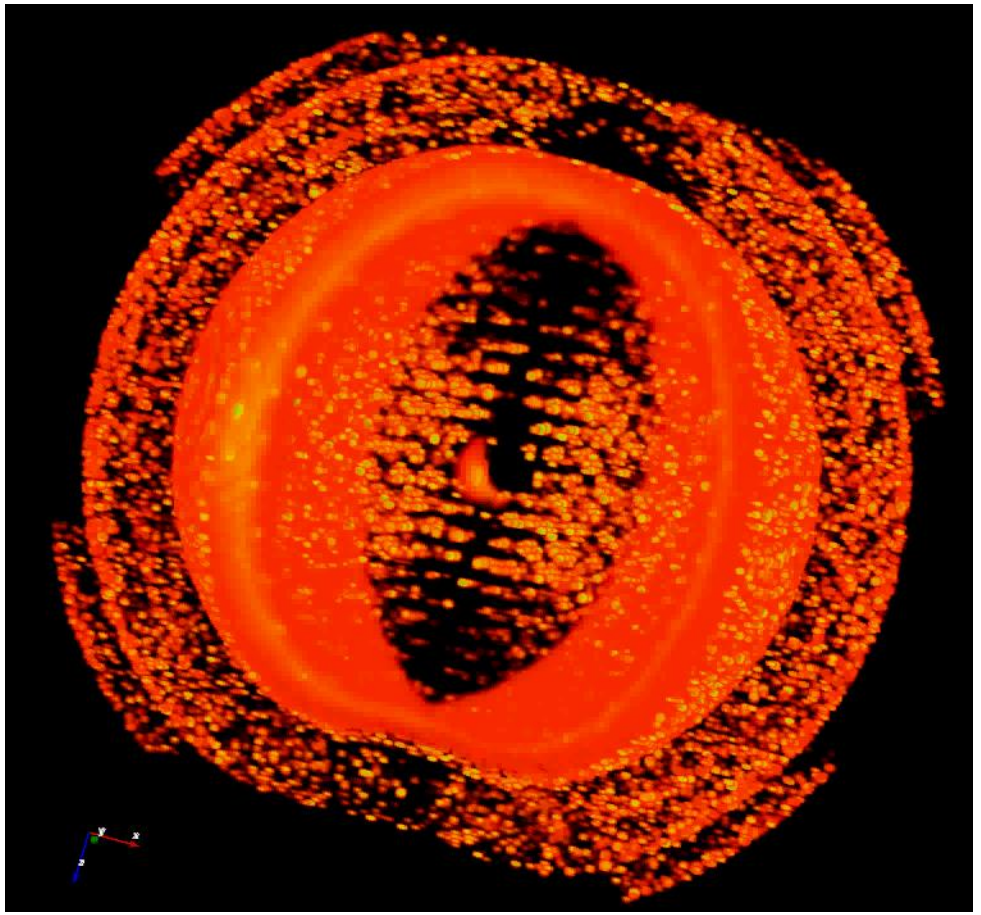


173C

Protein Single Crystal

Alba Guarne
Tamiza Nanji
McMaster

Rigaku
R-Axis4++
Image Plate



Visualization of area detector scans

Supercells

Incommensurate scattering

Diffuse scattering

Twinning

Thin films

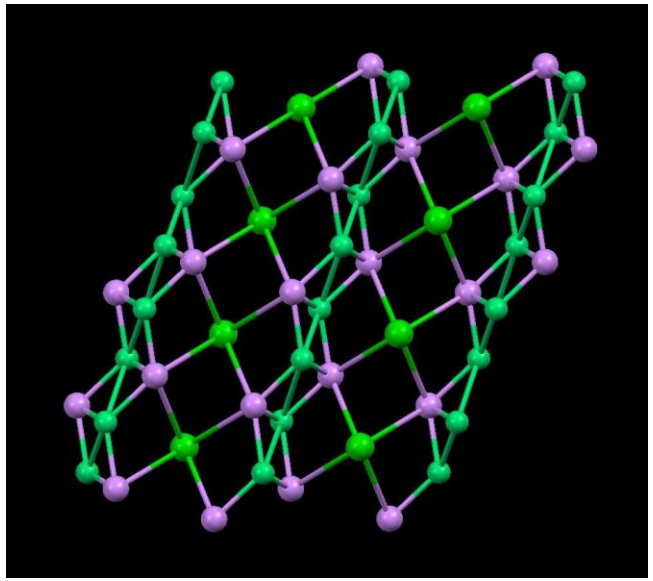
Teaching Crystallography

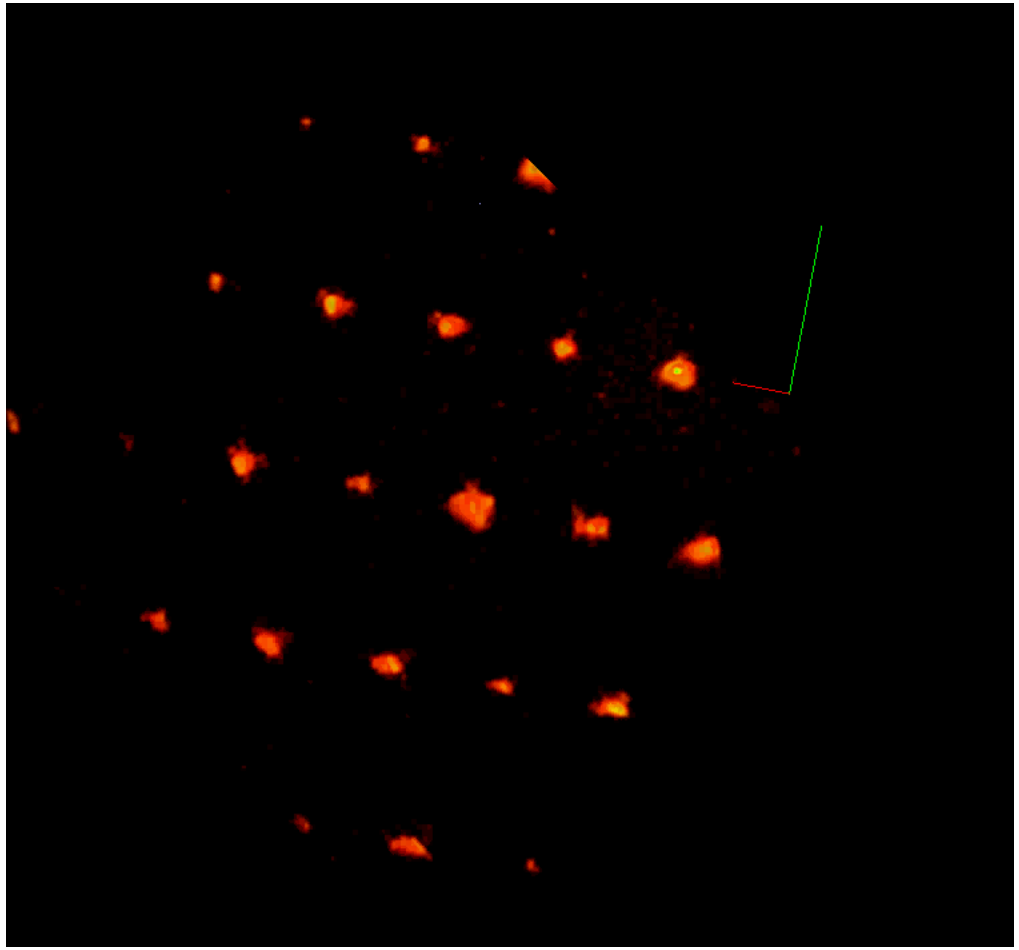
Other diffraction patterns worth saving

Supercell

Athena Safa-Sefat
Yurij Mozharivskij

Ba-As-Ni





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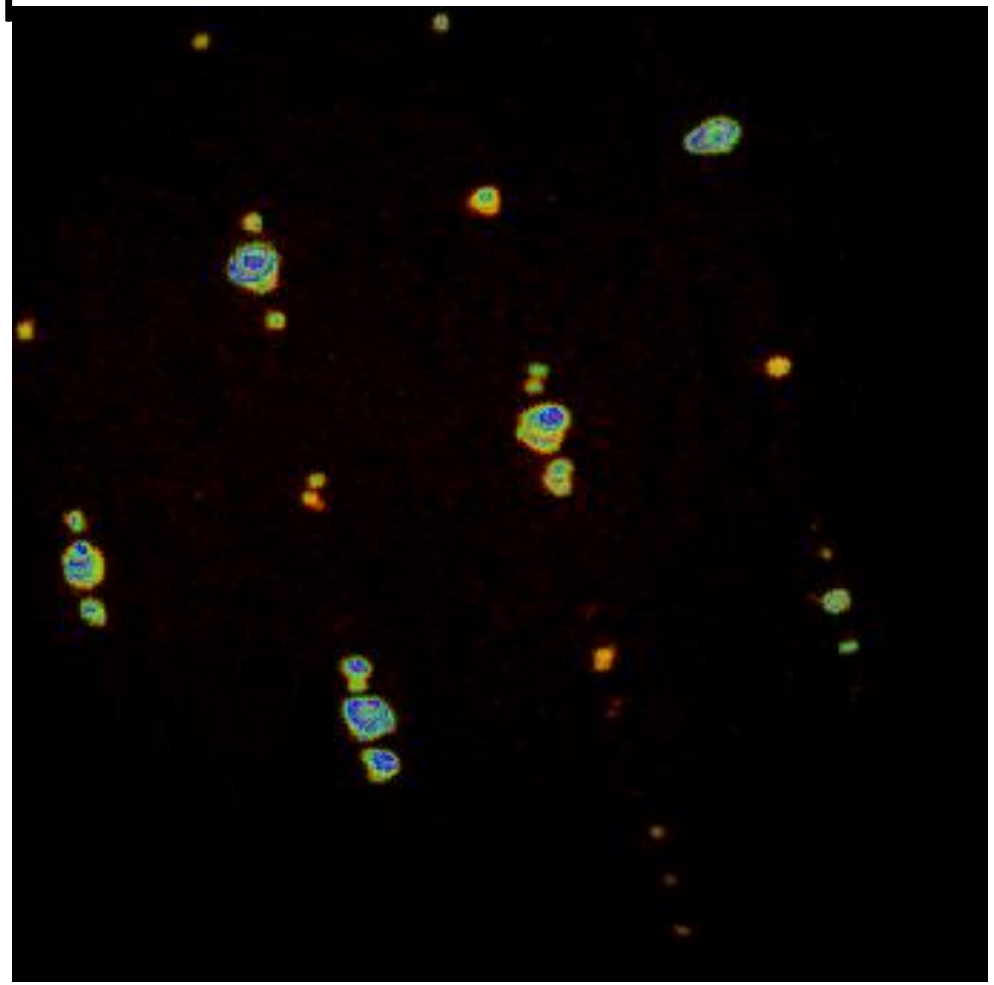
Aperiodic Incommensurate Crystal



