# JADE Software Spotlight: Insights from the Penn State Materials Community

The X-ray Scattering Team at Penn State's Materials Characterization Laboratory

https://www.mri.psu.edu/materials-characterization-lab

Nichole Wonderling, Anthony Richardella, Maria Hilse, Gino Tambourine, Joshua Carlson, Sahani Iddawela and Alex Leffel

MCL | Materials Characterization Lab

PennState Materials Research Institute

# Our Team



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# Instrumentation



**Malvern Panalytical**
**X'Pert$^3$ MRD and MRD XL**

- 4-circle open Eulerian cradle
- Cu or Cr
- Hybrid monochromator – Kα1 only (line focus)
- X-ray lens (point focus)
- X-ray mirror (reflectivity/in-plane)
- Triple-axis monochromator (diffracted)
- Anton Paar DHS 900 domed hot stage (900° C)
- PIXcel 3D and Xe prop detectors
- Up to 420 mm radius goniometer
- Capable of full wafer defect mapping up to 200 mm



**Malvern Panalytical**
**Empyrean II – 3rd Generation**

- Microdiffraction
- 50, 100 and 300 µm collimators
- Multiple λ (Cu, Co, Mo, Ag)
- GaliPIX 2D and X'Celerator 1D detectors
- Motorized x-y-z, spinner, capillary, and 3-axis cradle stages
- Anton Paar HTK 2000N and HTK 1200N furnaces



**Malvern Panalytical**
**Empyrean III – 3rd Generation**

- Multicore Optics - iCore/dCore
- Cu radiation
- PIXcel 3D detector
- Laser height alignment with programmable z and spinner stages
- Reflection or transmission geometry
- 45 position sample changer



**Malvern Panalytical**
**Empyrean IV – 3rd Generation**

- Multicore Optics - iCore/dCore
- Cu or Mo radiation
- PIXcel 3D and 1Der detector
- Laser height alignment with programmable z and spinner stages
- Reflection or transmission geometry
- 45 position sample changer

PennState Materials Research Institute

# Instrumentation

## Multiwire Laue



- Real-time single crystal orientation
- Back-reflection
- Indexing options

## Malvern Panalytical Aeris




- Benchtop diffractometer
- 6 position autosampler

## Xenocs Xeuss 2.0 SAXS/WAXS



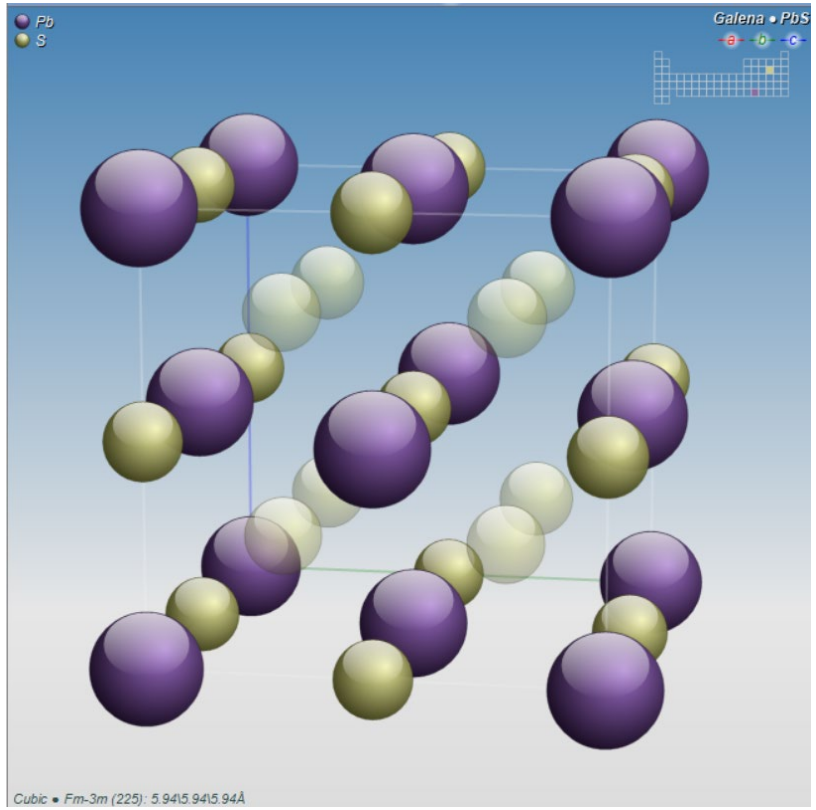

- Variable sample to detector distance for SAXS/WAXS
- Linkam HFSX 350 and CSS450 non ambient stages (-196 – 450° C)
- Transmission or GI-SAXS/WAXS for bulk, thin film, powder, liquid, slurry specimens in vacuum or air

# Project #1

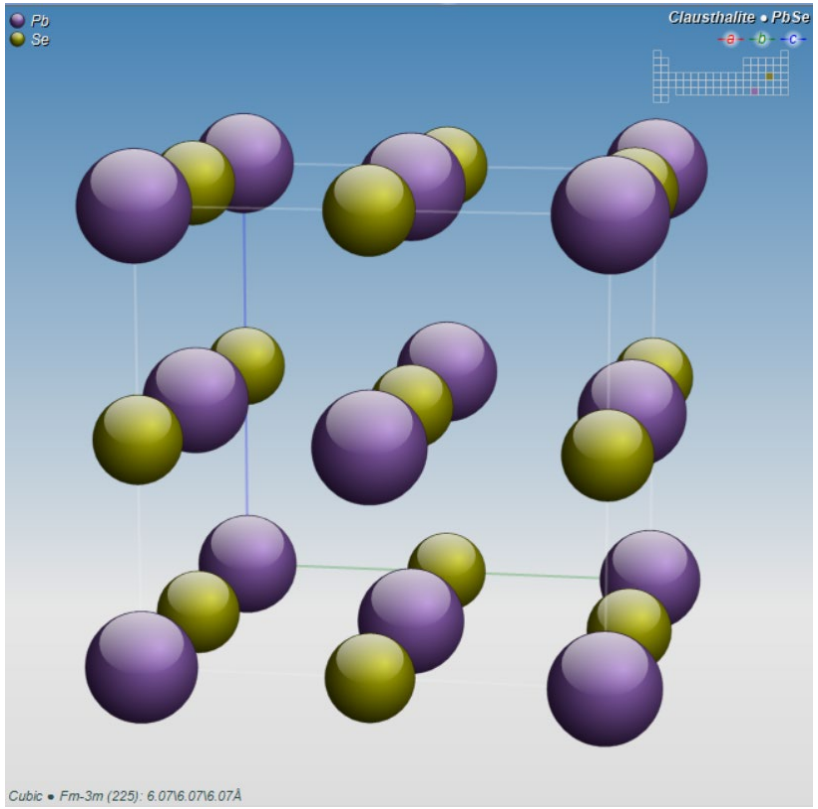
Find out the composition of S:Se in an unknown $PbS_xSe_{(1-x)}$ powder sample

# Substitutional solid solution

PbS PDF 04-025-8638
Cubic Fm-3m (225)




PbSe PDF 01-090-7762
Cubic Fm-3m (225)



# Vegard's Law

The lattice parameter of a **substitutional solid solution** varies linearly with the concentrations of its constituent components.

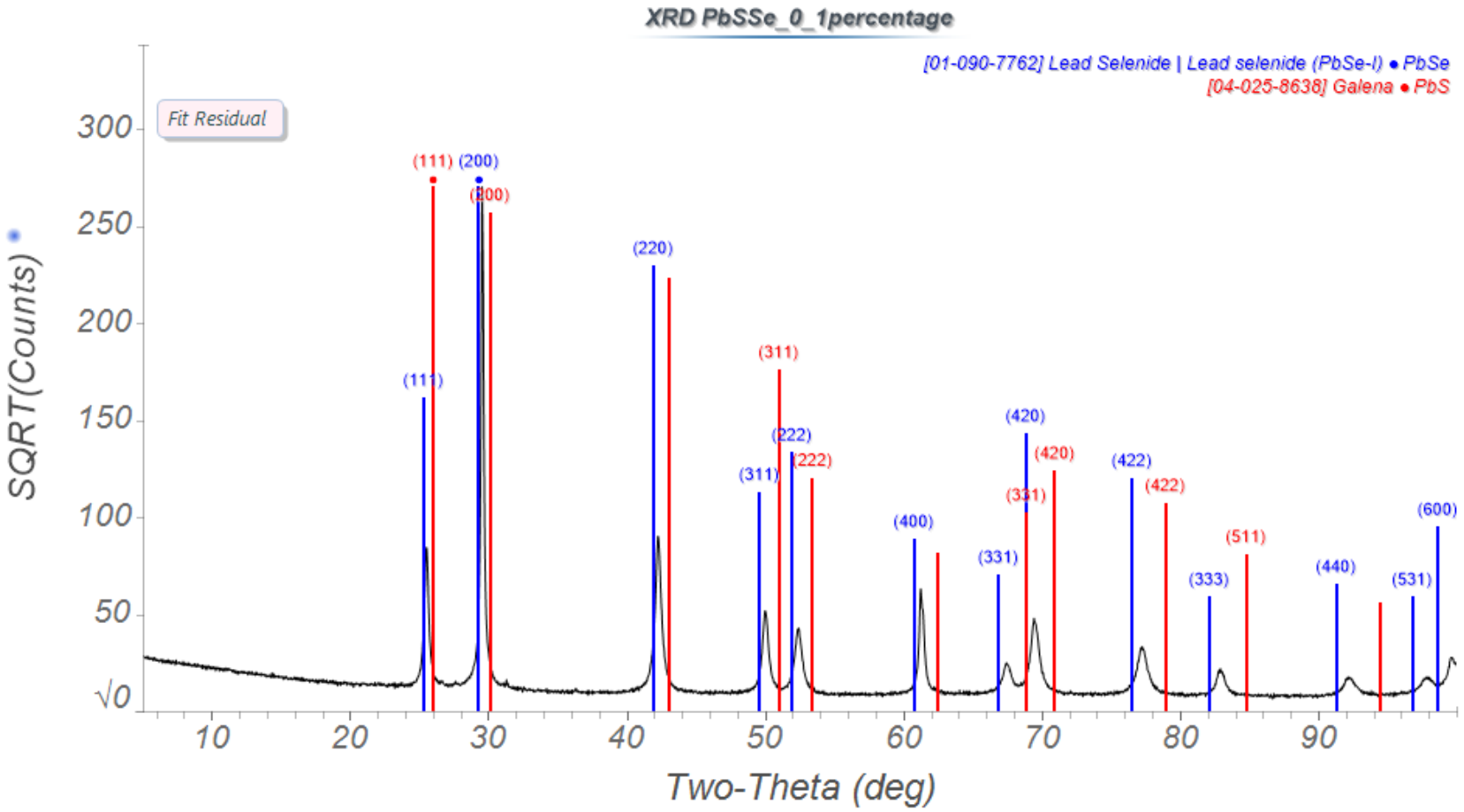
$$a(x) = x \cdot a_A + (1-x) \cdot a_B$$

