3D Visualization of XRD³ Texture Data as a Routine Research Tool and an Intuitive Teaching Aid

MAX diffraction facility

MAX3D

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Introduction

- XRD³ texture analysis is a non-destructive characterization tool which provides information on crystal orientation typically interpreted with pole figures
- Modern advances in instrumentation allow for rapid XRD³ texture data collection with wide reciprocal space (RS) coverage
- More RS coverage, more information. We need a way to look at the full diffraction pattern: MAX3D

Why Visualize in 3D?

- Provides a comprehensive examination of the data
- Reveals features beyond the scope of typical pole figures such as diffuse scattering, secondary phase behavior, etc.
- Complete data sets only take a few minutes to load

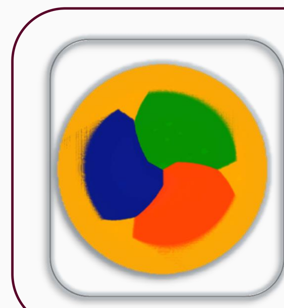
3D Visualization Software: MAX3D

- McMaster Analytical X-ray (**MAX**) Diffraction Facility **3D** visualization of area detector diffraction scans
- Create N³ voxels of average measured intensity and create a single 3D object representing the total measured diffraction pattern
- Object may include Bragg scattering, amorphous or diffuse scattering, hot detector pixels, beam artifacts, etc. – raw data.
- Adjustable 'transfer function' to set which intensities are transparent, translucent, or opaque.
- Inputting an orientation matrix can allow you to identify the position of diffraction features in terms of fractional HKL and relate crystal coordinates to sample coordinates
- Can isolate a 2θ range to view 3D pole figures and export pole figures for further analysis
  - for example: Orientation Distribution Function (ODF) calculation
- Integration of 3D to 1D (2θ vs. Intensity plot)- can be output to identify phases and peak widths

A sample area detector frame (left) and full data set 3D object in MAX3D(right) of InSb nanowires grown on Si (100) substrate. Sharp substrate peaks, broad oriented nanowire peaks, and polycrystalline diffraction shells are all observed simultaneously.

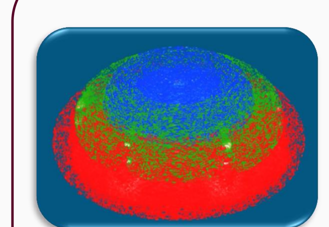
XRD³ Data Collection Strategy and RS Coverage

A combination of omega (ω), phi (ϕ), and psi (ψ) scans can be performed in order to obtain the appropriate reciprocal space (RS) coverage.



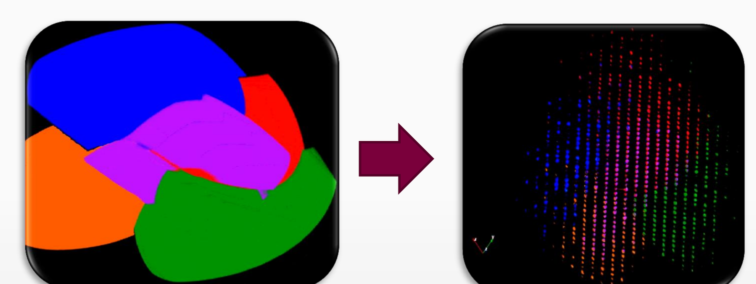
Scans:
3 ω (blue, green, red)
+ 1 ϕ (orange)

- Scans shown with opaque background



Scans:
3 ϕ (red, green, blue)

- Scans shown with semi-transparent background



Scans:
4 ω (blue, purple, red, green)
+ 1 ϕ (orange)