Bruce Gaulin – Bi Cu Oxide Superconductor

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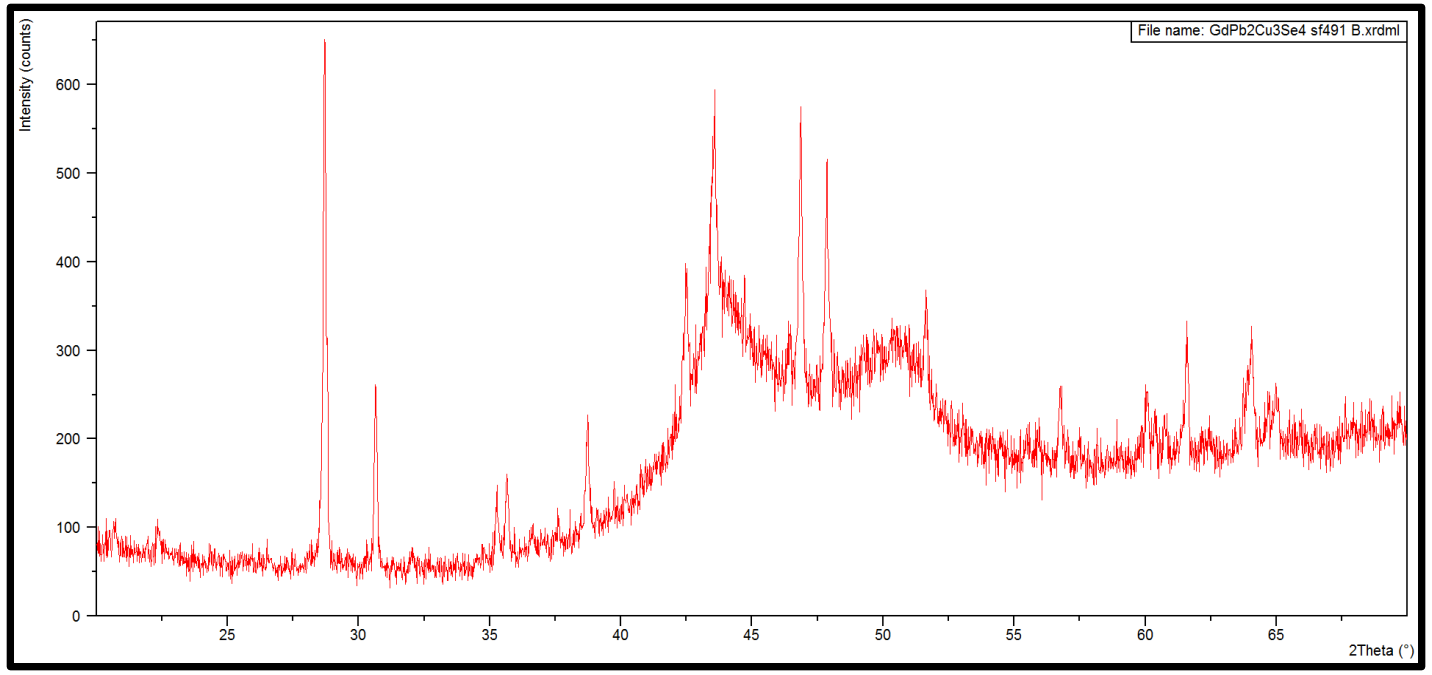
$GdPb_2Cu_3Se_4$ 1200°C for 4 hrs (Plates)

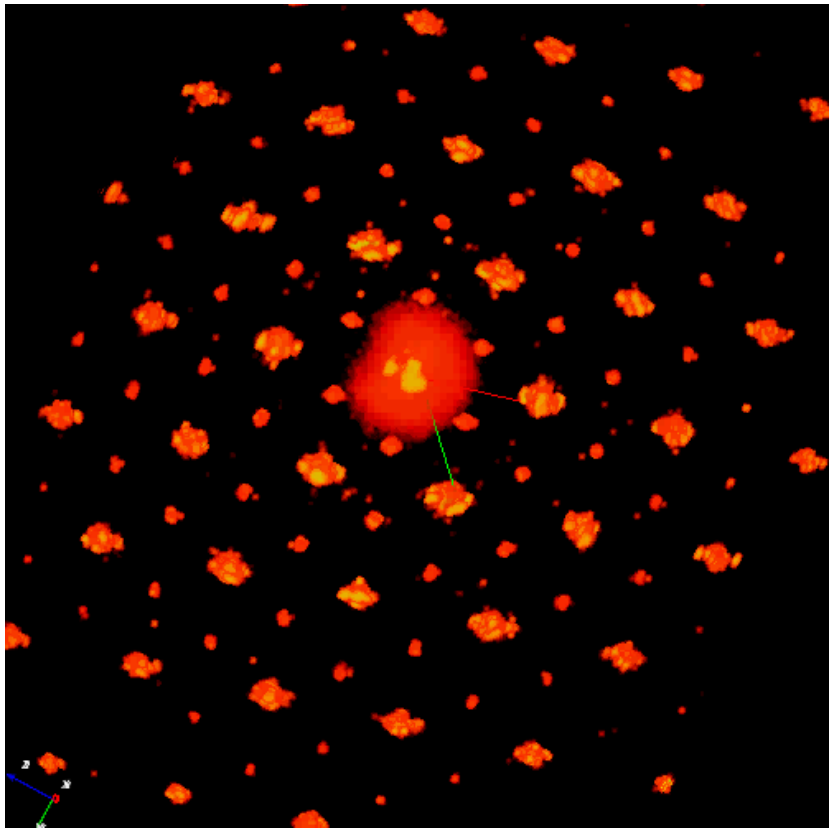
Mozharivskyj, McMaster

XRD pattern from Panalytical X'Pert Pro Diffractometer, Cu $K\alpha_1$



$GdPb_2Cu_3Se_4$

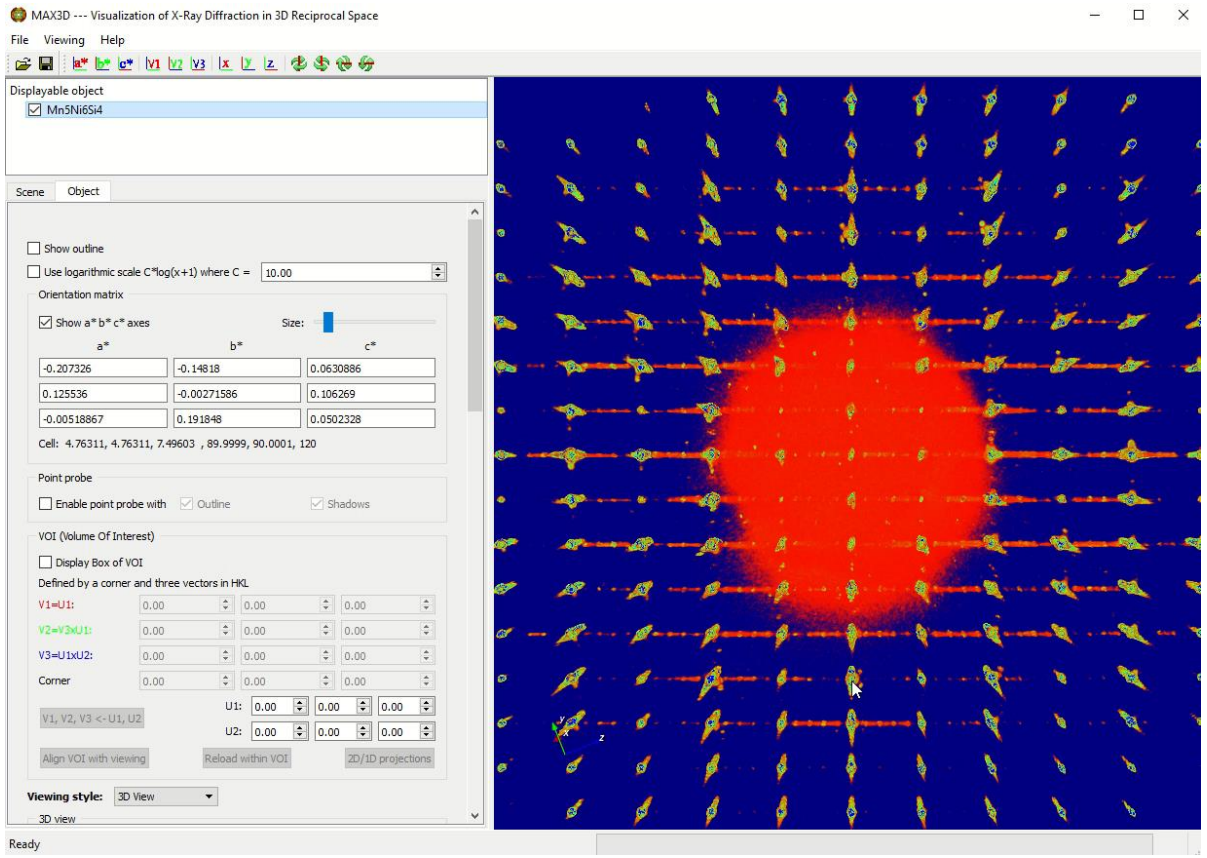
Look at a ‘single’ grain of the powder on a protein beamline.

