Visualization of 3D Diffraction: MAX3D

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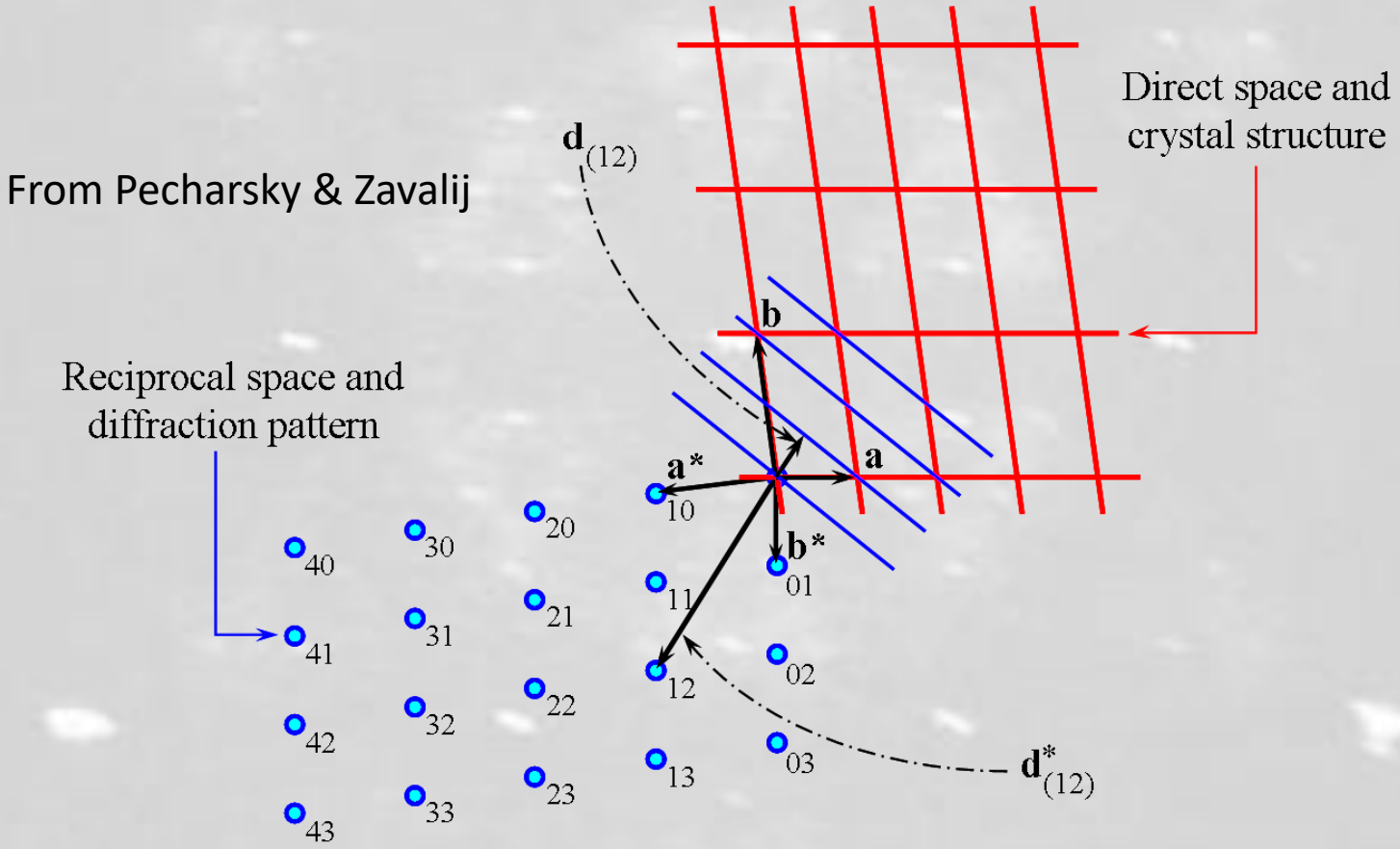
Abstract

MAX3D is a program for the **visualization of 3D diffraction data**. This includes everything seen by the area detector – not just harvested Bragg spots. The input may be transmission data from a single crystal, reflection data from a textured solid, or diffraction from a thin nanoparticle film on a substrate. **Tools** have been developed **for probing, imaging and exporting selected areas of reciprocal space** in terms of 2Theta or HKL. It helps you understand your sample, recognize diffuse scattering and troubleshoot difficult problems. It is a powerful tool for generating **teaching materials** from real samples. MAX3D is available at no charge for Academic researchers.

Quick X-ray Review

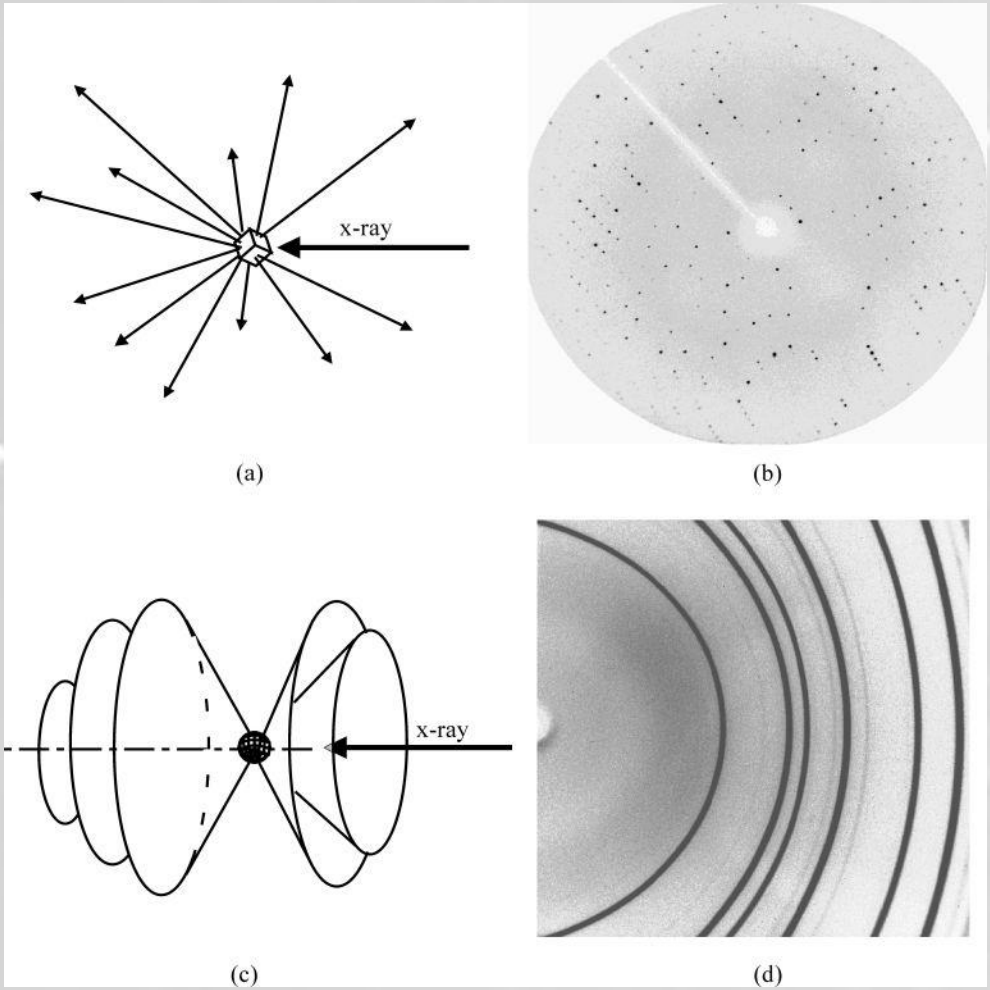
Reciprocal Space

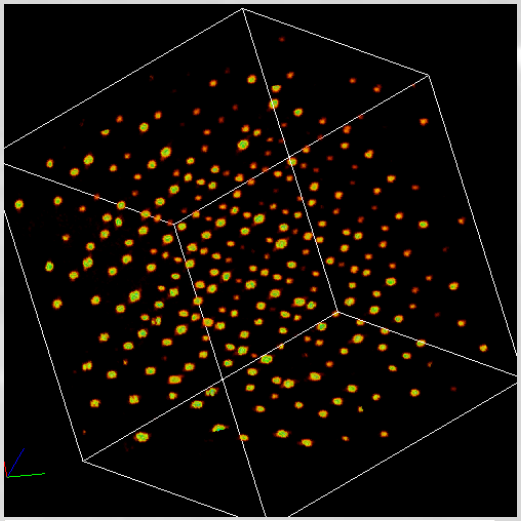
From Pecharsky & Zavalij



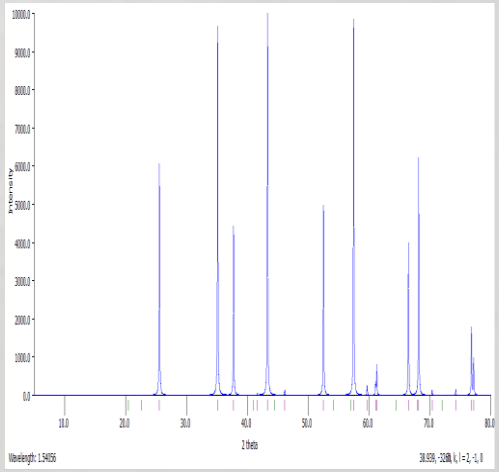
SCD - 2D image + scan –> 3D Int vs 2θ

XRD² - 2D image –> 1D Int vs 2θ





Single Crystal

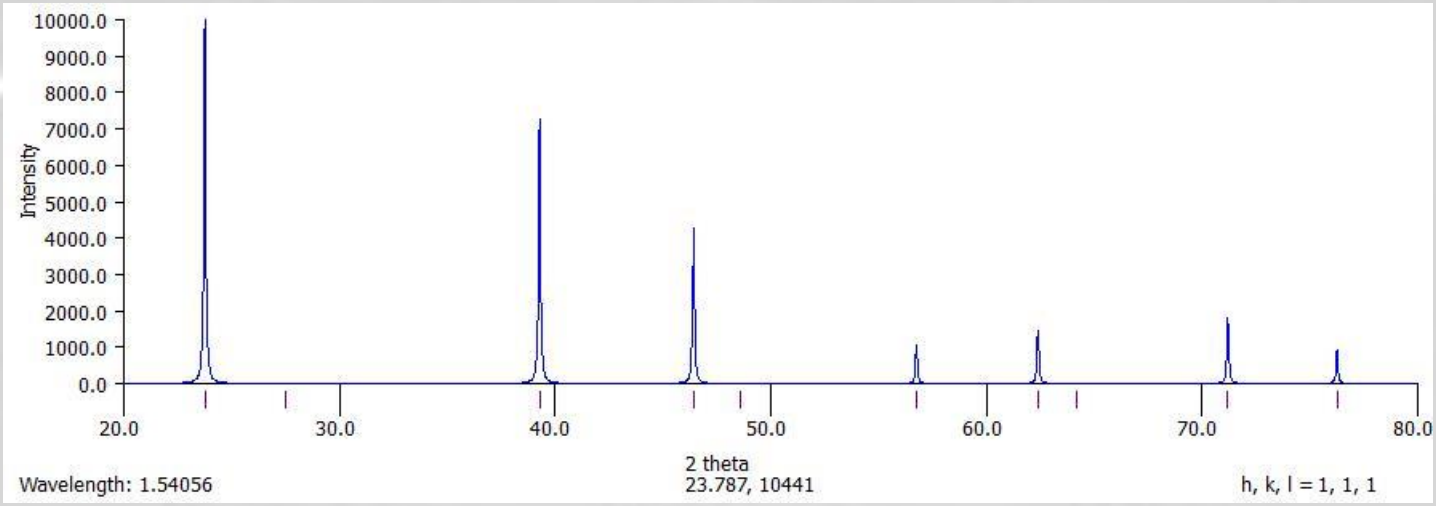


Powder or polycrystalline solid

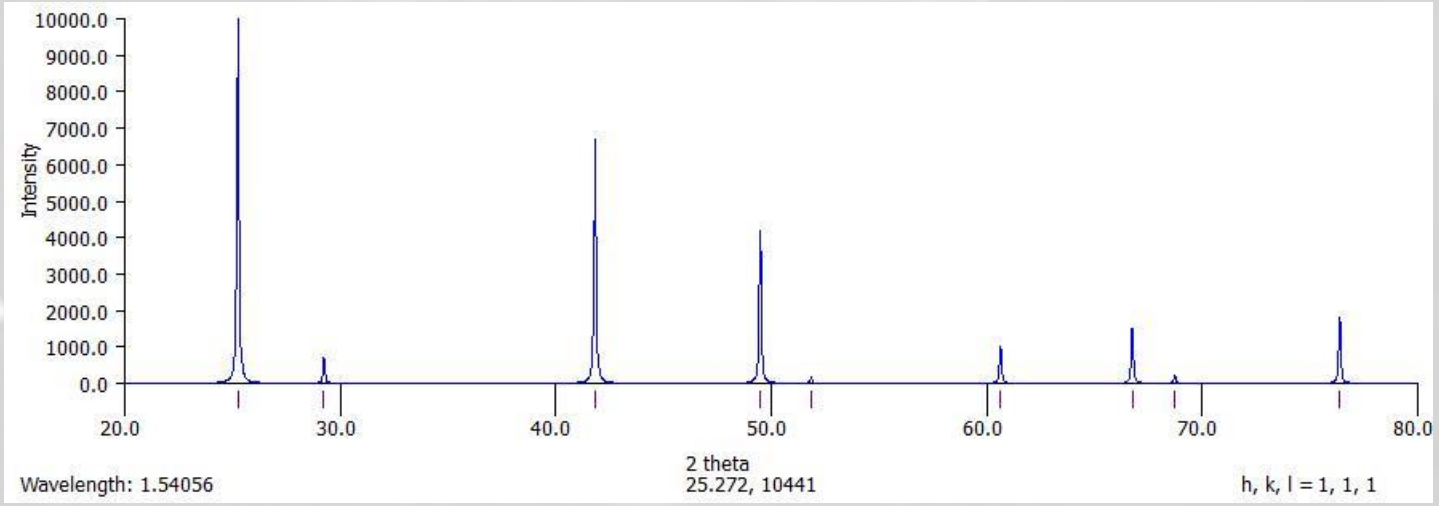
From Bob He's book: Two-Dimensional X-Ray Diffraction

Visualisation of 1D Reciprocal Space

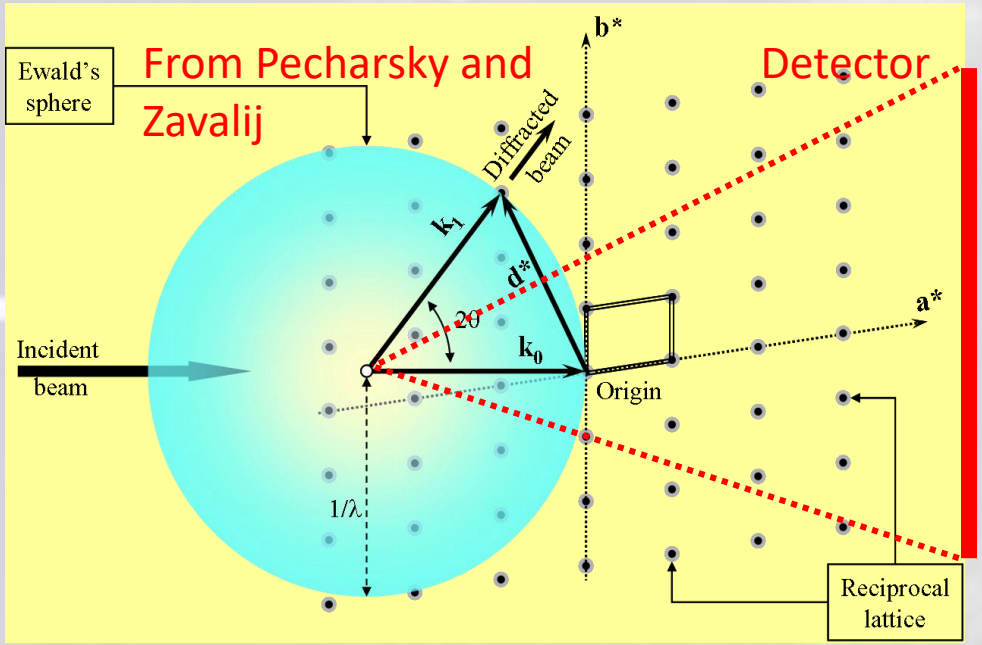
CdTe
a=6.4827
F-43m

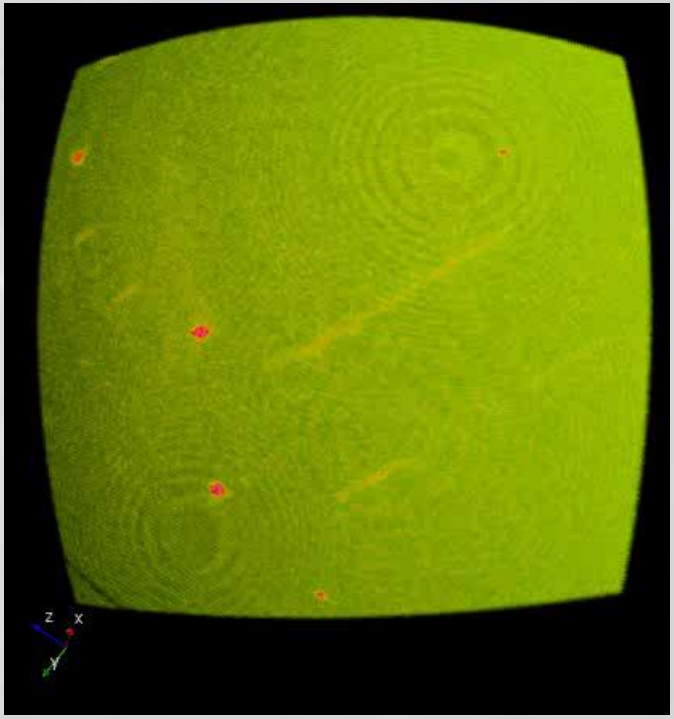


ZnTe
a=6.1034
F-43m



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

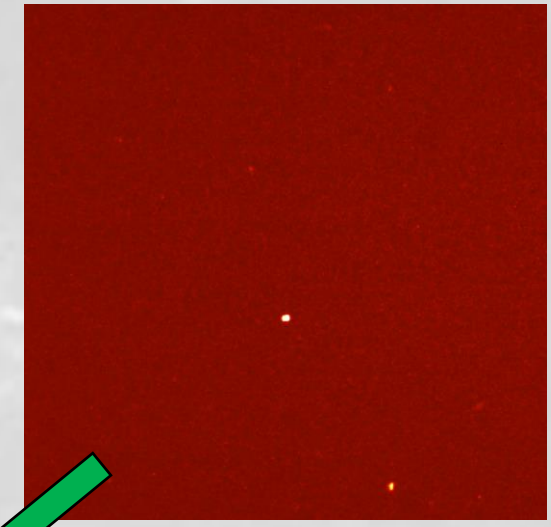




The 2D images can be mapped into reciprocal space – onto the surface of Ewald's Sphere

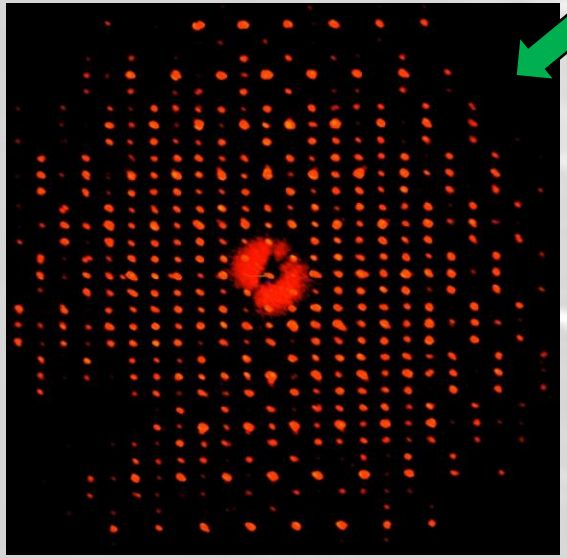
Single Crystal Structure Determination

Rotate 200µm crystal in X-ray beam



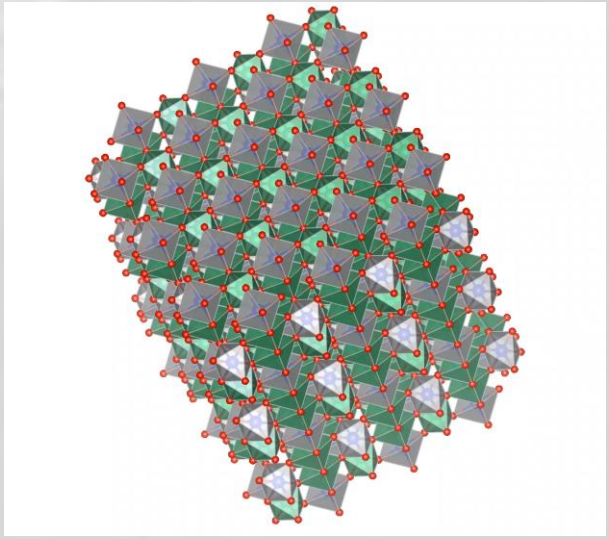
Every 0.5° store CCD image of diffraction.

