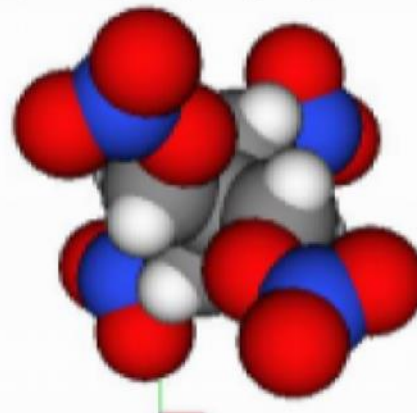


# GSAS-II OVERVIEW

**ROBERT VON DREELE**  
APS, ANL (retired)  
vondreele@anl.gov

# GSAS-2



# GSAS-II INSTALLATION

# GSAS-II INSTALLATION

Search web for “GSAS-II” → only thing out there:

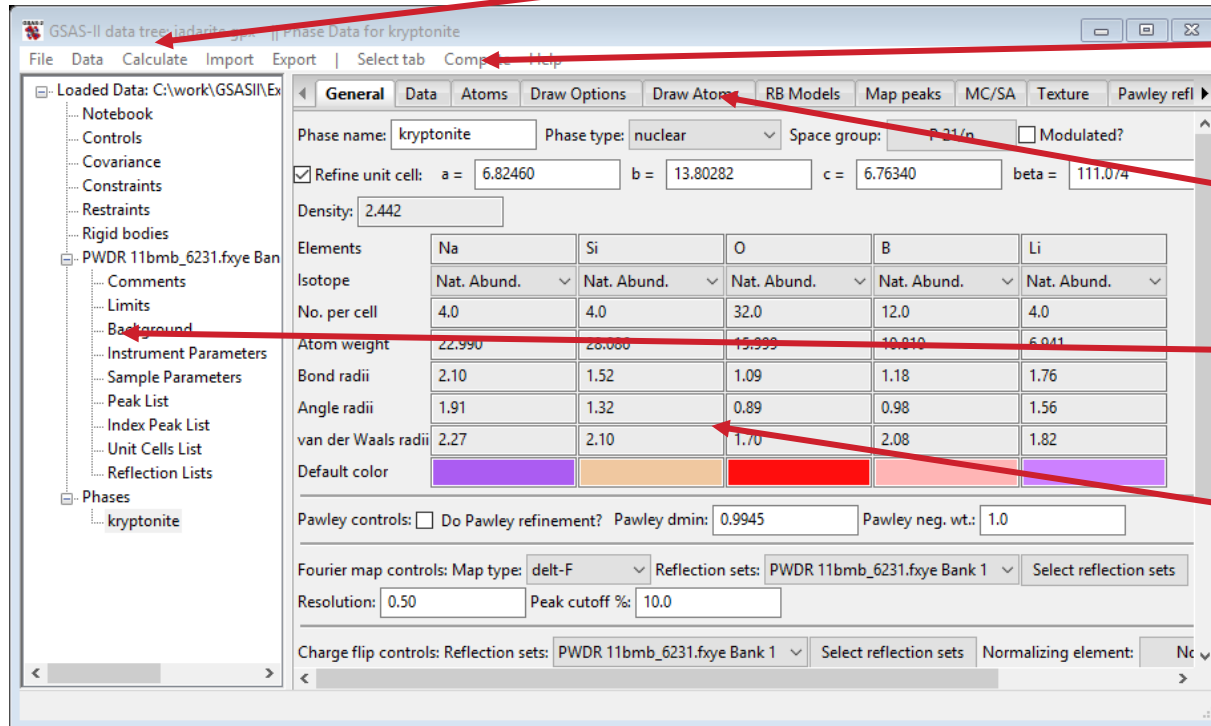
See GSAS-II “home page” <https://subversion.xray.aps.anl.gov/trac/pyGSAS>.

- Includes
  - Installation instructions – includes a 1-step for python/GSAS-II
  - Tutorials - ~50 of these; direct access via Help menu for GSAS-II.
  - **NB: Must have internet access!**
  - Usually installs in your personal space so no “authorizations” usually needed (unless you institution forbids it!)
  - Can be installed anywhere (even a memory stick – 5GB or more)
  - Don’t use the resulting GSAS-II directory for your project files; work elsewhere
  - **Update often!** Versions can change almost daily as we add/fix things. We work under the continuous software improvement model; not the fixed update schedule model commonly used by commercial software.

# GSAS-II – MODERN GUI APPLICATION FOR CRYSTALLOGRAPHY (IN PYTHON)

# GSAS-II: MODERN GUI

## – 2 FRAME LAYOUT + CONSOLE



Main menu

Submenu

Data tabs

Data tree

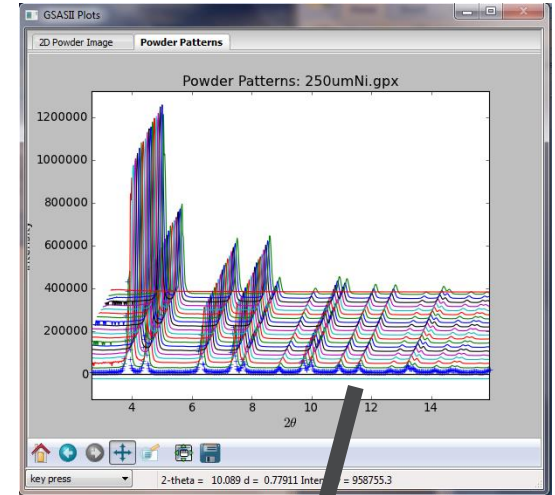
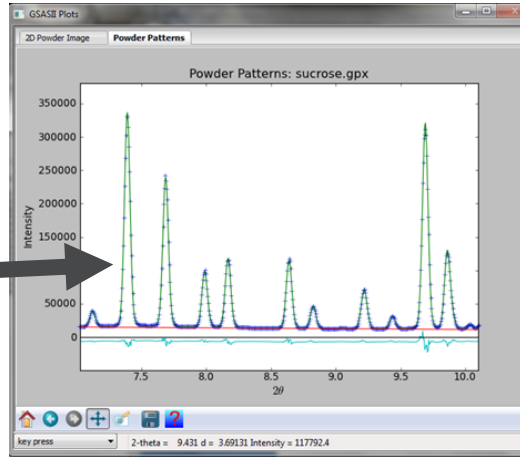
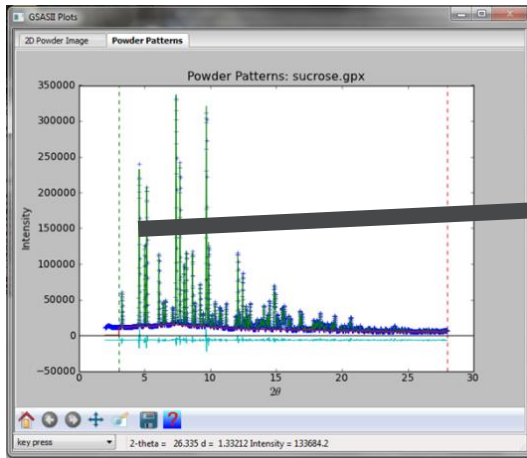
Data window