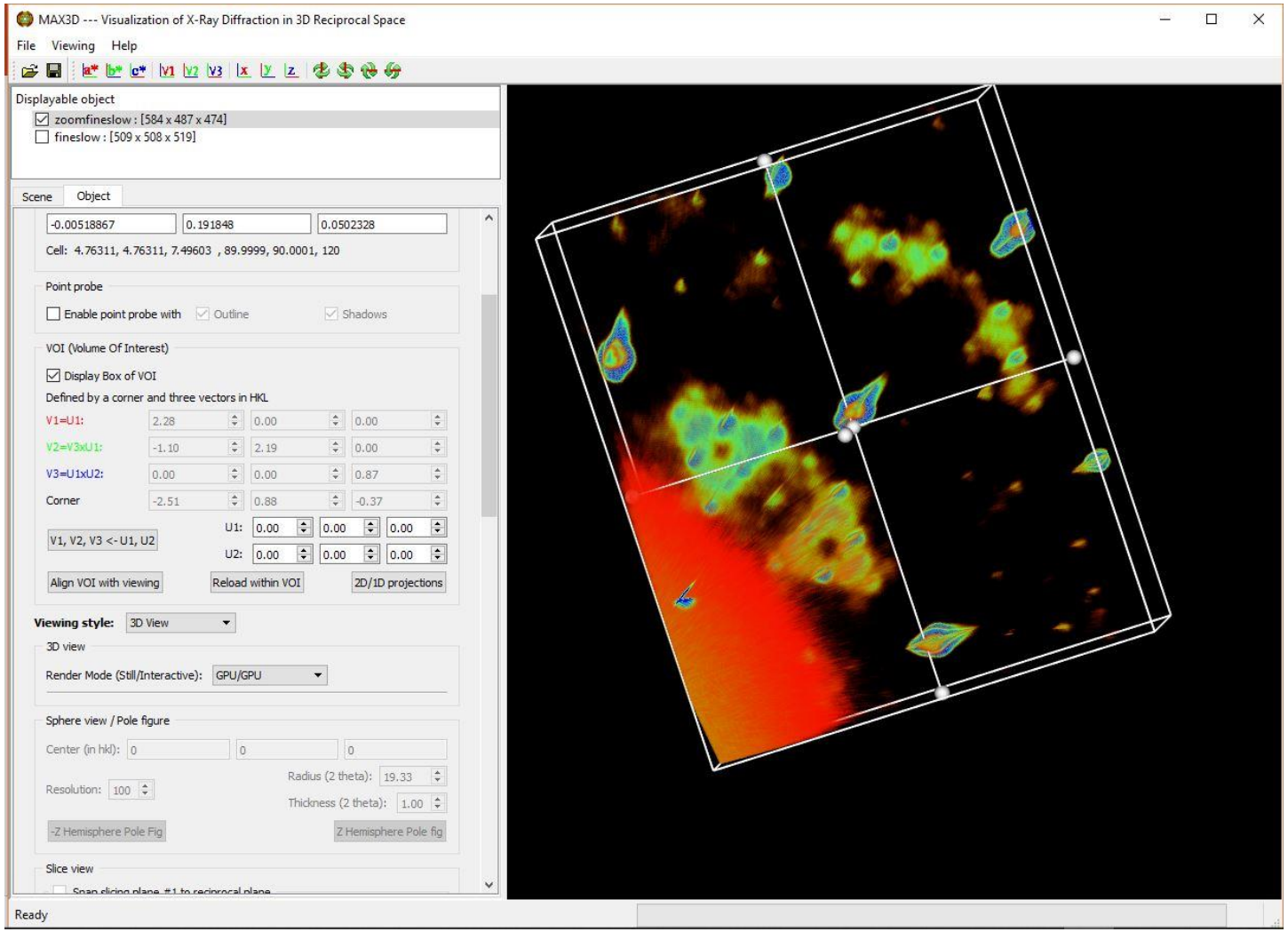


Canadian Macromolecular Crystallography Facility, 08B1-1 (CMCF-BM) Beamline

$Mn_5Ni_6Si_4$

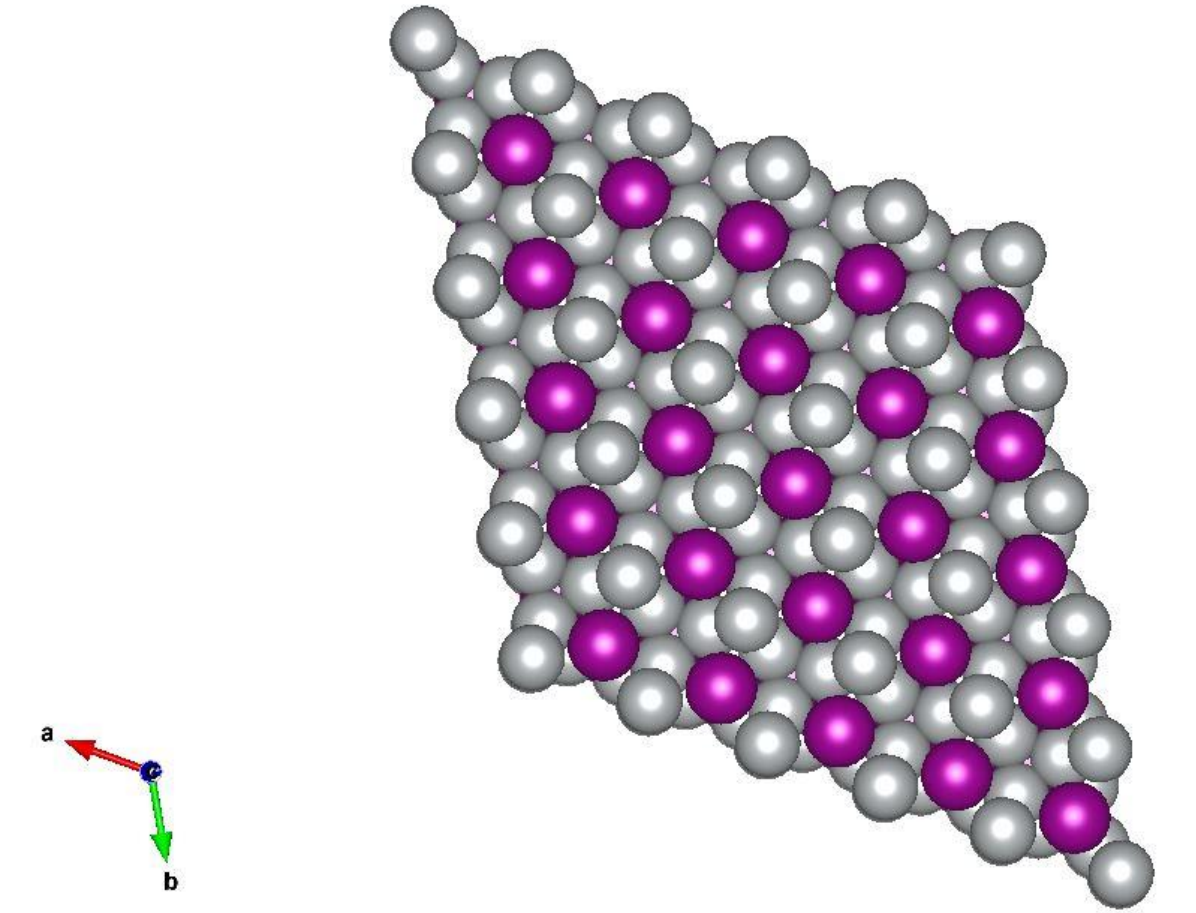
Marek Niewczas, Sheikh Ahmed, McMaster



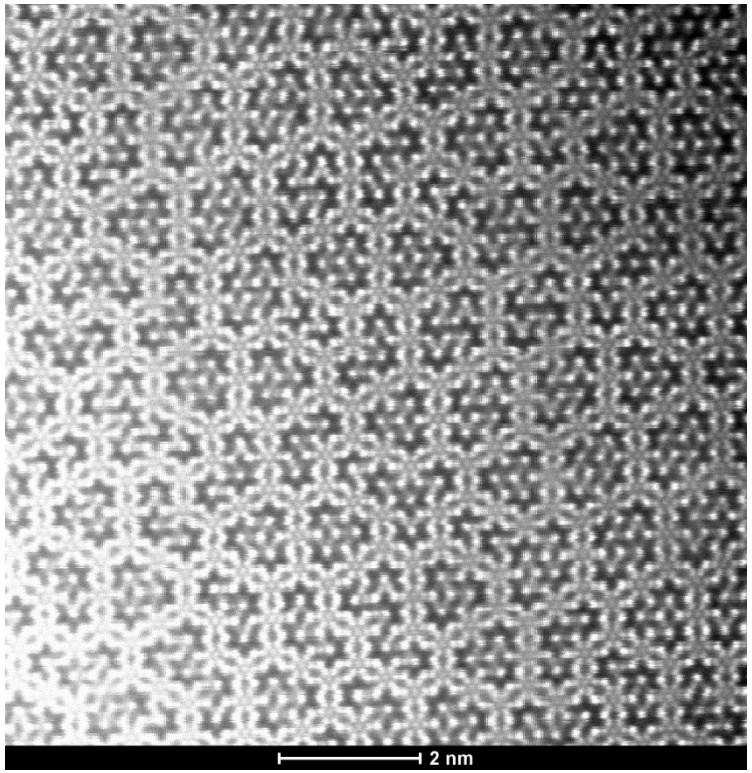

MAX3D --- Visualization of X-Ray Diffraction in 3D Reciprocal Space
File
Viewing
Help
Displayable object
zoomfineslow : [584 x 487 x 474]
fineslow : [509 x 508 x 519]
Scene
Object
-0.00518867
0.191848
0.0502328
Cell: 4.76311, 4.76311, 7.49603 , 89.9999, 90.0001, 120
Point probe
Enable point probe with
Outline
Shadows
VOI (Volume Of Interest)
Display Box of VOI
Defined by a corner and three vectors in HKL
V1=U1:
V2=V3xU1:
V3=U1xU2:
Corner
U1:
U2:
V1, V2, V3 <- U1, U2
Align VOI with viewing
Reload within VOI
2D/1D projections
Viewing style:
3D View
3D view
Render Mode (Still/Interactive):
GPU/GPU
Sphere view / Pole figure
Center (in hkl):
Resolution:
Radius (2 theta):
Thickness (2 theta):
-Z Hemisphere Pole Fig
Z Hemisphere Pole fig
Slice view
Ready

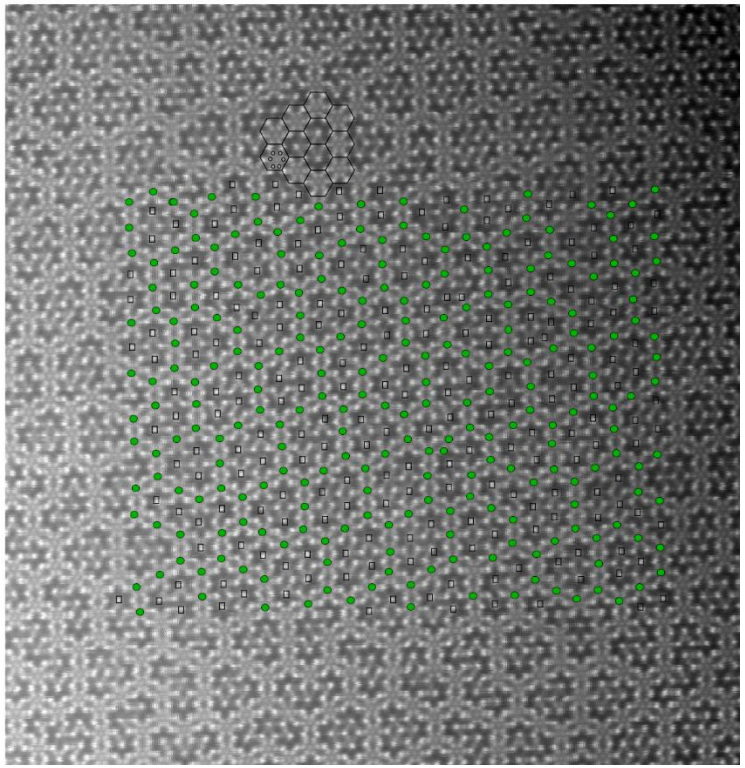
$Mn_5Ni_6Si_4$

Marek Niewczas, Sheikh Ahmed, McMaster

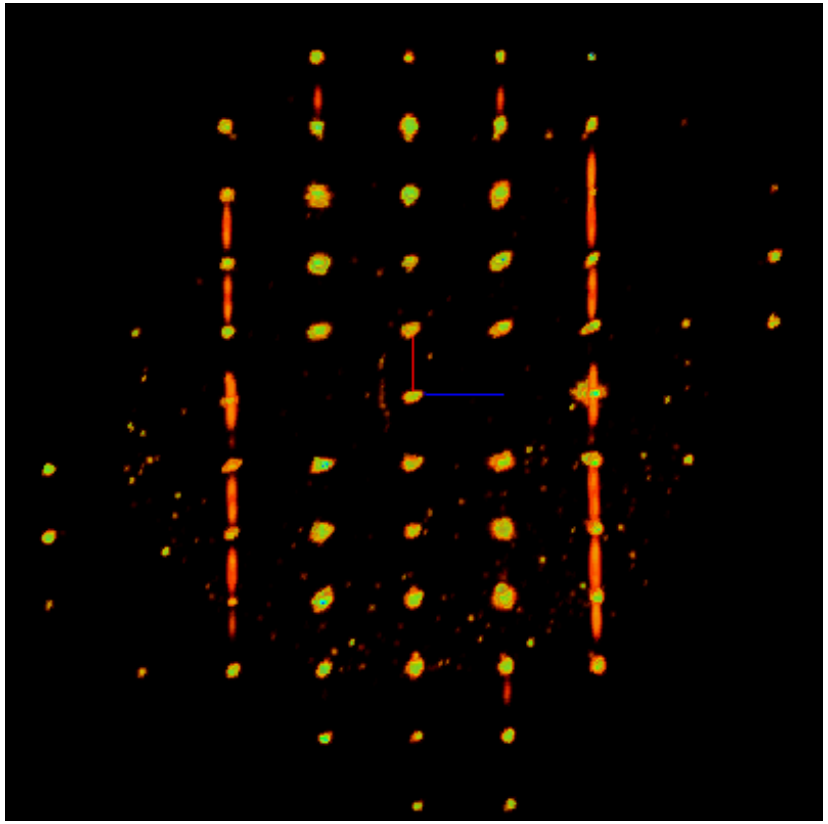


HRTEM





Diffuse Scattering



Columns of hexanapthylbenzene are ordered along the stacking axis. The columns have a partial rotational disorder relative to one another.
The refined structure shows a multiple orientations for the napthyls. The configuration of the molecule in the ordered stack have not been determined.