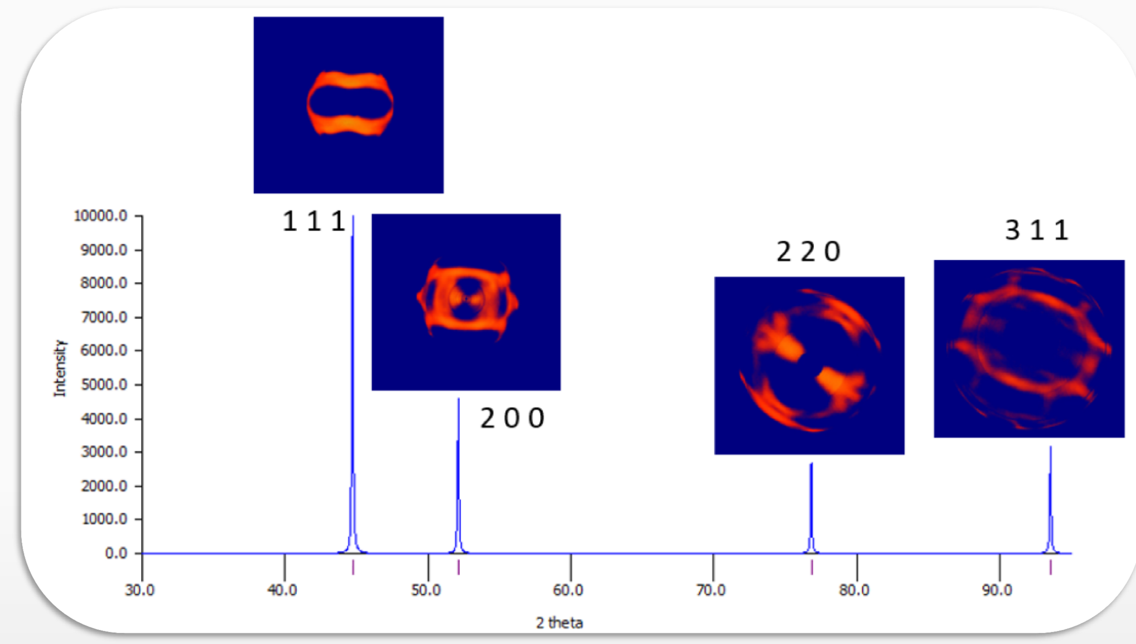
- Scans shown with opaque (left) and transparent (right) backgrounds of a single crystal data collection

Example 1- 3D Pole Figures

Pole figures can be viewed more intuitively, without stereographic projection.

Video Inside

- Reciprocal space mapping on a Ni Foil filter was performed to obtain 4 experimental pole figures
- Individual pole figures are extracted from the 3D object for viewing by restricting visible 2θ range



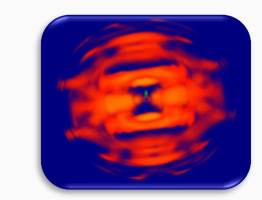


Figure: View of full dataset in MAX3D

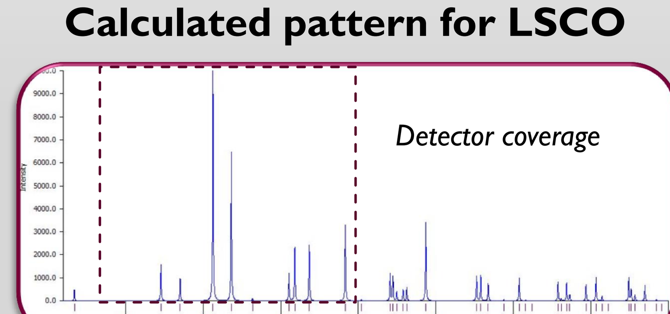
Background corrected pole figures generated in MAX3D can be exported for further analysis.

Example 2 – Orientation Matrix

A large single crystal sample of $La_{1.83}Sr_{0.17}CuO_4$, LSCO, (~3mm diameter) was measured to determine the crystal orientation with respect to the square base for magnetic measurements.