$a(x)$ = lattice constant of the alloy at composition $x$

$a_A$, $a_B$ = lattice constants of pure components A and B

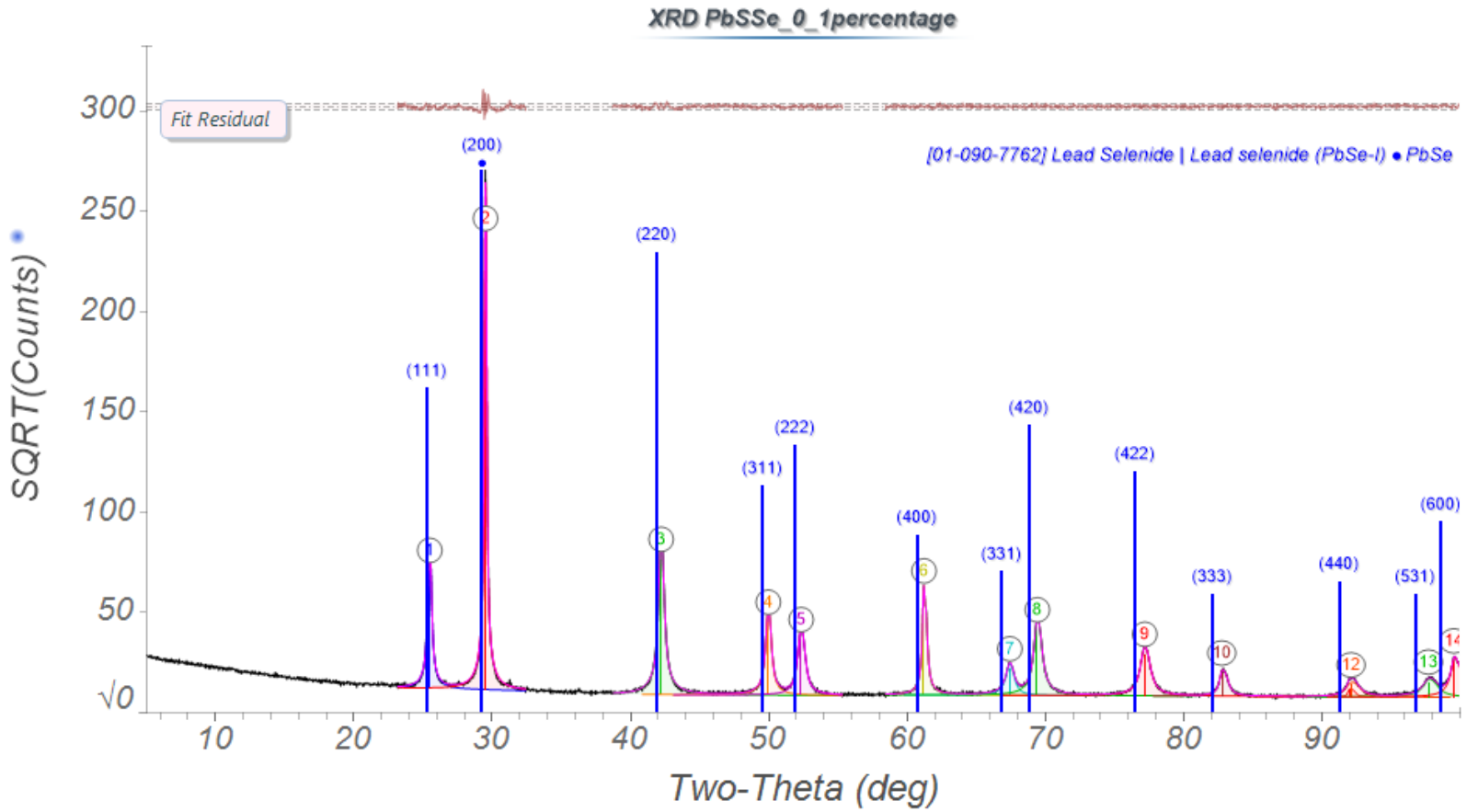
x = mole fraction of component A

# Applying Vegard's Law – End members




The known lattice parameters of the binary cubic Fm-3m (225) phases PbSe with a = 6.097 Å and PbS with a = 5.939 Å were used as interpolation points for Vegard's law. The reference XRD spectra are overlaid with the measured XRD scan in blue and red.

# Measure stoichiometry in $PbS_xSe_{1-x}$ - Profile Fit



- [C:\Users\nmw10\Downloads\XRD PbSSe_0_1percentage.xrdml]

| # | Angle(°) | d(Å) | H/σ | Area(α1) | Skew | FWHM(°) | XS(Å) | ( h k l) | Phase ID | Cell Type |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 25.4591 | 3.49579 | 160.6 | 1681.5 | 0.036 | 0.251 | 393 | ( 1 1 1) | PbSe ● Lead Selenide | Cubic (F) |
| 2 | 29.4924 | 3.02625 | 366.5 | 9727.0 | 0.067 | 0.135 | >1000 | ( 2 0 0) | PbSe ● Lead Selenide | Cubic (F) |
| 3 | 42.1929 | 2.14007 | 193.9 | 2586.5 | 0.006 | 0.328 | 294 | ( 2 2 0) | PbSe ● Lead Selenide | Cubic (F) |
| 4 | 49.9407 | 1.82470 | 99.6 | 908.3 | 0.060 | 0.329 | 301 | ( 3 1 1) | PbSe ● Lead Selenide | Cubic (F) |
| 5 | 52.3161 | 1.74730 | 100.1 | 759.5 | 0.018 | 0.445 | 214 | ( 2 2 2) | PbSe ● Lead Selenide | Cubic (F) |
| 6 | 61.1978 | 1.51328 | 96.5 | 997.1 | -0.096 | 0.189 | 674 | ( 4 0 0) | PbSe ● Lead Selenide | Cubic (F) |
| 7 | 67.4085 | 1.38815 | 42.9 | 295.1 | 0.221 | 0.488 | 209 | ( 3 3 1) | PbSe ● Lead Selenide | Cubic (F) |
| 8 | 69.3914 | 1.35325 | 103.0 | 1074.1 | 0.023 | 0.461 | 225 | ( 4 2 0) | PbSe ● Lead Selenide | Cubic (F) |
| 9 | 77.1468 | 1.23540 | 76.6 | 647.7 | 0.009 | 0.573 | 187 | ( 4 2 2) | PbSe ● Lead Selenide | Cubic (F) |
| 10 | 82.7877 | 1.16494 | 37.0 | 215.0 | -0.127 | 0.466 | 244 | ( 3 3 3) | PbSe ● Lead Selenide | Cubic (F) |
| 11 | 91.9968 | 1.07087 | 0.6 | 34.3 | -0.408 | 0.384 | 327 | ( 4 4 0) | PbSe ● Lead Selenide | Cubic (F) |
| 12 | 92.1321 | 1.06965 | 1.0 | 170.7 | -0.049 | 0.955 | 123 | ( 4 4 0) | PbSe ● Lead Selenide | Cubic (F) |
| 13 | 97.7333 | 1.02272 | 34.8 | 228.8 | 0.045 | 0.943 | 131 | ( 6 0 0) | PbSe ● Lead Selenide | Cubic (F) |
| 14 | 99.5722 | 1.00872 | 48.3 | 404.3 | 0.028 | 0.522 | 250 | ( 6 0 0) | PbSe ● Lead Selenide | Cubic (F) |