Plot & console in separate frames

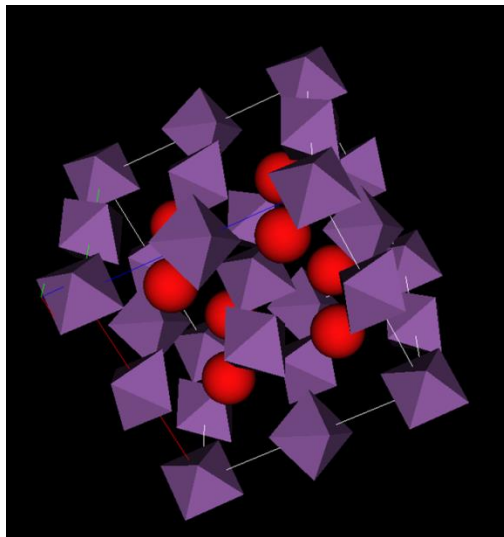


# THE PLOTS - ADVANCED VISUALIZATION

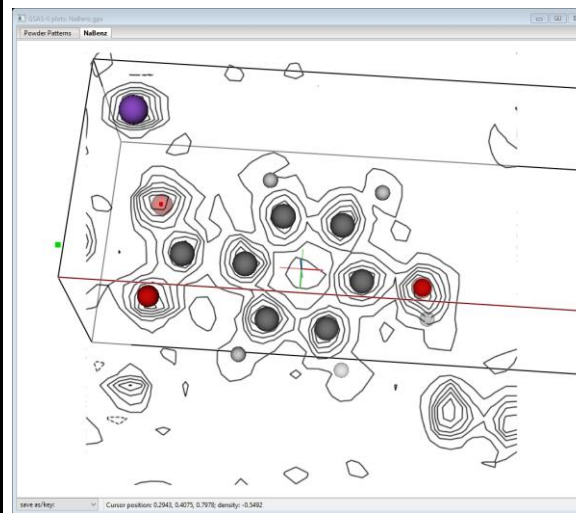
Powder profile – easy zoom



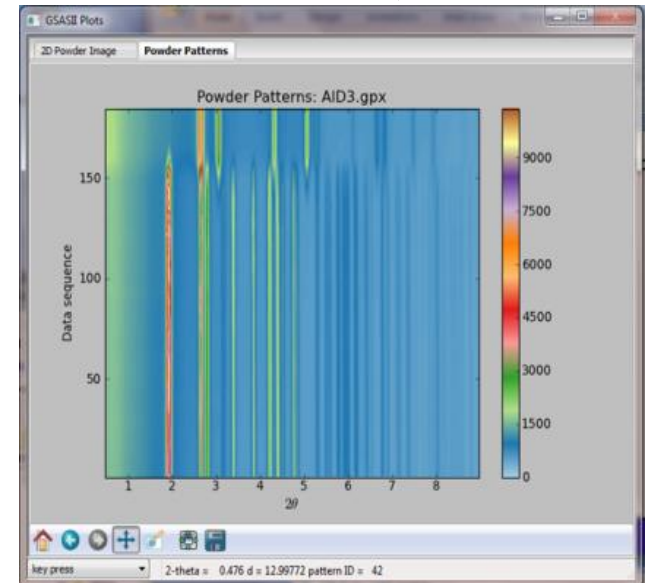
Waterfall plot



Structure drawing



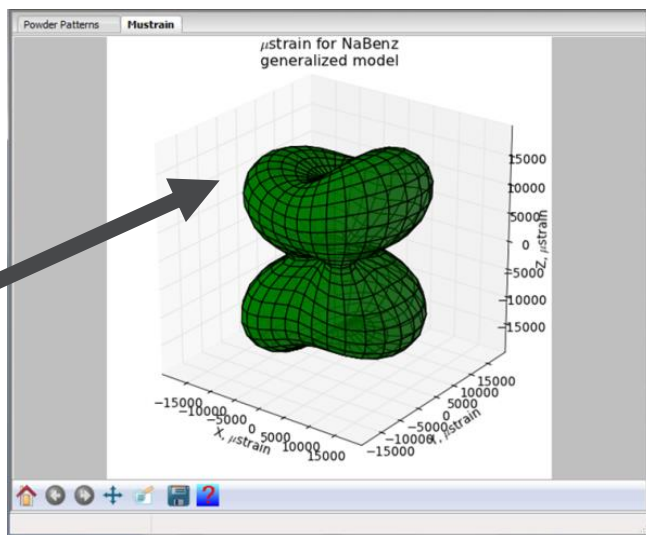
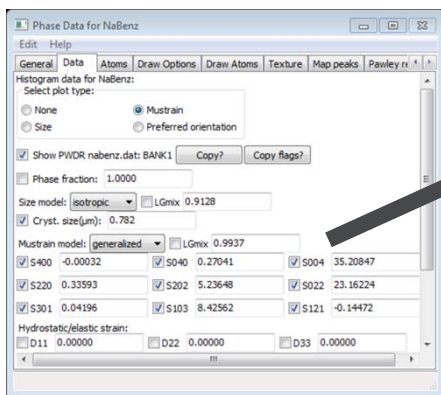
Contoured density thru any plane



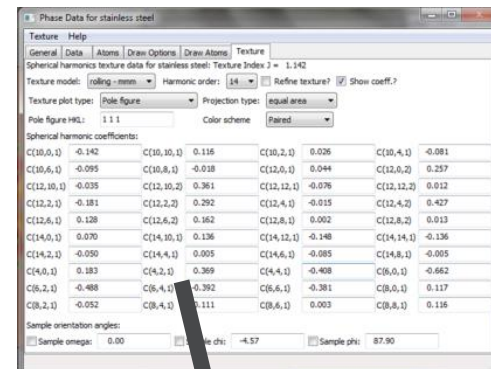
Contour plot

# ADVANCED VISUALIZATION IN GSAS-II: NUMBERS AS PICTURES

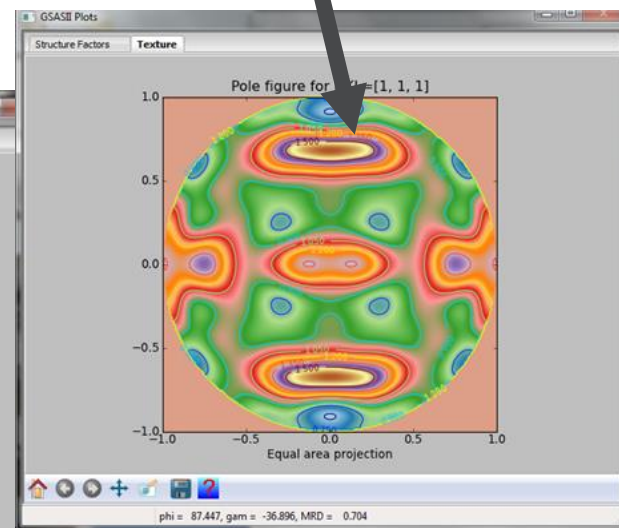
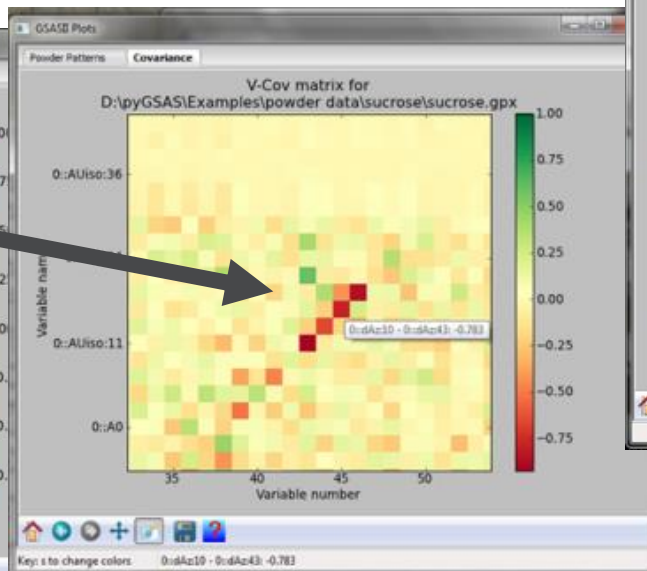
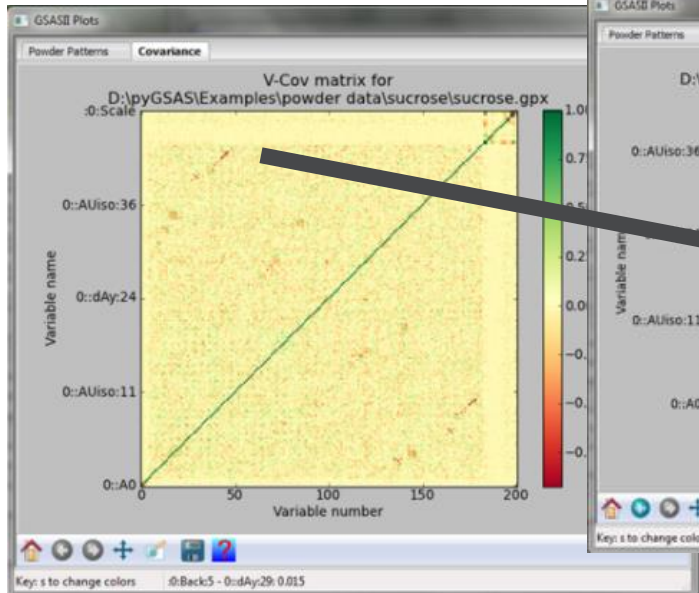
$\mu$ strain surface



Texture – sph. harmonics



v-cov matrix



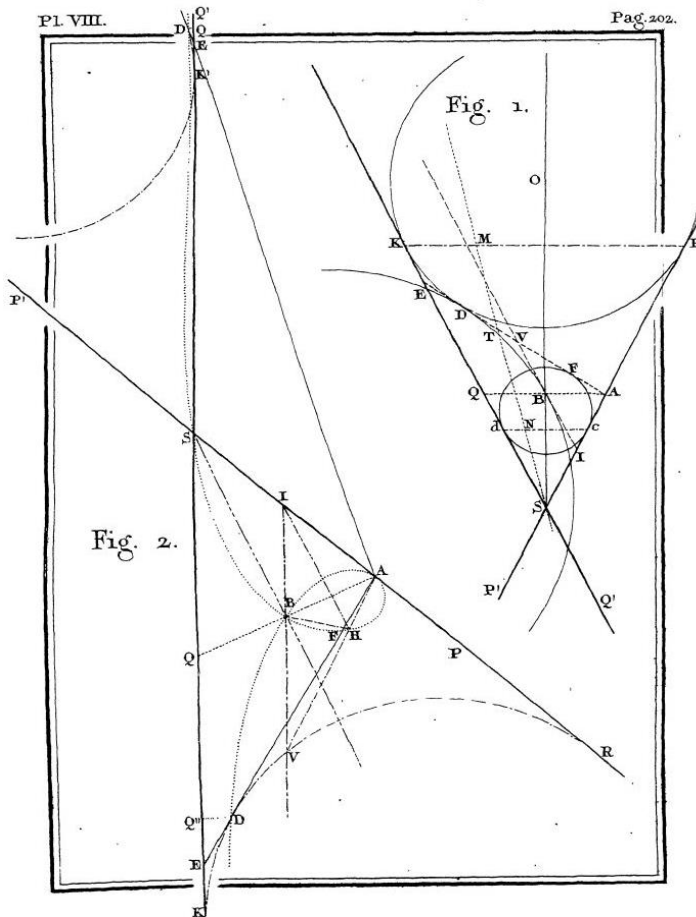
# CONIC SECTIONS

Where is the incident beam on the detector?

Fit2D (& DataSqueeze) – assumes center of the diffraction ellipse - False  
Analysis – G.P. Dandelin,

Nouveaux memories de l'Academie royal de Bruxelles, 2, 171-202 (1822)

Drawing by Dandelin p.202



Taken from Dandelin's original paper  
Fig. 1: Shows the 2 spheres in contact  
with plane EA

- line SO is cone axis
  - F&D are the ellipse foci on the plane
- He refers to a work by M. Quetlet as  
having previously made this construction  
- source?

This is not something new!