Identify unit cell and Space Group

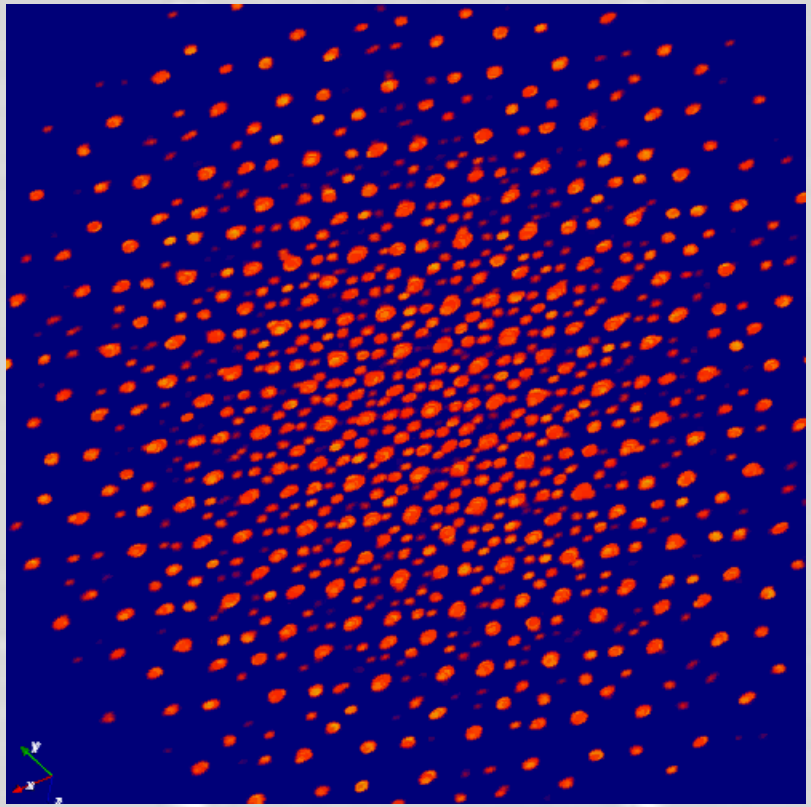


Collect full diffraction pattern

Phase and Fourier Transform to see structure

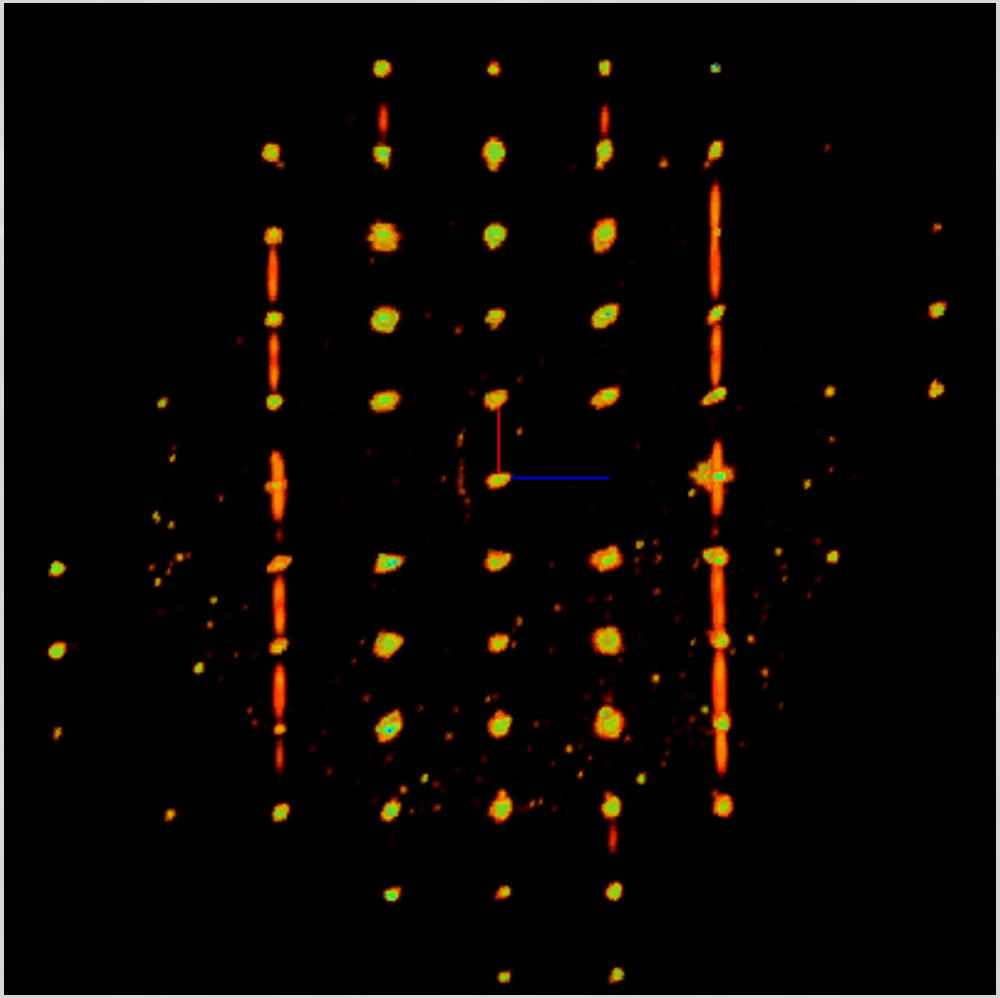


Visualisation of 3D Reciprocal Space
Quasicrystal



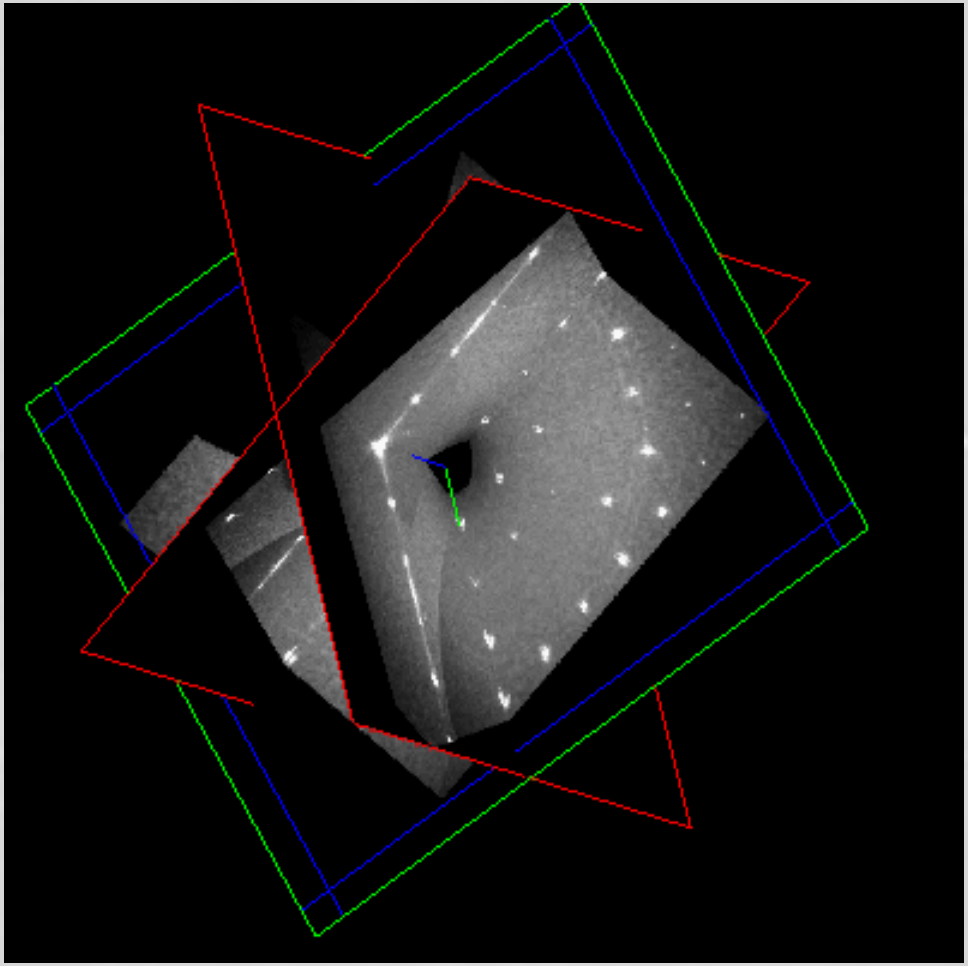
$Al_{70}Pd_{21}Mn_9$ - Geetha Balakrishnan, University of Warwick
Nathan Armstrong, Tom Timusk, McMaster

Diffuse Scattering



Hexanapthylbenzene. Laura Harrington, Mike McGlinchey

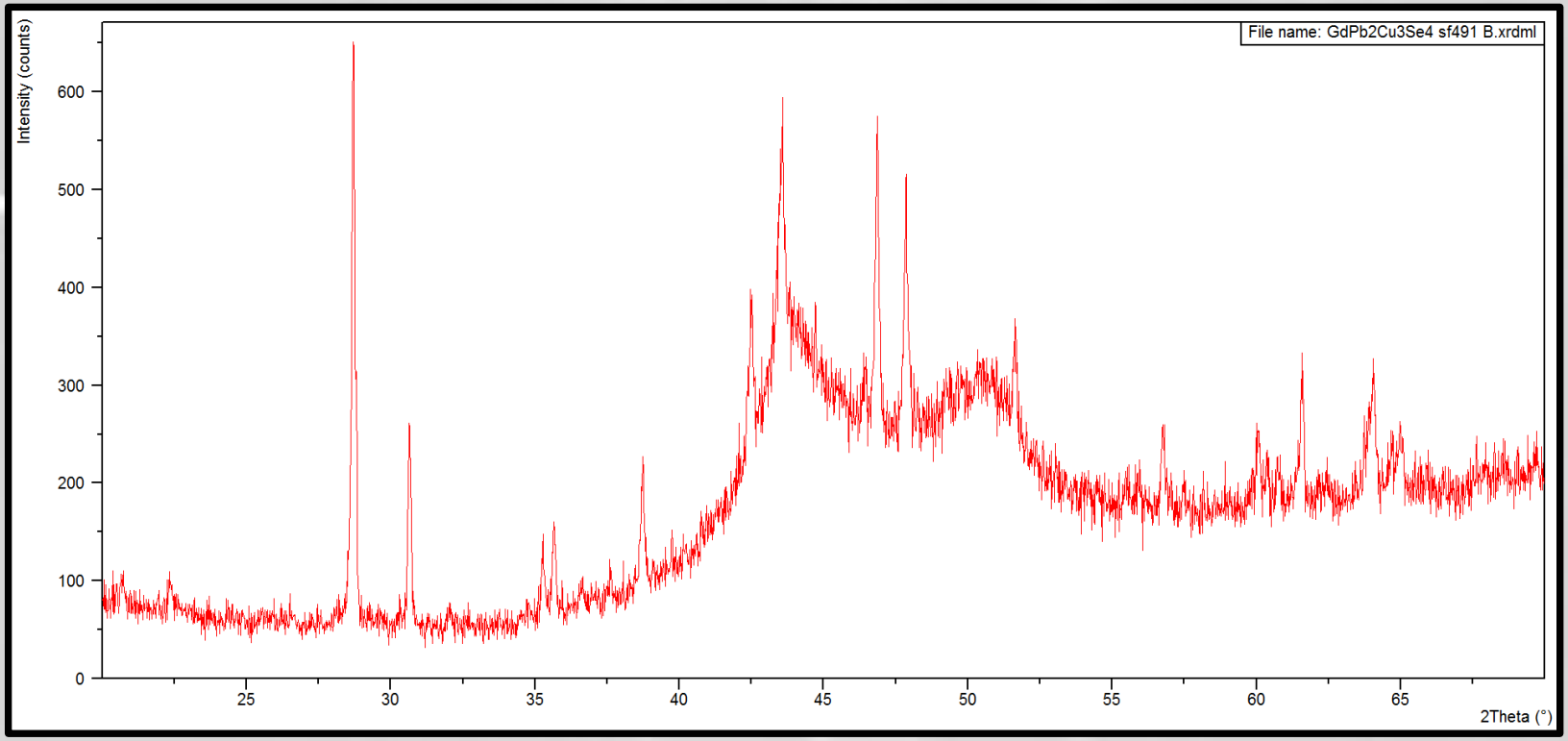
Diffuse Scattering



Hexanapthylbenzene. Laura Harrington, Mike McGlinchey

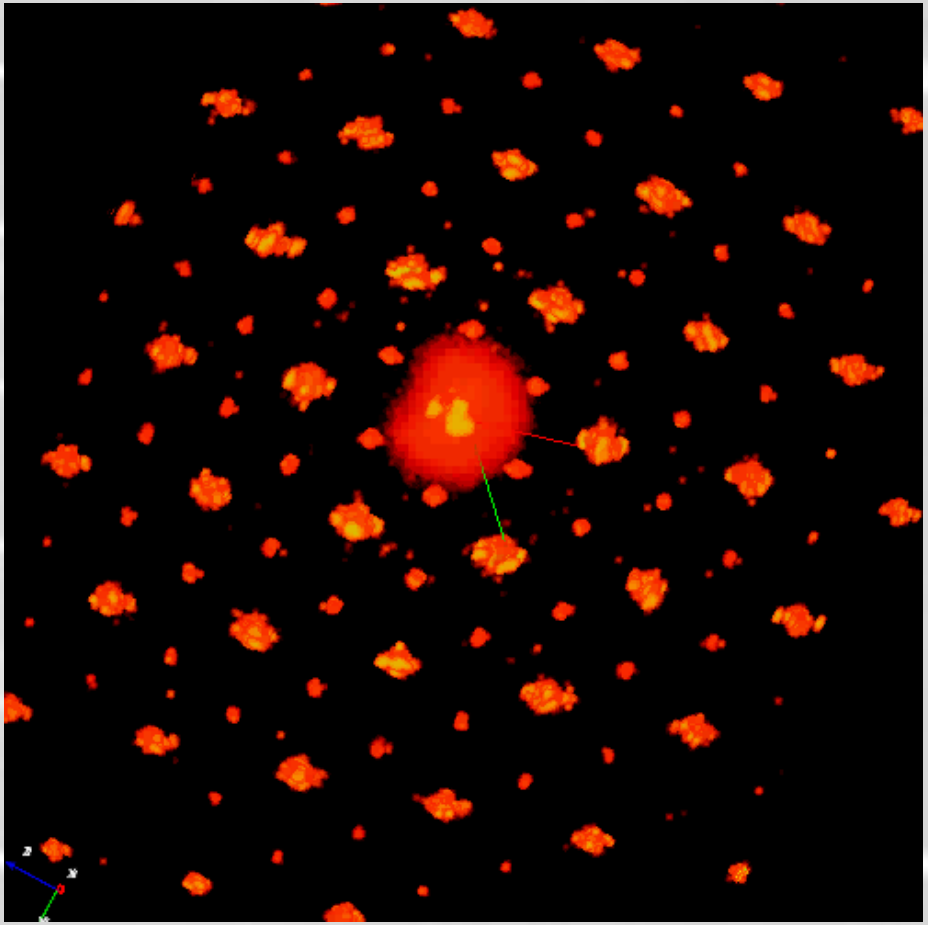
$GdPb_2Cu_3Se_4$ 1200°C for 4 hrs (Plates)

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



$GdPb_2Cu_3Se_4$

Phone a friend – Pawel Grochulski. Look at a single grain of the powder on a protein beamline.