# Determination of $PbS_xSe_{1-x}$ lattice parameter (1)

**Eq 1**

$$n\lambda = 2d\ sin\theta$$

**Eq 2**

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

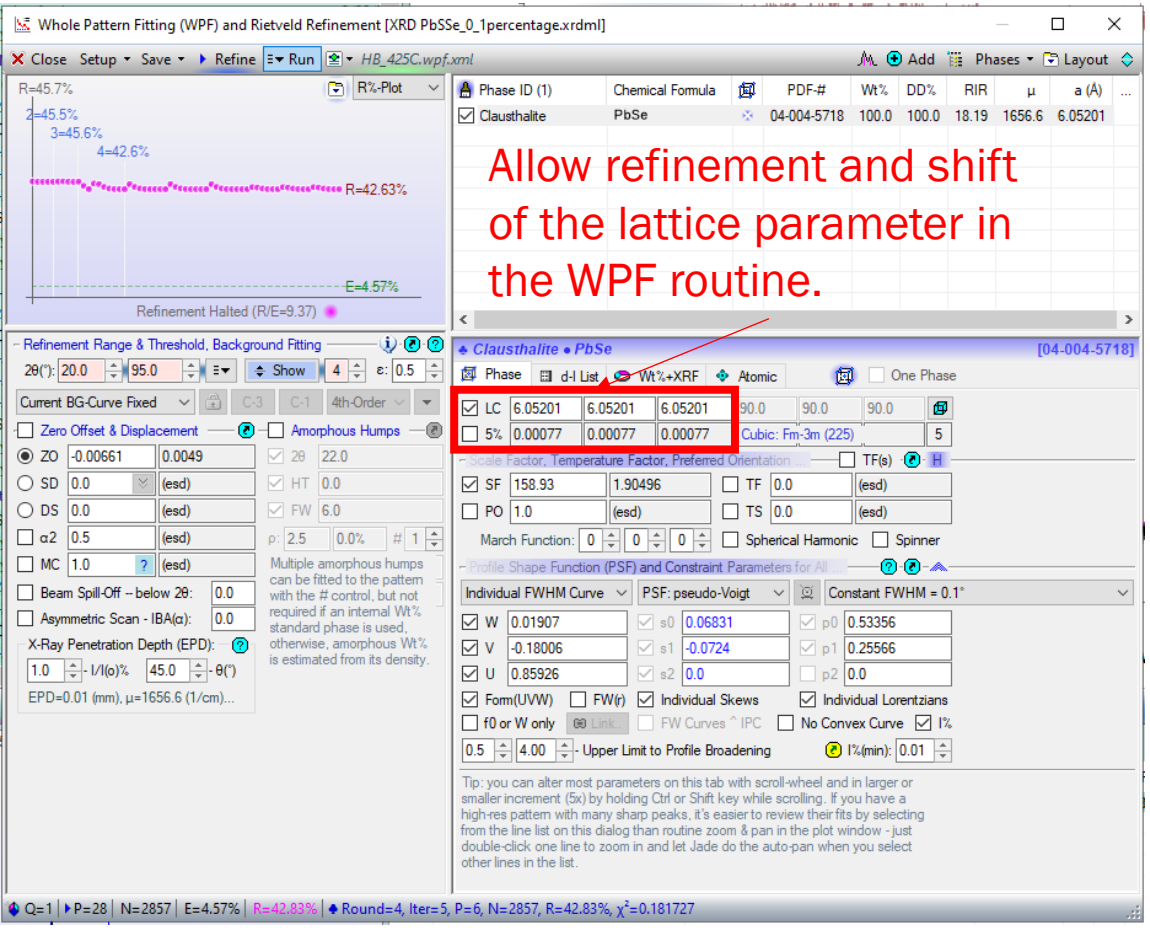
**Eq 1 +2**

$$a = \frac{\lambda\sqrt{h^2 + k^2 + l^2}}{2\ sin\theta}$$

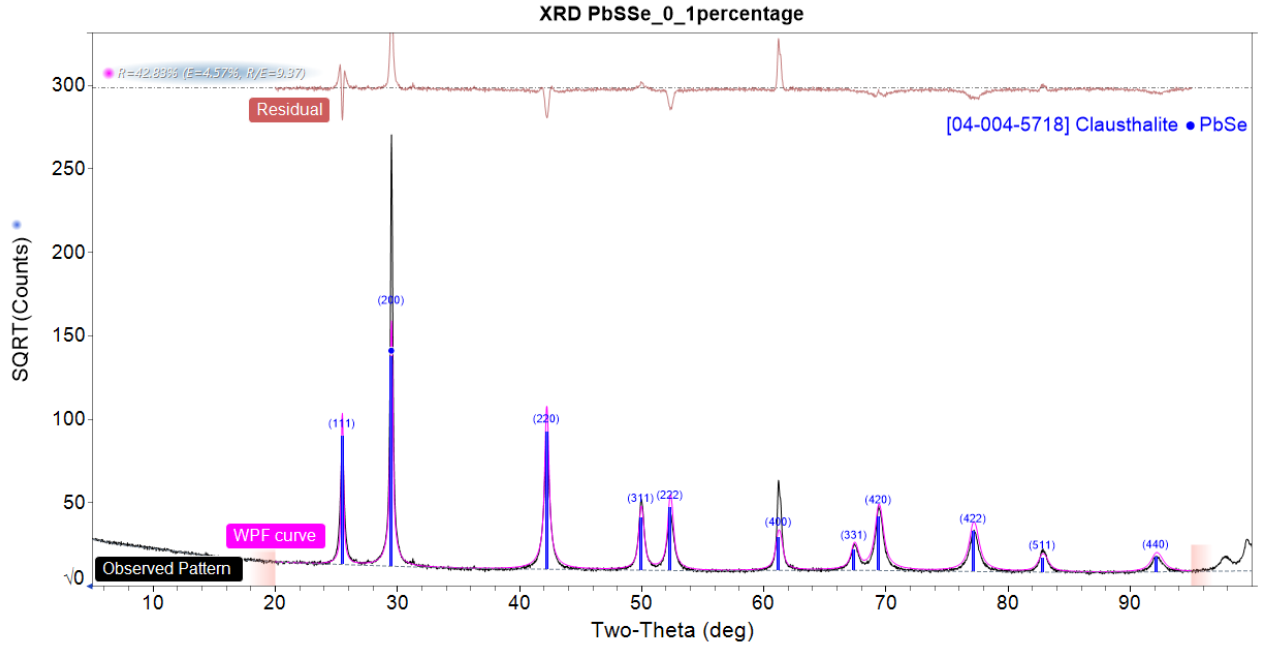
| Peak # | Angle(°) | h | k | l | Calculated lattice parameter [Å] |
|---|---|---|---|---|---|
| 1 | 25.461 | 1 | 1 | 1 | 6.054473846 |
| 2 | 29.4951 | 2 | 0 | 0 | 6.051989275 |
| 3 | 42.1966 | 2 | 2 | 0 | 6.052559579 |
| 4 | 49.9404 | 3 | 1 | 1 | 6.051905637 |
| 5 | 52.318 | 2 | 2 | 2 | 6.052651103 |
| 6 | 61.2075 | 4 | 0 | 0 | 6.052267957 |
| 7 | 67.3862 | 3 | 3 | 1 | 6.052619022 |
| 8 | 69.3907 | 4 | 2 | 0 | 6.05201054 |
| 9 | 77.1396 | 4 | 2 | 2 | 6.052726921 |
| 10 | 82.8201 | 3 | 3 | 3 | 6.051297572 |
| 11 | 92.093 | 4 | 4 | 0 | 6.052853171 |
| 12 | 97.6828 | 5 | 3 | 1 | 6.052871403 |
| 13 | 99.5751 | 6 | 0 | 0 | 6.052195585 |

Average lattice parameter (a) for the unknown sample = (6.0525 ± 0.0007) Å

# Determination of $PbS_xSe_{1-x}$ lattice parameter (2)




Alternatively, the sample's unknown lattice parameter was extracted from the XRD scan using the whole pattern fit refinement function in Jade starting from the manually shifted PbSe reference to match the peaks of the unknown composition.



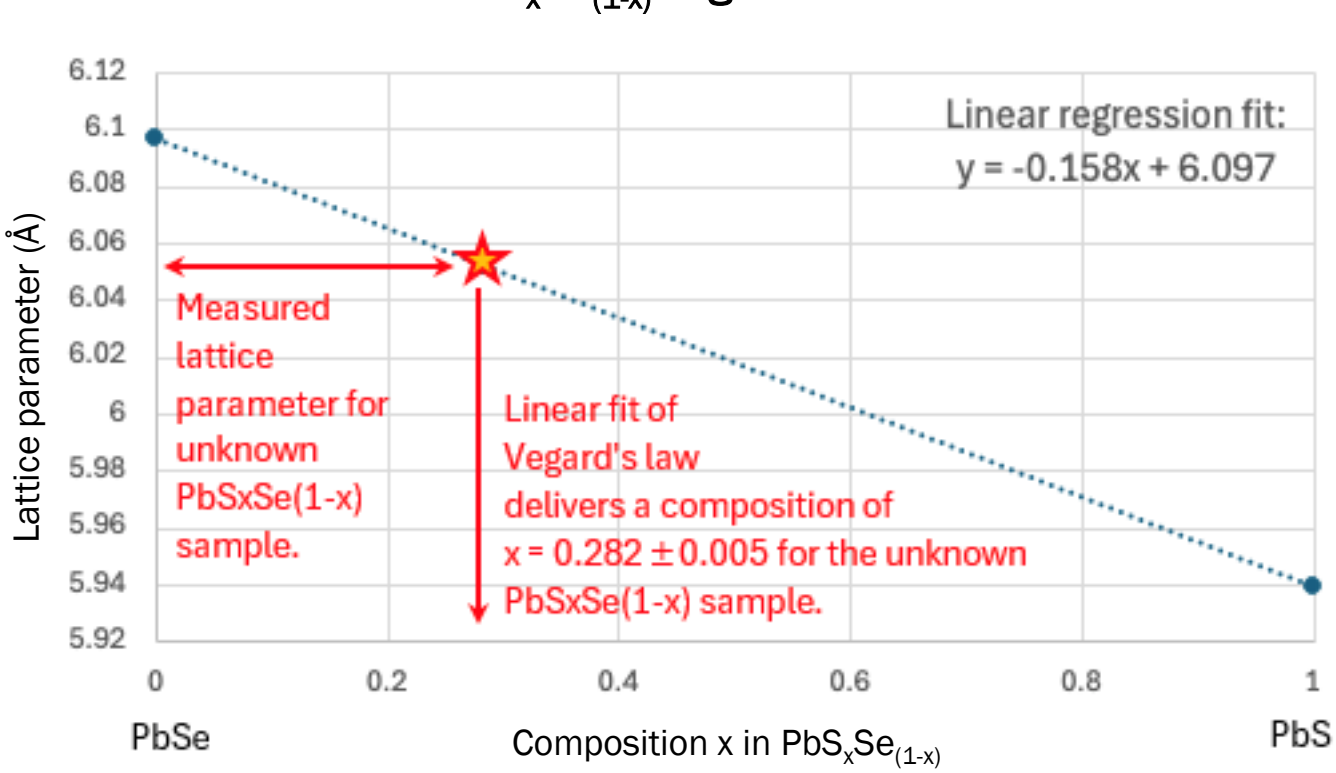

Average lattice parameter (a) for the unknown sample = 6.052 Å

# Vegard‘s law connects lattice parameter and stoichiometry

The linear regression between the two known binary reference phases links lattice parameter of the ternary compound to the stoichiometry.

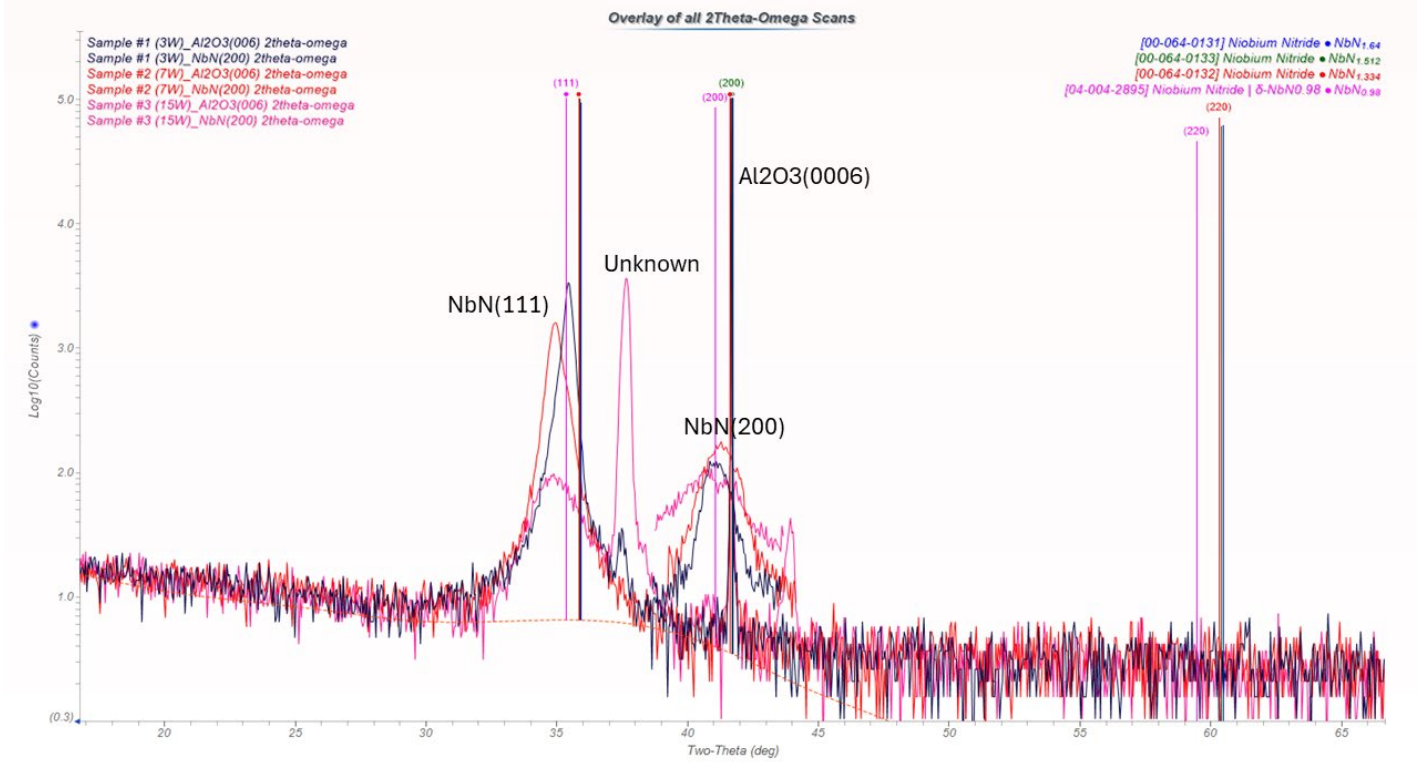
Solving the linear regression for the determined lattice parameter yields the S content in the unknown sample.

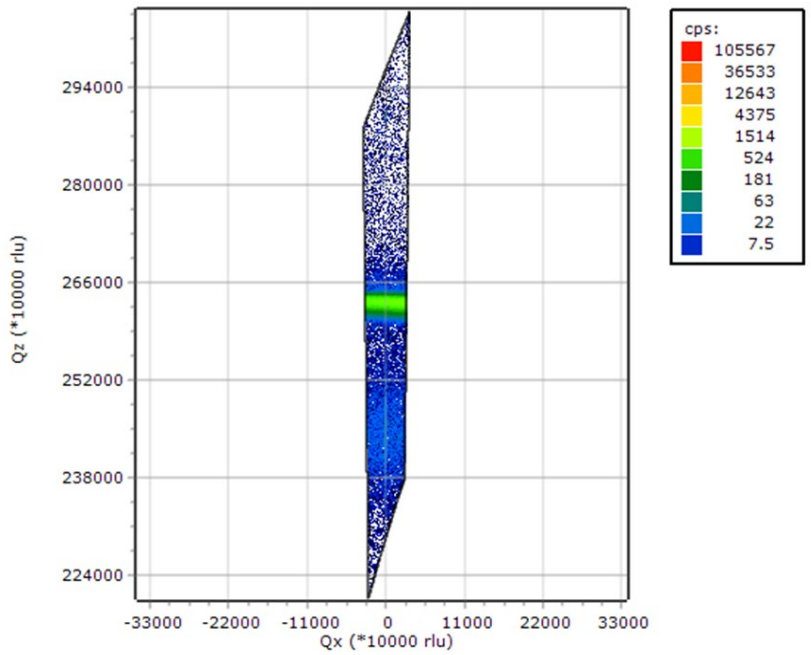
| Compound | Composition (x) | Lattice Parameter [Å] |
|---|---|---|
| PbS | 1 | 5.9390 |
| PbSe | 0 | 6.0970 |
| $PbS_xSe_{(1-x)}$ | 0.282 ± 0.005 | 6.0525 ± 0.0007 |
| | 0.285 | 6.052 |