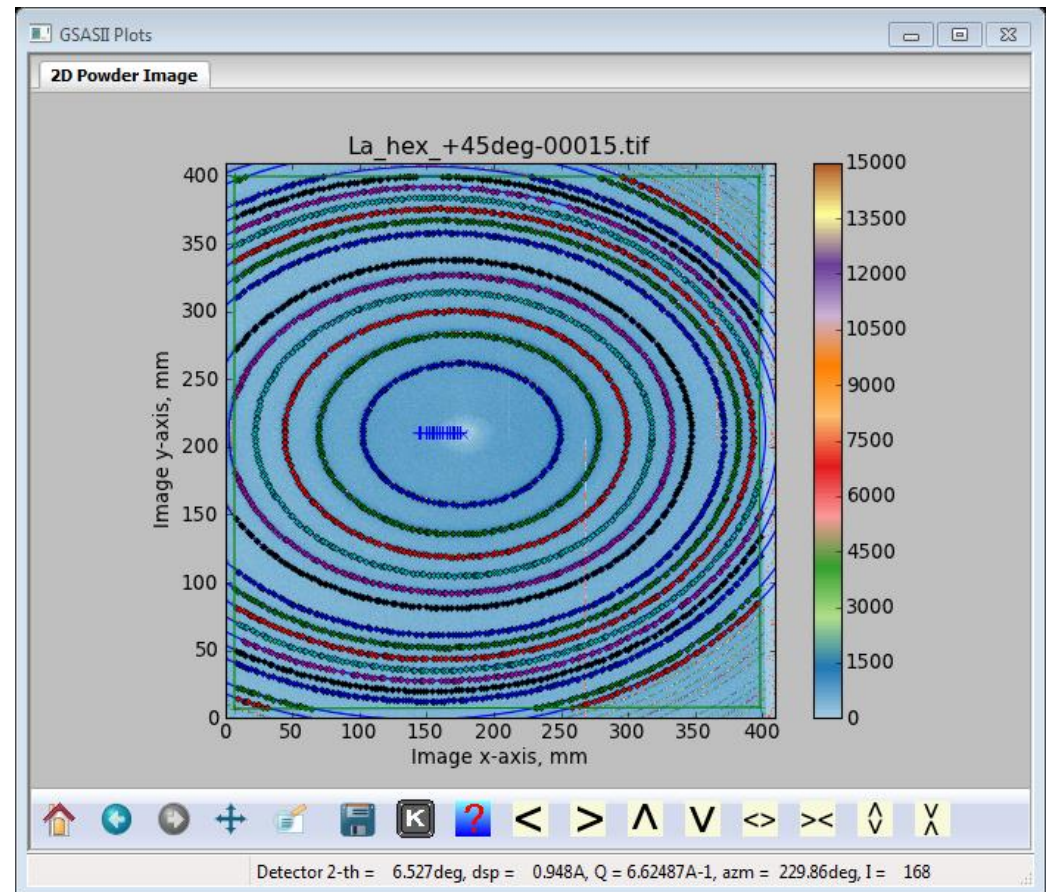
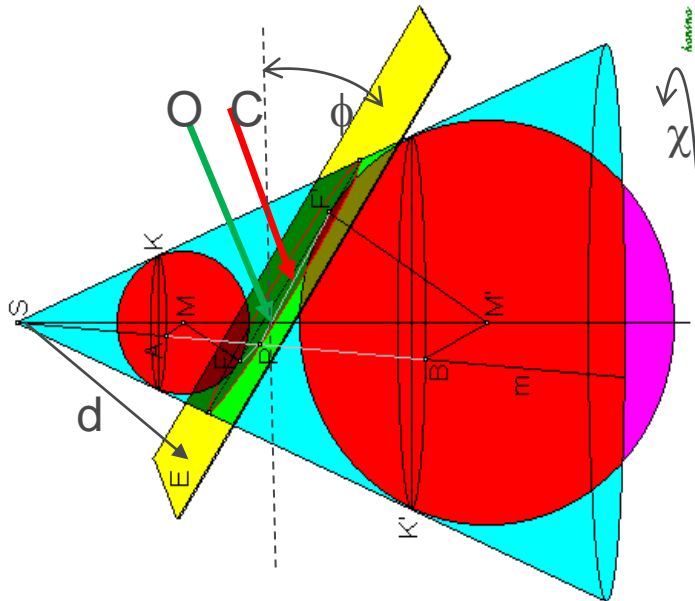
Dandelin sphere construction used in  
GSAS-II for image plate orientation  
calibration



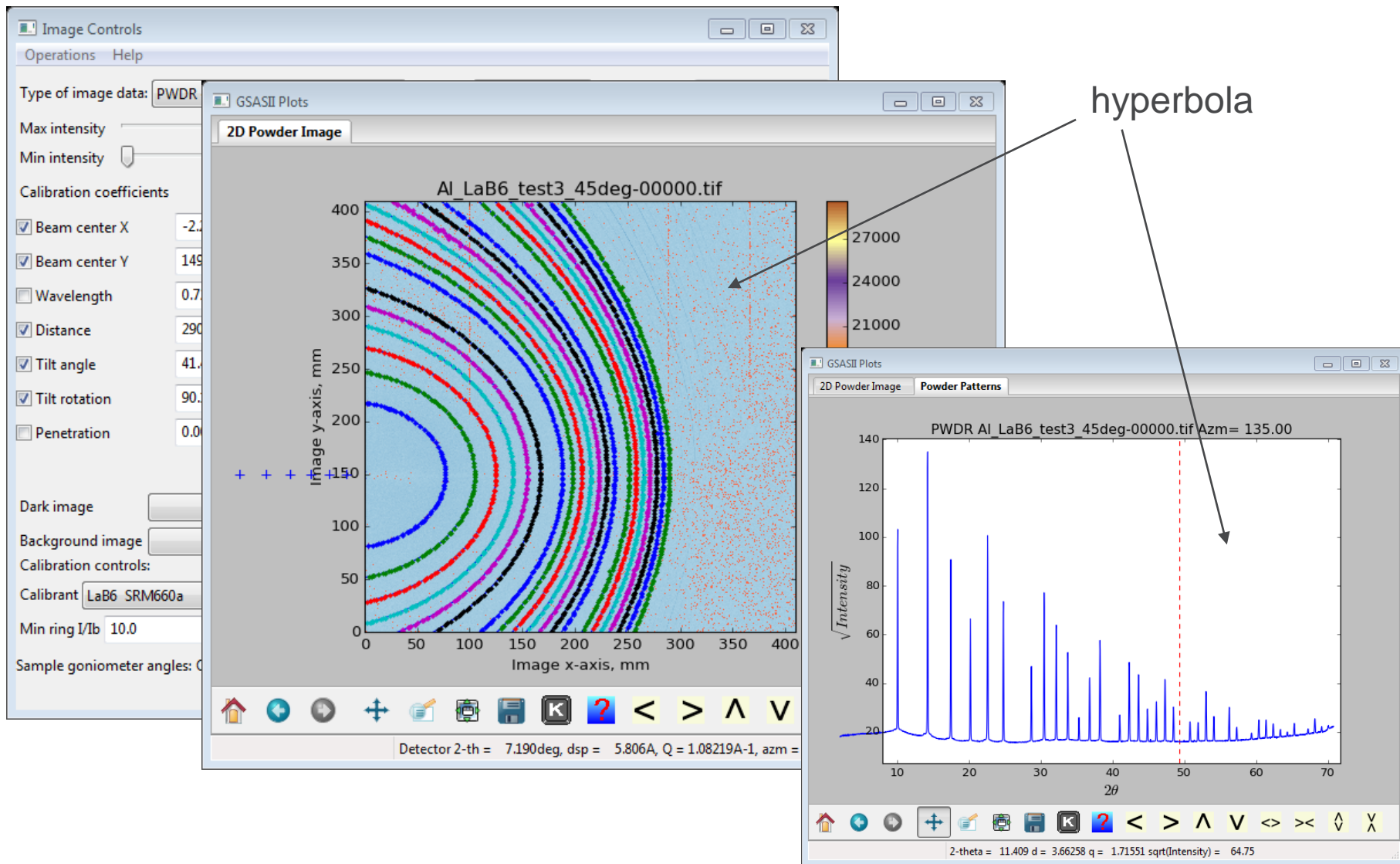
# 2D IMAGE CALIBRATION (REPLACE FIT2D)

## Correct calibration for tilt

- Dandelin sphere construction (1822)

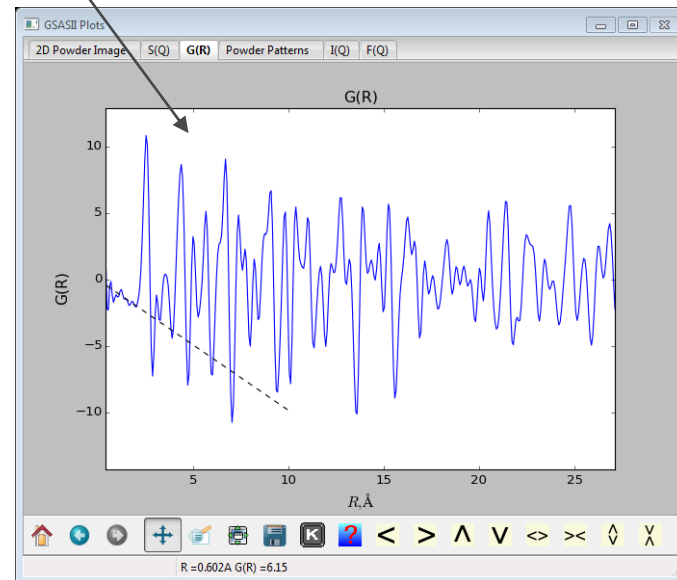
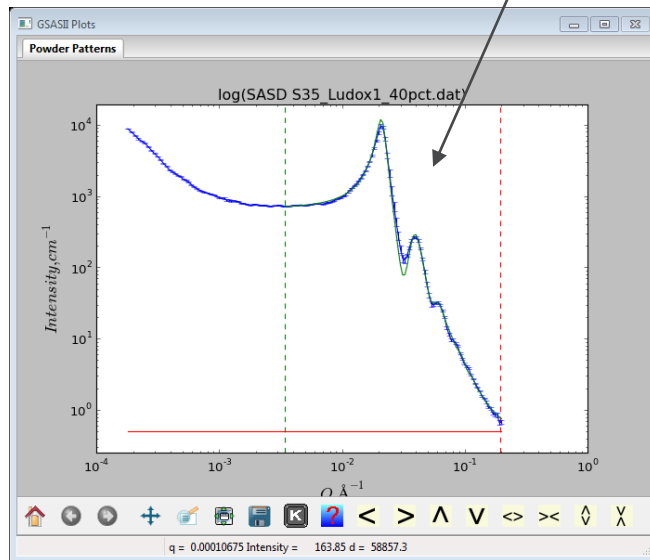
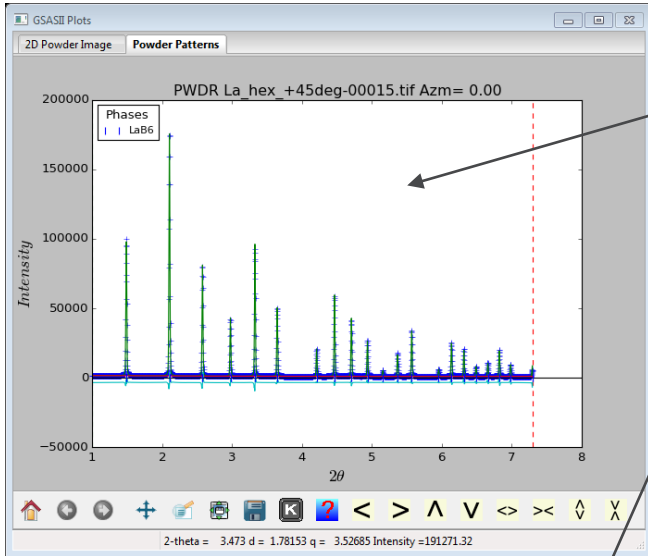