Hexanapthylbenzene. Laura Harrington, Mike McGlinchey, McMaster

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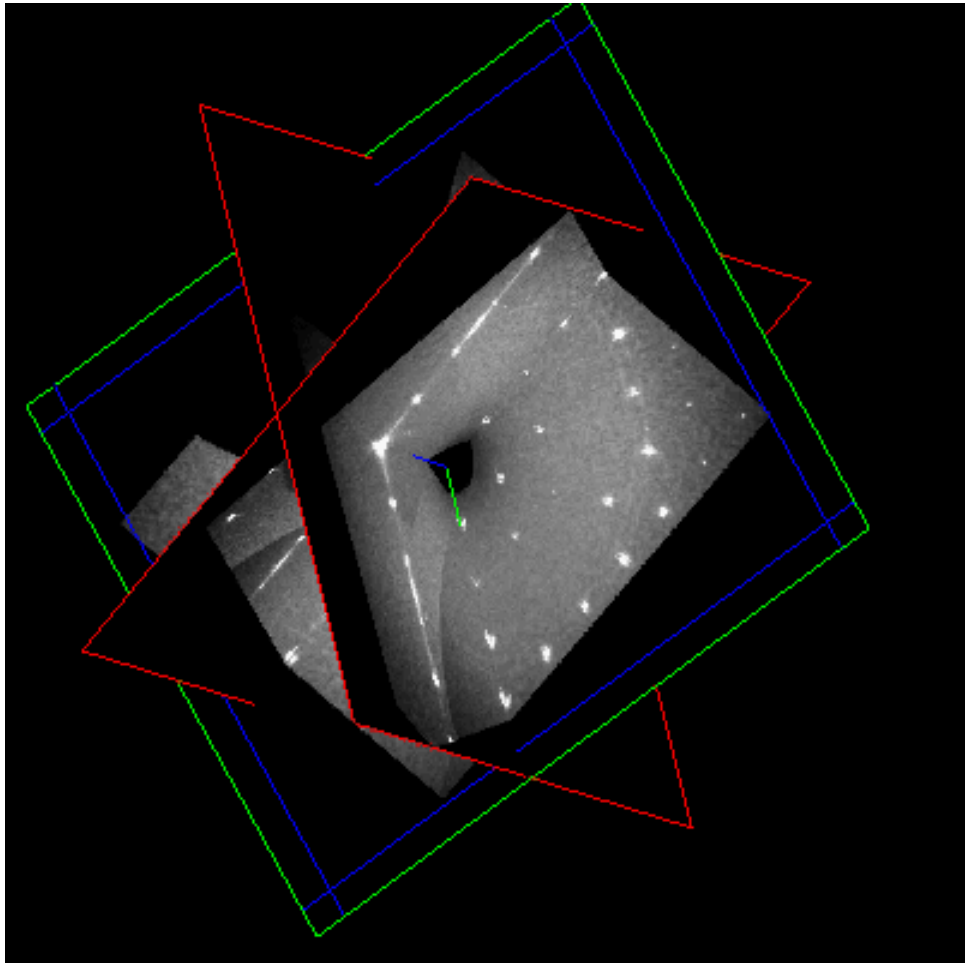
Diffuse Scattering



Hexanapthylbenzene. Laura Harrington, Mike McGlinchey, McMaster

Visualization of area detector scans

Supercells

Incommensurate scattering

Diffuse scattering

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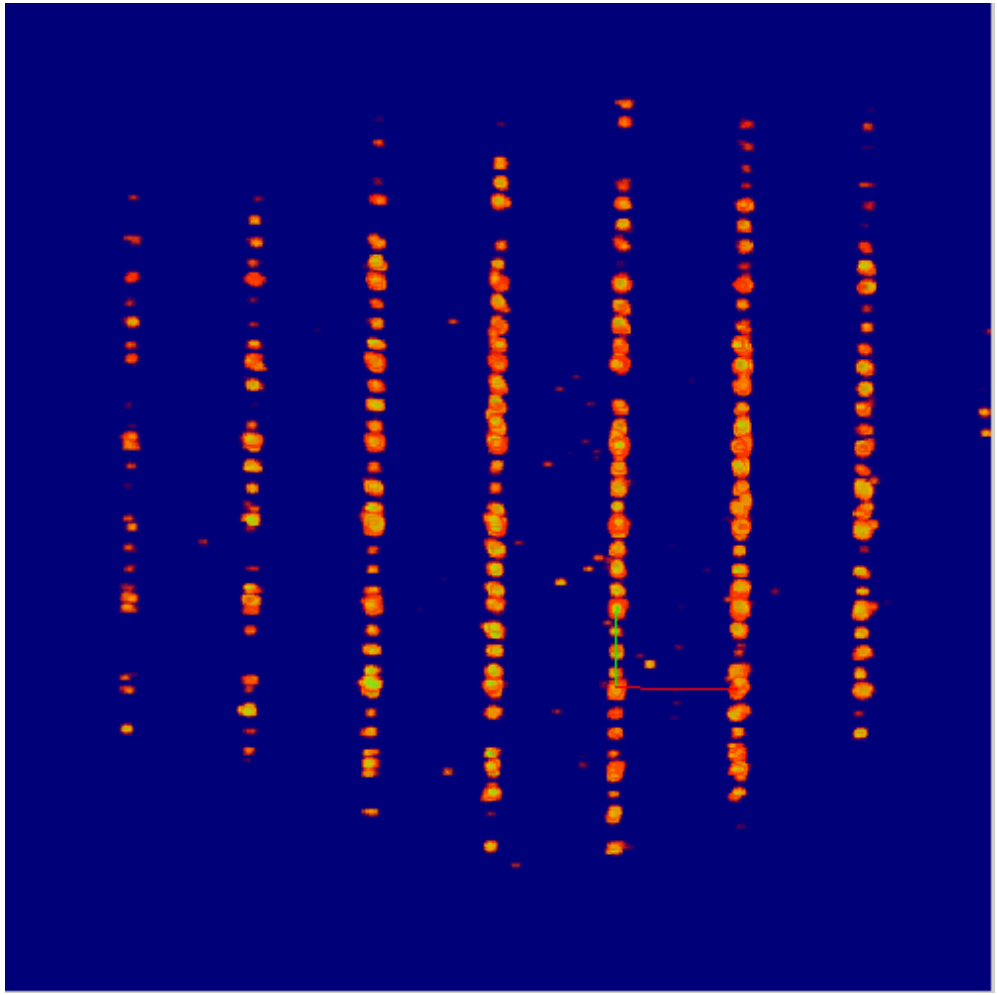
Thin films

Teaching Crystallography

Other diffraction patterns worth saving

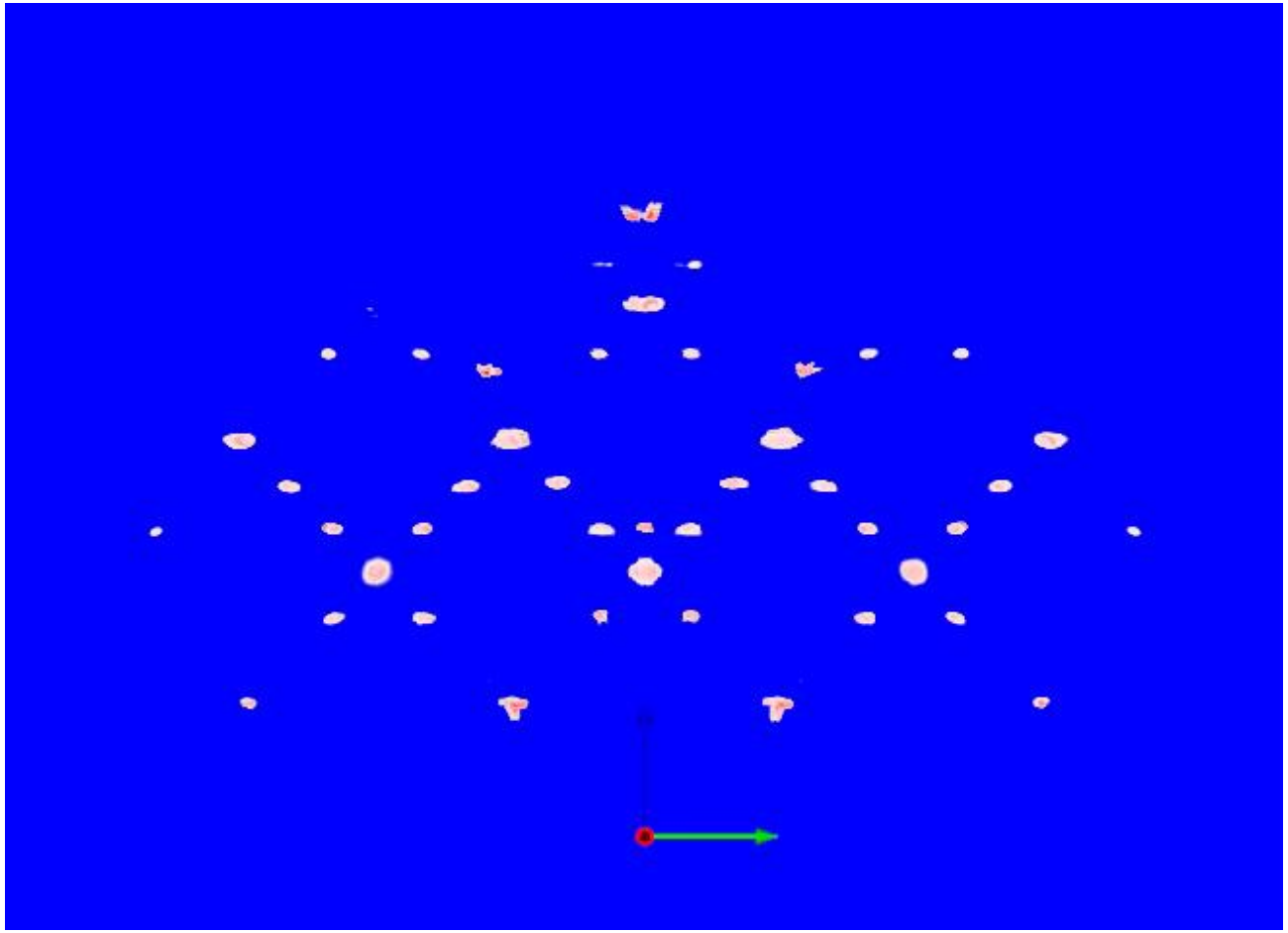
Small Molecule Twinned Crystal

Bruker Smart Apex2 CCD



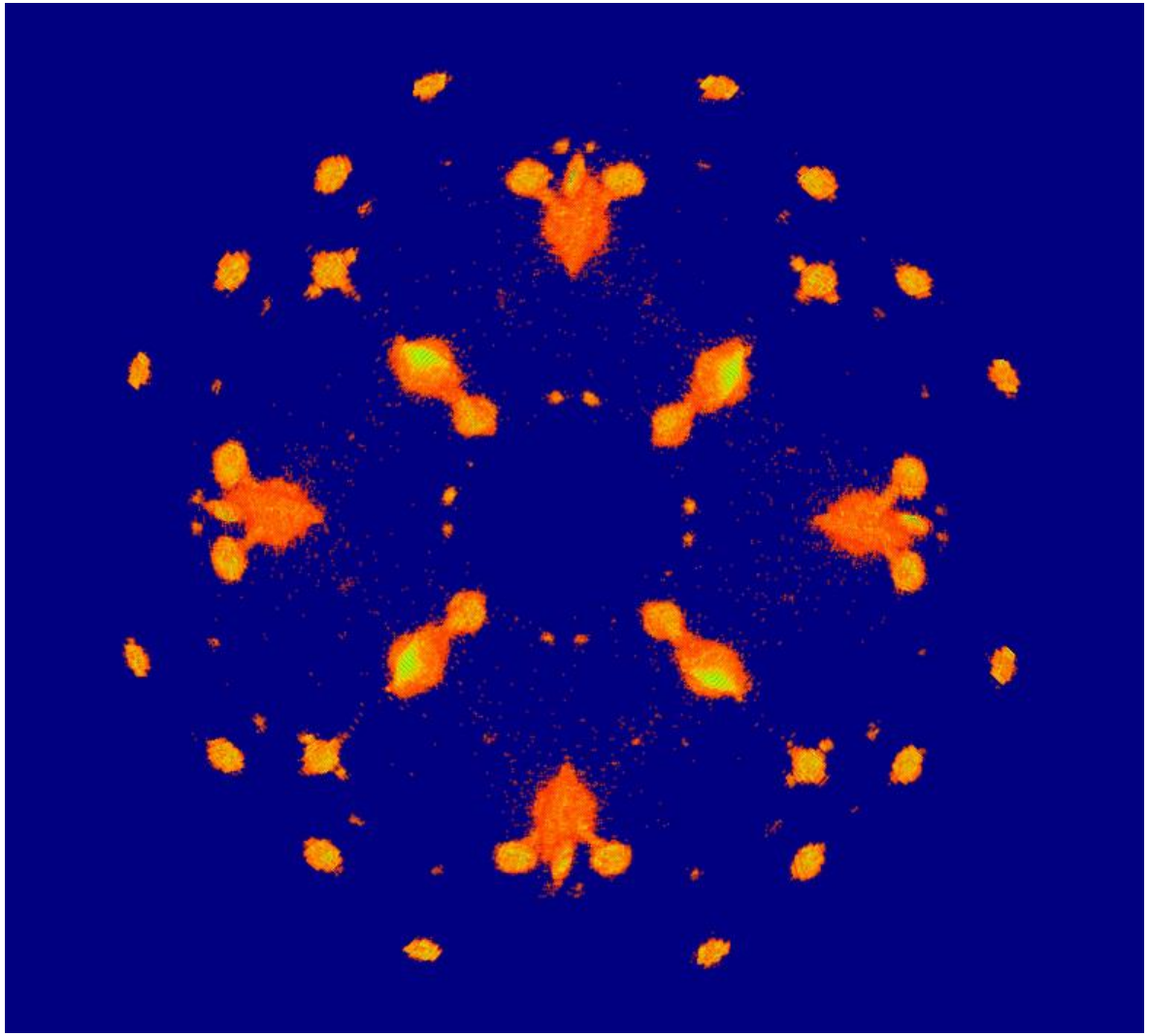
3D diffraction pattern from thin film of $InAs_{(1-x)}Sb_x$ nanowires (isolate (111) reflections)