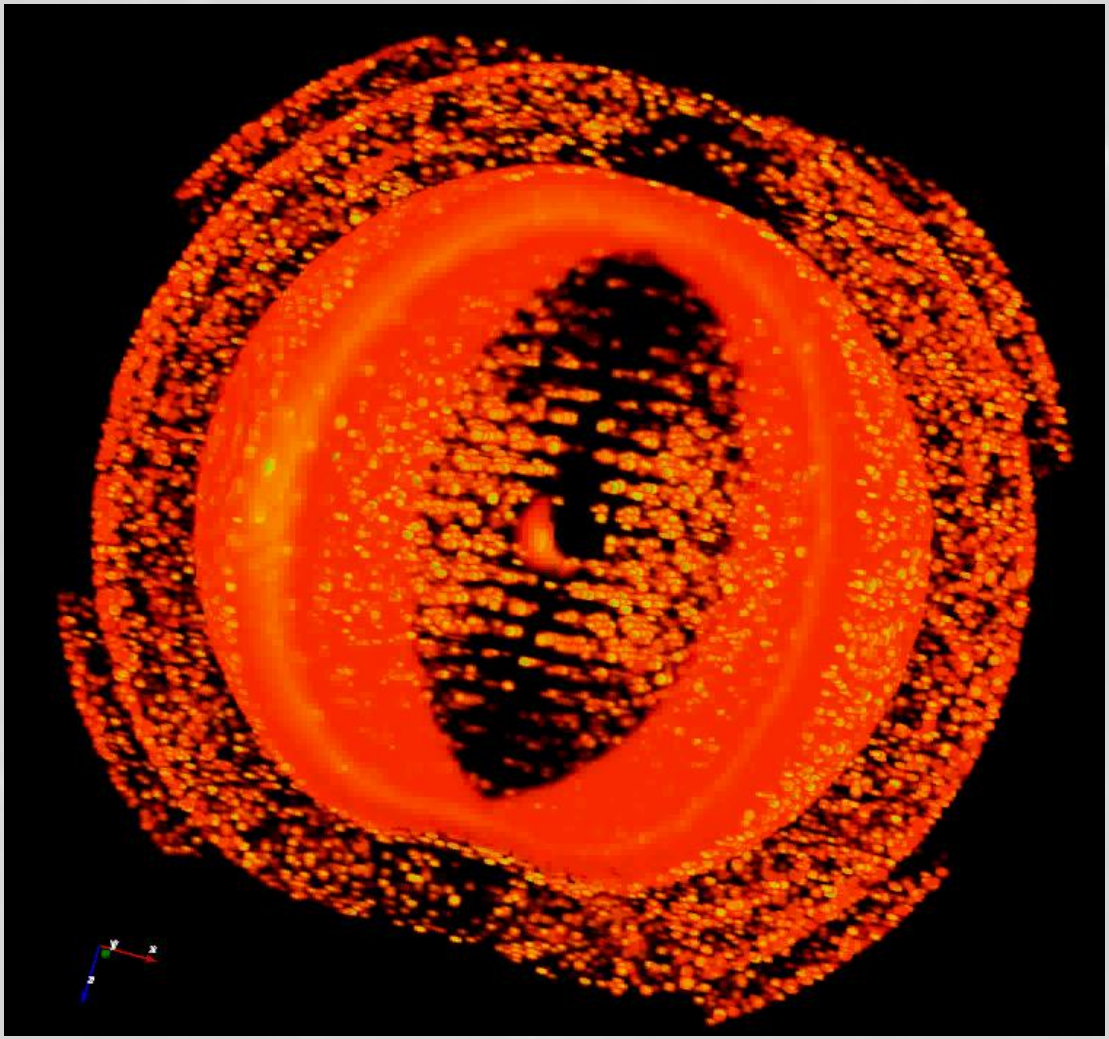


Canadian Macromolecular Crystallography Facility, 08B1-1 (CMCF-BM) Beamline

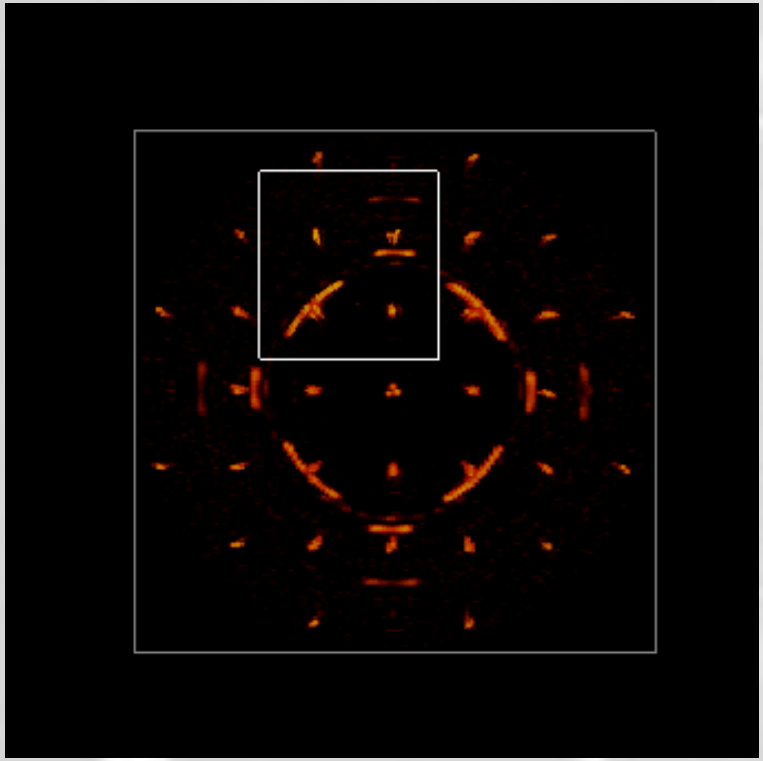
Protein SC Diffraction Pattern

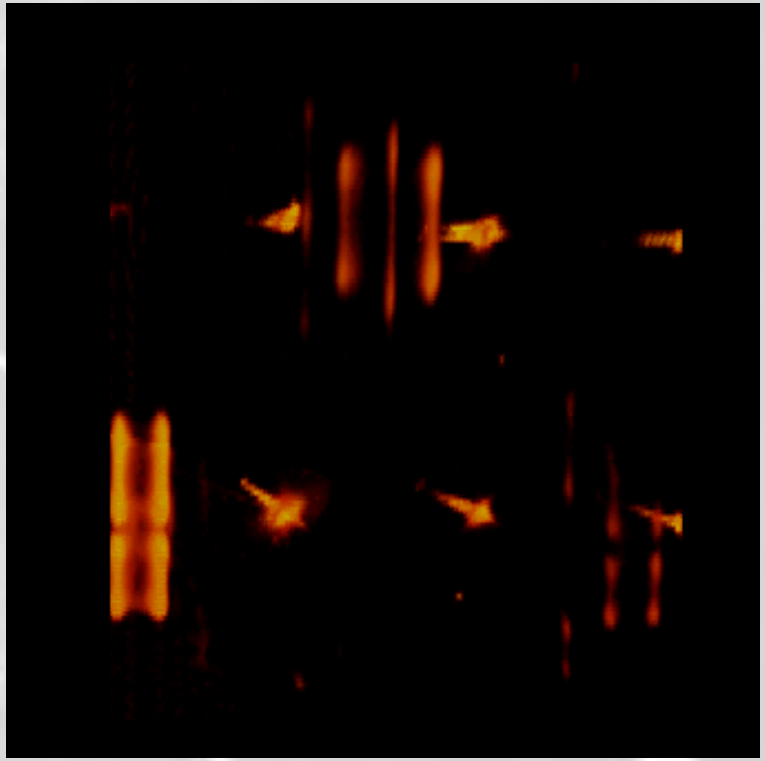
Alba Guarne
Tamiza Nanji

Rigaku
R-Axis4++
Image Plate



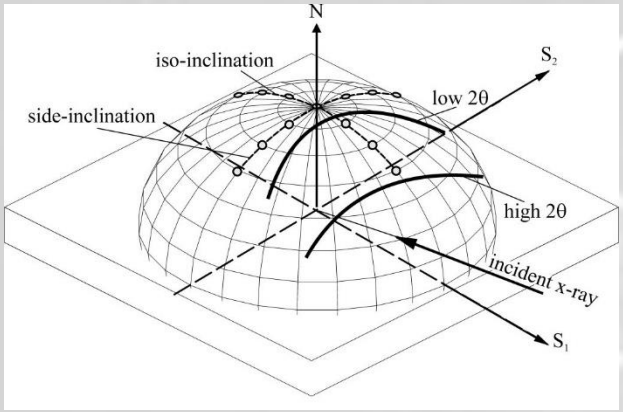
Follow Phase Changes



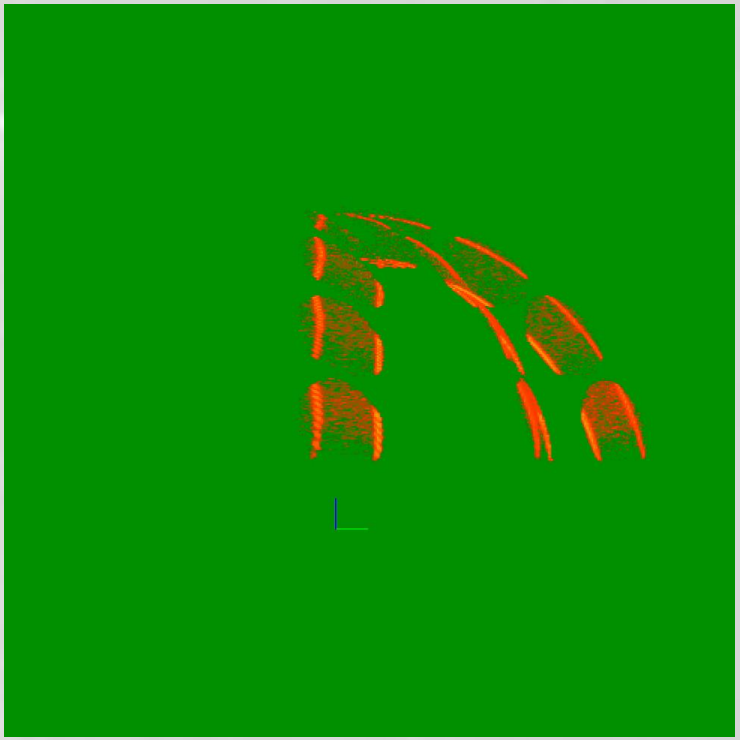


Zheng, Preston, McMaster U

Residual Stress – Sampling 3D RS

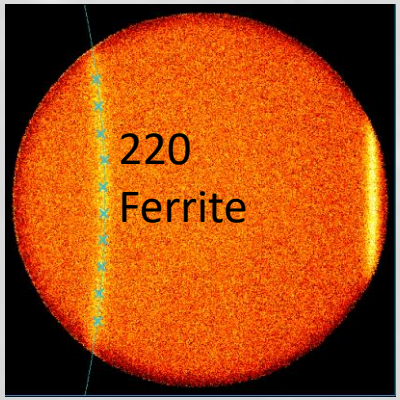


(Image from Bob He's book)



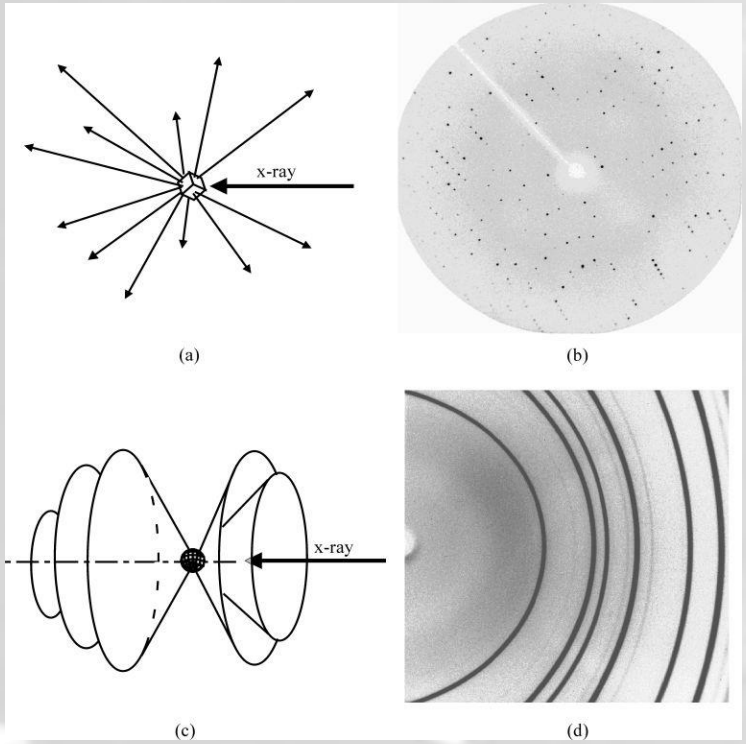
Looking for subtle changes in 2θ position of line/arc/shell to indicate orientation dependent residual stresses. Hard to see visually – need mathematical analysis.

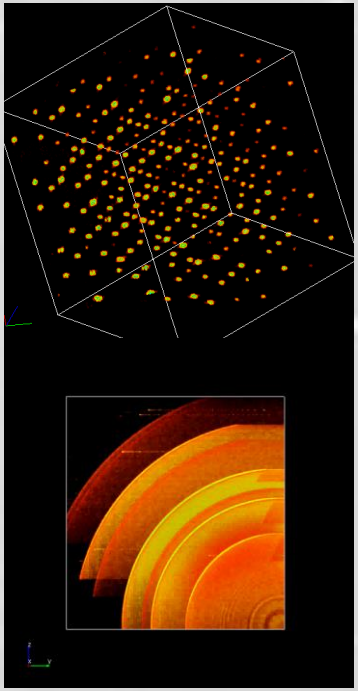
High angle snapshots of diffraction shell segments in four series of ϕ steps at different ψ tilt angles. Looking for elliptical deviation from spheres where r = $1/d_0$



SCD - 2D image + scan –> 3D Int vs 2θ

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



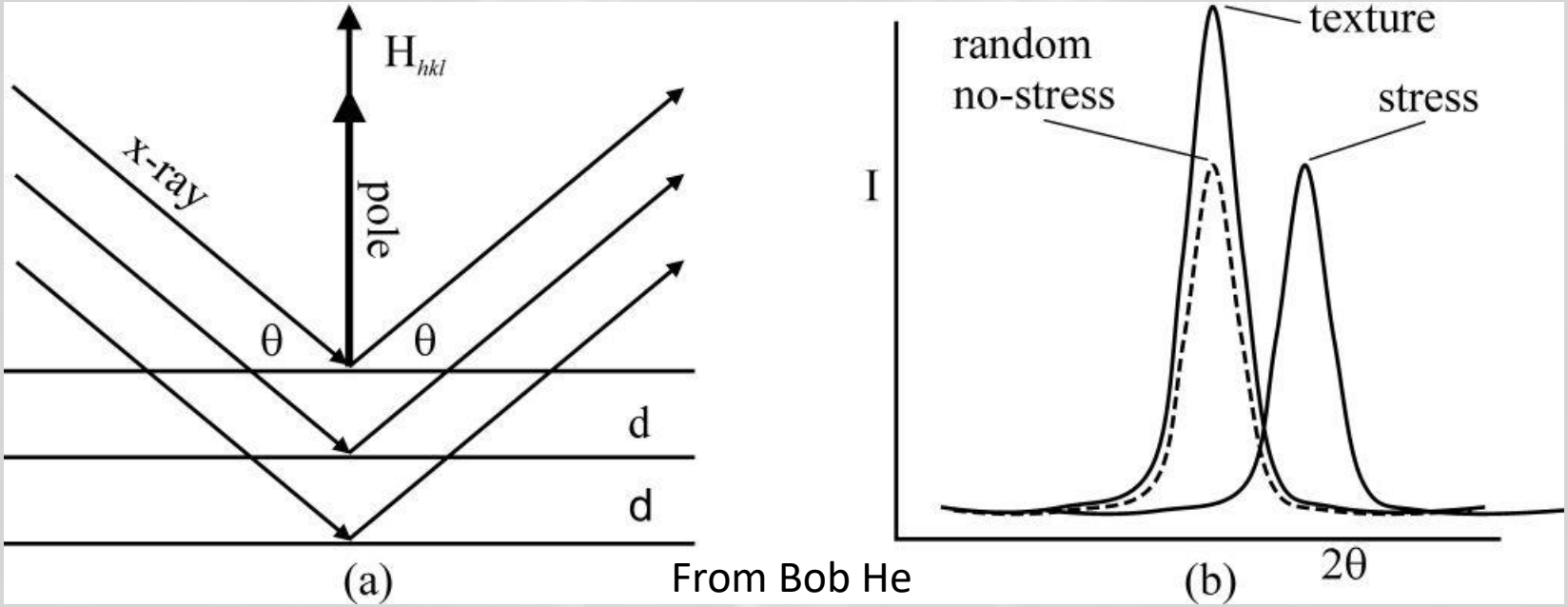


Single Crystal

Powder or polycrystalline solid

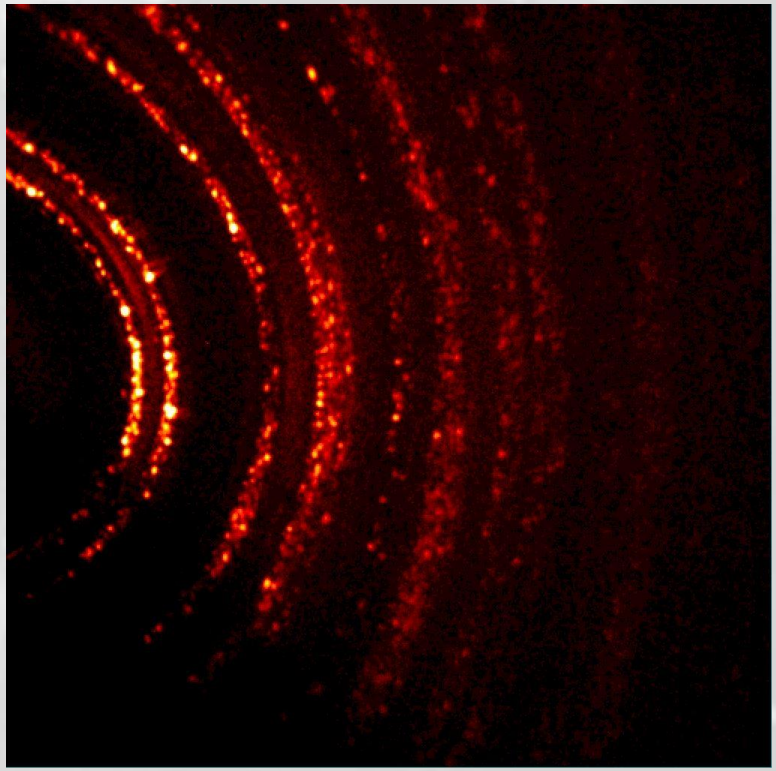
From Bob He's book: Two-Dimensional X-Ray Diffraction

A ‘**pole**’ is a unit vector along a diffraction vector representing one grain. For a random powder the number of poles is normalized to 1. Textured samples show a variation in normalized pole density with orientation.