# OFFSET & TILTED DETECTORS – AN EXAMPLE

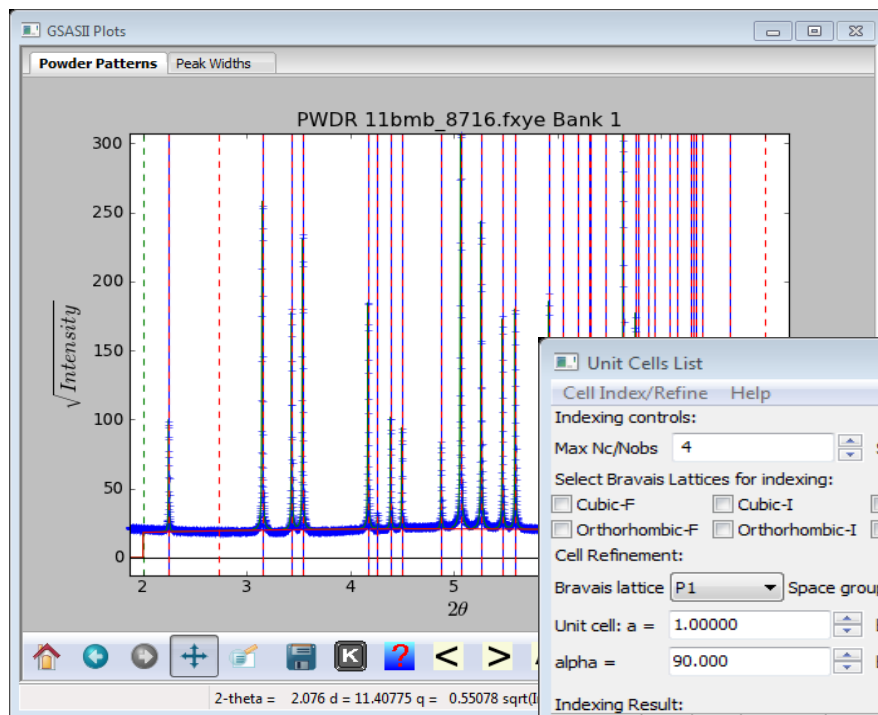


# AFTER IMAGE PROCESSING IN GSAS-II – STAY IN PROJECT FILE

- Powder diffraction analysis
- Small angle data analysis
- PDF calculation (needs some development & enthusiastic users)



# PEAK PICKING, FITTING & INDEXING



Indexing – Coehlo algorithm  
(same as topas)

Unit Cells List

Cell Index/Refine Help

Indexing controls:

Max Nc/Nobs 4 Start Volume 25  Use M20/(X20+1)?

Select Bravais Lattices for indexing:

Cubic-F  Cubic-I  Cubic-P  Trigonal-R  Trigonal/Hexagonal-P  Tetragonal-I  Tetragonal-P  
 Orthorhombic-F  Orthorhombic-I  Orthorhombic-C  Orthorhombic-P  Monoclinic-C  Monoclinic-P  Triclinic

Cell Refinement:

Bravais lattice P1 Space group P -1 Zero offset 0.0000  Refine?  Super lattice? Show hkl positions

Unit cell: a = 1.00000 b = 1.00000 c = 1.00000 Vol = 1.000  
 alpha = 90.000 beta = 90.000 gamma = 90.000

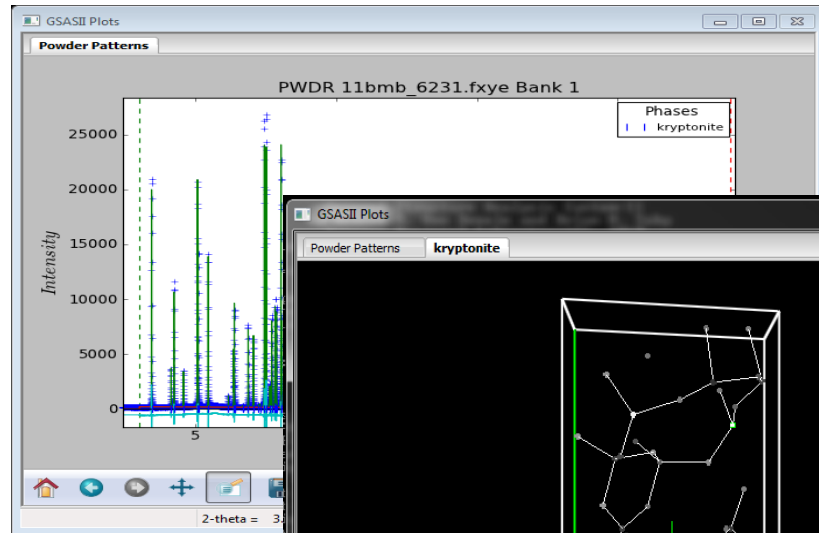
Indexing Result:

M20	X20	use	Bravais	a	b	c	alpha	beta	gamma	Volume	Keep
874.25	0	<input checked="" type="checkbox"/>	P2/m	7.71409	8.66281	10.80844	90.000	77.017	90.000	703.82	<input type="checkbox"/>
874.25	0	<input type="checkbox"/>	P2/m	7.71409	8.66281	10.80844	90.000	77.017	90.000	703.82	<input type="checkbox"/>
874.25	0	<input type="checkbox"/>	P2/m	7.71409	8.66281	10.80844	90.000	102.983	90.000	703.82	<input type="checkbox"/>
874.25	0	<input type="checkbox"/>	P2/m	7.71409	8.66281	10.80844	90.000	102.983	90.000	703.82	<input type="checkbox"/>
874.25	0	<input type="checkbox"/>	P2/m	7.71409	8.66281	10.80844	90.000	102.983	90.000	703.82	<input type="checkbox"/>
27.33	10	<input type="checkbox"/>	P2/m	7.71447	3.66989	10.80913	90.000	102.991	90.000	298.19	<input type="checkbox"/>
27.33	10	<input type="checkbox"/>	P2/m	7.71447	3.66989	10.80913	90.000	102.991	90.000	298.19	<input type="checkbox"/>
5.31	10	<input type="checkbox"/>	P2/m	7.71864	3.90344	10.80061	90.000	103.013	90.000	317.06	<input type="checkbox"/>

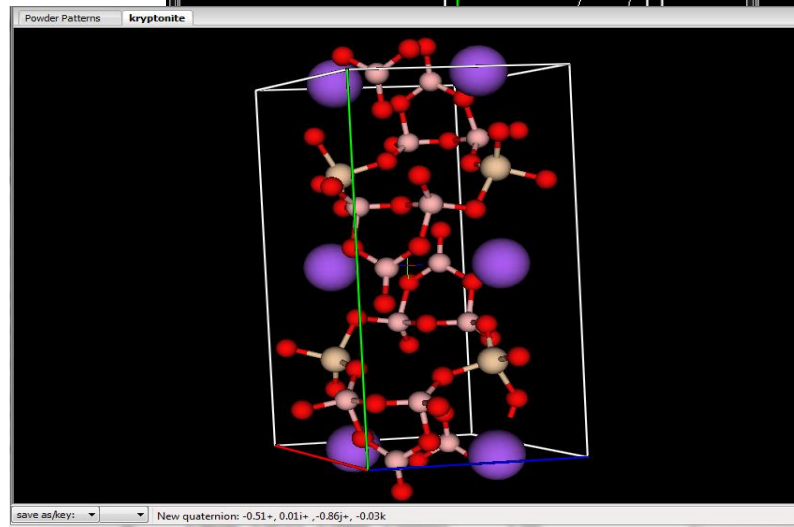
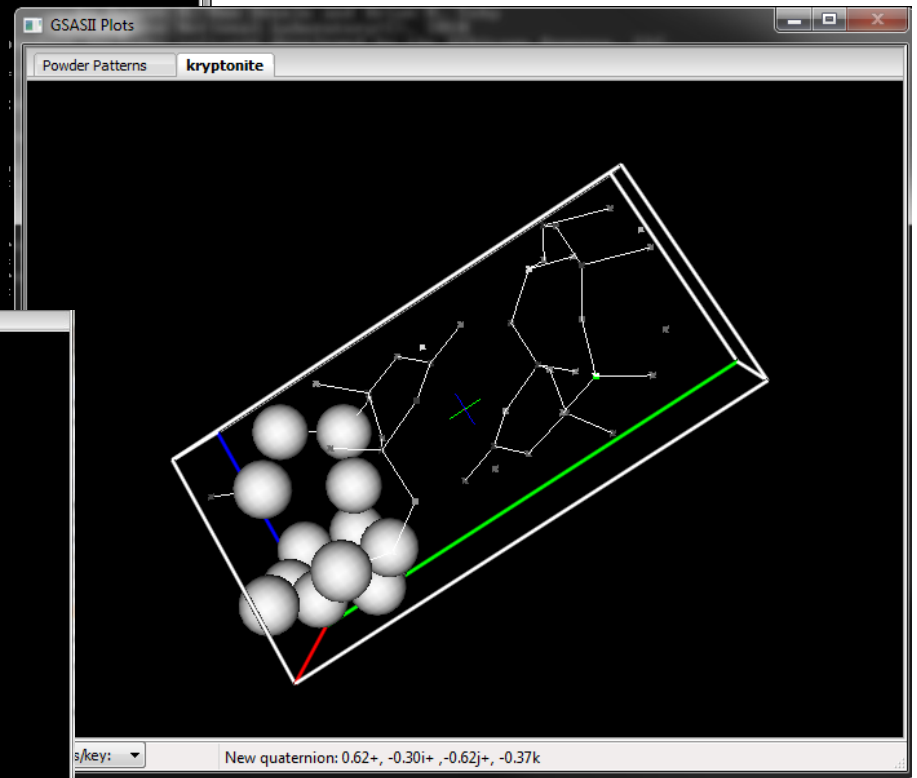
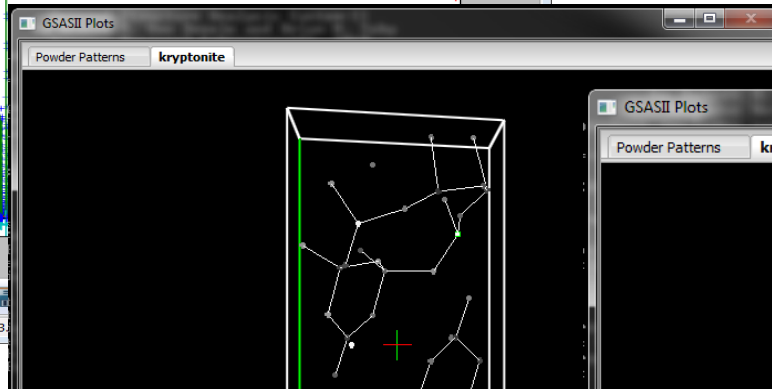