Goosney, Jarvis, Britten, Lapierre, McMaster



(220) and
(311) shells

Diffuse lines
connect twins



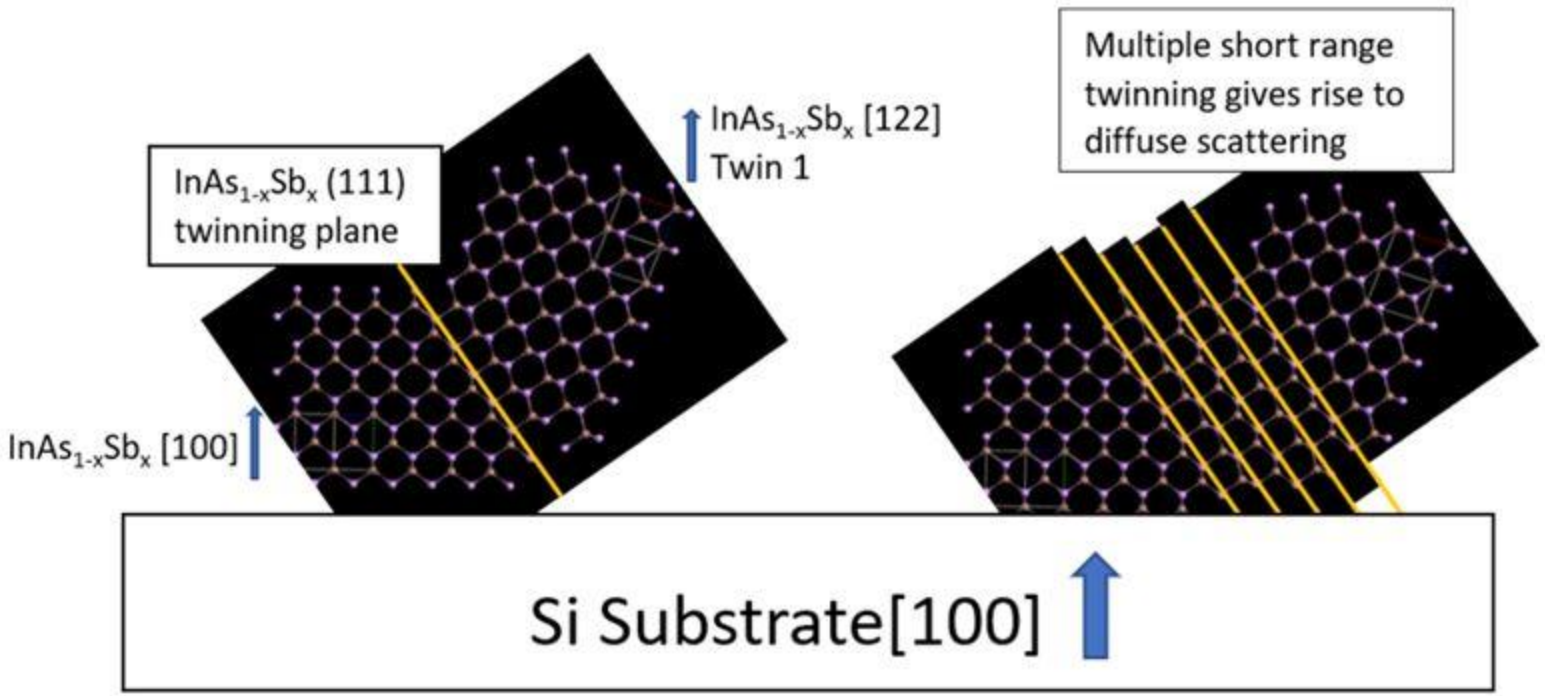


Figure 3. Twinning by 180° (or ±60°) rotation about the [111] face of $InAs_{1-x}Sb_x$. Regions of multiple layer twinning account for the diffuse scattering observed in the 3D diffraction pattern. Twin planes are indicated by yellow lines.

Goosney, Jarvis, Britten, Lapierre, Infrared Physics and Technology

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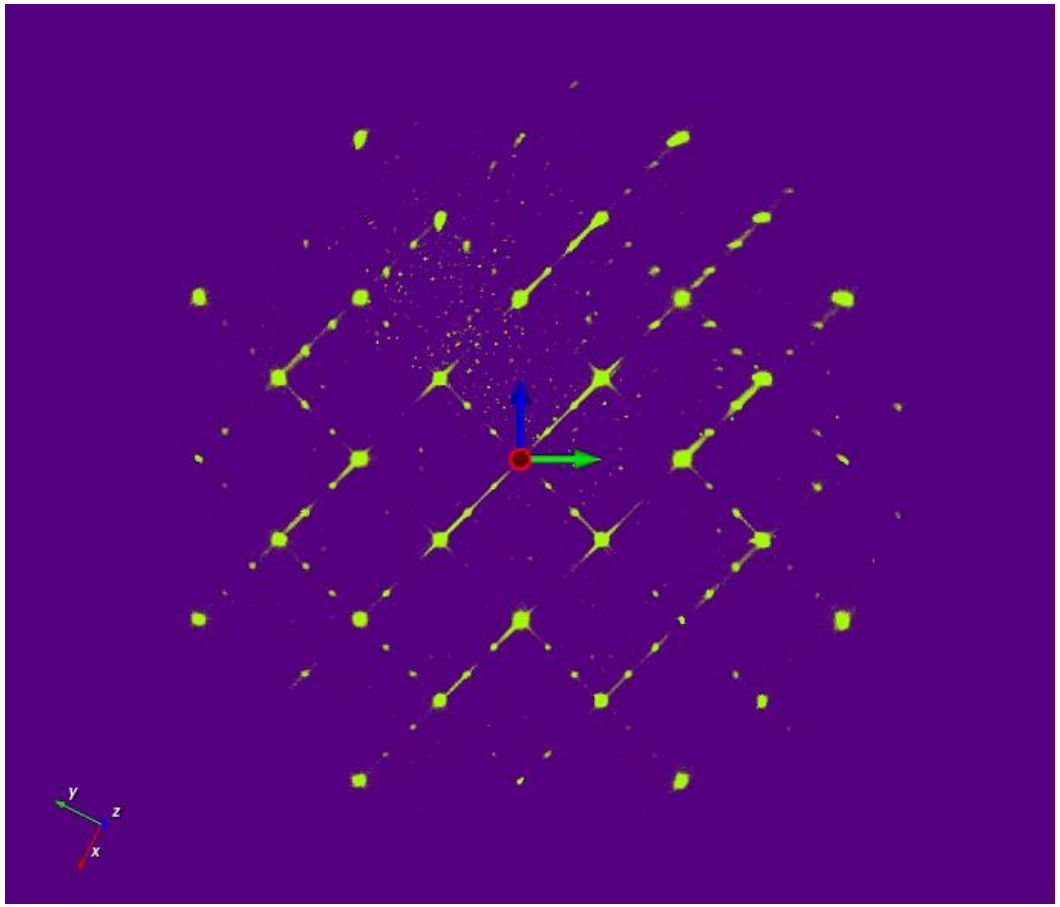
$Ga_{1-x}In_xAs$ film on 100 GaAs Substrate

Ryan Lewis, Spencer McDermott, McMaster

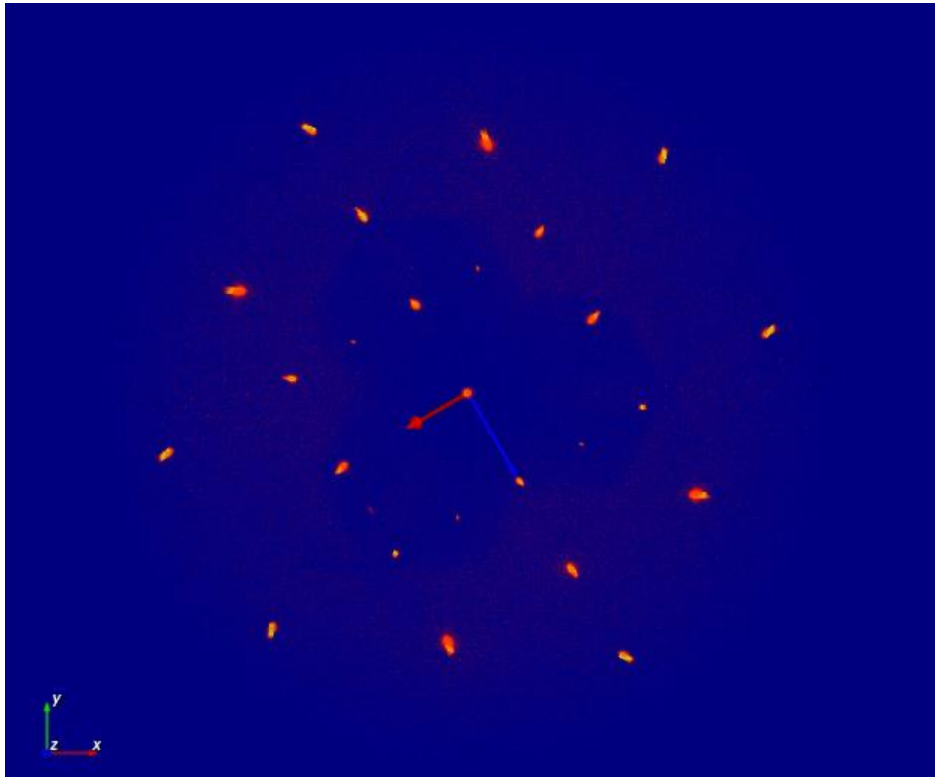
Evidence of film twinning and nano-twinning along <111>



Thin Film Strain – Tuning the Band Gap

30nm film
($Pn\bar{3}m$) on
110 Substrate
($Fm\bar{3}m$)