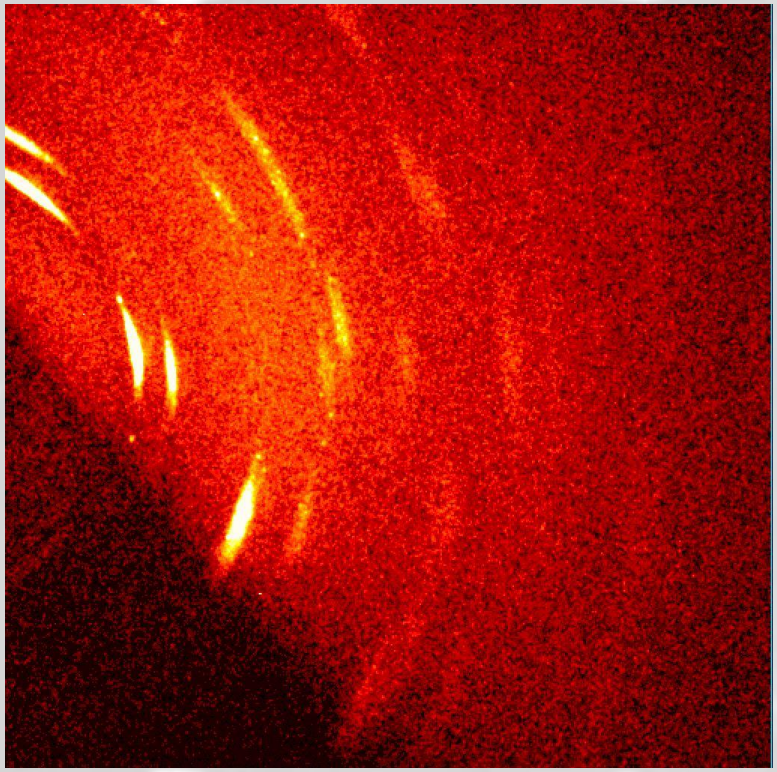


From Bob He

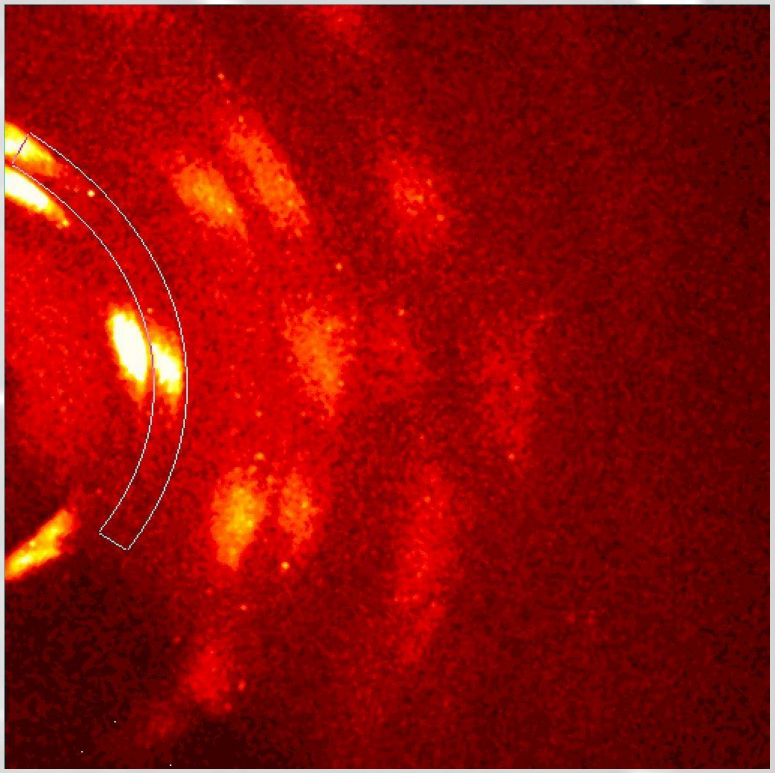
Mo diffraction from surface of tab of an Aluminum weighing dish



Mo diffraction from Al foil (ALCAN)

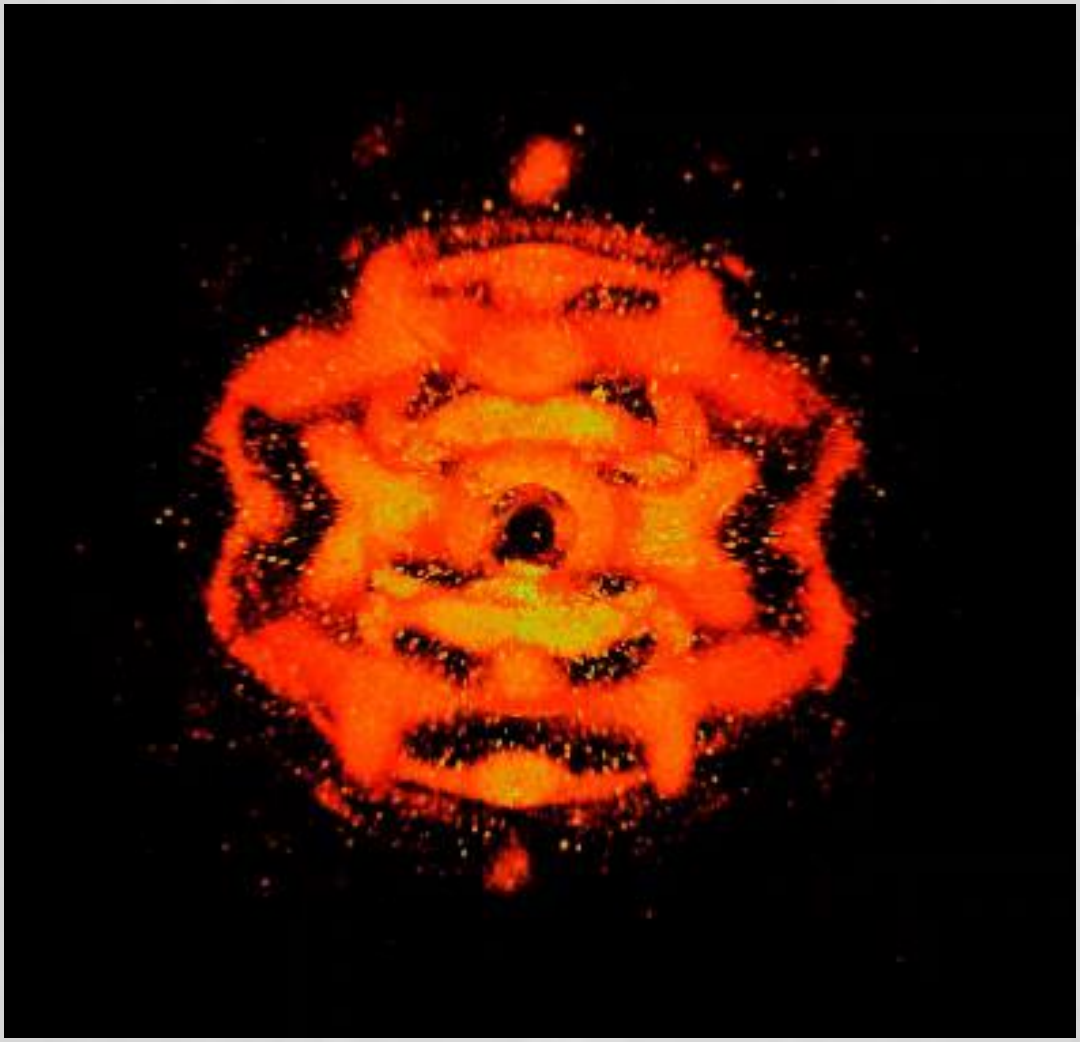


ϕ-scan, 2θ=-40, ω=168, χ=54.74, 2s

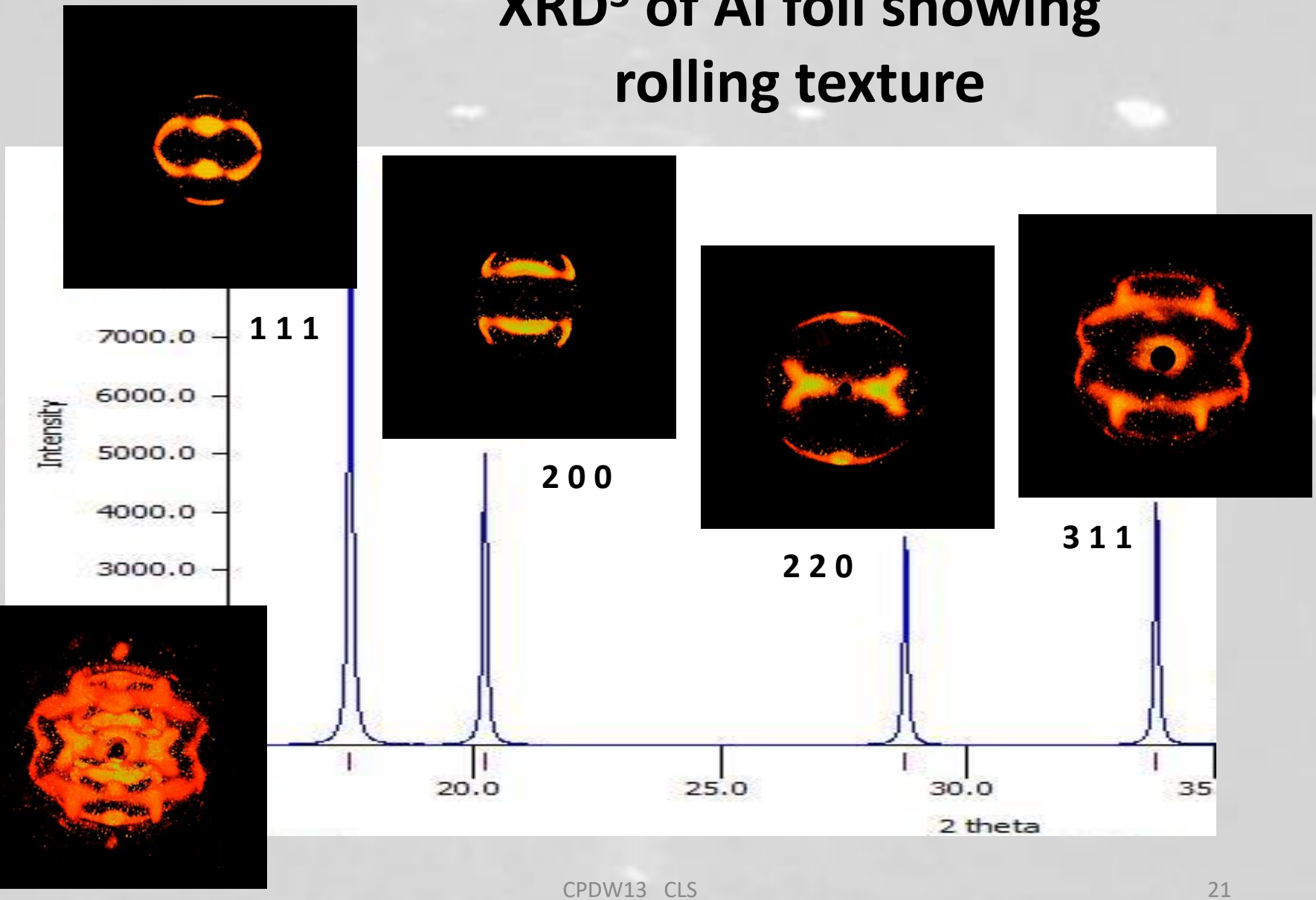


ϕ-scan, 2θ=-40, ω=175, χ=54.74, 2s

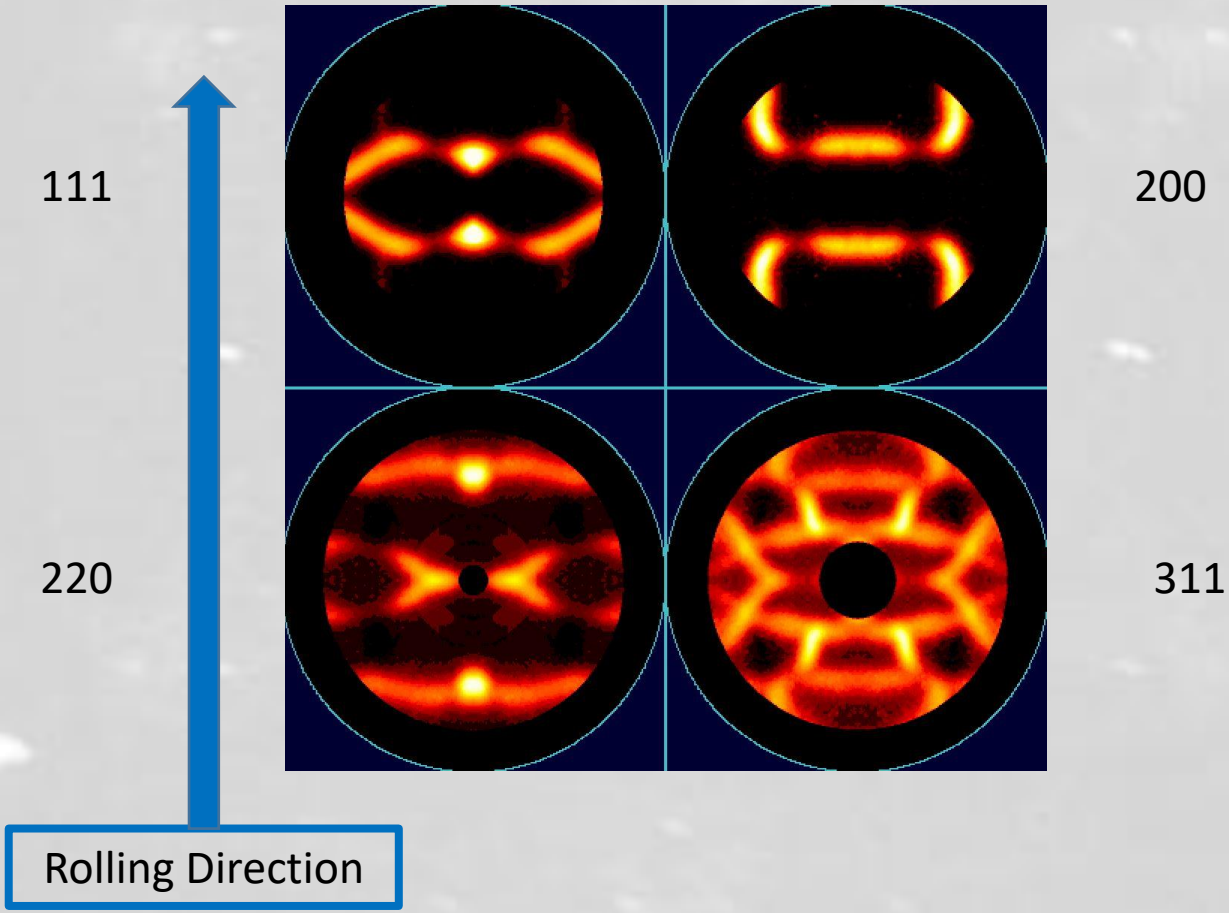
Mo diffraction from Al foil (ALCAN)



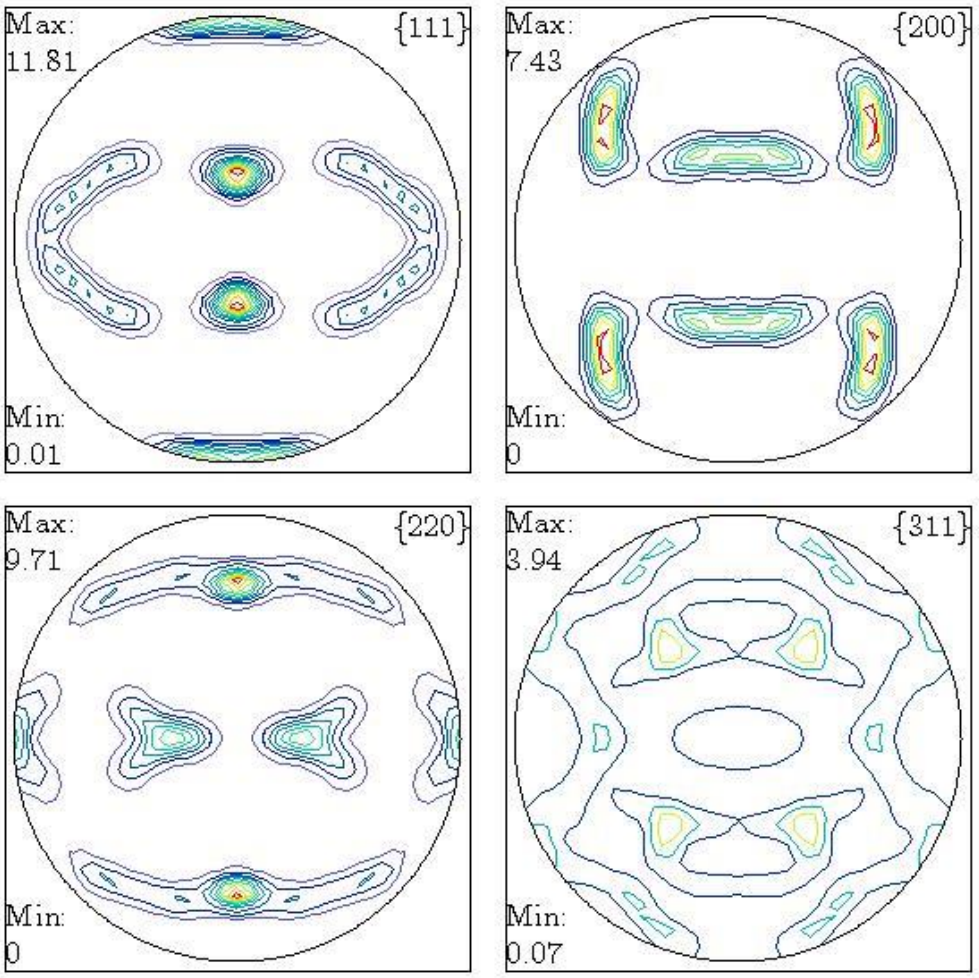
XRD[3] of Al foil showing rolling texture