# STRUCTURE SOLUTION

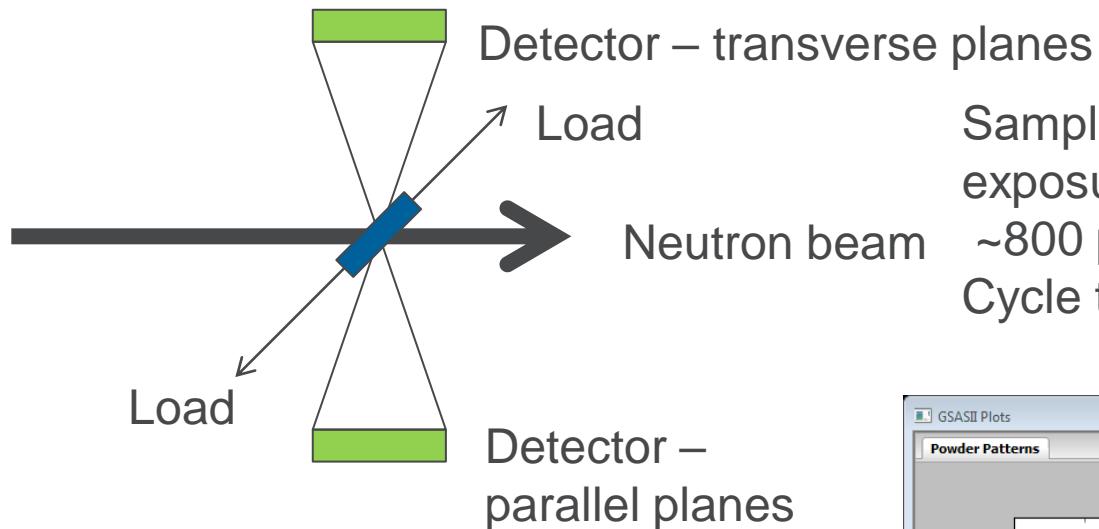
## Charge Flipping 3D & 4D



Powder – Pawley refinement  
Charge flip (NB: no symmetry)  
Atom selection & identification

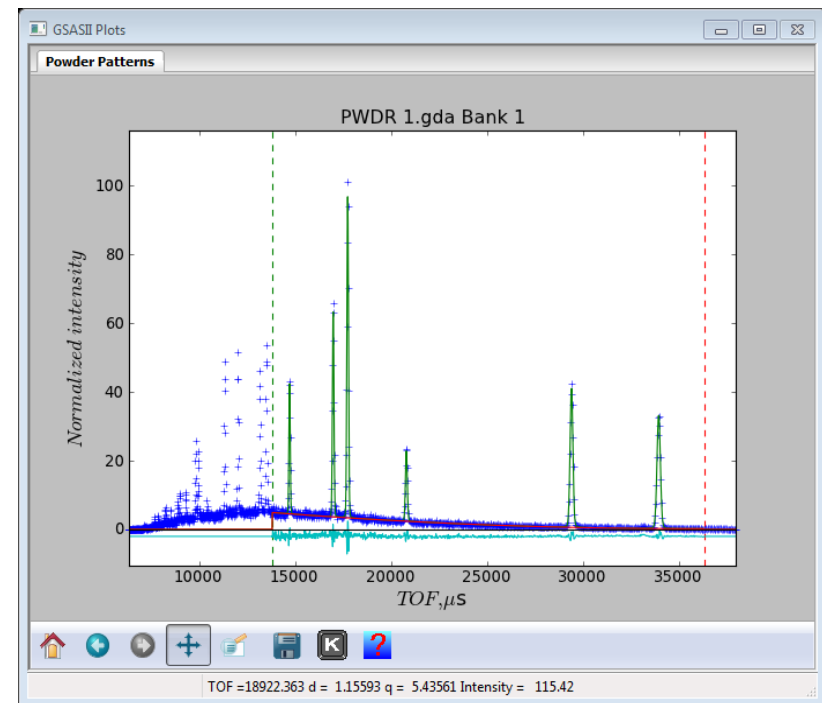


# SEQUENTIAL PEAK FITTING – OBSERVATION OF STRAIN – SNS VULCAN DIFFRACTOMETER

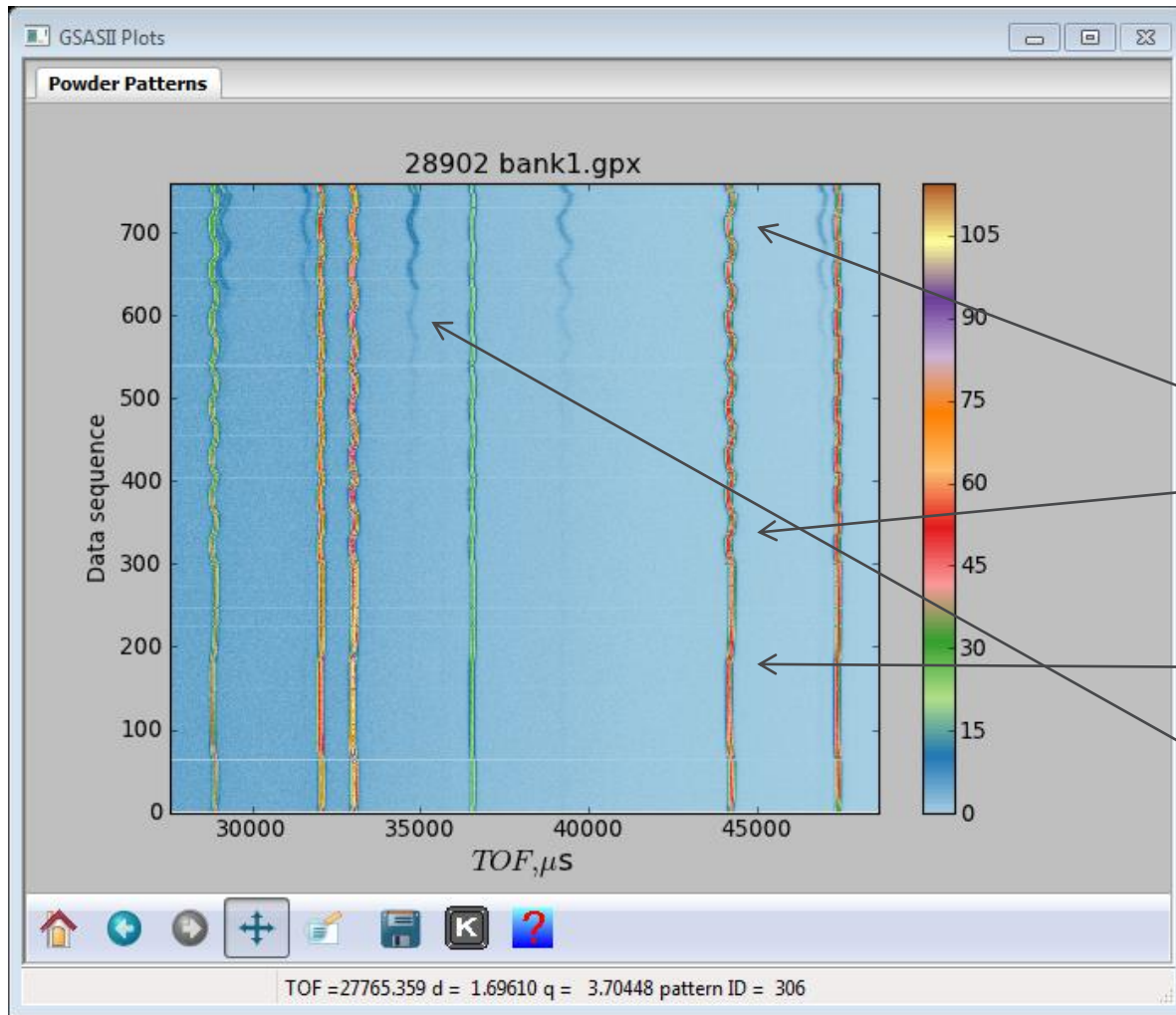


Sample: ¼” stainless steel rod, 1 min exposures over 13+ hrs  
~800 patterns  
Cycle tension – compression loading

One pattern –  
single peak fits: 6 lines  
Follow vs time & loading



# ~800 TOF POWDER PATTERNS IN GSAS-II



8-9 fast load cycles

1 Slow load cycle

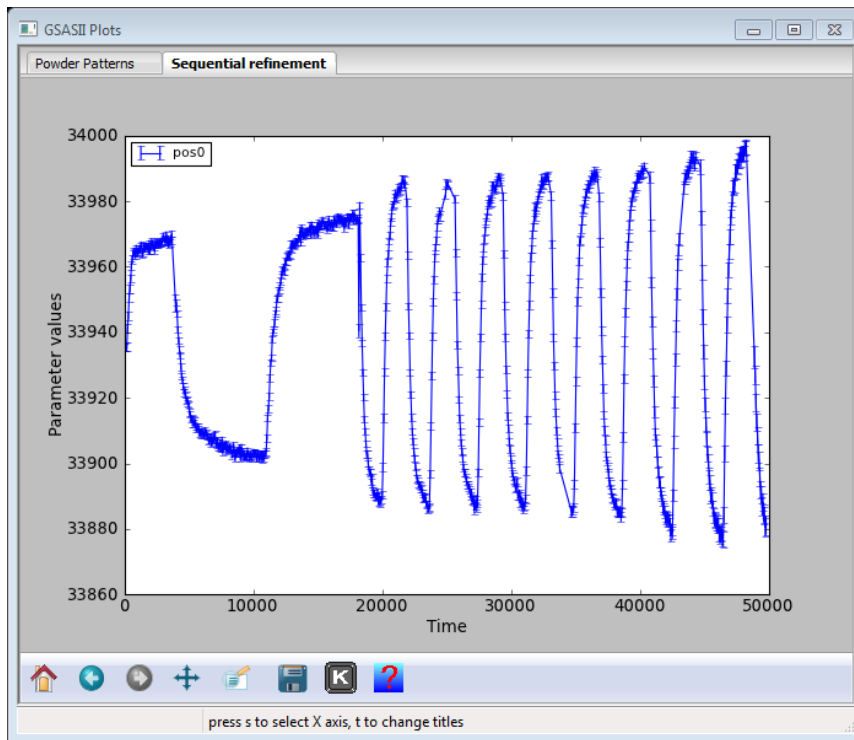
2<sup>nd</sup> phase appears  
(ferrite - BCC)