Yimin Wu
Jeffrey Chen
Waterloo
Canada

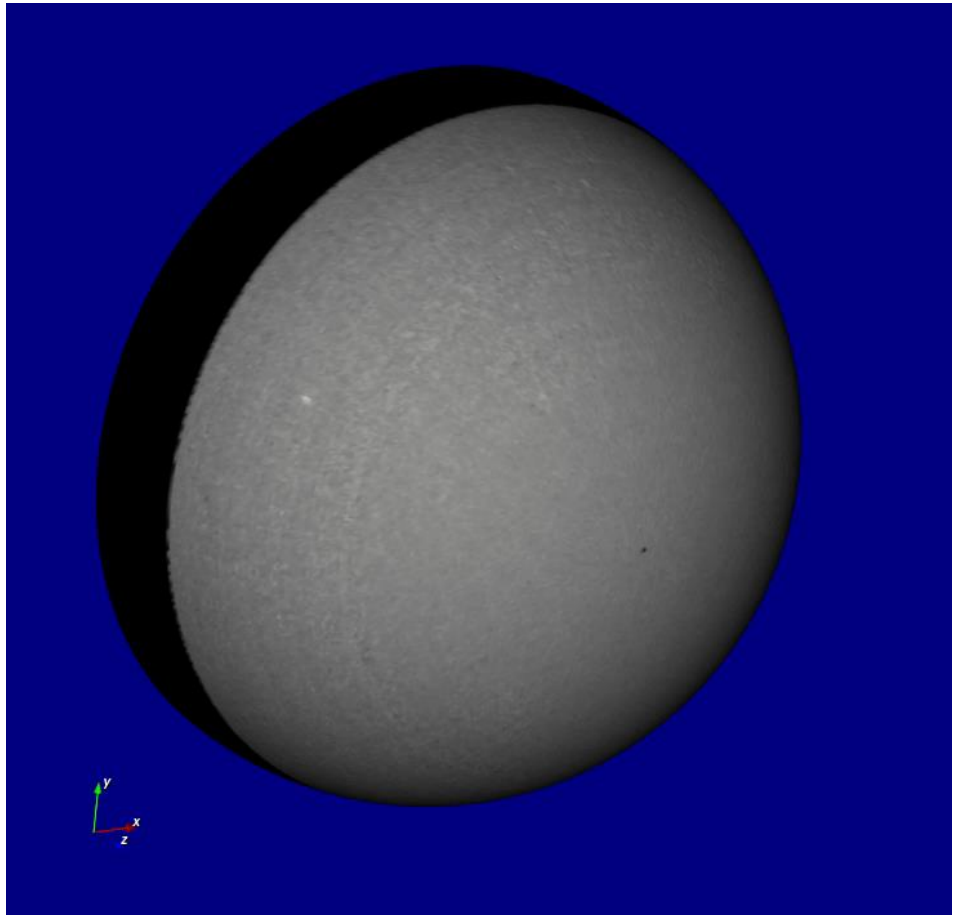


Bruker Venture D8
Cu IμS source
Photon III detector
65 minute
collection

Thin Film Strain – Tuning the Band Gap

30nm film ($Pn\bar{3}m$) on 110 Substrate ($Fm\bar{3}m$)

Yimin Wu
Jeffrey Chen
Waterloo
Canada



Scanning through 2θ for film { 1 1 0} reflections.

You can see the 2θ difference for the peak normal to the plane compared to the others.

A longer exposure would give us enough intensity to calculate the strained cell of the film.

Thin Film Strain – Tuning the Band Gap

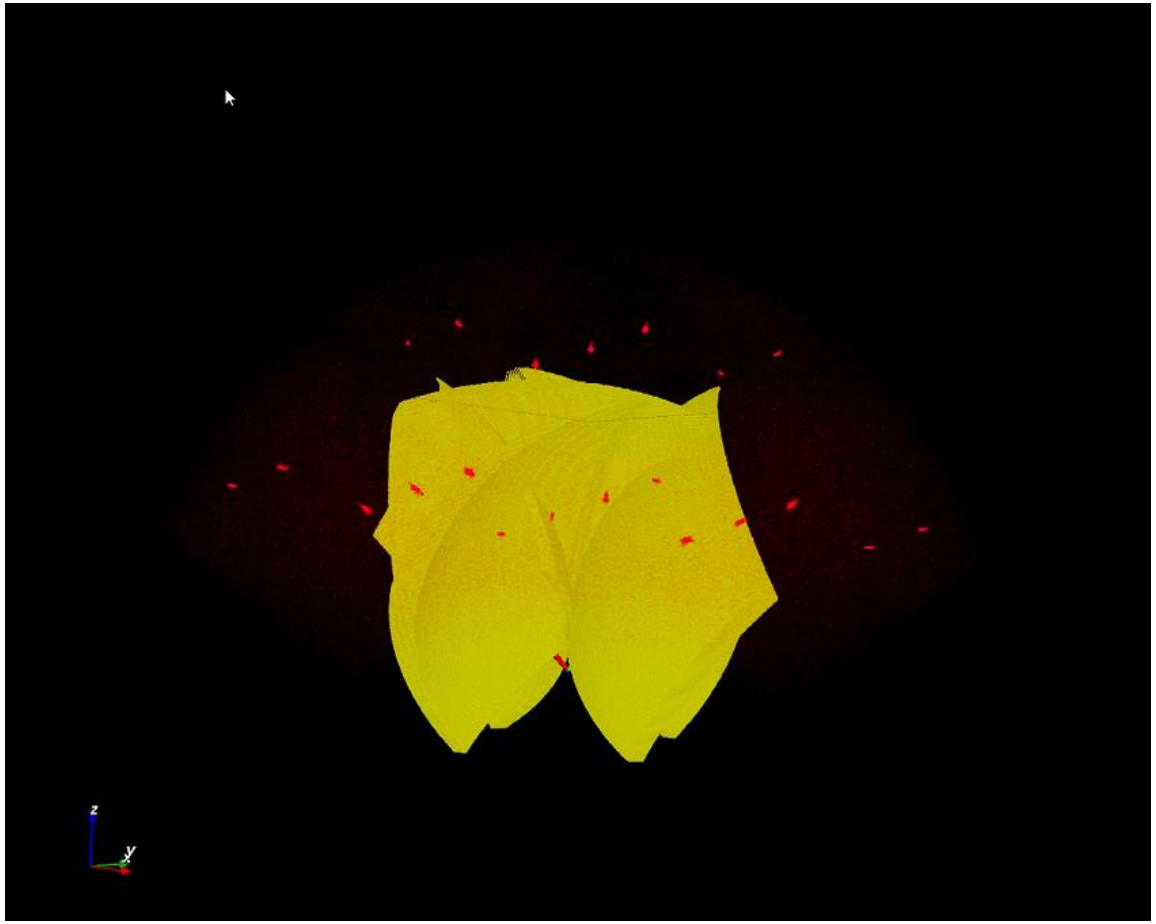
Use Substrate lattice to predict positions of [110] set of film reflections.

5 separate 12 min scans.

Refine film cell based only on the [110] set of reflections.

CELL 4.2982 4.2982 4.1689
90.0000 90.0000 90.0000 77.018

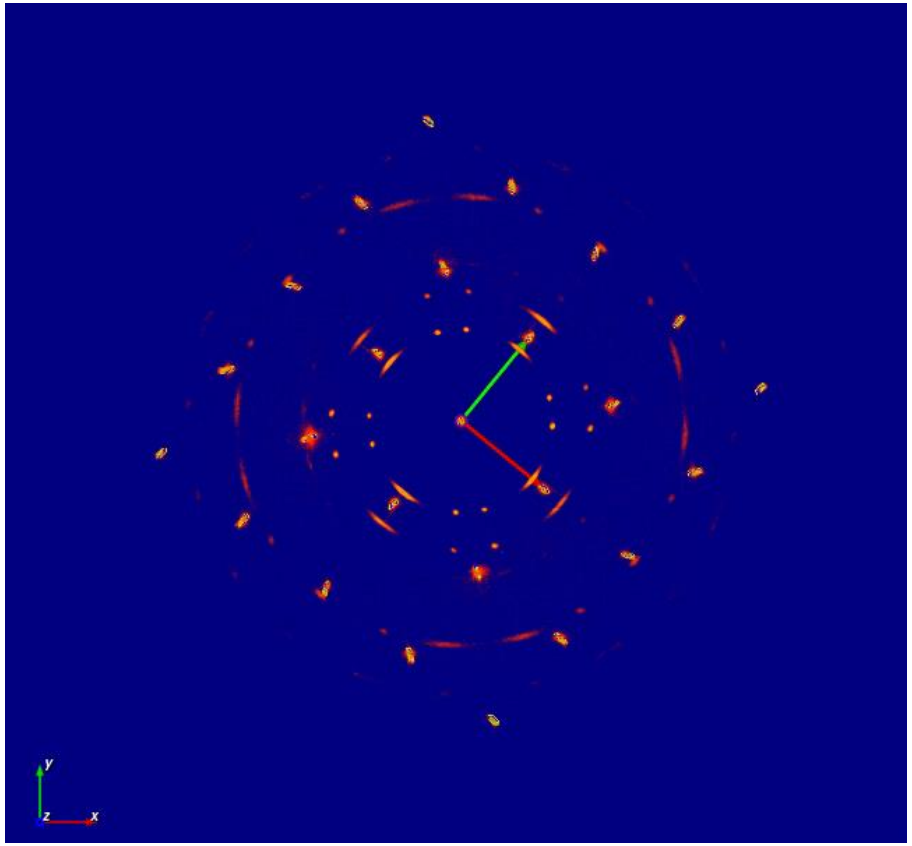
CELLSD 0.0117 0.0000 0.0143
0.0000 0.0000 0.0000 0.270



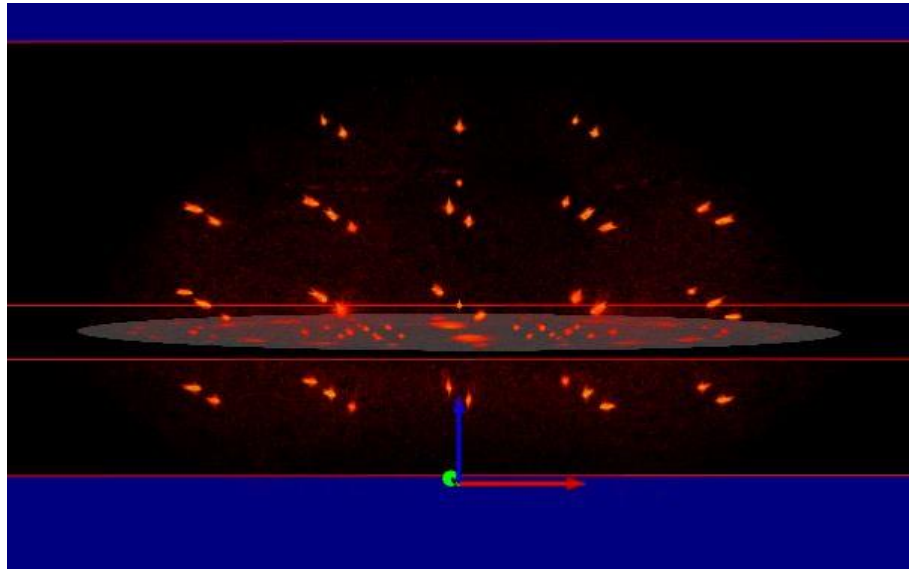
Thin Film Twinning and Rotational Spread