A composition of x = 0.282 ± 0.005, i.e., a S content of (28.2 ± 0.5)%

# Export Peaks from Multiple Scans – NbN on Al2O3

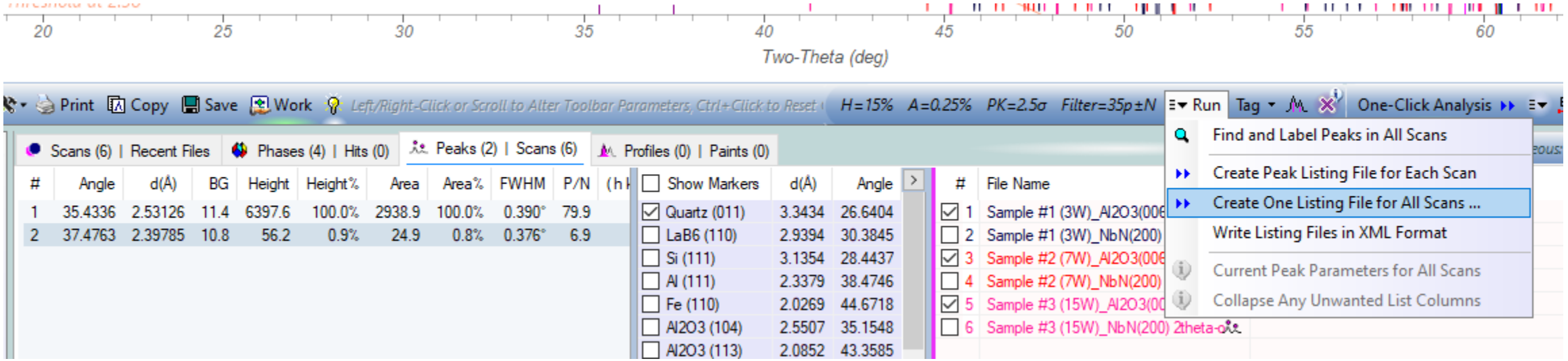



We were asked to determine in-plane lattice parameters for NbN films on sapphire (Al2O3).
Used a 4-circle diffractometer to collect gonio scans, rocking curves and reciprocal space maps to understand film quality, composition, epitaxy and twinning. Generated a ton of data!
In the end need accurate peak positions from multiple scans to calculate the lattice parameters.
Note that these scans don't all cover the same range!

# Export Peaks from Multiple Scans

- Get export file for peaks: Run->Create On Listing File for All Scans…




If you have a set of scans and need a peak list for all of them…
Overlay scans in the scan tab. In this example, three scans were selected.
Then, select the "Run" button on the Peaks tab and choose "Create One Listing File for all Scans."
A text file will be generated with all peaks listed that can be pasted into Excel (next slide)

# Output

| # (Scan ID) | Angle(°) | d(Å) | Centroi | Centroid(Å) | BG | Height | H/σ | Area(α1) | A |
|---|---|---|---|---|---|---|---|---|---|
| Sample 1 | 35.4474 | 2.53 | 35.405 | 2.53324 | 11.4 | 4050 | 139.8 | 1989.4 | |
| Sample 2 | 37.495 | 2.4 | 37.465 | 2.39855 | 10.8 | 21.5 | 6.4 | 5.7 | |
| Sample 3 | 41.0045 | 2.2 | 41.0645 | 2.19623 | 18.9 | 143.7 | 47.1 | 165.7 | |
| Sample 4 | 34.8909 | 2.57 | 34.919 | 2.56738 | 10.8 | 1894.3 | 135.9 | 1491.1 | |
| Sample 5 | 41.2102 | 2.19 | 41.235 | 2.18754 | 33.1 | 186.7 | 61.1 | 271.8 | |
| Sample 6 | 35.0922 | 2.56 | 34.8986 | 2.56884 | 5 | 110.7 | 51.7 | 243.4 | |
| Sample 7 | 37.6369 | 2.39 | 37.6109 | 2.38958 | 3.8 | 5245.7 | 136.9 | 1360.2 | |
| Sample 8 | 43.8981 | 2.06 | 43.8721 | 2.06198 | 2.2 | 56.7 | 8.2 | 19.5 | |
| Sample 9 | 40.748 | 2.21 | 40.822 | 2.2087 | 61.9 | 77.1 | 8.1 | 170.3 | |

# Laue Back Reflection

A polychromatic source of X-rays (S) is collimated onto the surface of a single crystal (SC)

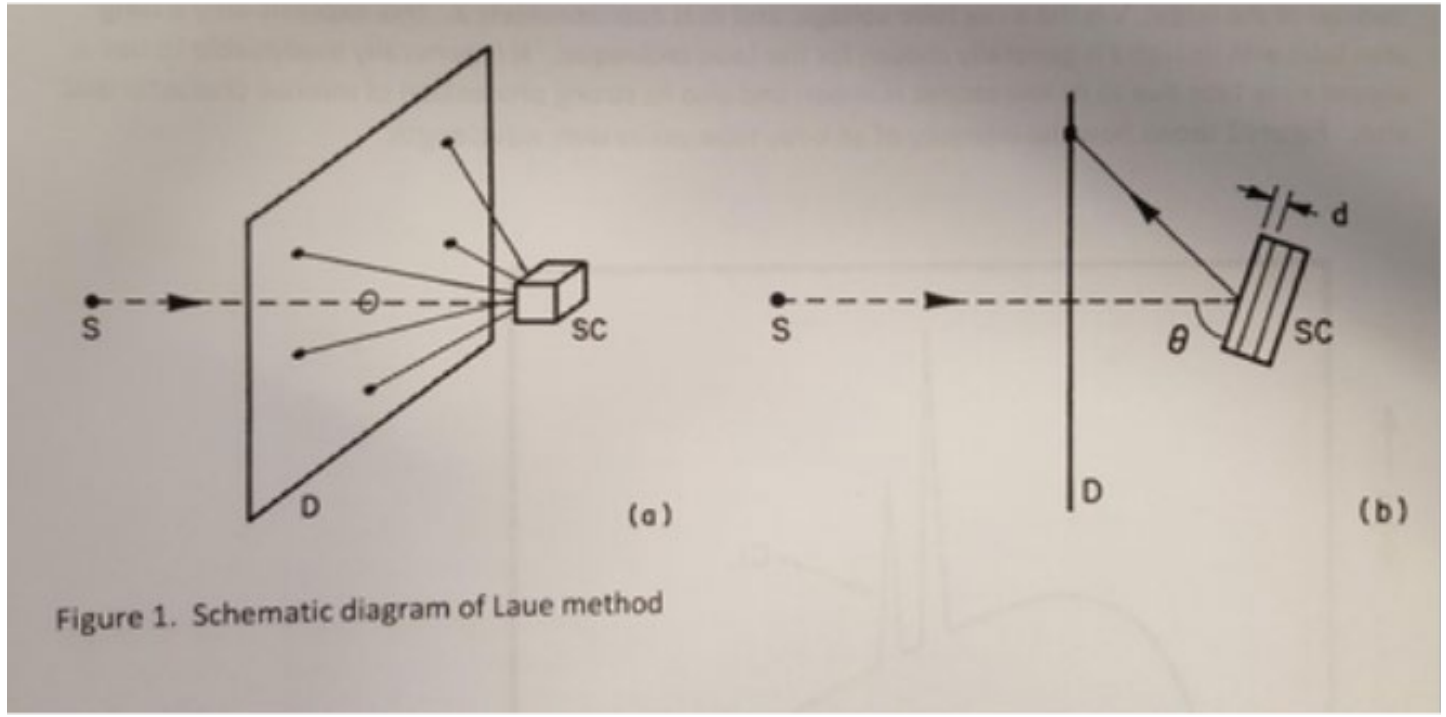



Figure 1. Schematic diagram of Laue method

*This Figure is copied from the Multiwire "MWL120 Real-Time Back Reflection Laue Camera" System documentation version 5.4, page 3.*

Bragg's law $n\lambda = 2d \sin \theta$

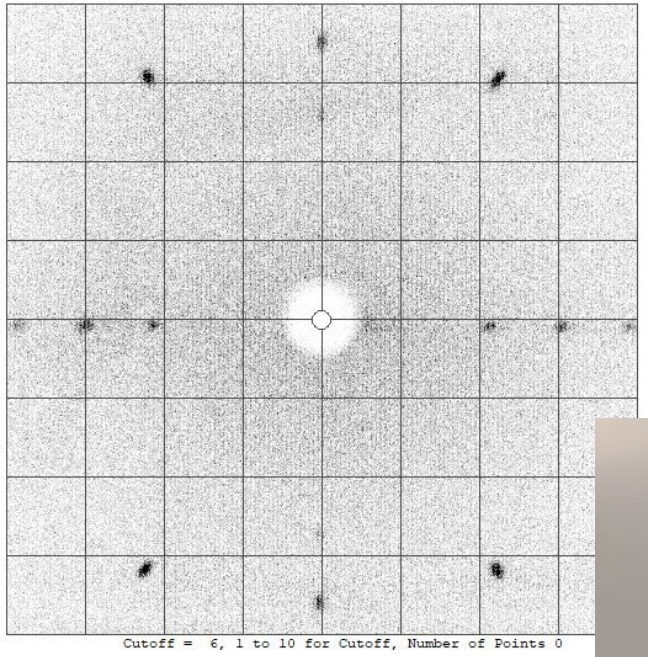
Where λ = wavelength,
d= interplanar lattice spacing,
θ = incidence angle

Θ and d are fixed
Bragg's law is fulfilled at specific λ's

Results in a spot pattern
Each spot is a specific (hkl)

# Use Jade to help with orienting single crystals – part 1

We confirmed the face of a crystal is (110) oriented cubic material using back reflection Laue.




Harder way – use dot product

$$\cos(\theta) = \frac{[110] \cdot [111]}{|[110]||[111]|}$$

1. Compute the dot product

$$A \cdot B = (1)(1) + (1)(1) + (0)(1) = 2$$

2. Compute the magnitudes

$$|A| = \sqrt{1^2 + 1^2 + 0^2} = \sqrt{2}$$

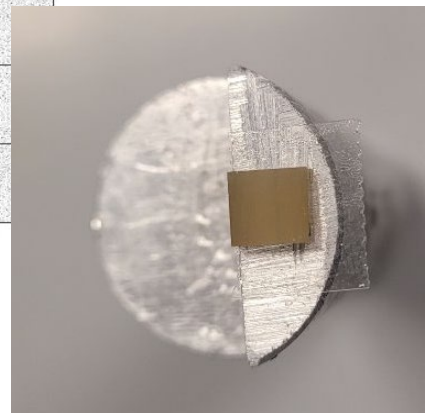
$$|B| = \sqrt{1^2 + 1^2 + 1^2} = \sqrt{3}$$

3. Use the dot product formula

$$\cos(\theta) = \frac{A \cdot B}{|A||B|} = \frac{2}{\sqrt{2} \cdot \sqrt{3}} = \frac{2}{\sqrt{6}}$$

4. Calculate the angle

$$\theta = \cos^{-1}\left(\frac{2}{\sqrt{6}}\right) \approx \cos^{-1}(0.8165) \approx 35.26^\circ$$

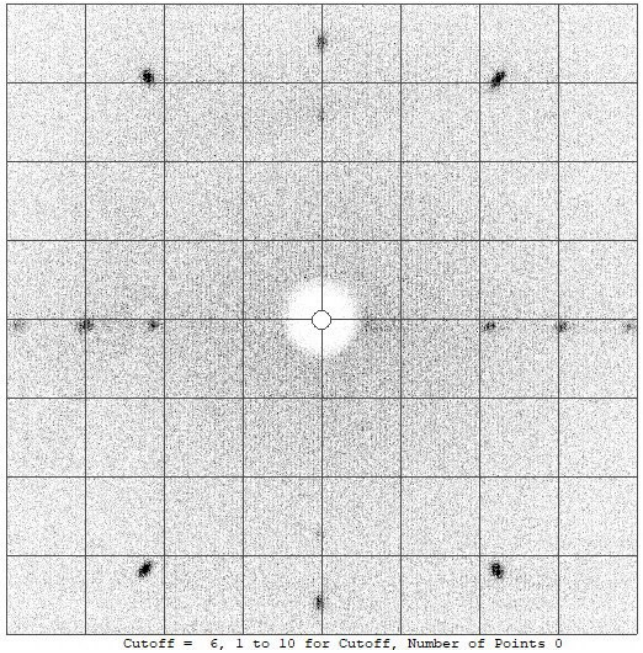


Student wants to know where the (111) face is located.