Main peaks –  
austenite - FCC

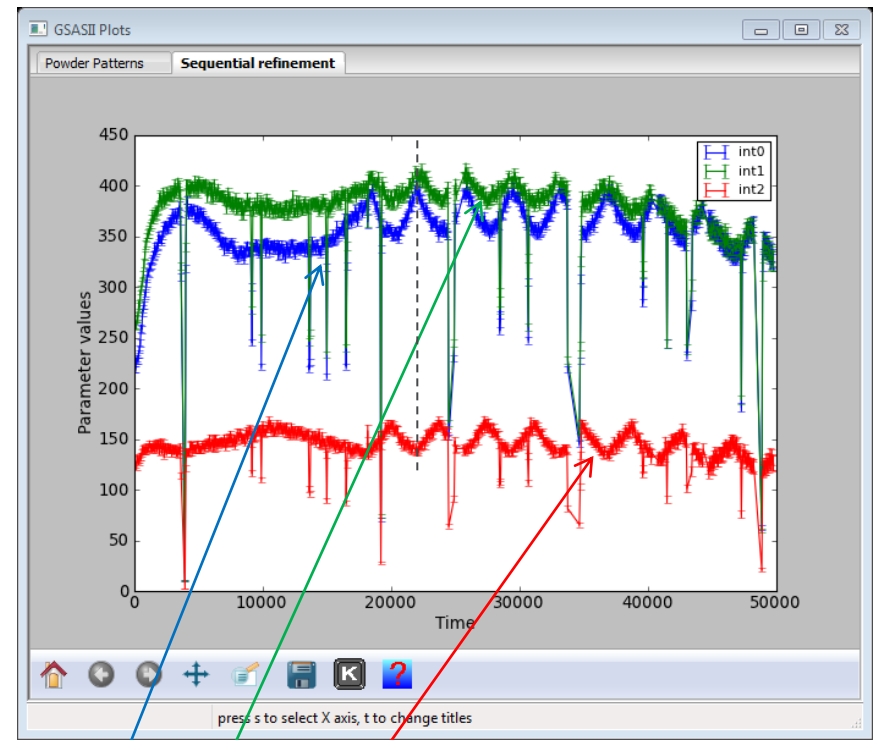
Do sequential peak fitting – 6 peaks + background  
~1.5 min to complete!

# SOME SEQUENTIAL PEAK FIT RESULTS

Lots to explore here – all within GSAS-II



Austenite 111 position



Austenite 111, 200 & 210 intensity

NB: note misalignment of 111, 200 vs 210

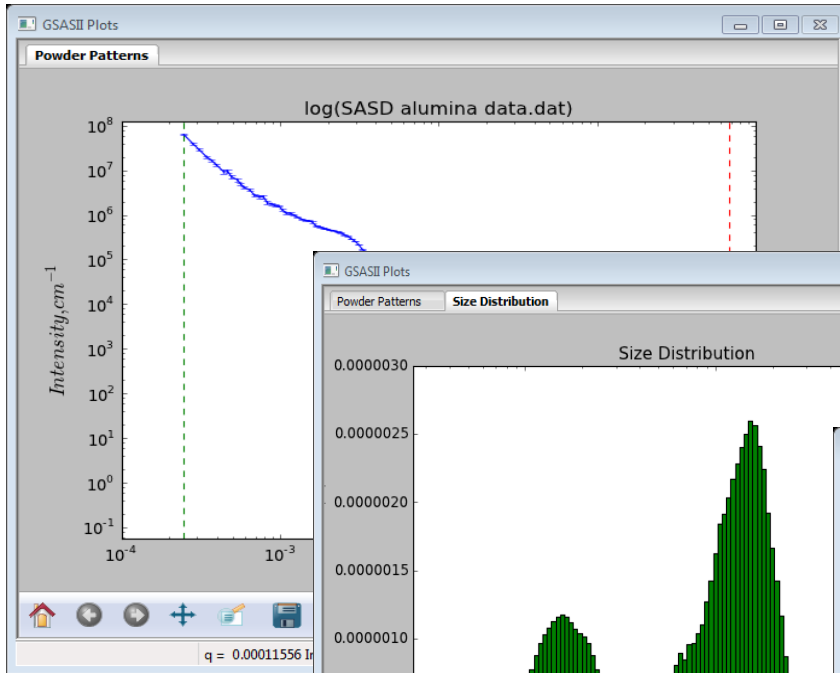
Crystallite reorientation under load

Spikes (down) – beam dropouts

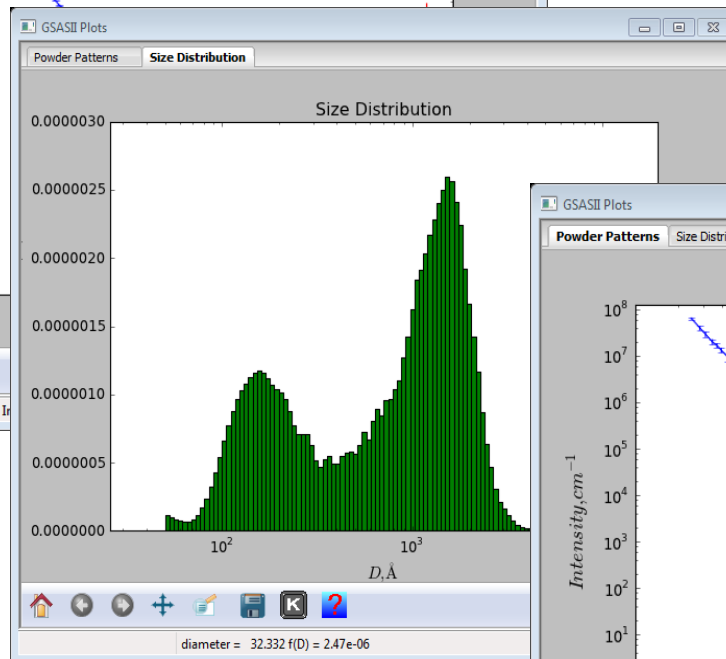


# SMALL ANGLE SCATTERING – SIZE DISTRIBUTION

Alumina polishing powder – bimodal particle size  $\sim 1600\text{\AA}$  &  $\sim 160\text{\AA}$  (not  $500\text{\AA}$  &  $1\mu\text{m}$  as advertised!)

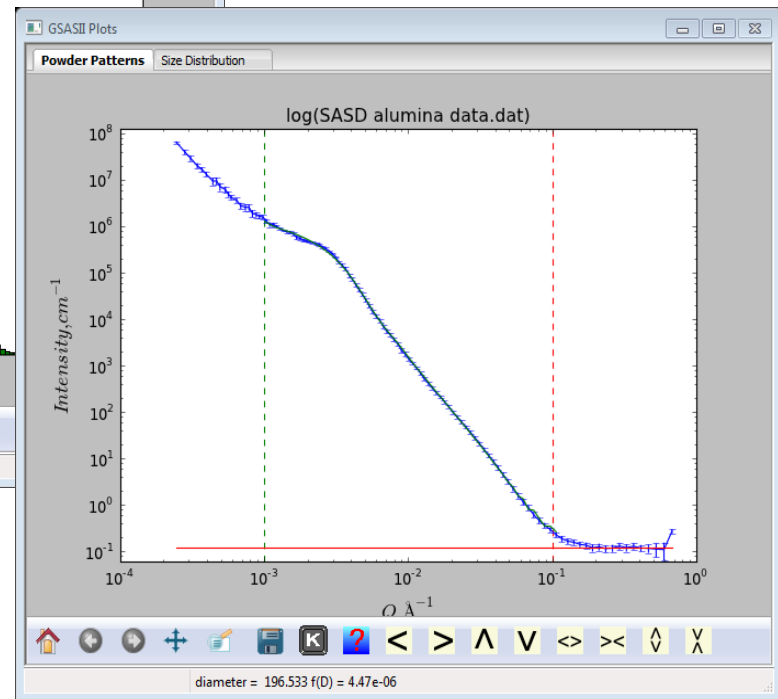


data



distribution

fit

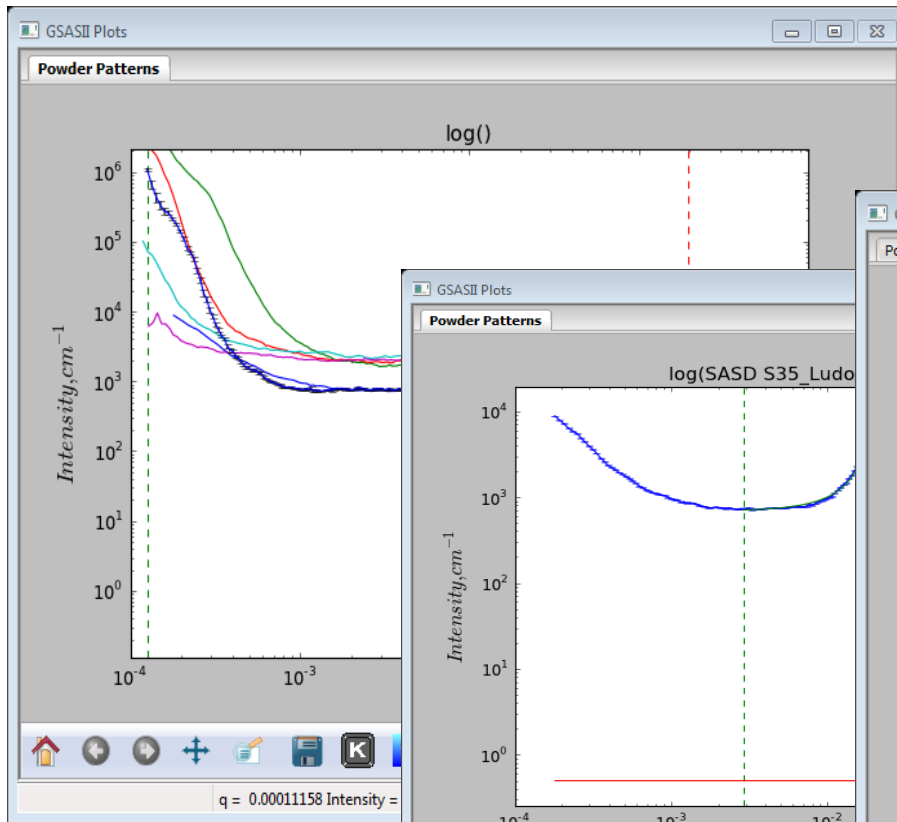


# SMALL ANGLE SCATTERING – SEQUENTIAL DATA ANALYSIS

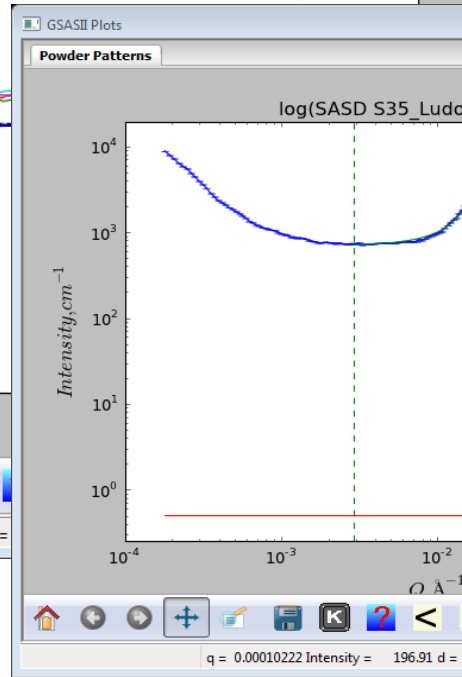
## Ludox colloidal silica from Aldrich – dilution range

Fit – hard sphere; log normal distribution, size, mean, vol. fr.