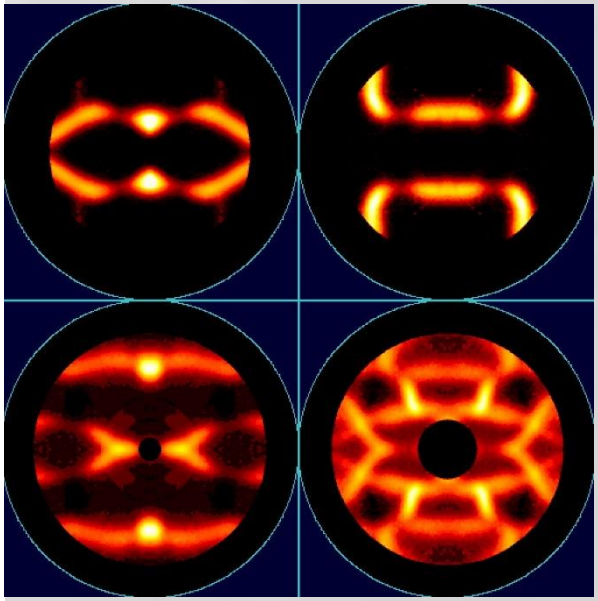


Pole figures from Al foil on Mo CCD



Al Foil Pole Figures calculated from ODF (MTEX in Matlab)





. . . from GADDS User Guide

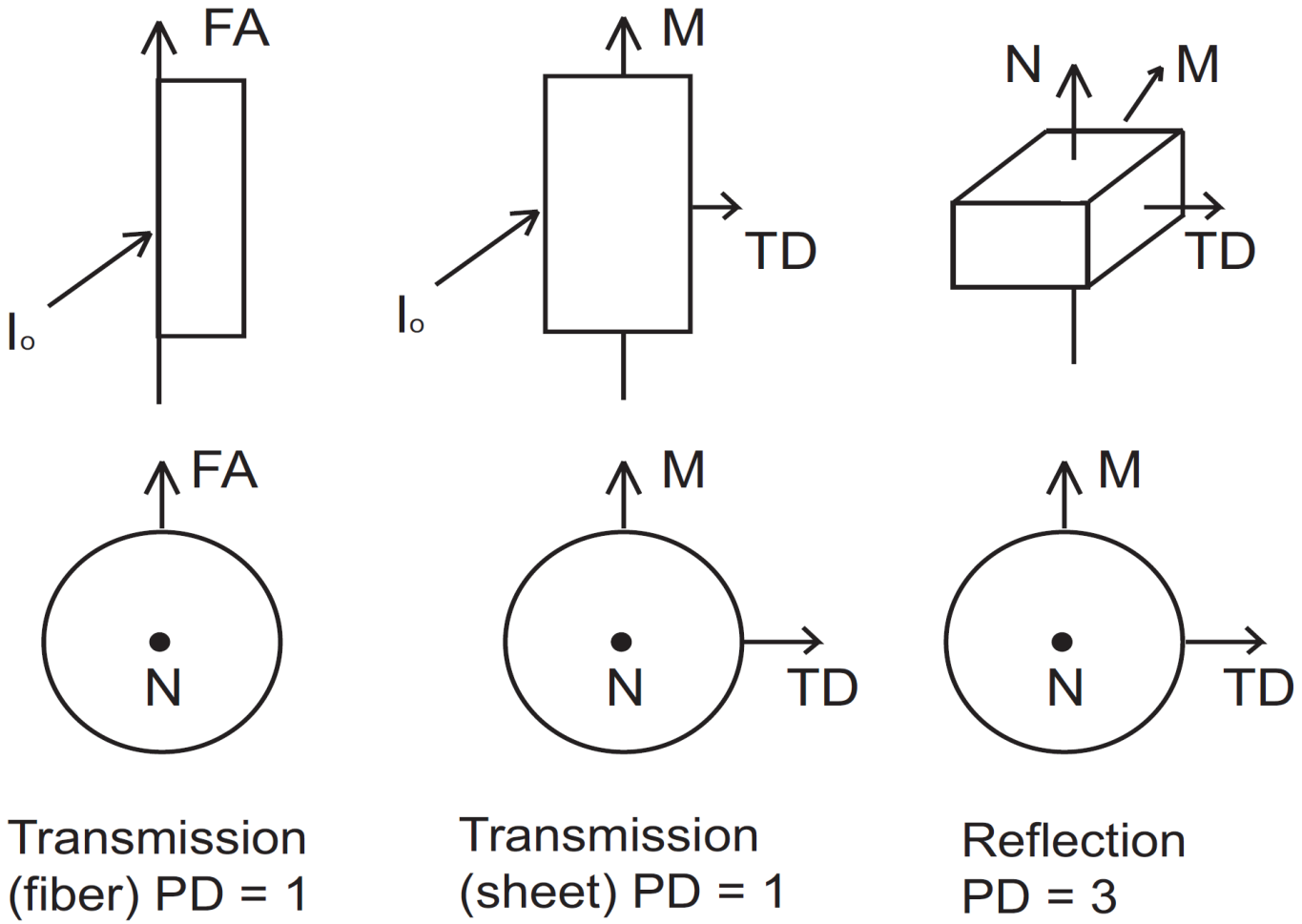


Figure 5.6 - Relationship between the significant directions in texture specimens and their associated pole figure

Texture Example: Au nanoparticle film grown on $MgAl_2O_4$ substrate

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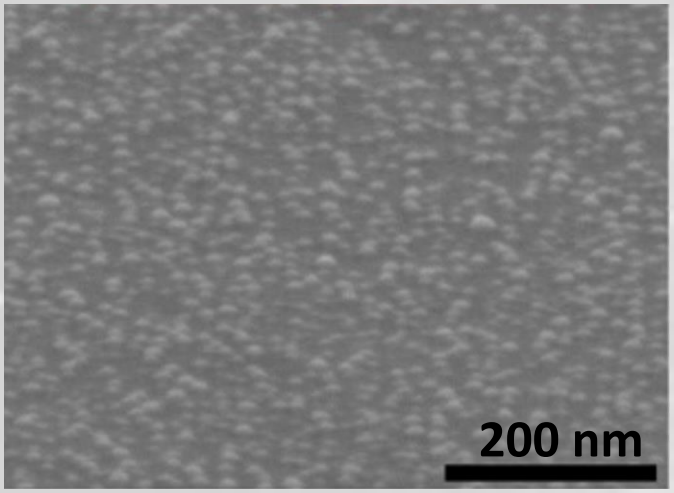
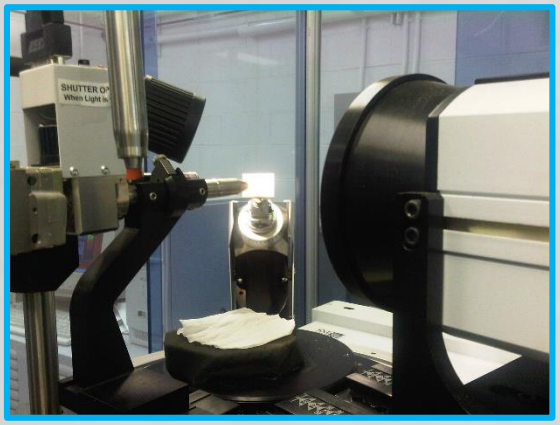
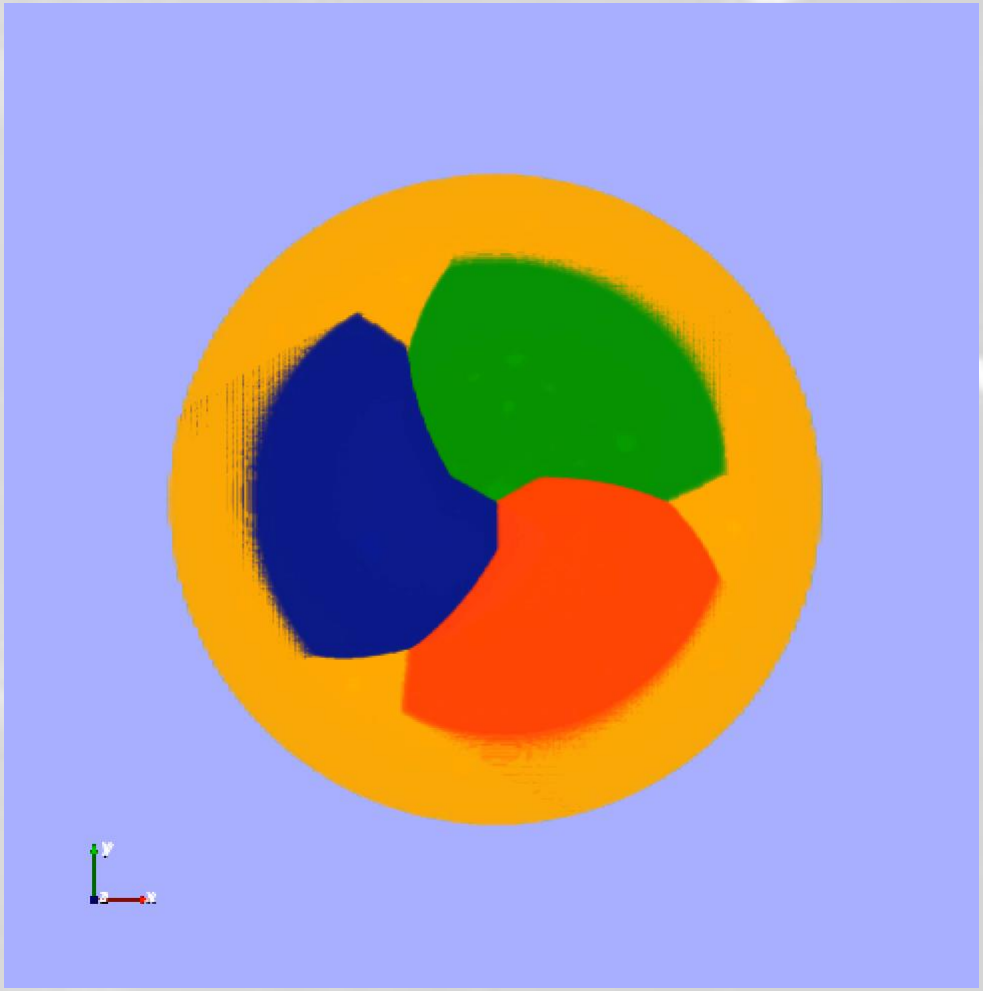
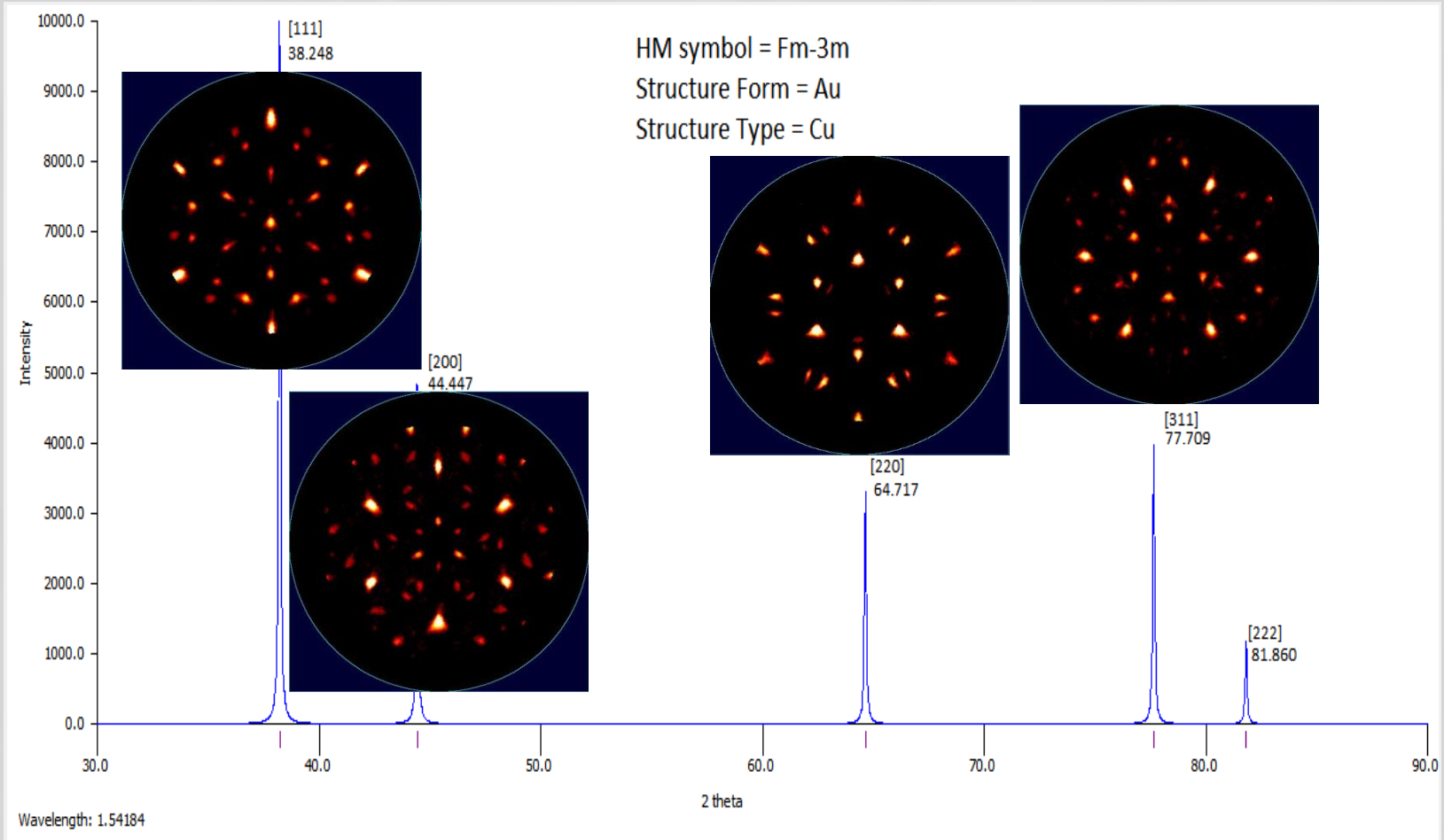






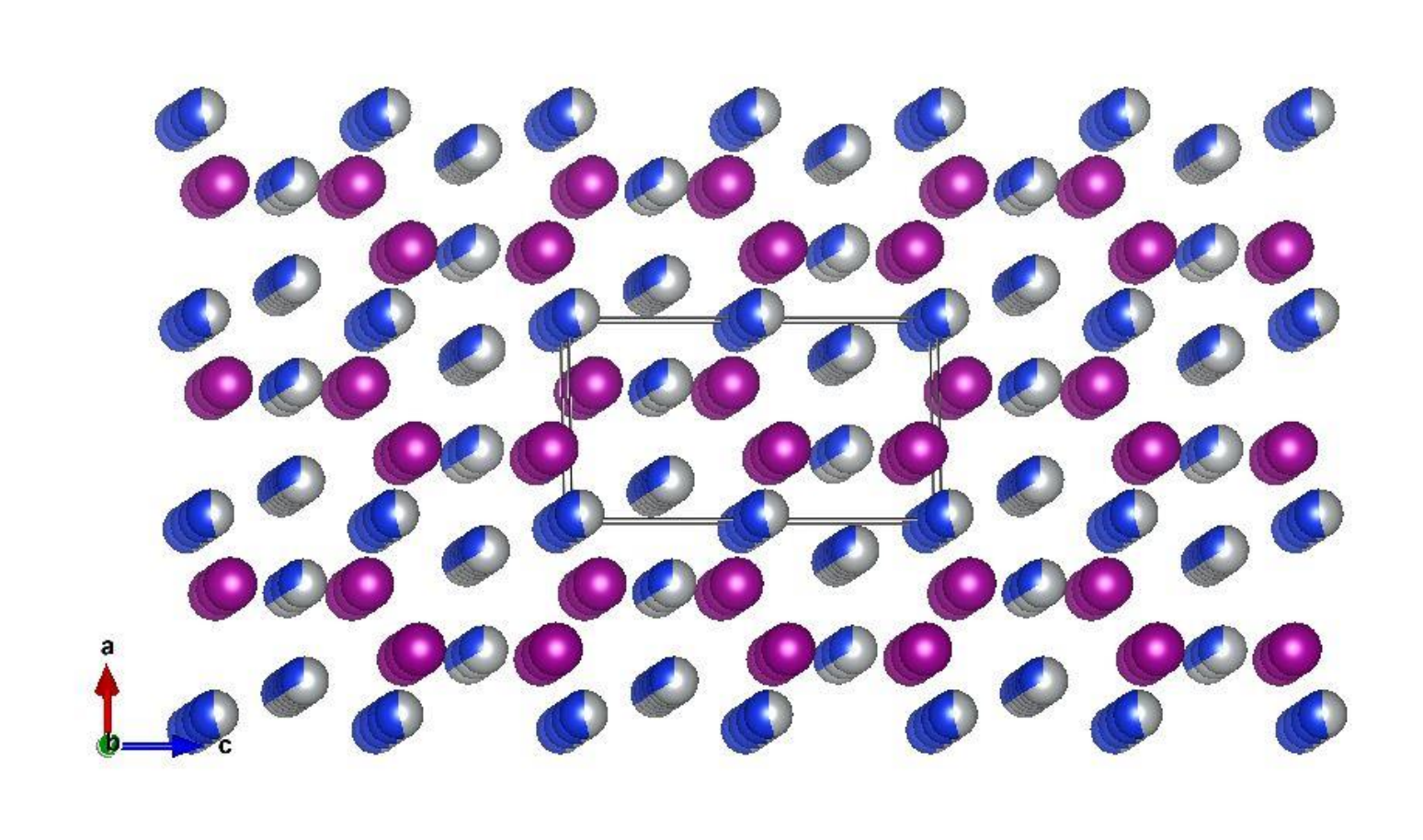
Pole Figures from Au film
Stereographic projections of individual 2θ hemispheres