30nm film ($Pn\bar{3}m$) on second substrate

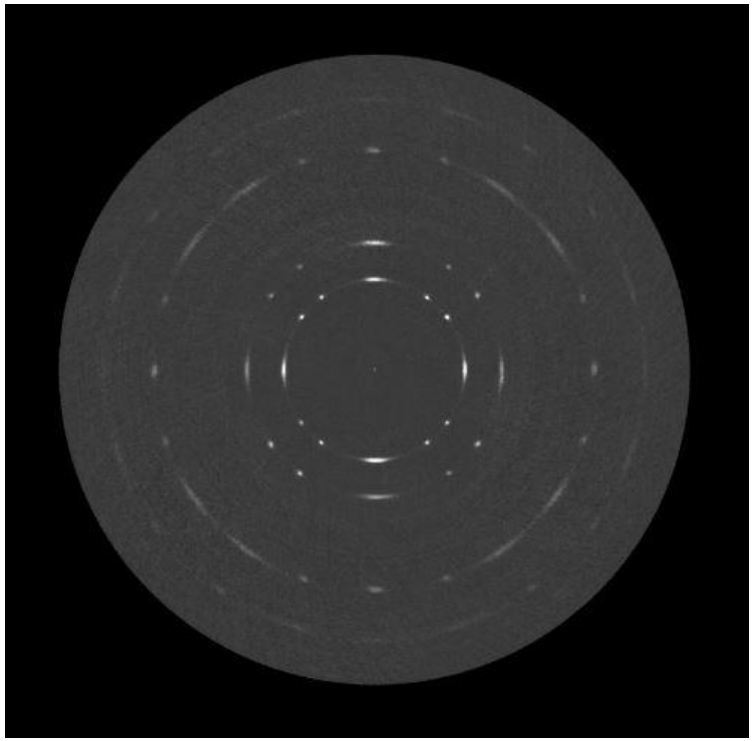
Yimin Wu
Jeffrey Chen
Waterloo
Canada



Bruker Venture D8
Cu IμS source
Photon III detector
65 minute collection

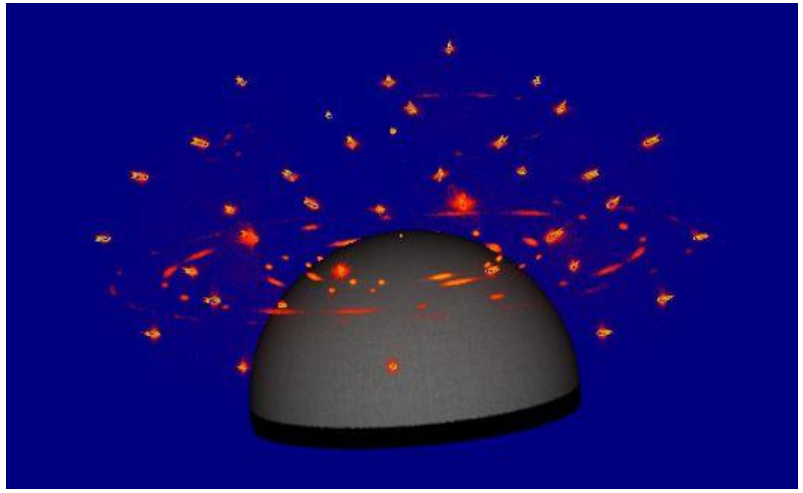


Reciprocal Space Layer showing twinning and orientation spread



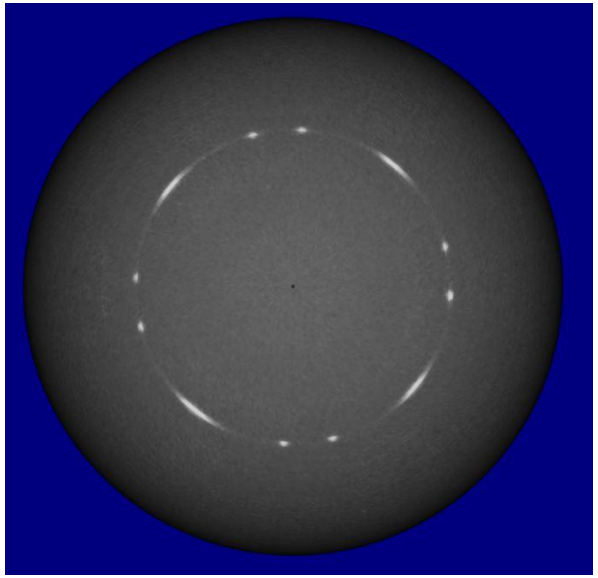
30nm film ($Pn\bar{3}m$) on second substrate

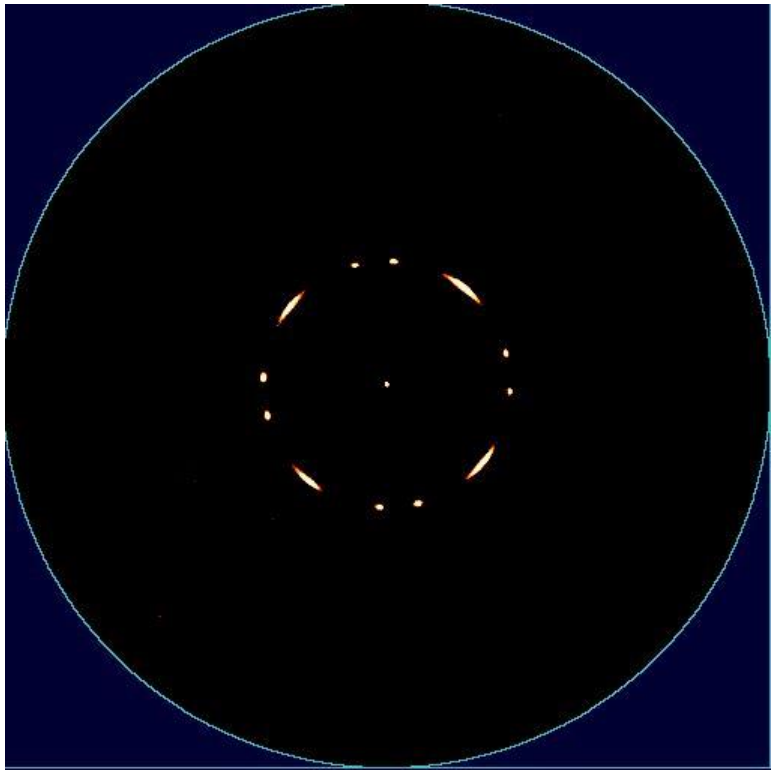
Yimin Wu
Jeffrey Chen
Waterloo
Canada



Reciprocal Space shell and stereographic projection (pole figure) of film 111, [110] normal.

30nm film ($Pn\bar{3}m$) on second substrate





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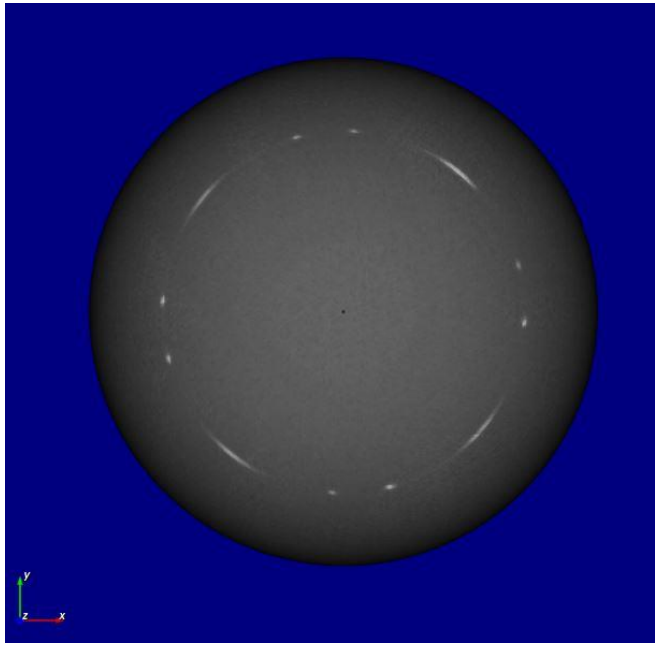
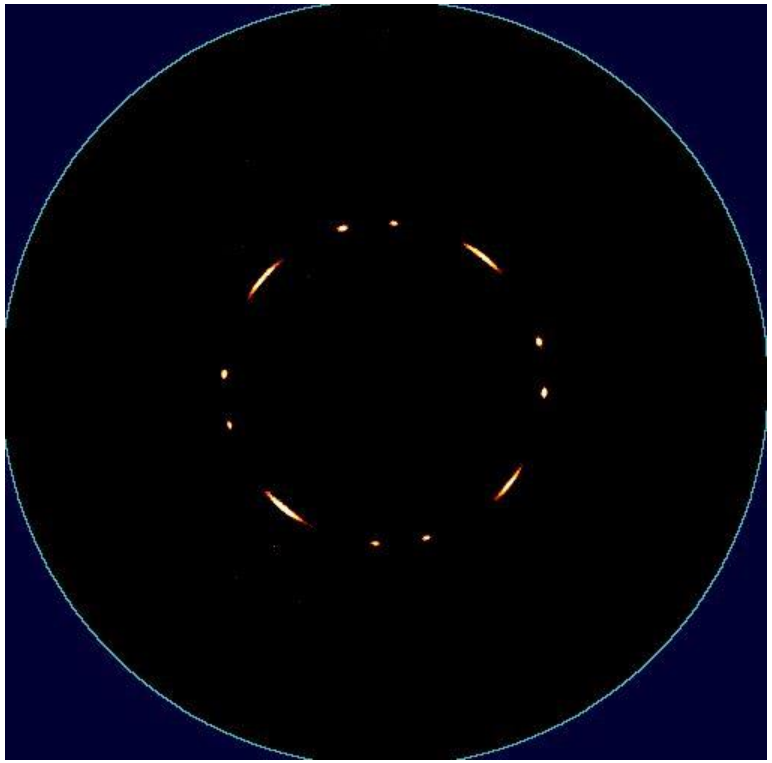
Jeffrey Chen
Waterloo
Canada



Reciprocal Space shell and stereographic projection (pole figure) of film 200



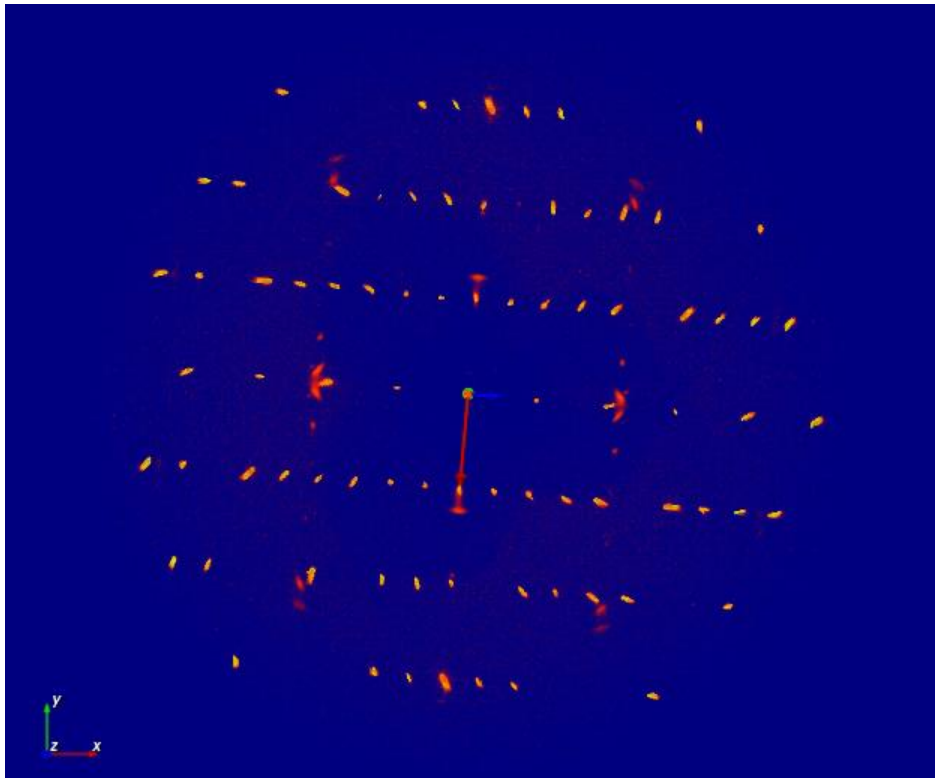
30nm film ($Pn\bar{3}m$) on second substrate