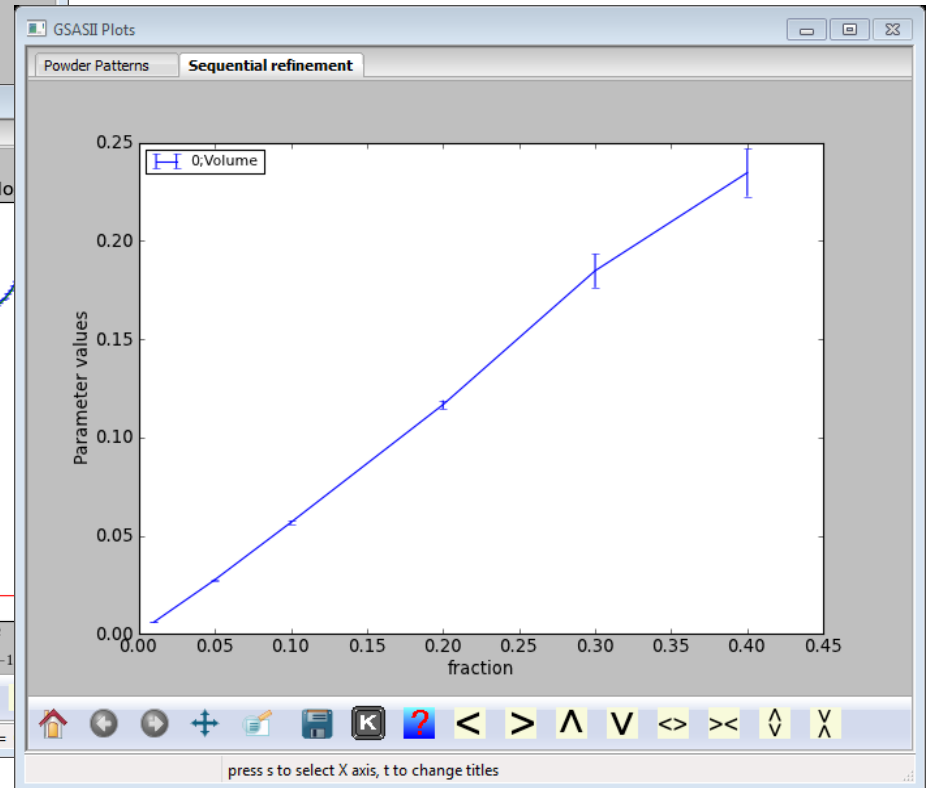
Sequential result – find slope/intercept



data

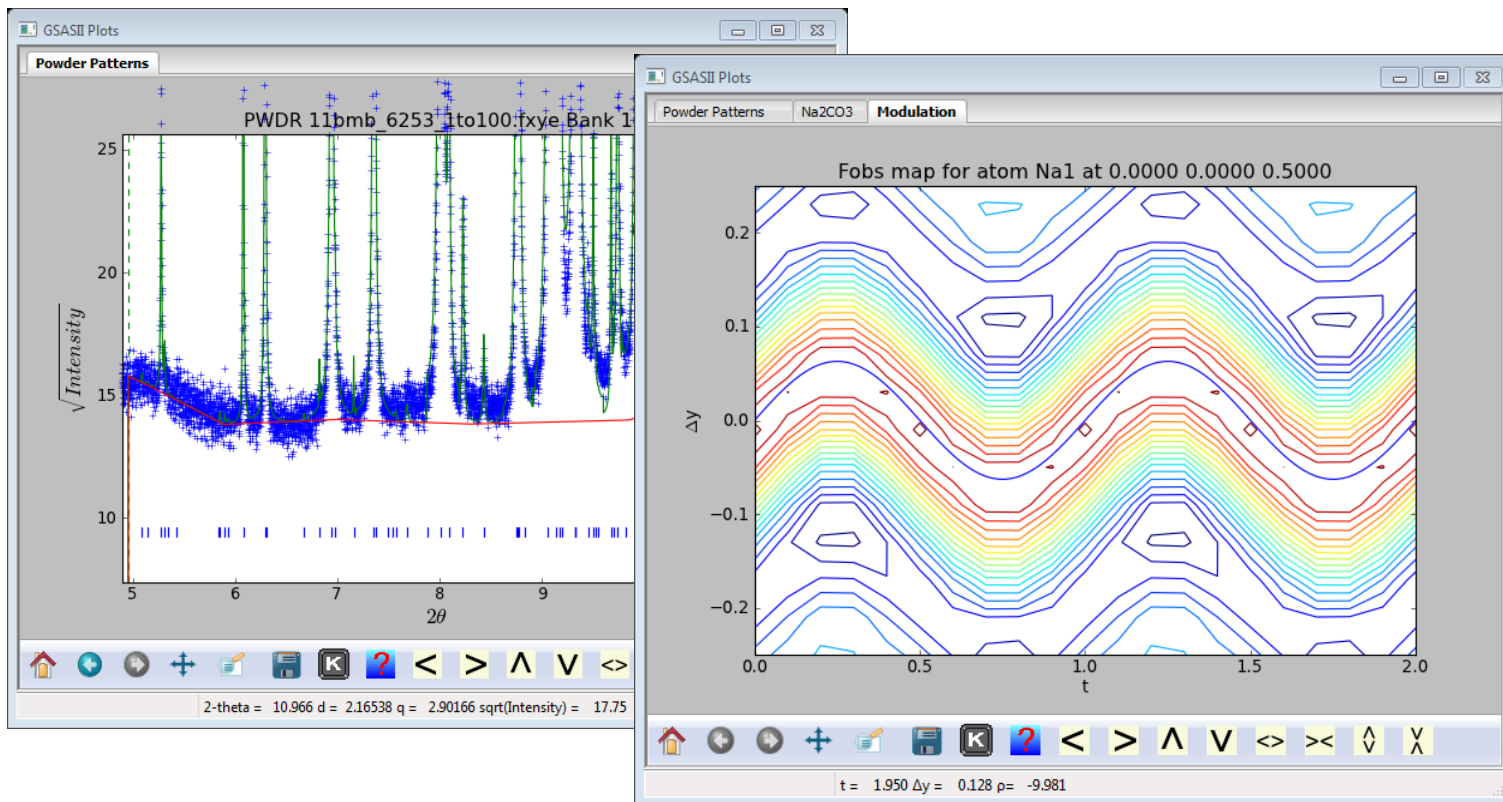


One fit



# INCOMMENSURATE STRUCTURES

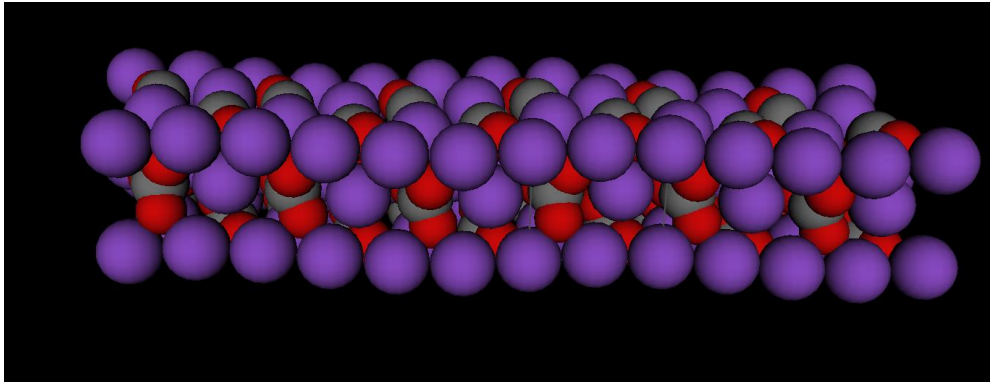
Simple materials – ex.  $\text{Na}_2\text{CO}_3$   $C 2/m (\alpha 0 \gamma) 0s$  super space group  
 $M = (0.1833, 0, 0.3191)$



# INCOMMENSURATE STRUCTURES IN GSAS-II

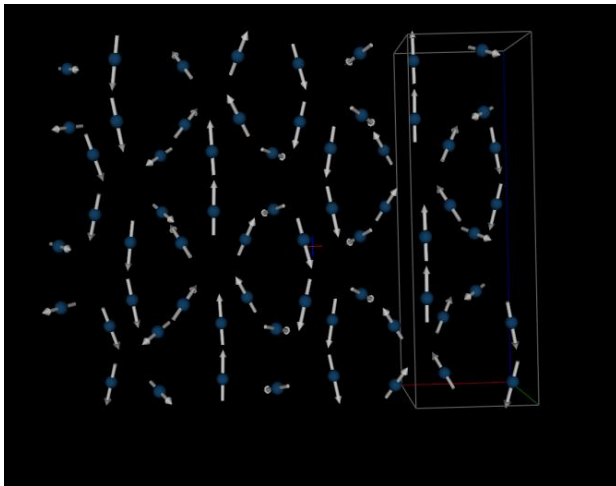
## Chemical & magnetic – object: full structure analysis

- Import incommensurate cif & mcif files & draw them – example:  $\text{Na}_2\text{CO}_3$



SC & powder data  
collected at APS  
Structure solution test  
both with GSAS-II

- Incommensurate magnetic mcif file & draw them – example:  $\beta\text{-Li}_2\text{IrO}_3$