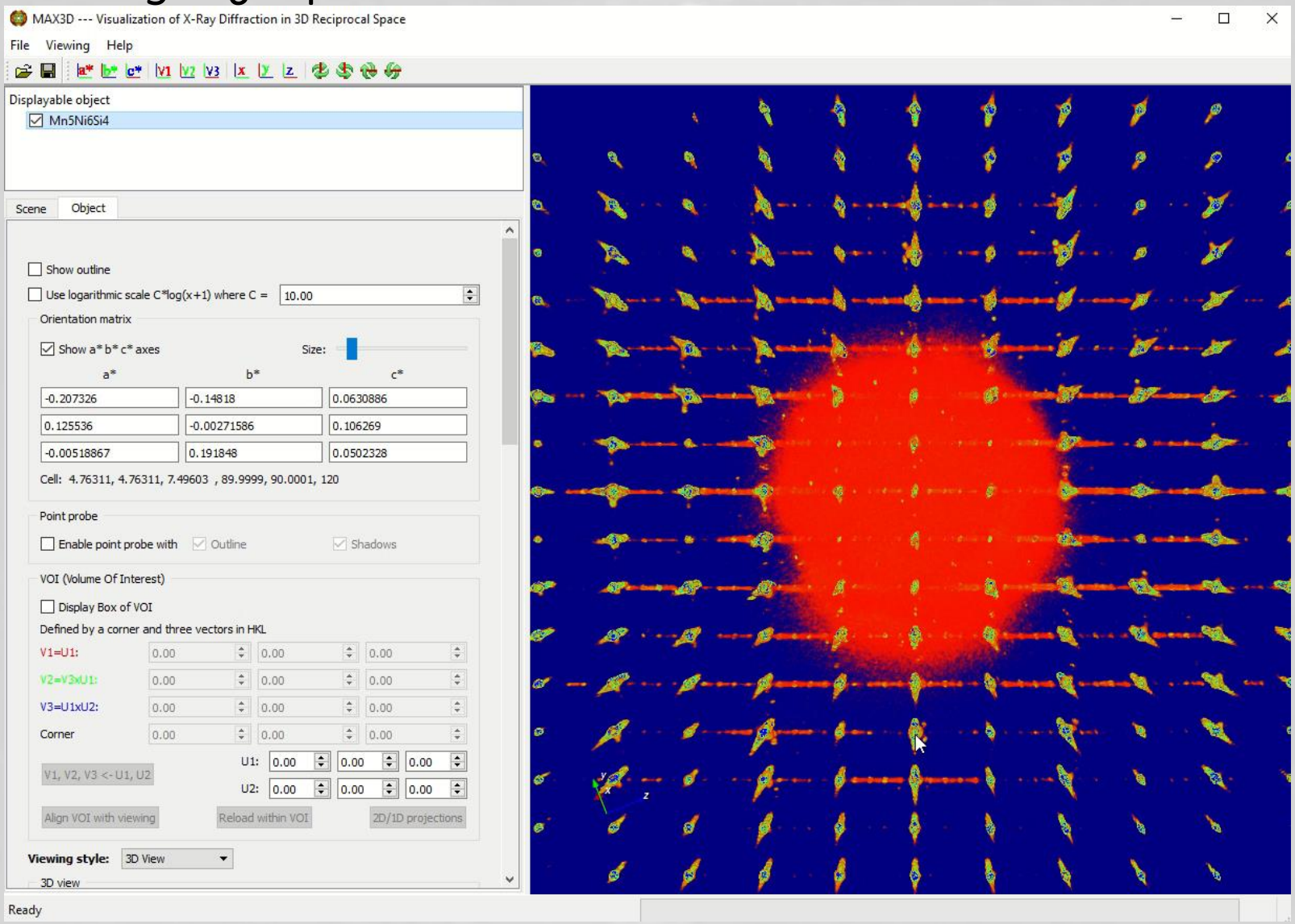
Recent Modifications to MAX3D

- Update VTK and QT libraries
- Merge frame readers into a Unified Reader
- 3D shells of $\Delta 2\theta$ thickness for pole figures
- 3D Clipping
- 2D and 1D projection output
- 3D pointer to identify diffraction features in terms of (fractional) hkl
- Linux version
- Supercomputer cluster version

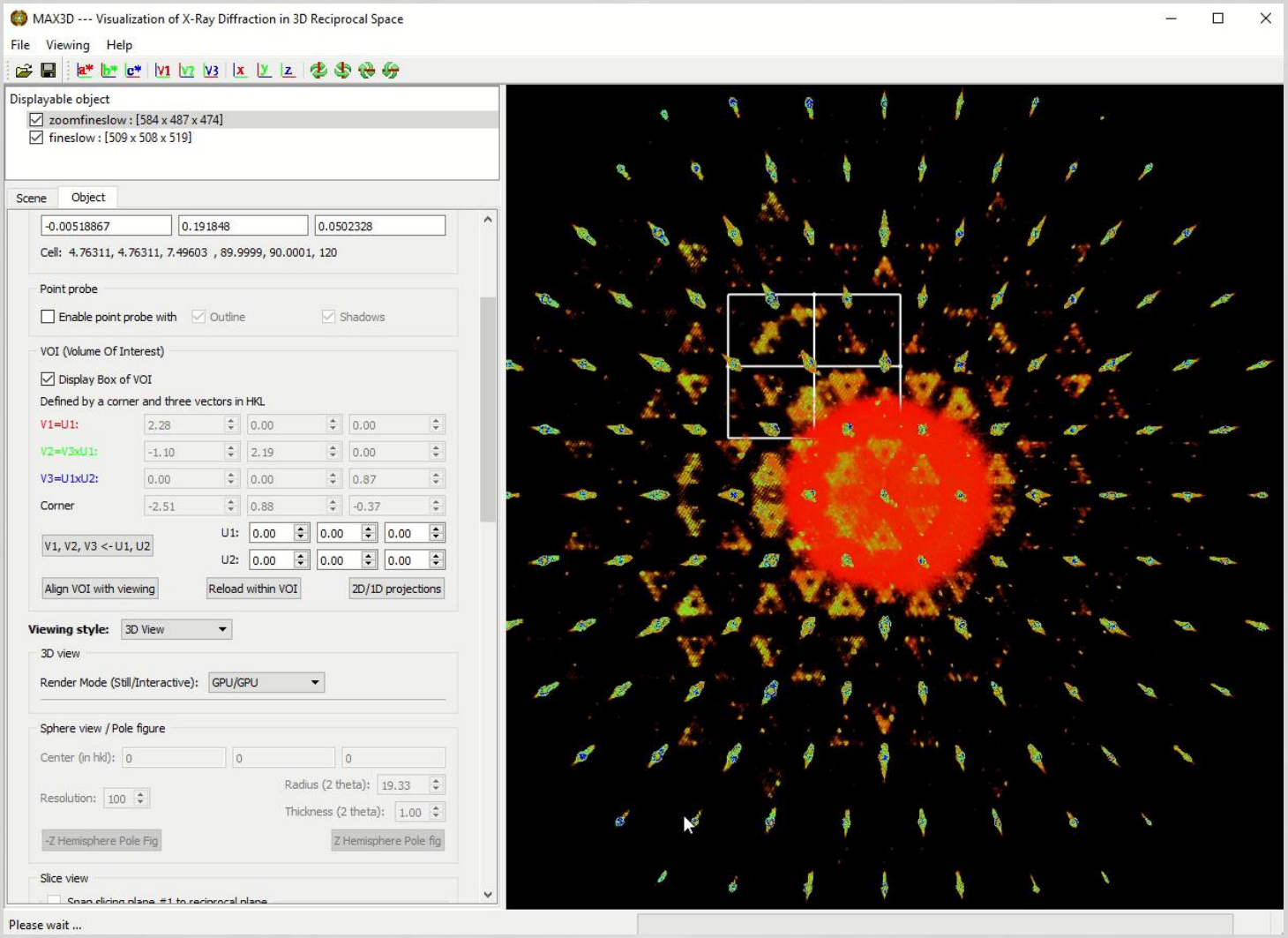
$Mn_5Ni_6Si_4$; Marek Niewczas, Sheikh Ahmed

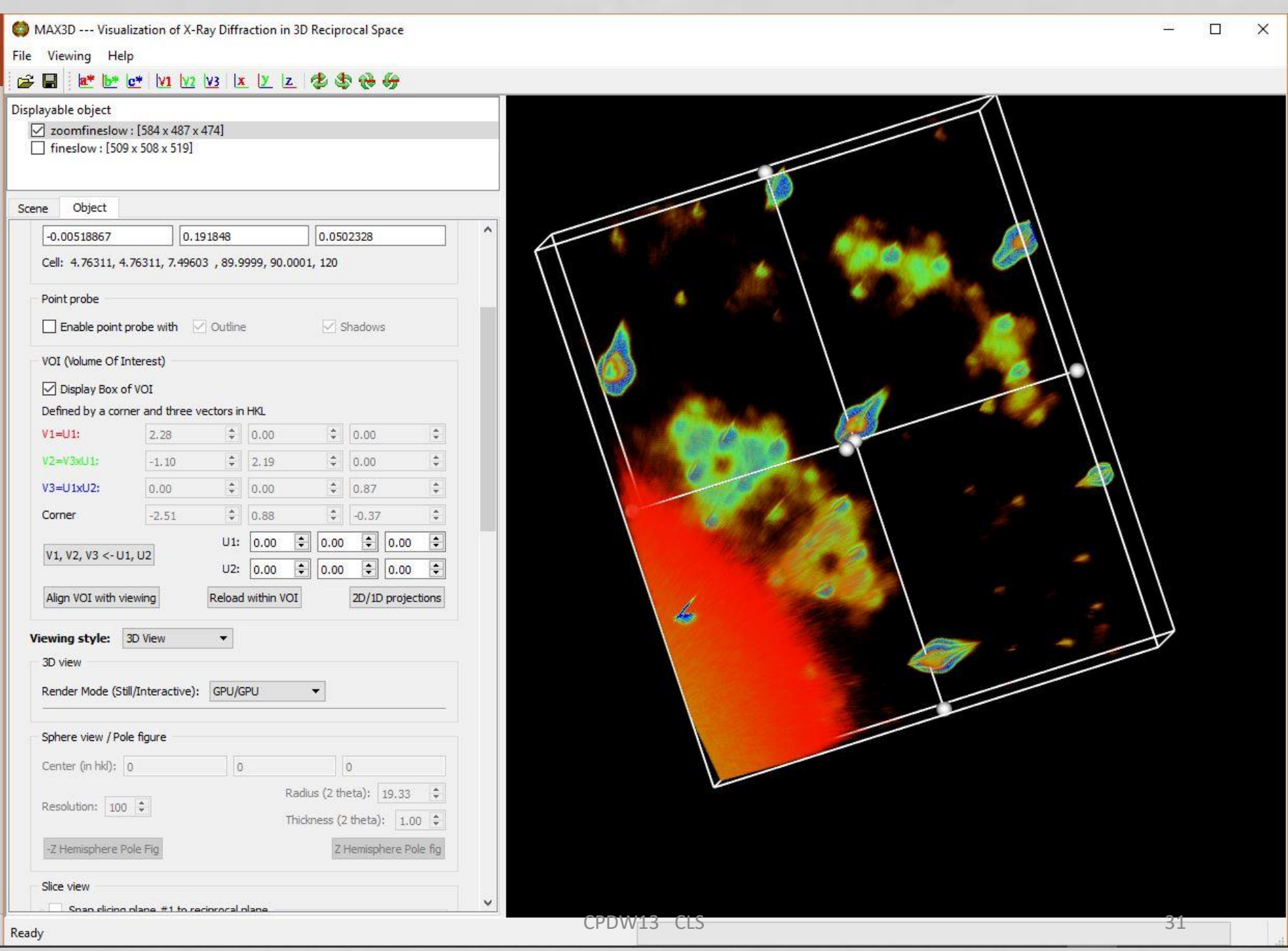


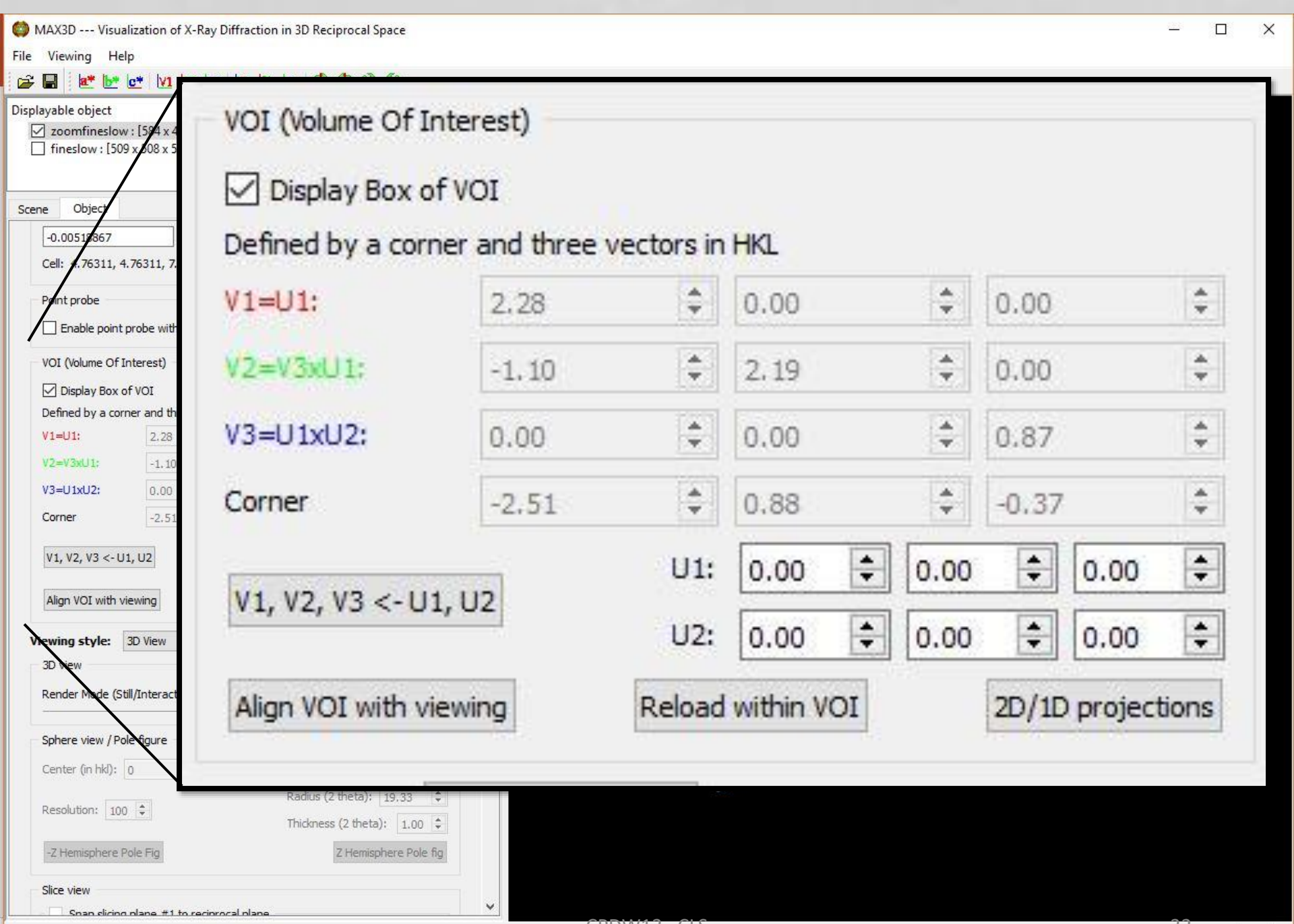
$Mn_5Ni_6Si_4$; Marek Niewczas, Sheikh Ahmed