Sample

Calculated pattern for LSCO

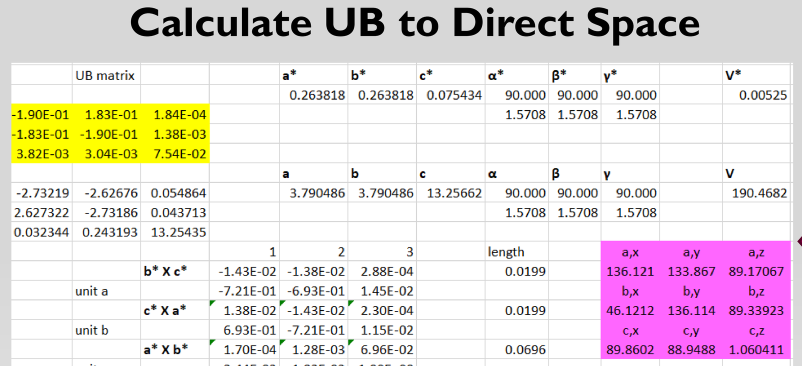


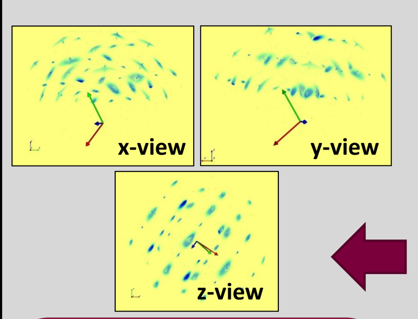
Data Collection:

- Cu Source (1.54056Å)
- 8cm detector distance (~ 42° 2θ coverage)
- One ϕ scan- 30°2θ, 15°ω (360° coverage, 1° slicing)

- **Determine unit cell** (Bruker Apex3 Single Crystal Software)
- **Obtain Orientation Matrix**

Calculate UB to Direct Space





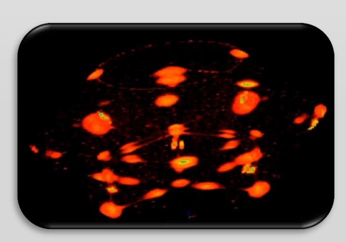
3D visualization to confirm that the sample is single crystal and to view reciprocal lattice coordinates (near centre) relative to sample coordinates (bottom left)

Example 3- Diffuse Scattering

An epitaxial thin film of CdTe on ZnTe, c-sapphire (0001) substrate, was measured:

- Co Source (1.79026Å), Bruker Vantec 500 Area Detector, 2 ϕ-scans, 1° slicing

Figure: Full data set loaded into MAX3D as a single 3D object. Diffraction from the sapphire substrate, ZnTe, and CdTe are visible simultaneously. Diffuse scattering is observed.



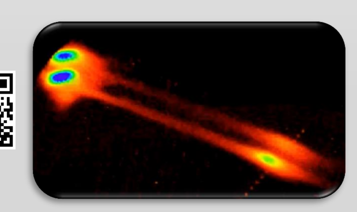
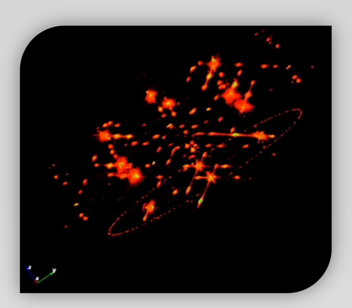


Figure: Volume of interest focused on a region of diffuse scattering . Diffuse lines along vectors between the (111) and (001) orientations

- CdTe adopts orientation, twinning, and strain behaviour of the underlying ZnTe film

A sample of GaAs nanowires grown on GaAs (100) substrate was measured:

Video Inside



- Two nanowire growth directions identified (approximately 35° off of the GaAs 100 substrate surface)
- Diffuse scattering along each nanowire direction
  - 2D ordered phase with stacking faults

Conclusion

3D visualization of texture data, obtained by XRD³ measurement with an area detector, is a comprehensive tool which allows users to quickly identify features which otherwise may be overlooked (i.e. diffuse scattering). Furthermore, it is a valuable teaching aid which provides users with an interactive learning platform for diffraction theory.

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References

J. Britten, W. Guan, MAX3D – a program for the visualization of reciprocal space, http://max3d.mcmaster.ca, *IUCr Commission on Crystallographic Computing Newsletter*, No. 8, November 2007, 96-108.

CCDC. 2016. "Mercury." http://www.ccdc.cam.ac.uk/mercury/.