Yimin Wu
Jeffrey Chen
Waterloo
Canada

Thin Film Twinning, Strain and Diffuse Scatter

30nm film
($Pn\bar{3}m$) on
third substrate

Yimin Wu
Jeffrey Chen
Waterloo
Canada



Bruker Venture D8
Cu IμS source
Photon III detector
65 minute
collection

Thin Film Twinning, Strain and Diffuse Scatter

30nm film
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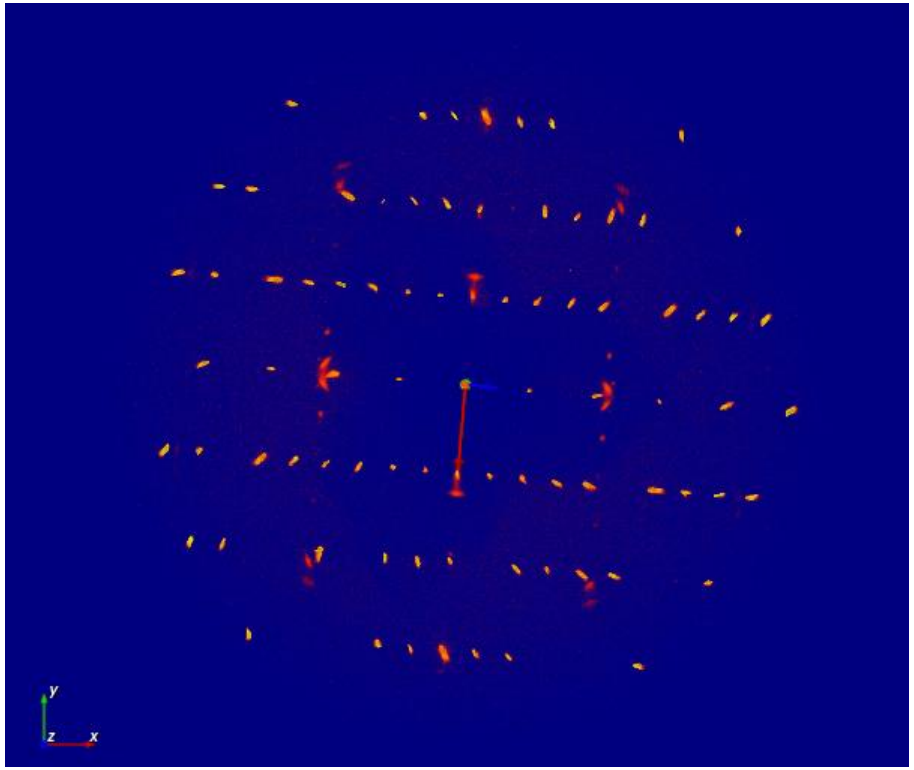
third substrate

Yimin Wu
Jeffrey Chen
Waterloo
Canada

Focus on
(-1 -1 0)



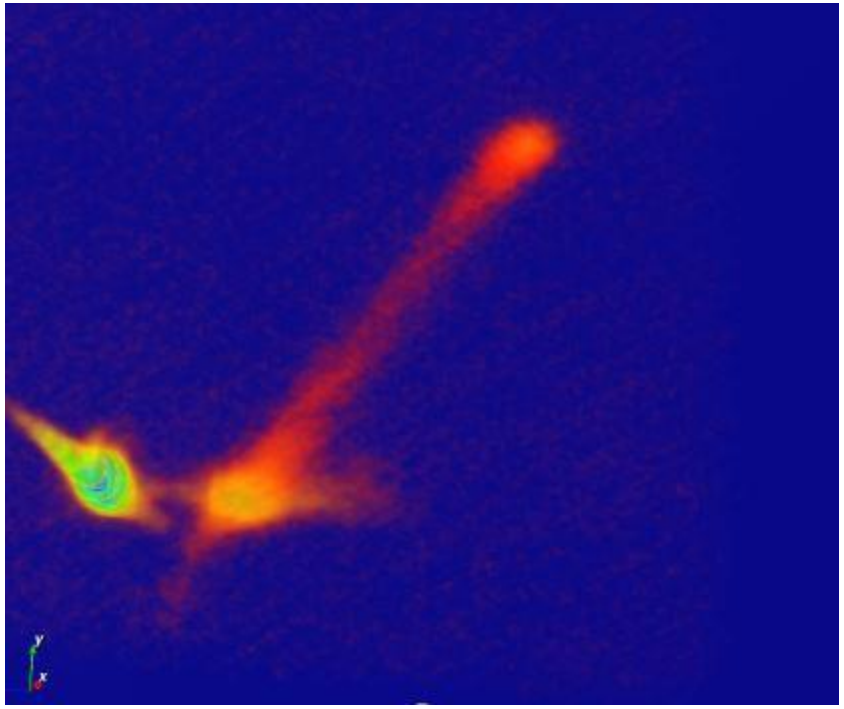
Bruker Venture D8
Cu IμS source
Photon III detector
65 minute
collection

11 minute rescan
of (-1 -1 0)

Thin Film Twinning, Strain and Diffuse Scatter

30nm film
($Pn\bar{3}m$) on
third substrate

Yimin Wu
Jeffrey Chen
Waterloo
Canada



Bruker Venture D8
Cu IμS source
Photon III detector
65 minute
collection

11 minute rescan
of (-1 -1 0)

Focus on
(-1 -1 0)

3D Rietveld refinement including instrument, substrate, film, twinning and diffuse scattering contributions in the model would be really nice at this point.

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Other diffraction patterns worth saving

Everything you have seen here and more . . .

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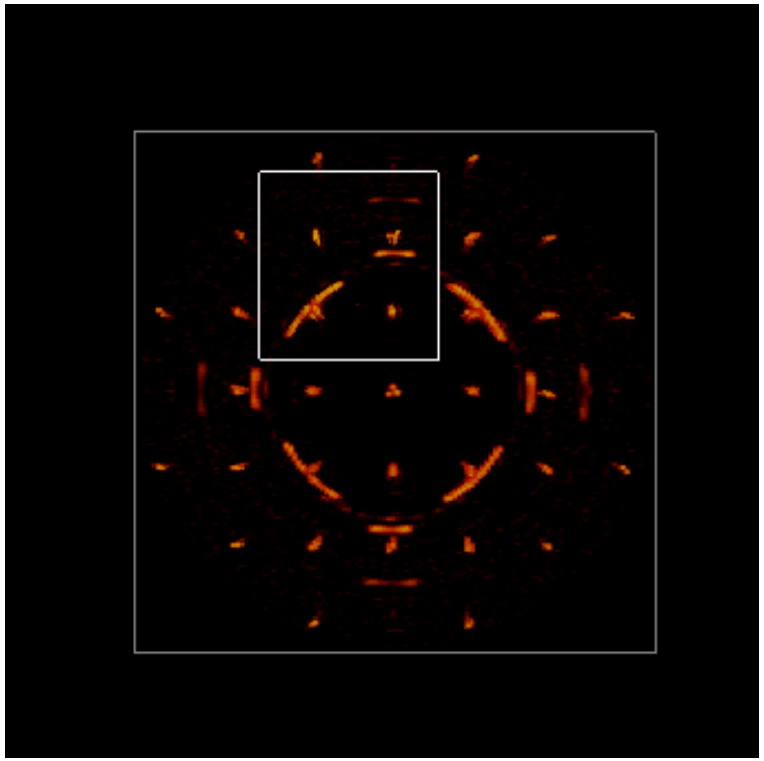
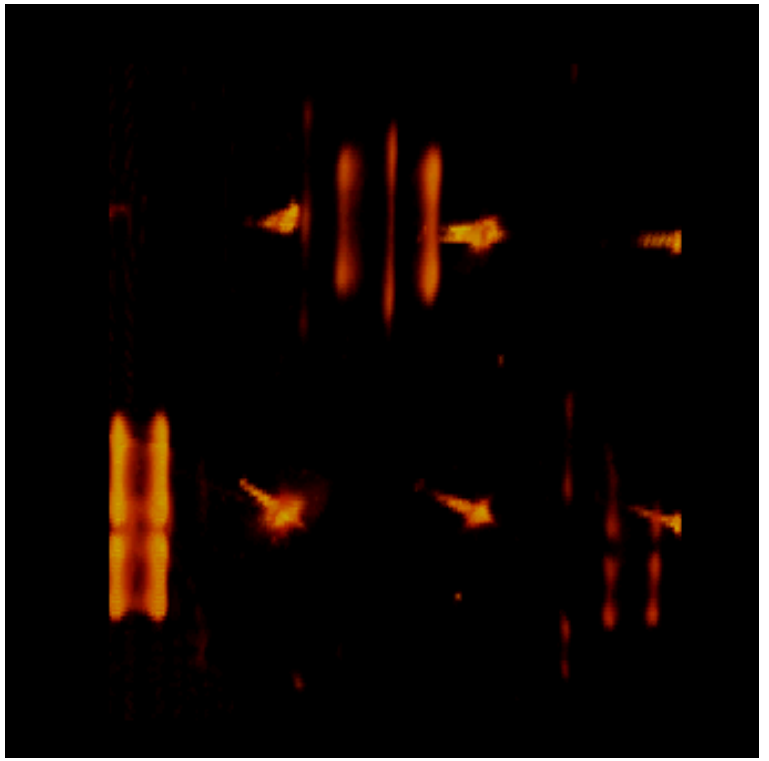
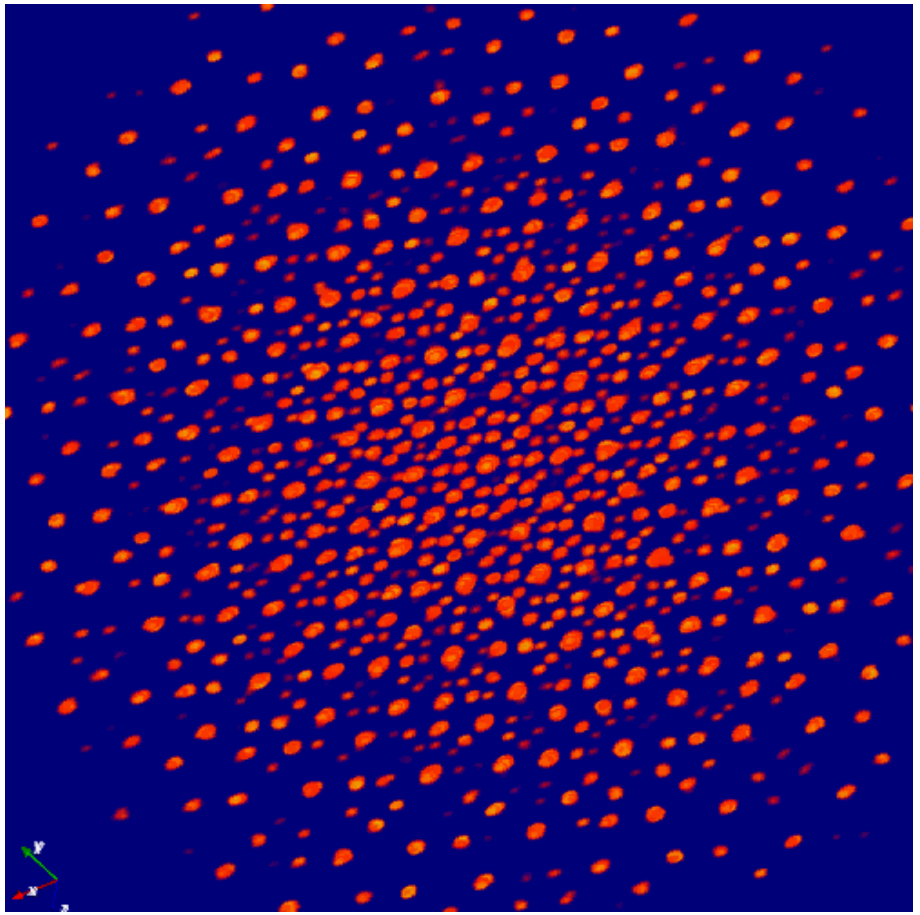
Follow Phase Changes





What do we do with beautiful single crystal data from a quasicrystal?



$Al_{70}Pd_{21}Mn_9$ - Geetha Balakrishnan, University of Warwick
Nathan Armstrong, Tom Timusk, McMaster

Software:

MAX3D : Jim Britten and Weiguang Guan, McMaster University, Canada

Thank you for your attention.