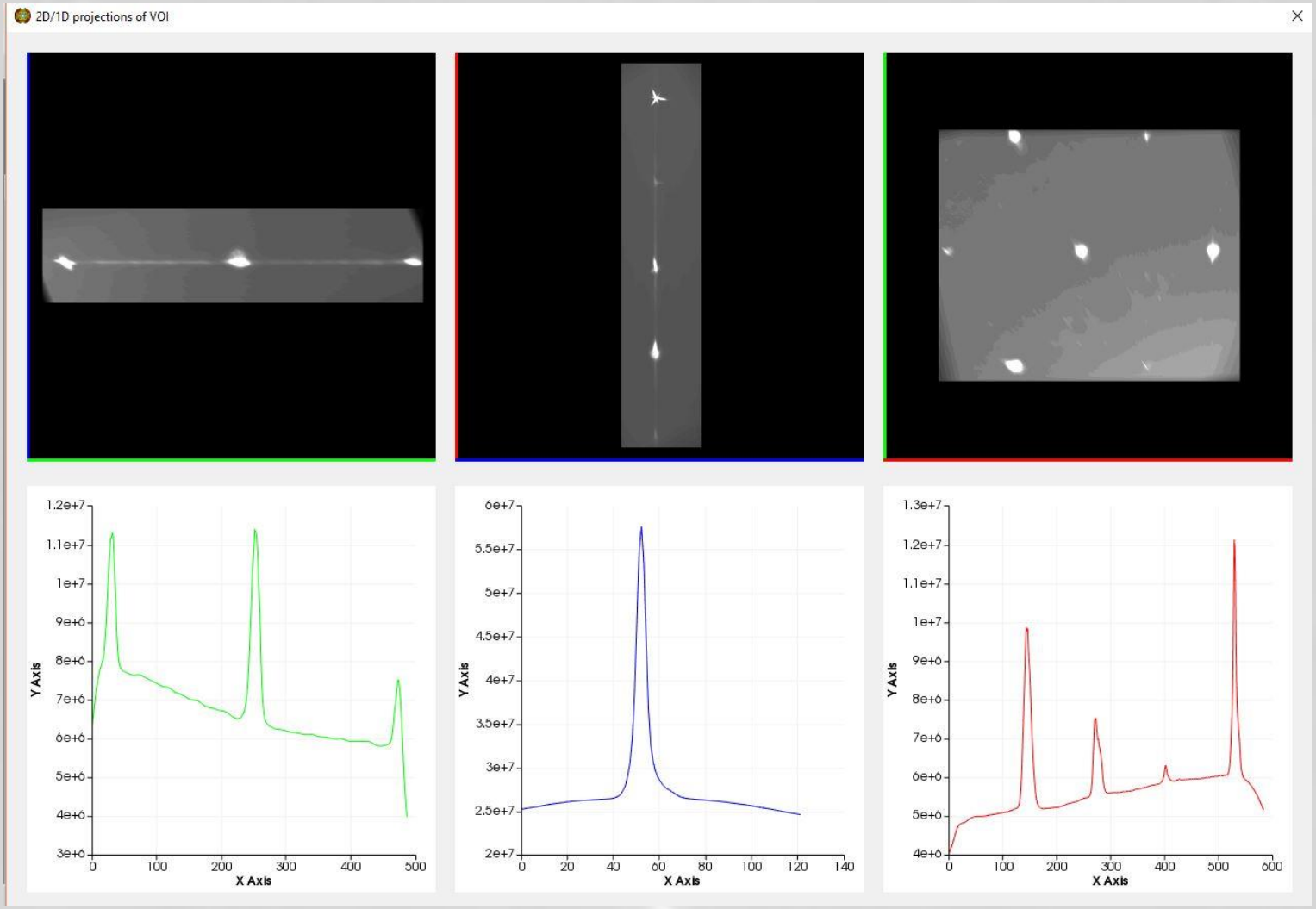
3D Clipping, Reloading at Higher Res



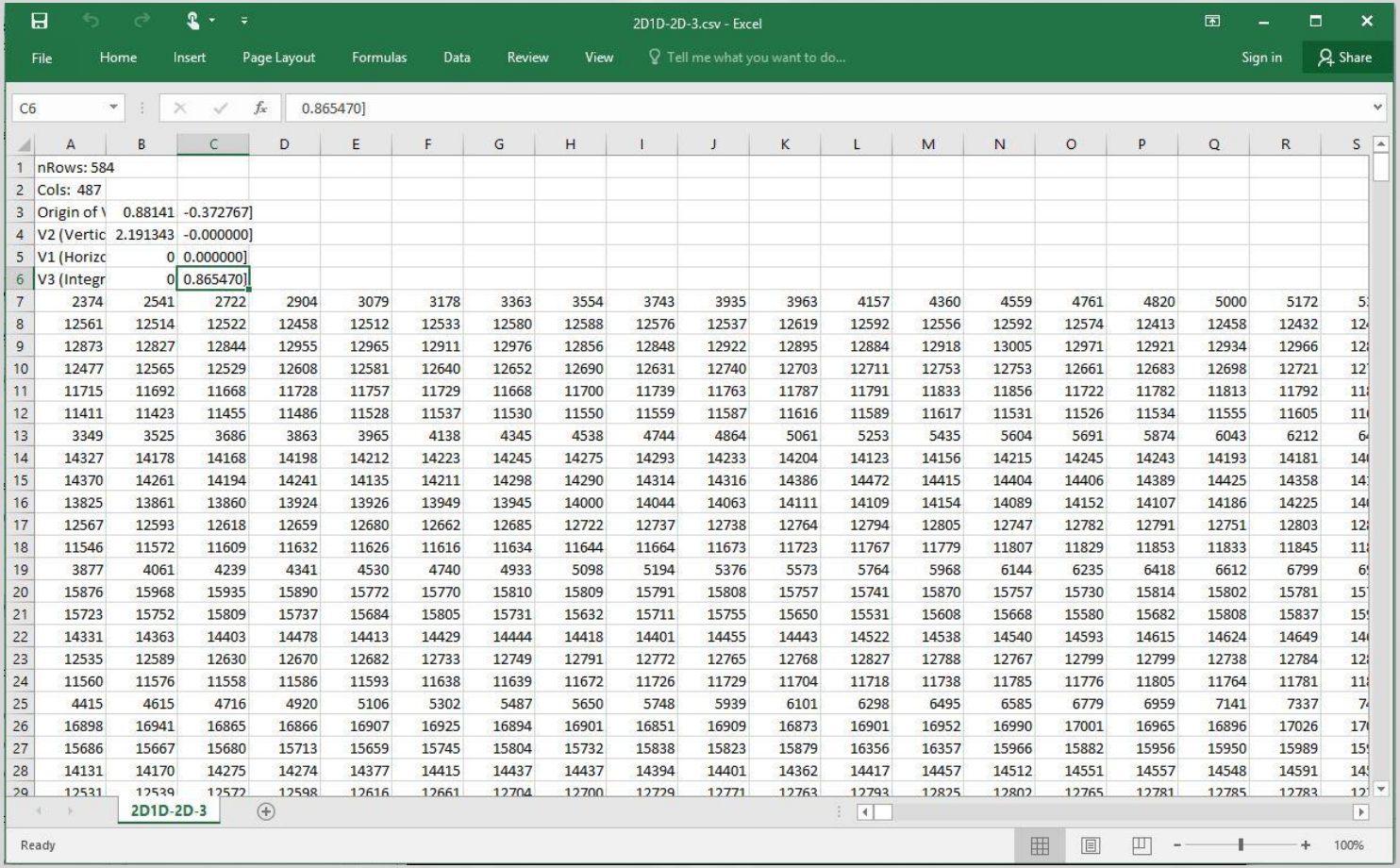

MAX3D --- Visualization of X-Ray Diffraction in 3D Reciprocal Space
File
Viewing
Help
Displayable object
zoomfineslow : [584 x 487 x 474]
fineslow : [509 x 508 x 519]
Scene
Object
-0.00518867
0.191848
0.0502328
Cell: 4.76311, 4.76311, 7.49603 , 89.9999, 90.0001, 120
Point probe
Enable point probe with
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


MAX3D --- Visualization of X-Ray Diffraction in 3D Reciprocal Space
File Viewing Help
Displayable object
zoomfineslow : [584 x 4
fineslow : [509 x 508 x 5
Scene Object
-0.00518867
Cell: 4.76311, 4.76311, 7.
Point probe
Enable point probe with
VOI (Volume Of Interest)
Display Box of VOI
Defined by a corner and th
V1=U1: 2.28
V2=V3xU1: -1.10
V3=U1xU2: 0.00
Corner -2.51
V1, V2, V3 <- U1, U2
Align VOI with viewing
Viewing style: 3D View
3D view
Render Mode (Still/Interact
Sphere view / Pole figure
Center (in hkl): 0
Resolution: 100
Radius (2 theta): 19.33
Thickness (2 theta): 1.00
-Z Hemisphere Pole Fig
Z Hemisphere Pole fig
Slice view
Ready
VOI (Volume Of Interest)
Display Box of VOI
Defined by a corner and three vectors in HKL
V1=U1: 2.28 0.00 0.00
V2=V3xU1: -1.10 2.19 0.00
V3=U1xU2: 0.00 0.00 0.87
Corner -2.51 0.88 -0.37
U1: 0.00 0.00 0.00
U2: 0.00 0.00 0.00
V1, V2, V3 <- U1, U2
Align VOI with viewing
Reload within VOI
2D/1D projections

2D and 1D Projections of the VOI



Example: 2D Projection Output



2D1D-2D-3.csv - Excel

File Home Insert Page Layout Formulas Data Review View Tell me what you want to do... Sign in Share

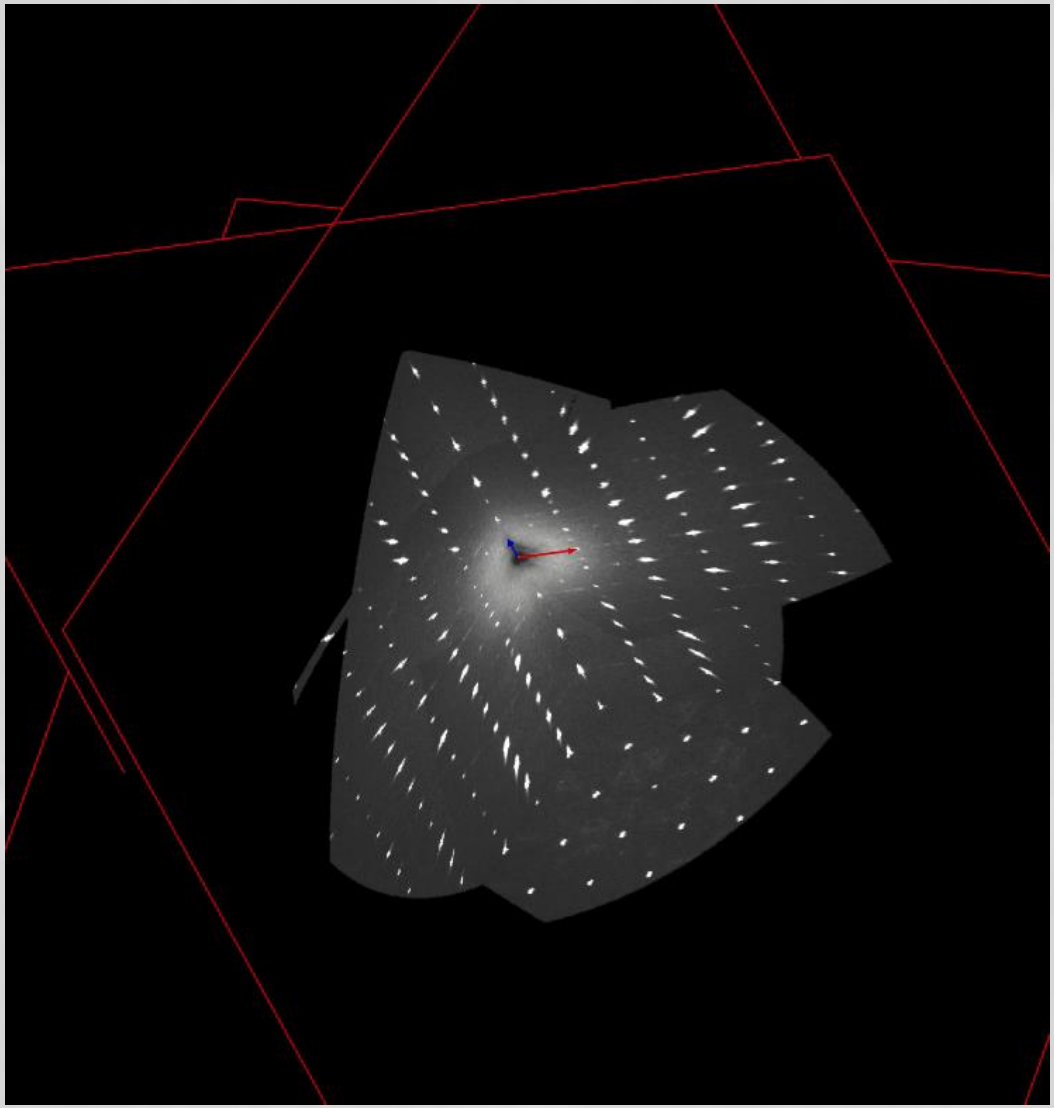
C6 0.865470]

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S
1	nRows: 584																		
2	Cols: 487																		
3	Origin of V	0.88141	-0.372767]																
4	V2 (Vertic	2.191343	-0.000000]																
5	V1 (Horizc	0	0.000000]																
6	V3 (Integr	0	0.865470]																
7	2374	2541	2722	2904	3079	3178	3363	3554	3743	3935	3963	4157	4360	4559	4761	4820	5000	5172	5
8	12561	12514	12522	12458	12512	12533	12580	12588	12576	12537	12619	12592	12556	12592	12574	12413	12458	12432	12
9	12873	12827	12844	12955	12965	12911	12976	12856	12848	12922	12895	12884	12918	13005	12971	12921	12934	12966	12
10	12477	12565	12529	12608	12581	12640	12652	12690	12631	12740	12703	12711	12753	12753	12661	12683	12698	12721	12
11	11715	11692	11668	11728	11757	11729	11668	11700	11739	11763	11787	11791	11833	11856	11722	11782	11813	11792	11
12	11411	11423	11455	11486	11528	11537	11530	11550	11559	11587	11616	11589	11617	11531	11526	11534	11555	11605	11
13	3349	3525	3686	3863	3965	4138	4345	4538	4744	4864	5061	5253	5435	5604	5691	5874	6043	6212	6
14	14327	14178	14168	14198	14212	14223	14245	14275	14293	14233	14204	14123	14156	14215	14245	14243	14193	14181	14
15	14370	14261	14194	14241	14135	14211	14298	14290	14314	14316	14386	14472	14415	14404	14406	14389	14425	14358	14
16	13825	13861	13860	13924	13926	13949	13945	14000	14044	14063	14111	14109	14154	14089	14152	14107	14186	14225	14
17	12567	12593	12618	12659	12680	12662	12685	12722	12737	12738	12764	12794	12805	12747	12782	12791	12751	12803	12
18	11546	11572	11609	11632	11626	11616	11634	11644	11664	11673	11723	11767	11779	11807	11829	11853	11833	11845	11
19	3877	4061	4239	4341	4530	4740	4933	5098	5194	5376	5573	5764	5968	6144	6235	6418	6612	6799	6
20	15876	15968	15935	15890	15772	15770	15810	15809	15791	15808	15757	15741	15870	15757	15730	15814	15802	15781	15
21	15723	15752	15809	15737	15684	15805	15731	15632	15711	15755	15650	15531	15608	15668	15580	15682	15808	15837	15
22	14331	14363	14403	14478	14413	14429	14444	14418	14401	14455	14443	14522	14538	14540	14593	14615	14624	14649	14
23	12535	12589	12630	12670	12682	12733	12749	12791	12772	12765	12768	12827	12788	12767	12799	12799	12738	12784	12
24	11560	11576	11558	11586	11593	11638	11639	11672	11726	11729	11704	11718	11738	11785	11776	11805	11764	11781	11
25	4415	4615	4716	4920	5106	5302	5487	5650	5748	5939	6101	6298	6495	6585	6779	6959	7141	7337	7
26	16898	16941	16865	16866	16907	16925	16894	16901	16851	16909	16873	16901	16952	16990	17001	16965	16896	17026	17
27	15686	15667	15680	15713	15659	15745	15804	15732	15838	15823	15879	16356	16357	15966	15882	15956	15950	15989	15
28	14131	14170	14275	14274	14377	14415	14437	14437	14394	14401	14362	14417	14457	14512	14551	14557	14548	14591	14
29	12531	12539	12572	12598	12616	12661	12704	12700	12729	12771	12763	12793	12825	12802	12765	12781	12785	12783	12