Rotate crystal 35.26 degrees up-down and left-right until the (111) face is located.

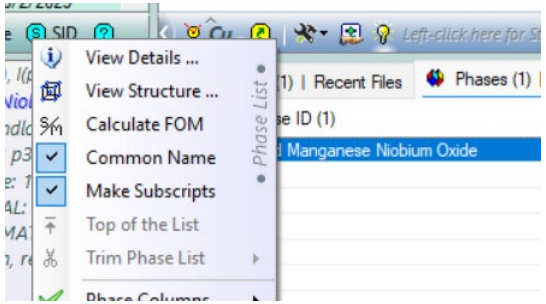
# Use Jade to help with orienting single crystals – part 2

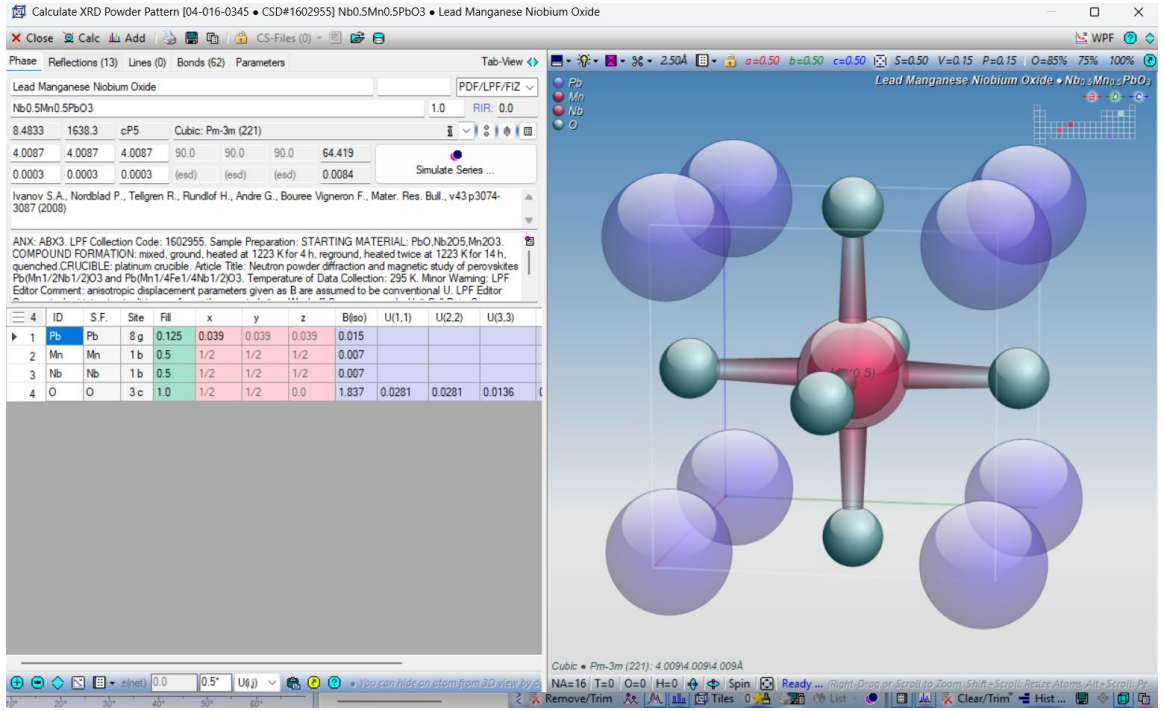
(110) oriented PMN-PT




Student wants to know where the (111) face is located.

Easy way – Use Jade

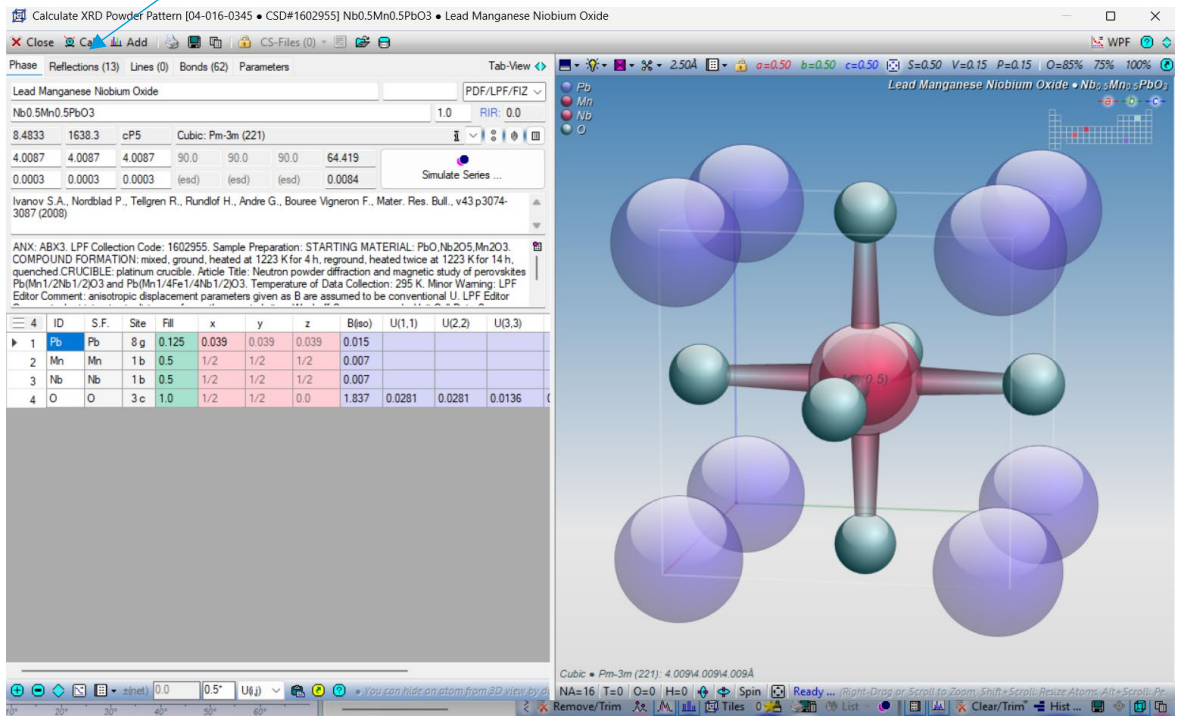


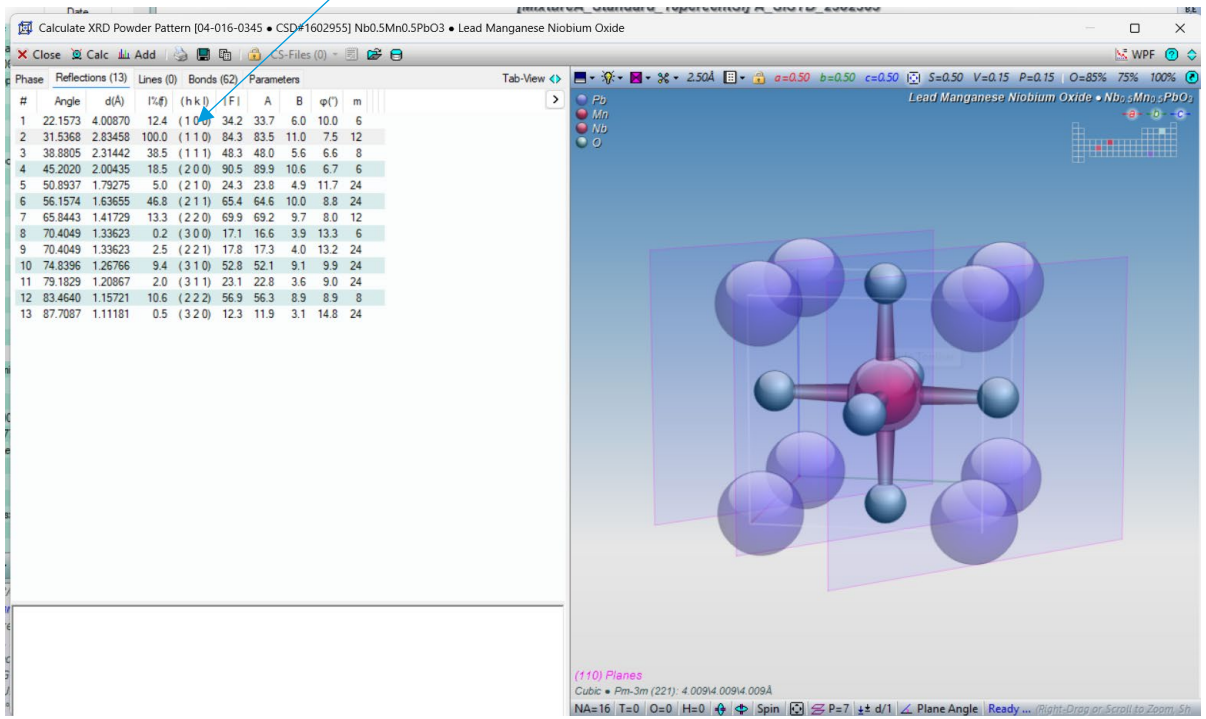

1. Locate a PDF file for your material (or similar structure). 04-016-0345
2. Right click on the Phase ID file name and choose "View Structure."