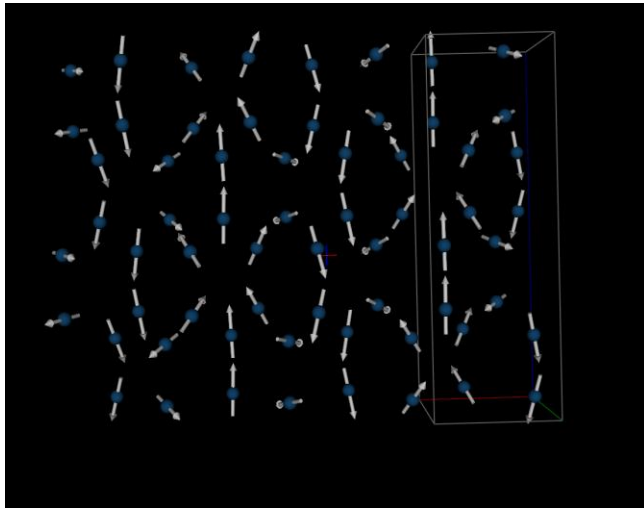


Determined from SC x-ray  
resonant diffraction (Diamond) &  
neutron powder data (ISIS)

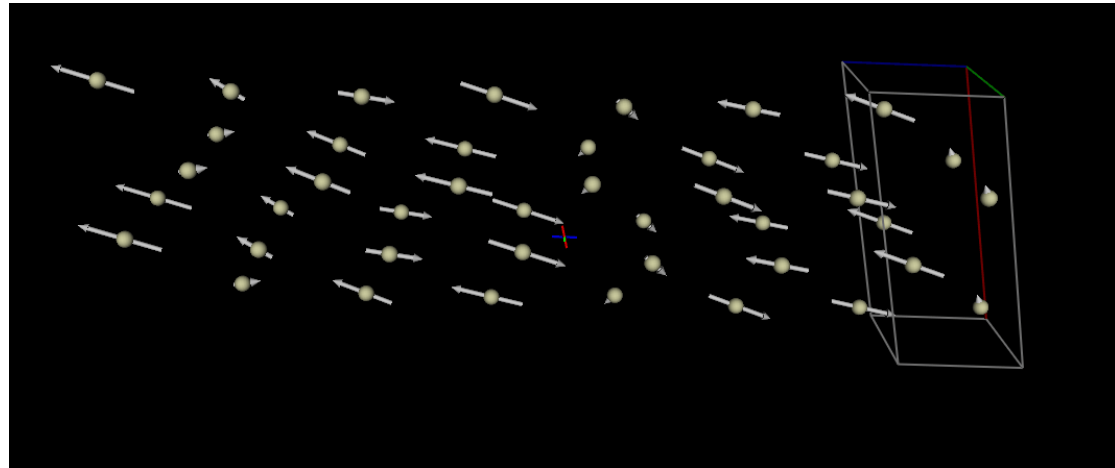


# MAGNETIC INCOMMENSURATE STRUCTURES

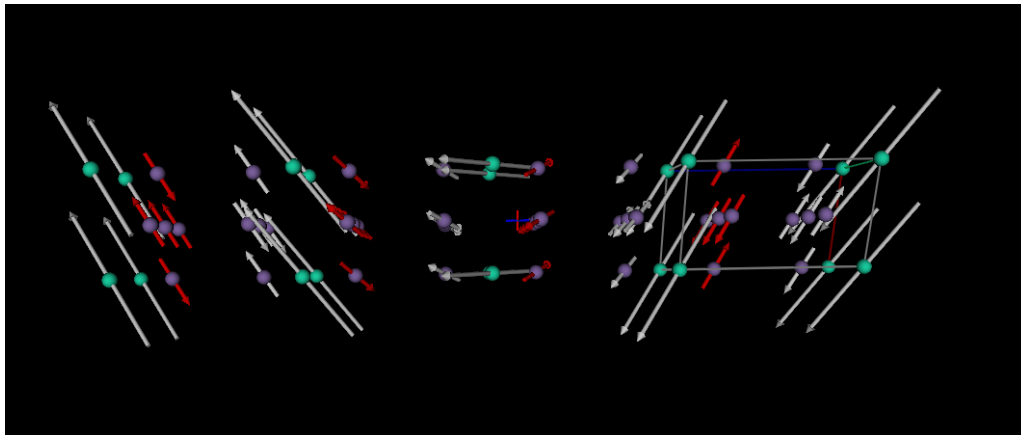
Some examples:



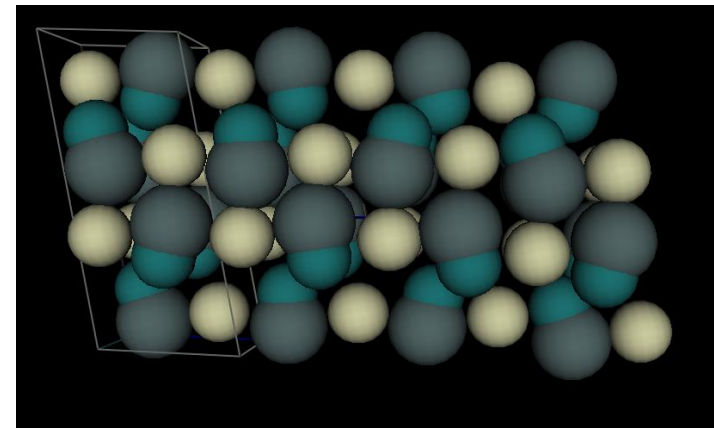
$\beta\text{-Li}_2\text{IrO}_3$



CeRuSn – Ce moment



$\text{DyMn}_6\text{Ge}_6$  – residual moment



CeRuSn –  
structure modulation

# GSAS-II CAPABILITIES

# GSAS-II CAPABILITIES & EXAMPLES

## Powder data

- 2D Images:
  - calibration & integration → 1D patterns
  - Direct strain fitting → 3 strain tensor elements\*
- 1D patterns
  - Peak picking & fitting\*
  - Indexing & space group selection → make new phase
- Multidata X-ray/neutron, CW/TOF → all combinations possible
- Structure solution
  - Stochastic – Monte Carlo/Simulated Annealing
  - Deterministic – Charge Flipping (3D & 4D)
- Structure Refinement – Rietveld Method\*
  - Pawley & LeBail refinement (needed for Structure Solution)
  - (3+1) Incommensurate structures
  - Constraints & restraints
  - Rigid bodies (2 kinds)
  - Texture Analysis → spherical harmonics\*
- Stacking Faults → DIFFaX simulations (NB: no refinement)
- Pair Distribution Function → data transformation (e.g. make PDF)
- PDF fitting – Large Box & Small Box modelling
- Cluster analysis – Powder data & PDF patterns (New)

\* Can use Sequential Analysis

# GSAS-II CAPABILITIES & EXAMPLES

## Single Crystal Data

- Multidata X-ray/neutron → all combinations possible
- Structure solution
  - Stochastic – Monte Carlo/Simulated Annealing
  - Deterministic – Charge Flipping (3D & 4D)
- Structure Refinement –Levenberg-Marquardt least squares\*
  - (3+1) Incommensurate structures
  - Constraints & restraints
  - Rigid bodies (2 kinds)
  - Merohedral & pseudomerohedral twinning
  - Extinction (Gaussian/Lorentzian Primary & Secondary I & II)
- Stacking Faults → DIFFaX simulations (NB: no refinement)

\* Can use Sequential Analysis



# GSAS-II CAPABILITIES & EXAMPLES

## Small Angle & Reflectometry Diffraction Data

- 2D Images:
  - calibration & integration → 1D patterns
- 1D Small Angle Data
  - Scaling to glassy carbon standard
  - Size Analysis
    - Maximum Entropy Analysis
    - Total Non-negative Least Squares
  - Model fitting – components\*
    - Particle shapes – e.g. spheres, disks, hollow spheres,...
    - Porod scattering
    - Bragg peaks
    - Protein shape determination
- 1D Reflectometry Data
  - Layer sequence definition & repeats
  - Surface “roughness”, i.e. mixing for all surfaces

\* Can use Sequential Analysis

# SEQUENTIAL DATA ANALYSIS

## Multiple data sets – no maximum number

- 2D Images:
  - Direct strain fitting → 3 strain tensor elements
- 1D patterns
  - Peak picking & fitting
  - Structure Refinement – Rietveld Method
  - Small angle data – model fitting
  - Reflectometry data – model fitting
- Results table
  - Parameter plotting vs experiment variable (e.g.. Temperature)
  - Parametric equation modeling & fitting

# GSAS-II TUTORIALS

# BEST WAY TO LEARN GSAS-II IS BY RUNNING SELECTED TUTORIALS

Most major sections of the program are demonstrated by tutorial examples – best for today is to pick one/two for now

## Basic GSAS-II tutorials

- Starting GSAS-II describes how the user interface works
- Fitting laboratory X-ray powder data for fluoroapatite
- CW Neutron Powder fit for Yttrium-Iron Garnet
- Combined X-ray/CW-neutron refinement of PbSO<sub>4</sub>
- Combined X-ray/TOF-neutron Rietveld refinement

# MORE ADVANCED TUTORIALS

## Parametric Rietveld fitting

- Sequential refinement of multiple datasets (prerequisite for next)
  - Parametric Fitting and Pseudo Variables for Sequential Fits

## Structure solution

- Fitting individual peaks & autoindexing (prerequisite for next two)
  - Charge Flipping structure solution for jadarite
  - Charge Flipping structure solution for sucrose
- Charge Flipping structure solution with Xray single crystal data
- Charge flipping with neutron TOF single crystal data
- Monte-Carlo simulated annealing structure determination



# MORE ADVANCED TUTORIALS (II)

## Stacking Fault Modeling

- Stacking fault simulations for diamond
- Stacking fault simulations for Keokuk kaolinite
- Stacking fault simulations for Georgia kaolinite

## Image Calibration/Integration

- Calibration of an area detector
- Integration of area detector data
- Calibration of a Neutron TOF diffractometer

# MORE ADVANCED TUTORIALS (III)

## Small-Angle Scattering

- Small angle x-ray data size distribution (alumina powder)
- Fitting small angle x-ray data (alumina powder)
- Image Processing of small angle x-ray data
- Sequential refinement with small angle scattering data

## Other

- Texture analysis of 2D data
- Rietveld Refinement detail:
  - Fitting the Starting Background using Fixed Points
- Merohedral twin refinements
- Single crystal refinement from TOF data
- Scripting a GSAS-II Refinement from Python
- Strain fitting of 2D data