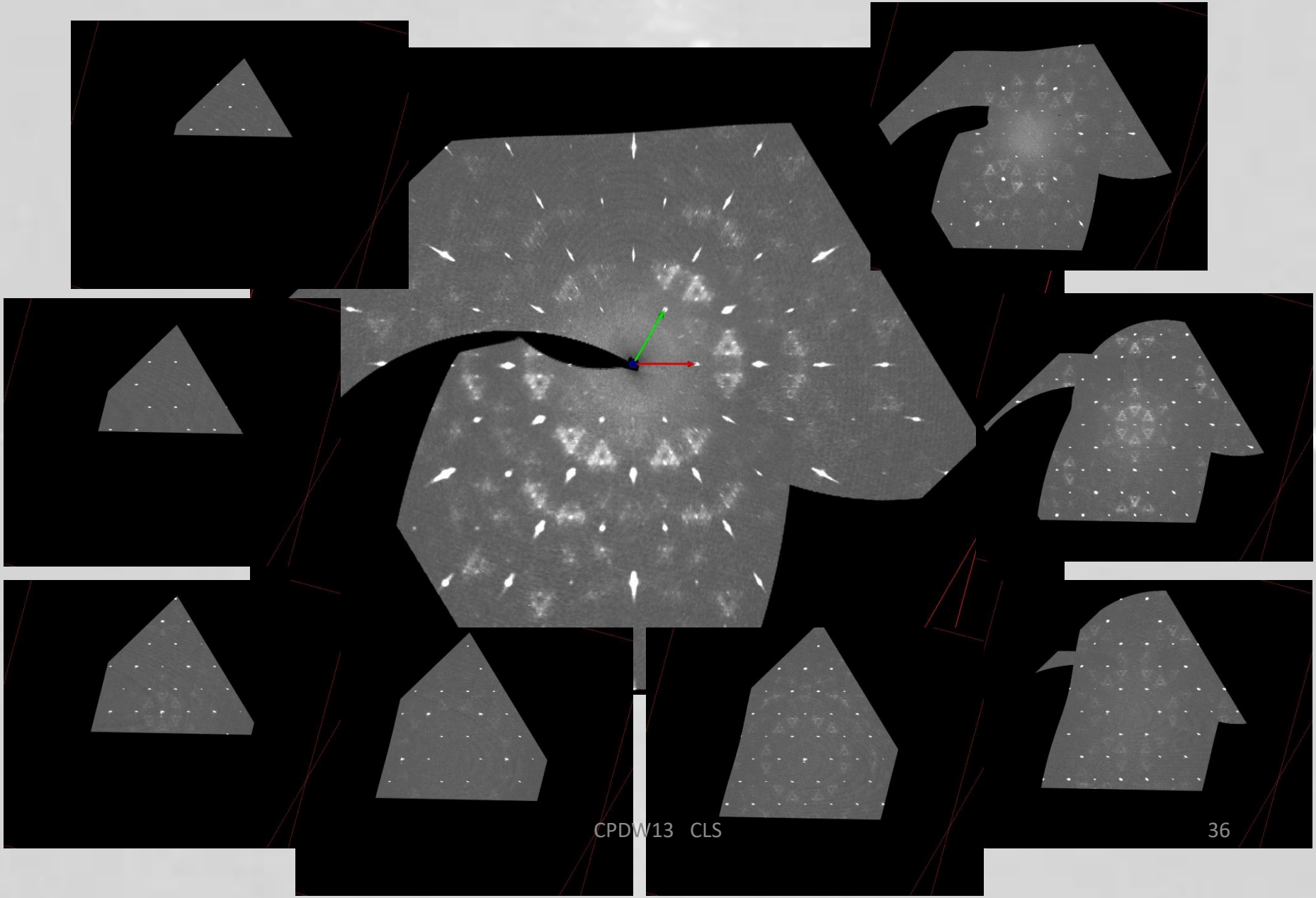
2D1D-2D-3

Ready 100%

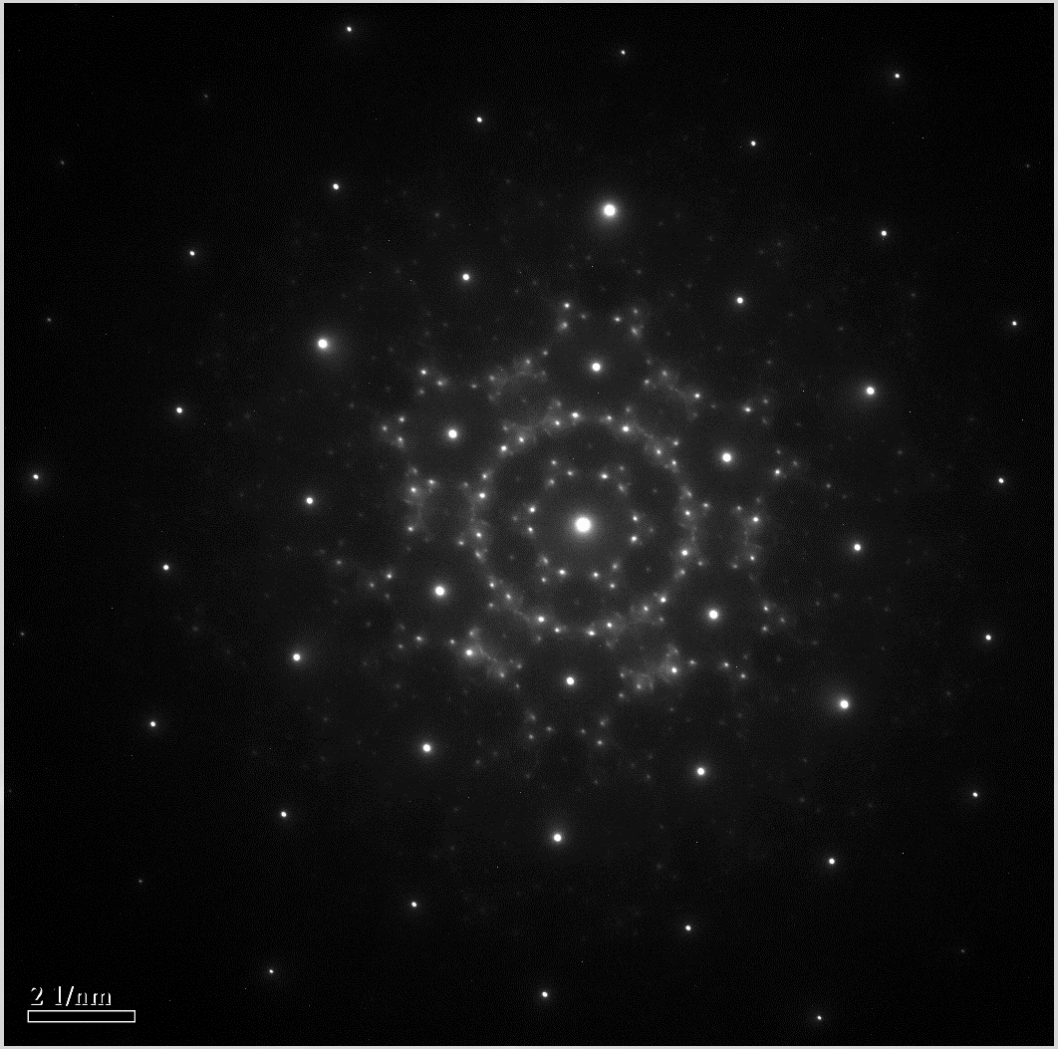
Slice View



HK0 > HK8

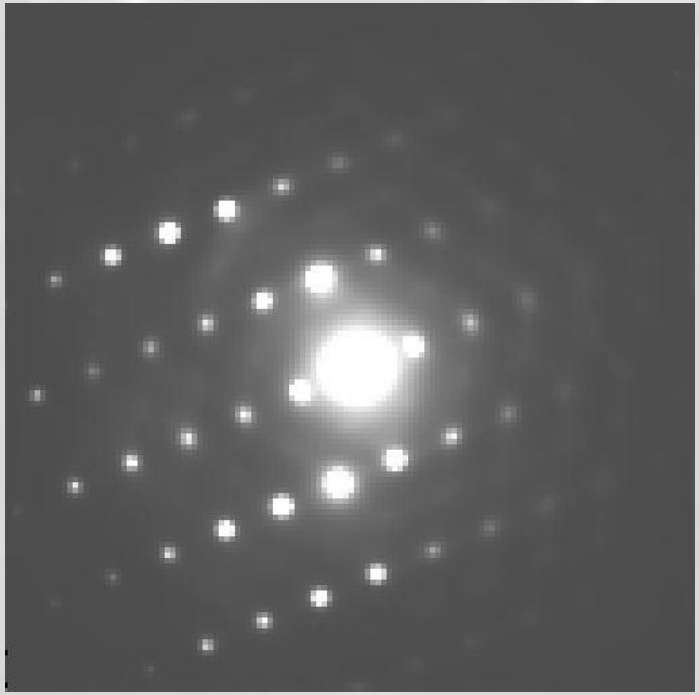


Electron Diffraction of $Mn_5Ni_6Si_4$



Current MAX3D project

- Reader for 3D Electron Diffraction Data
  - EMPAD detector
    - Jo Etheridge, Monash University



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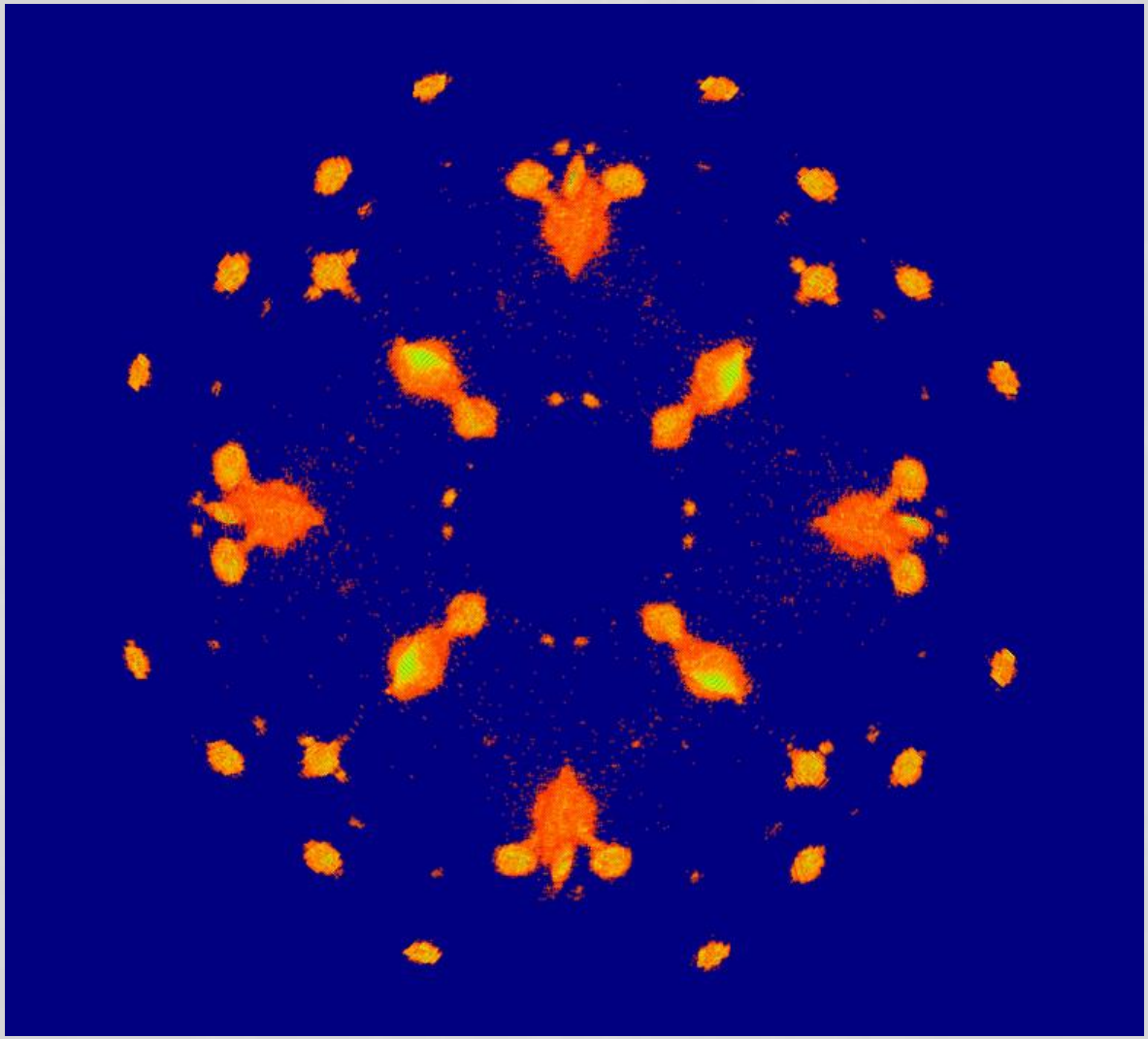
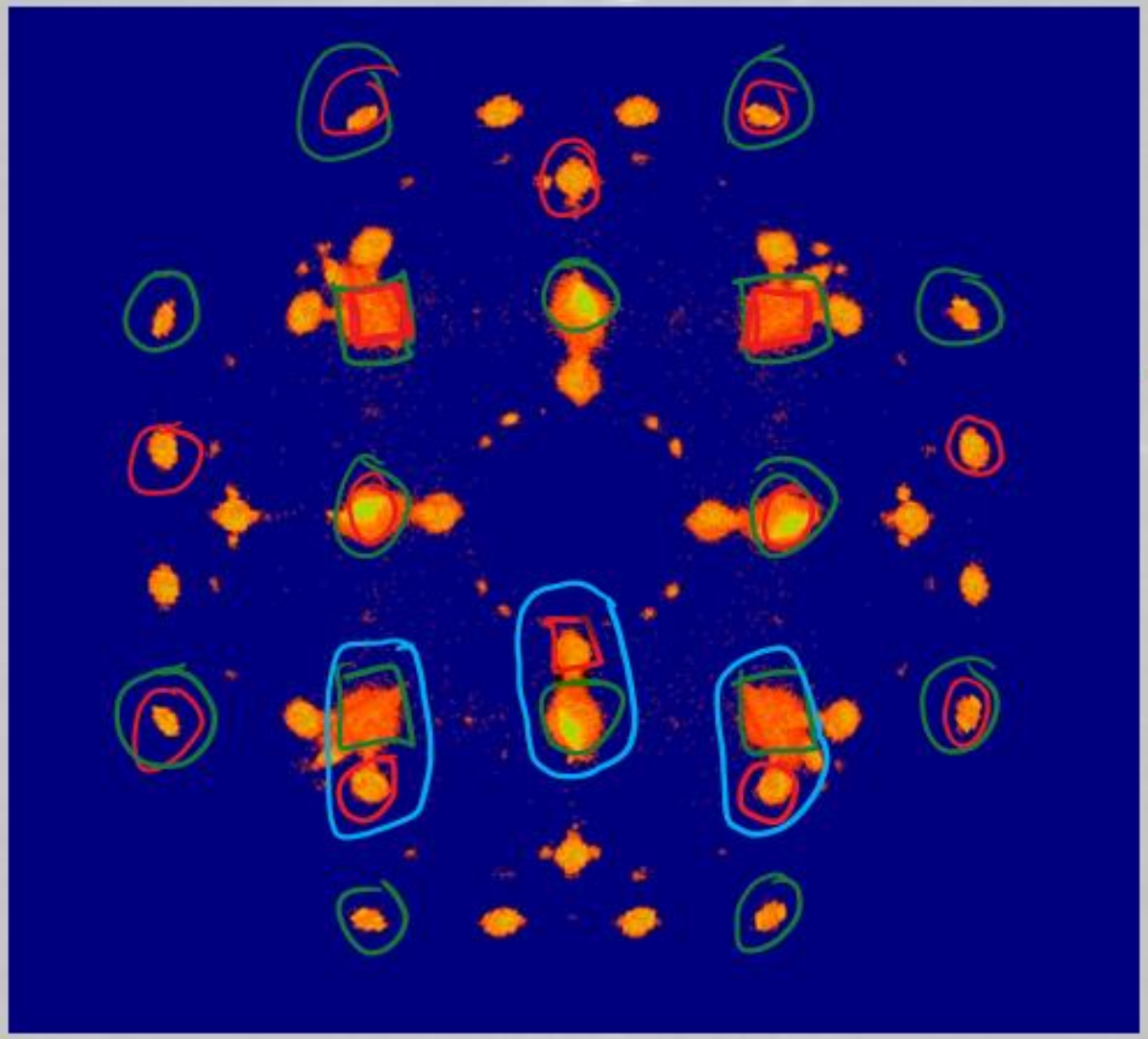
  - Ranil Sonnadara
- Mark Hollingsworth, KSU
- Joe Ferrara, Rigaku

- Thank You
  - . . . and for the tutorial . . .

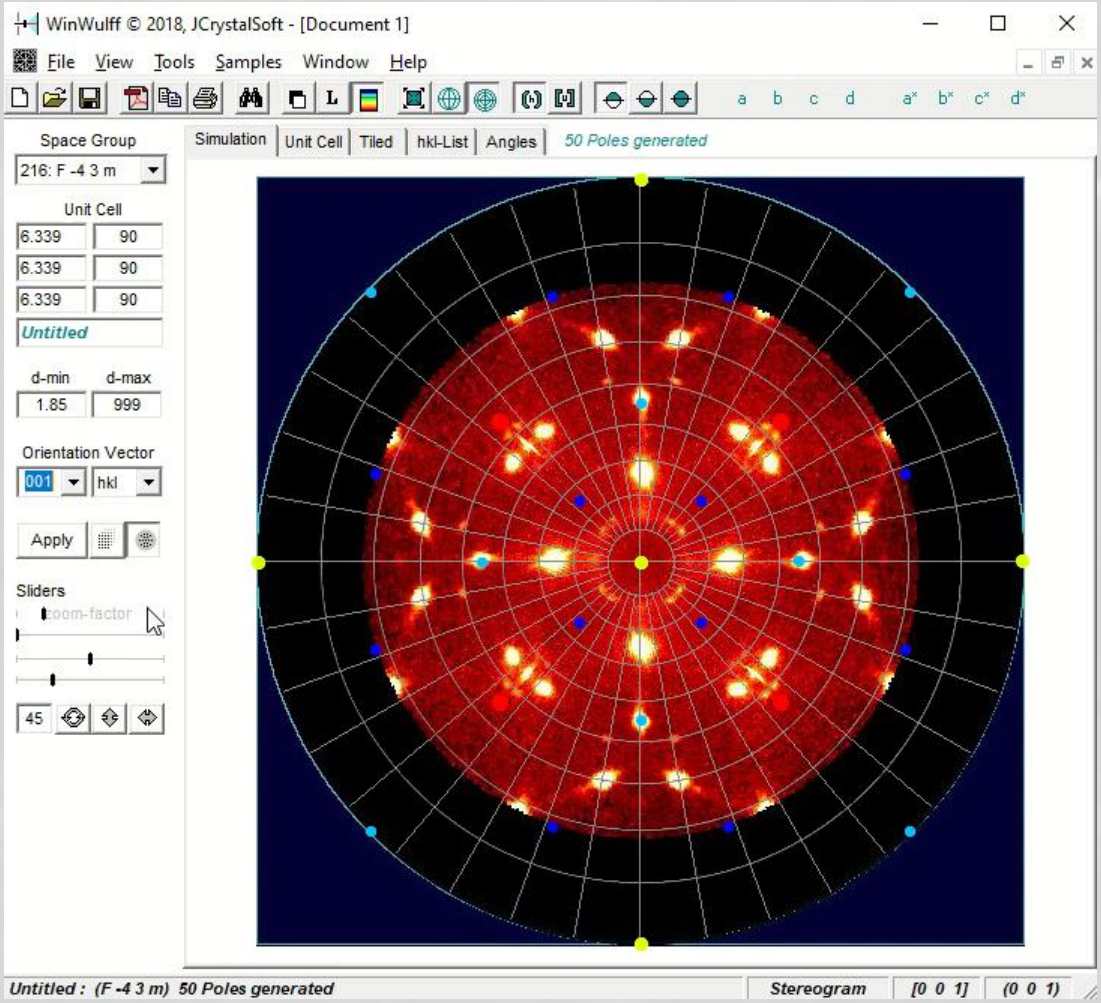
InAsSb Pillars for Multispectral Long-wavelength Infrared Absorption

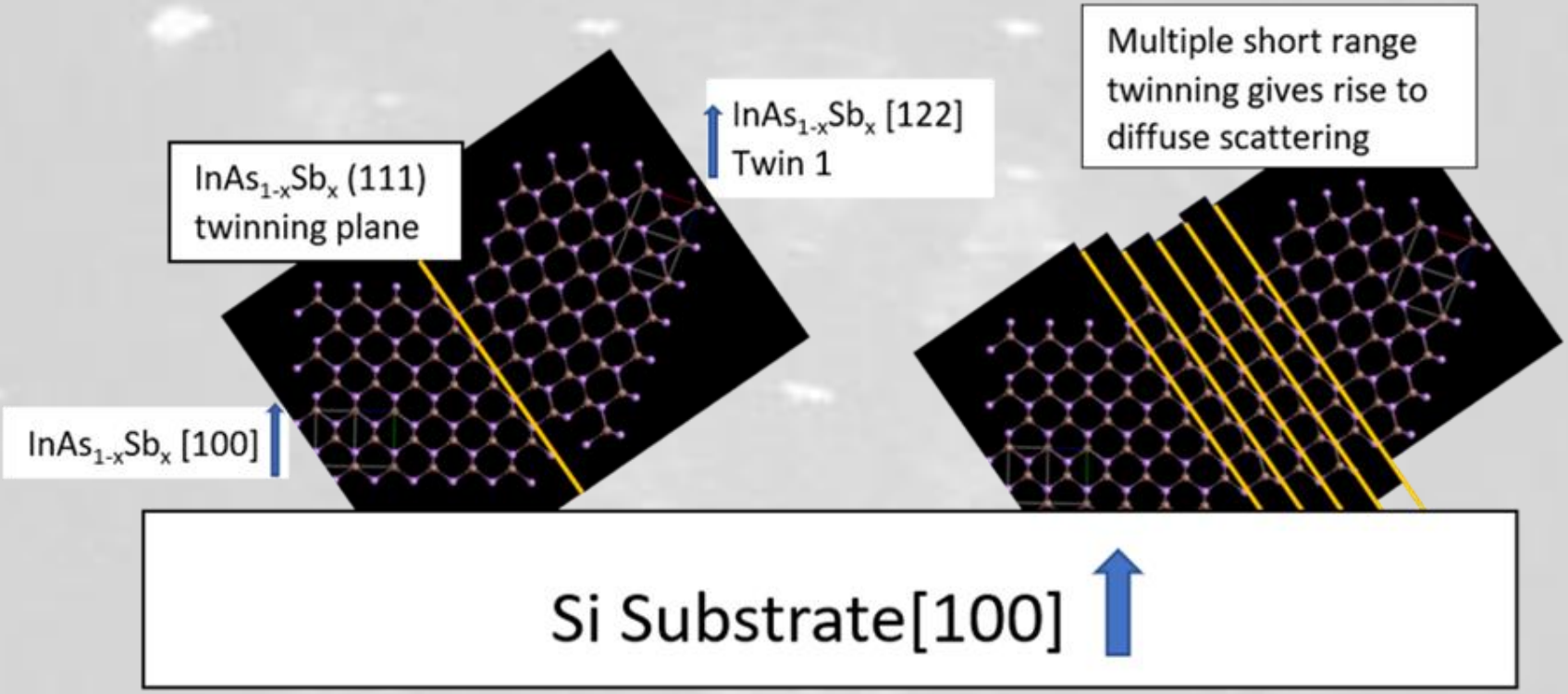
Curtis J. Goosney, Victoria M. Jarvis, James F. Britten, Ray R. LaPierre




100
113
220
221
113
220
DIFFUSE
SCATTERING

311 Pole Figure




Multiple short range twinning gives rise to diffuse scattering
InAs1-xSbx [122] Twin 1
InAs1-xSbx (111) twinning plane
InAs1-xSbx [100]
Si Substrate[100]