# Use Jade to help with orienting single crystals – part 2

3. Select Reflections

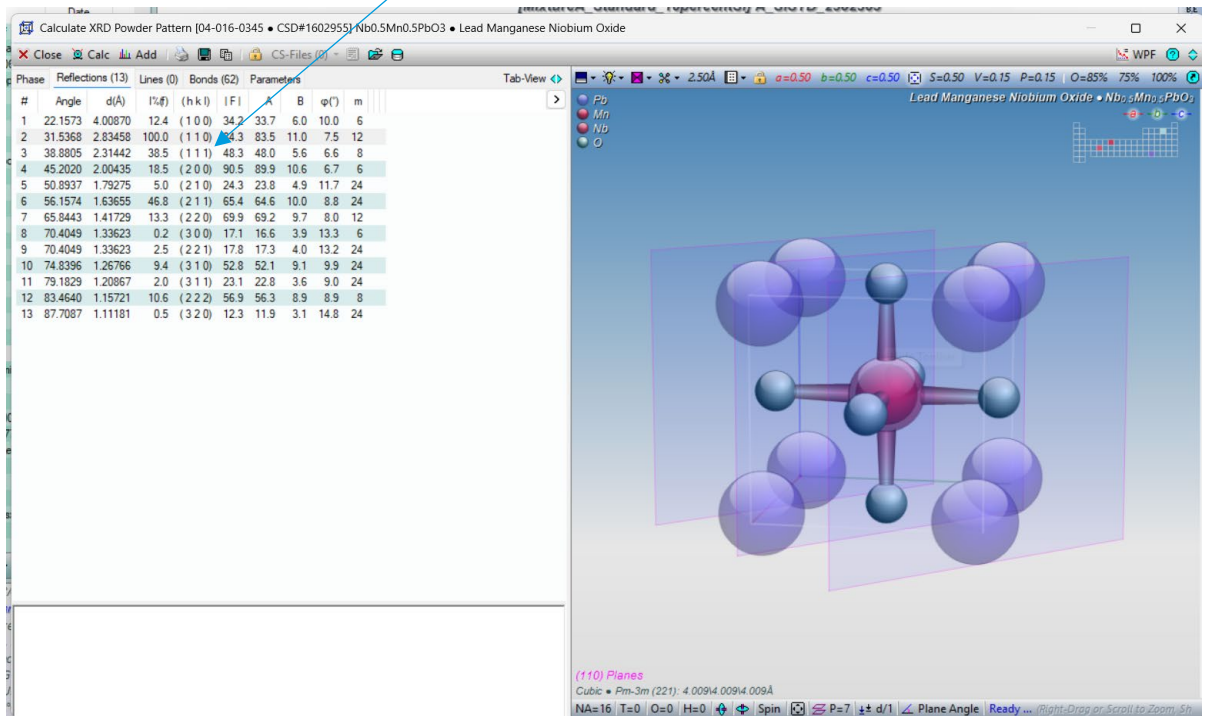



4. Click on the (110) row



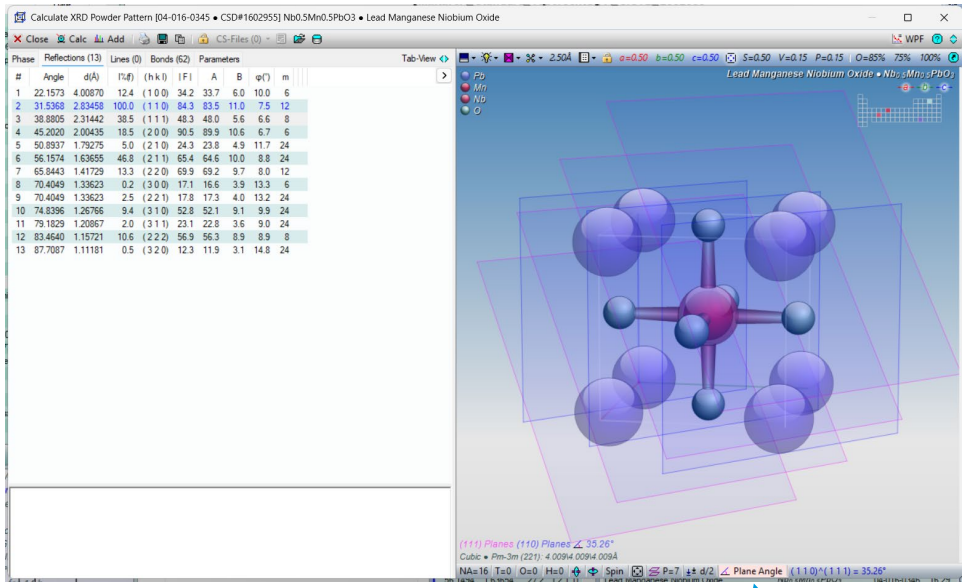
# Use Jade to help with orienting single crystals – part 2

5. Select the (111) row

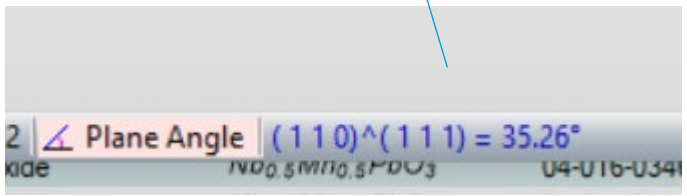



6. Click "< Plane Angle"

7. Both sets of planes will be shown.

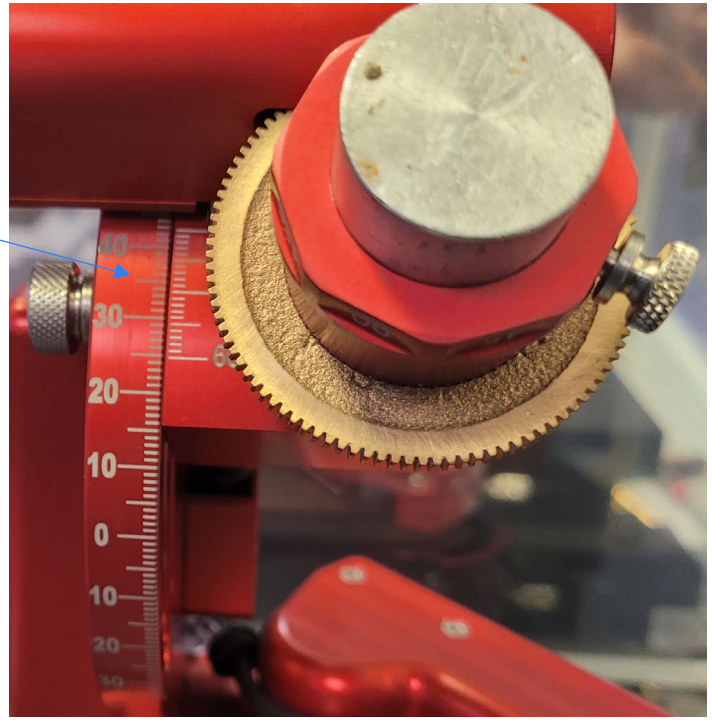



8. Angle between the planes is shown here.

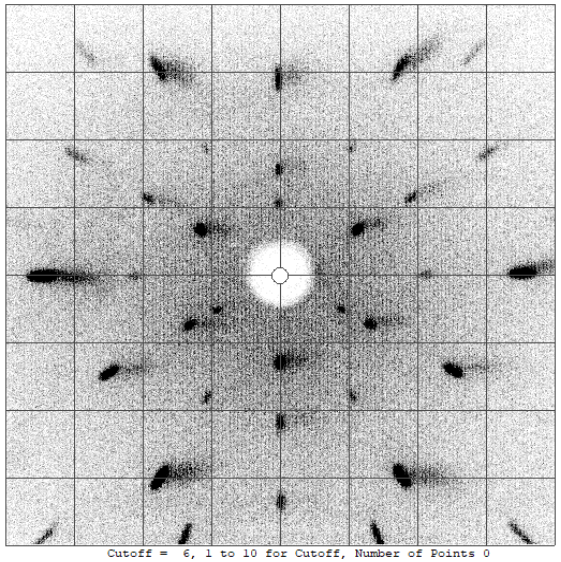


# Laue tilt into 111 zone axis

We confirmed (111) zone axis of the PMN-PT using back reflection Laue and tilting the sample 35deg.

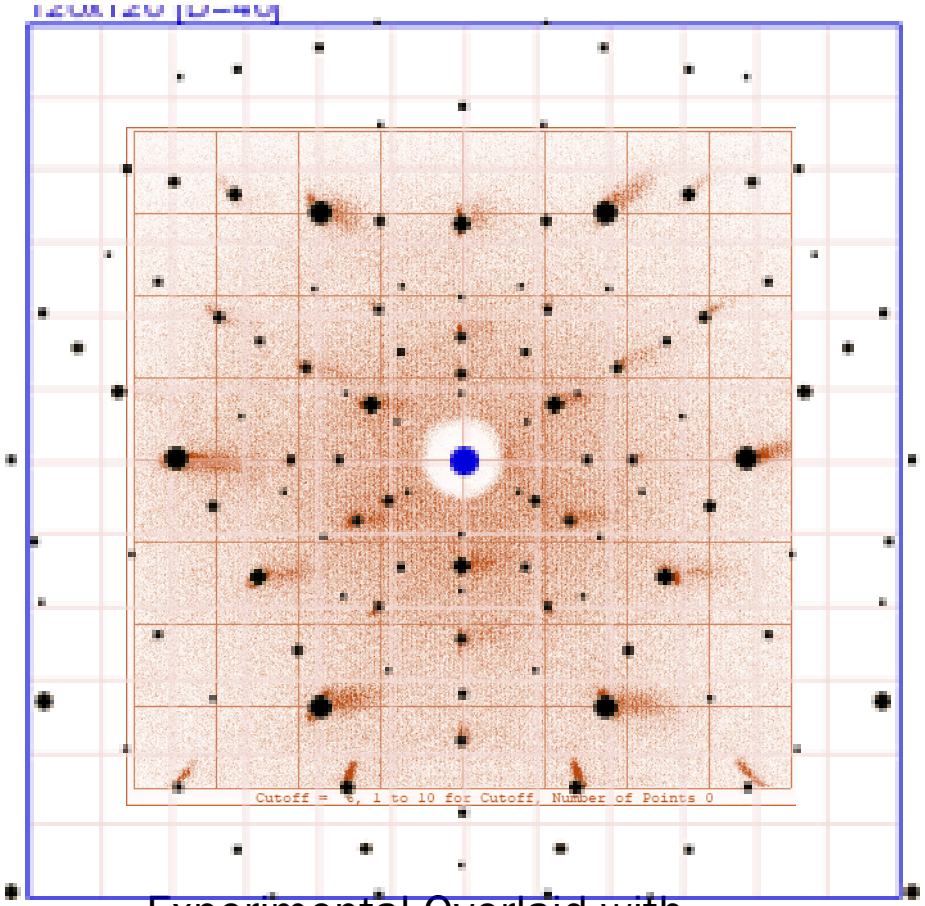



Gonio shows tilt




Experimental Back Reflection Laue

JPowd simulated (111)

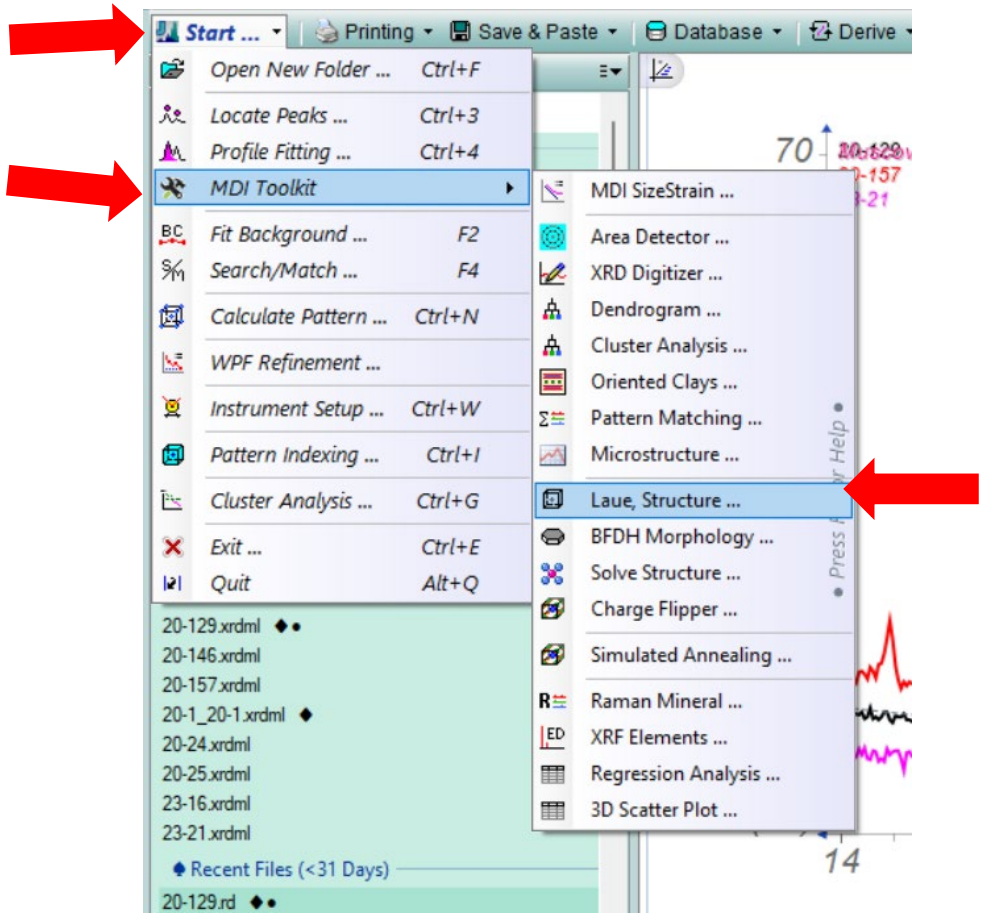



Experimental Overlaid with Calculated (111) Laue Pattern

# Laue Pattern Calculation in Jade

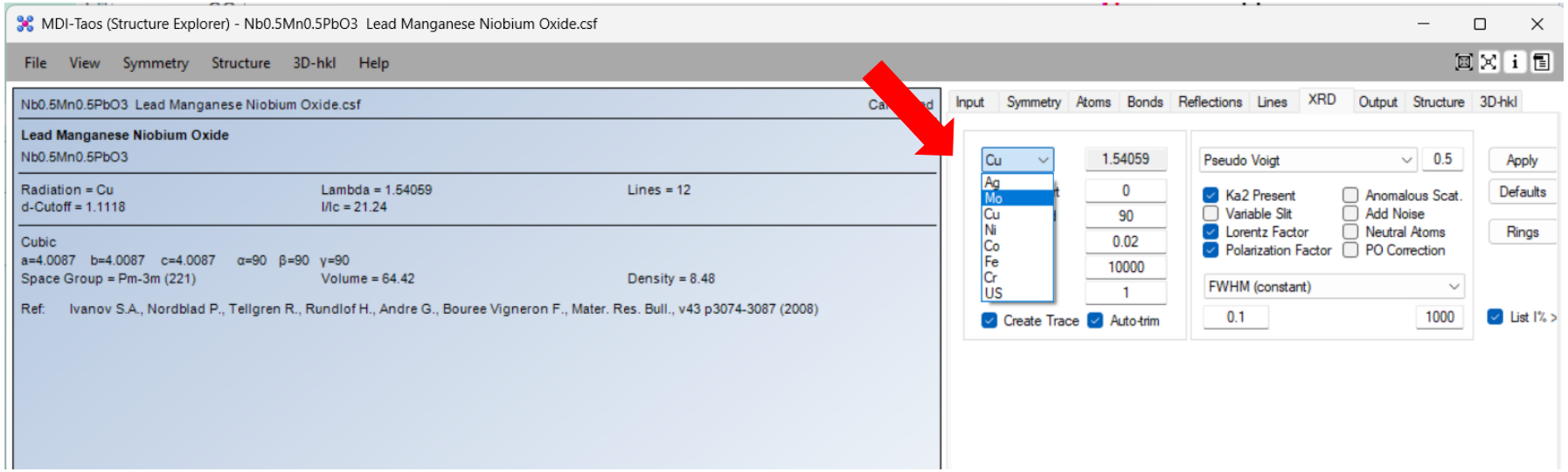
1. Select:

Start --> MDI Toolkit --> Laue Structure



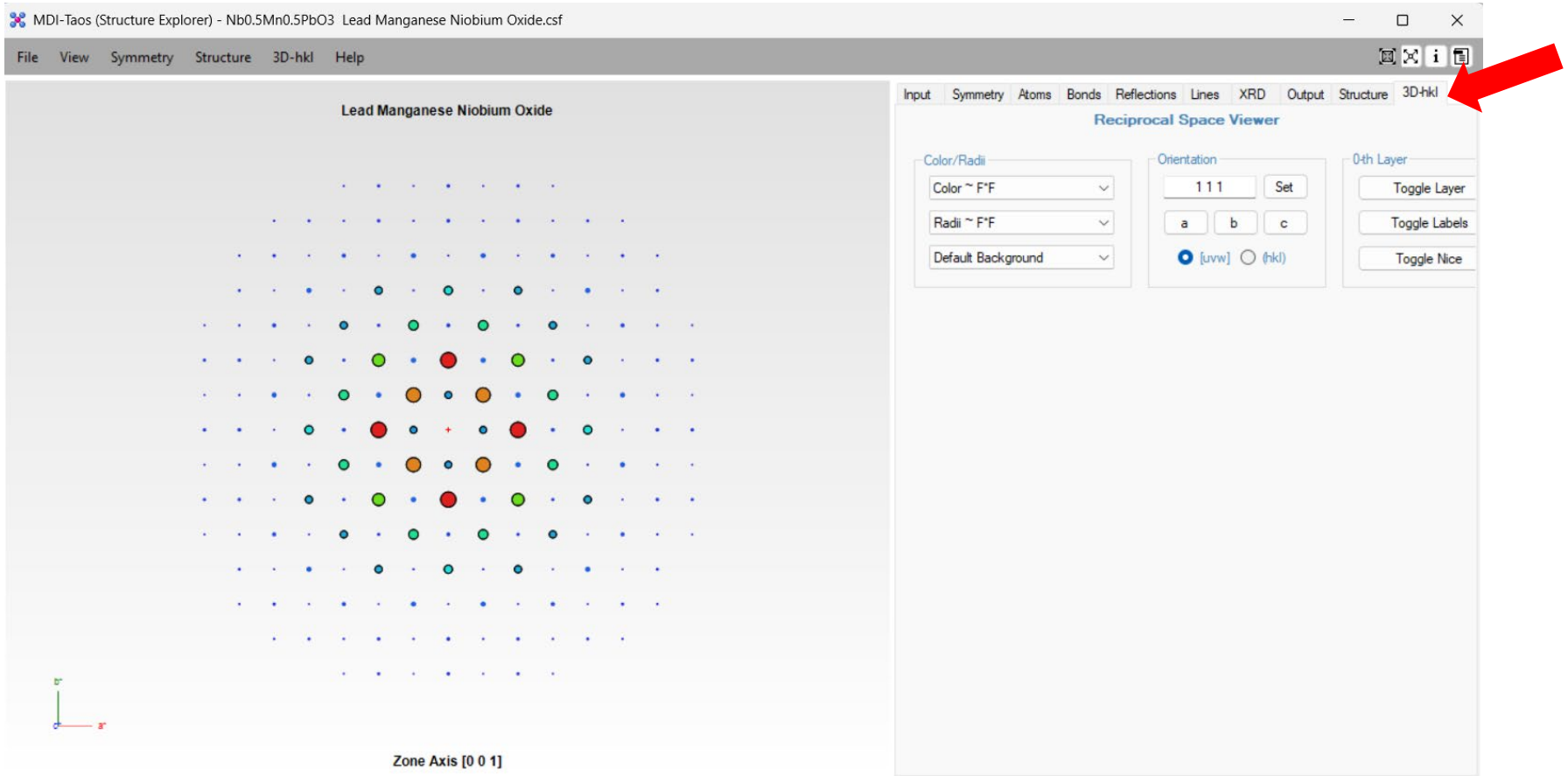
# Laue Pattern Calculation in Jade

2. Select XRD and change the wavelength to Mo or Ag – this will allow you to see more spots.



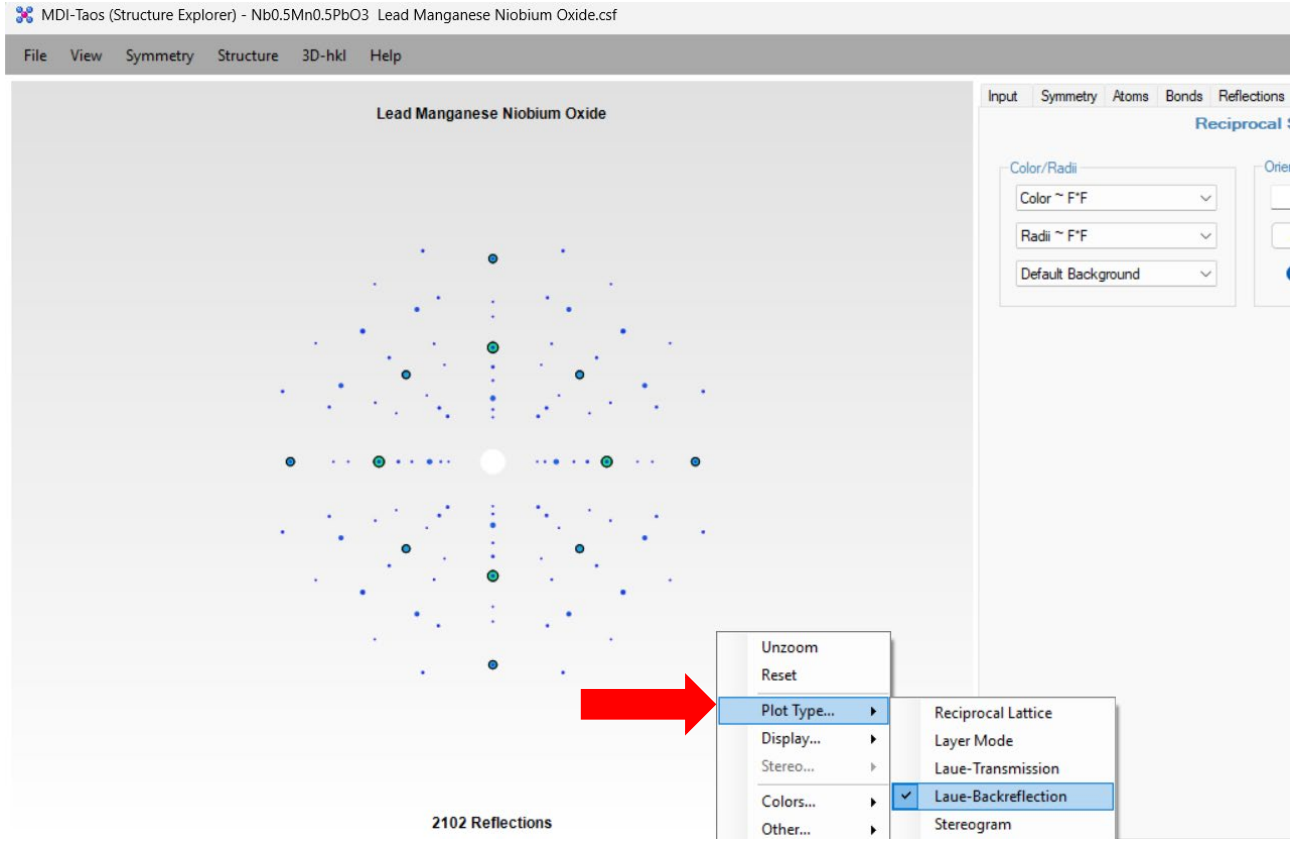
# Laue Pattern Calculation in Jade

3. Choose 3D hkl tab



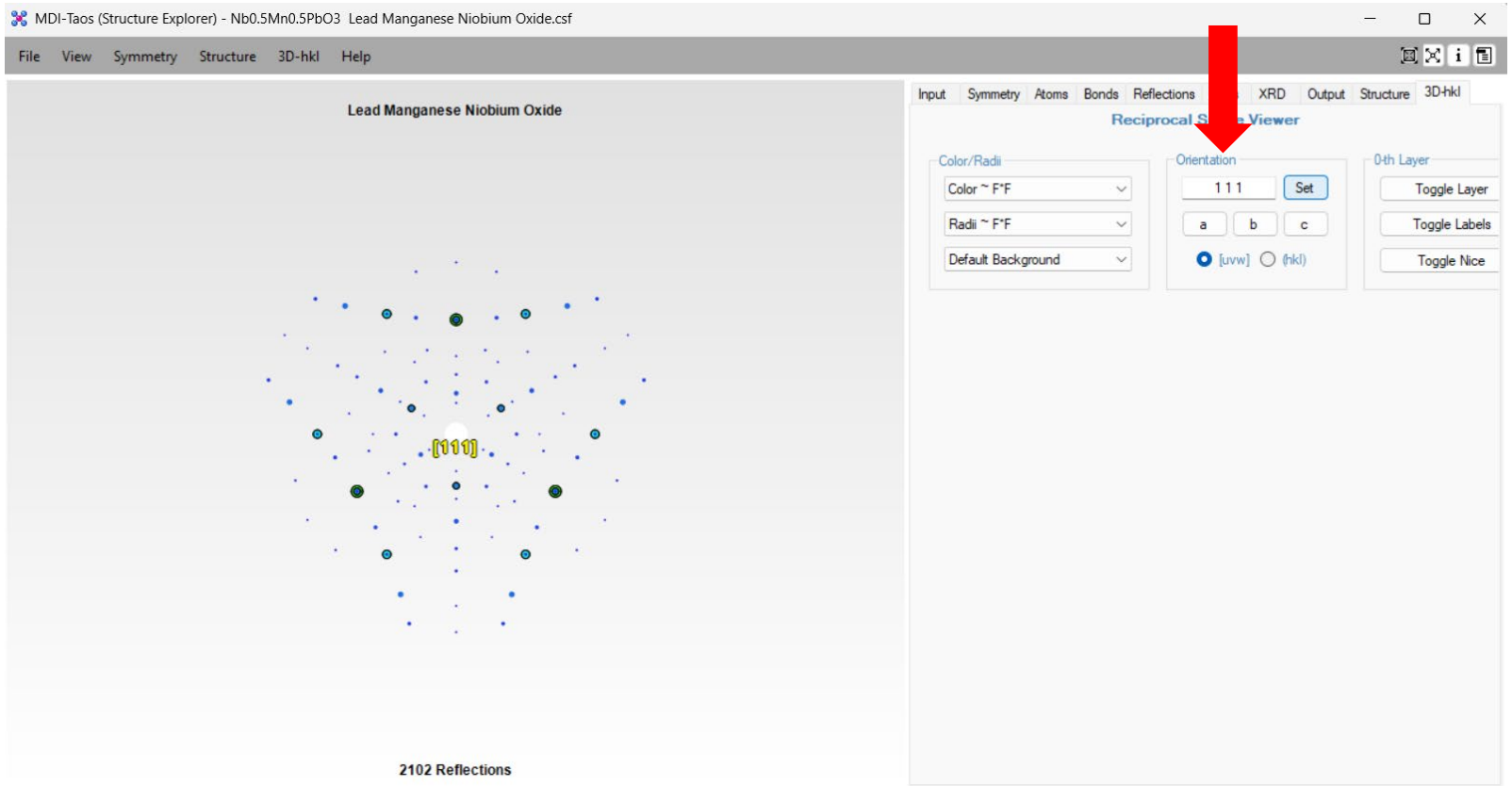
# Laue Pattern Calculation in Jade

4. Right click on the image, select "Plot Type" and "Laue backreflection."



# Laue Pattern Calculation in Jade

5. Select the orientation of interest and select "set."



# Laue Pattern Calculation in Jade

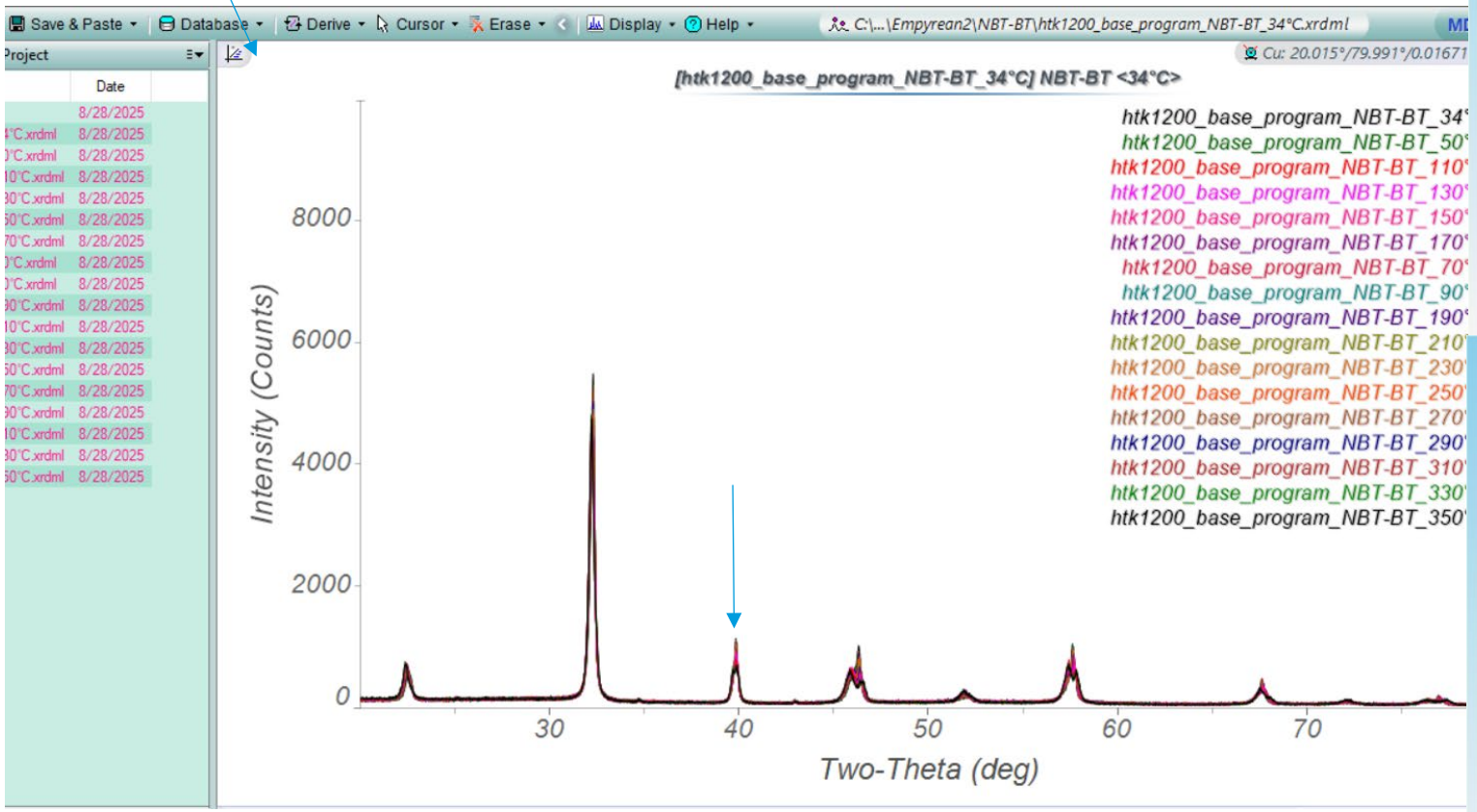
6. Toggle background options if you wish.

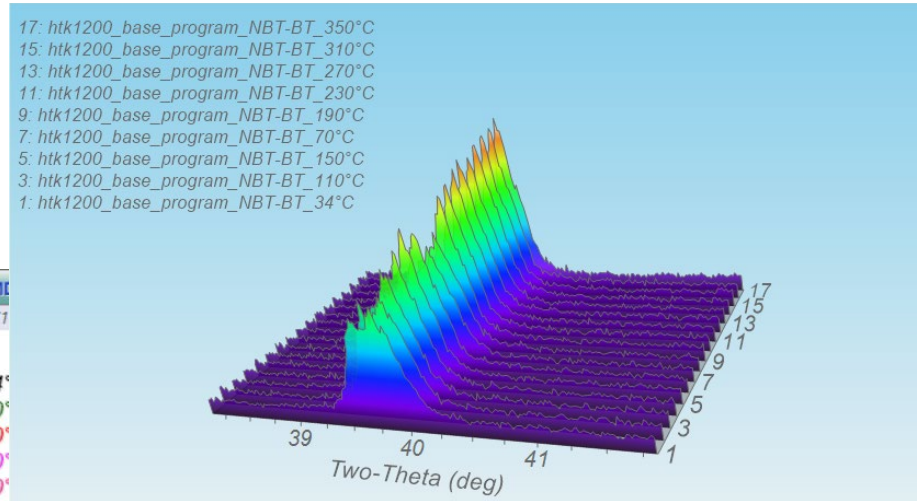


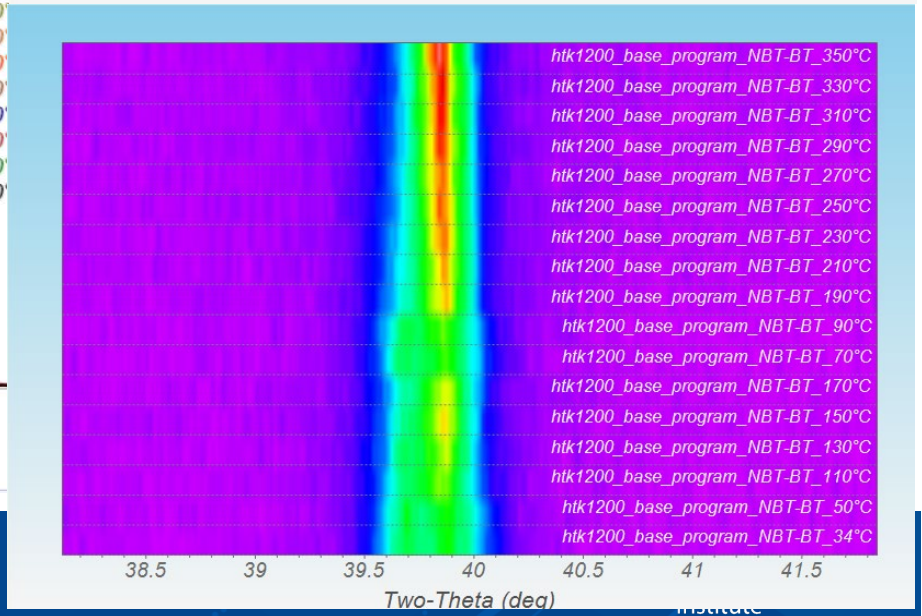
# Non-ambient Diffraction of NBT-BT



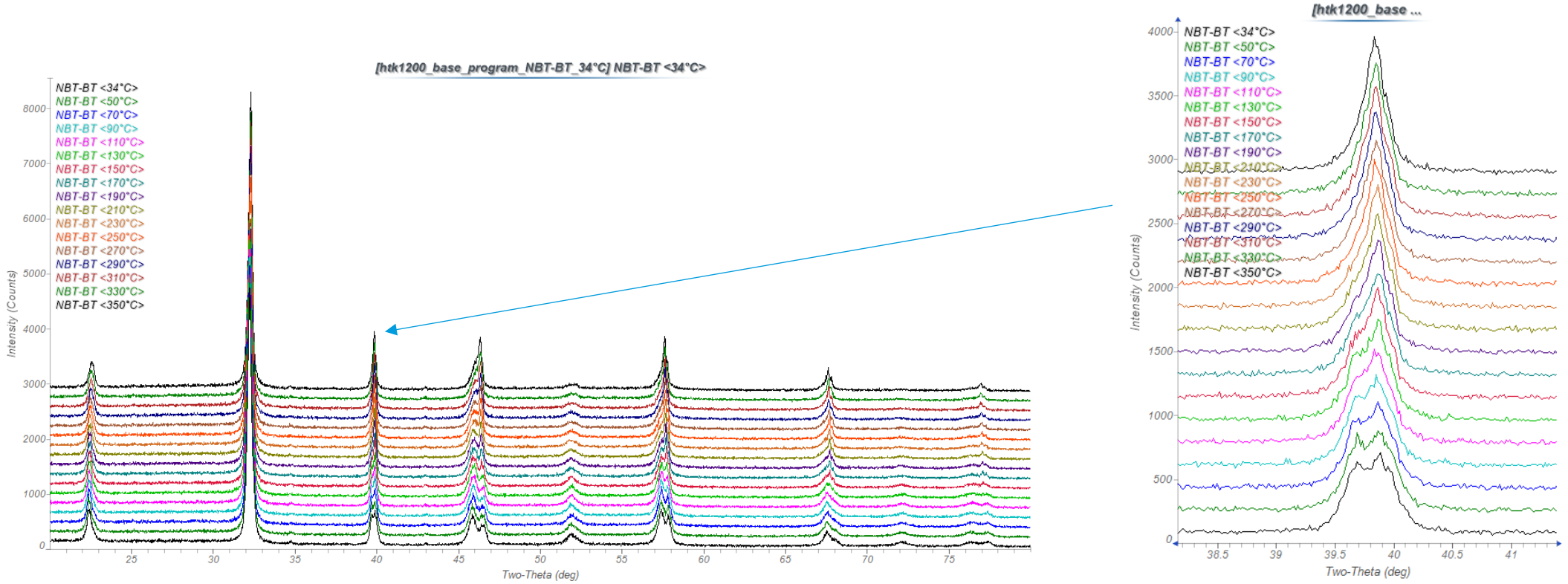
By selecting the 3D Perspective View or 2D Intensity Map – additional views can be accessed. Here we can show in two ways how the peak at 39 deg. is changing from 2 peaks to 1 peak.



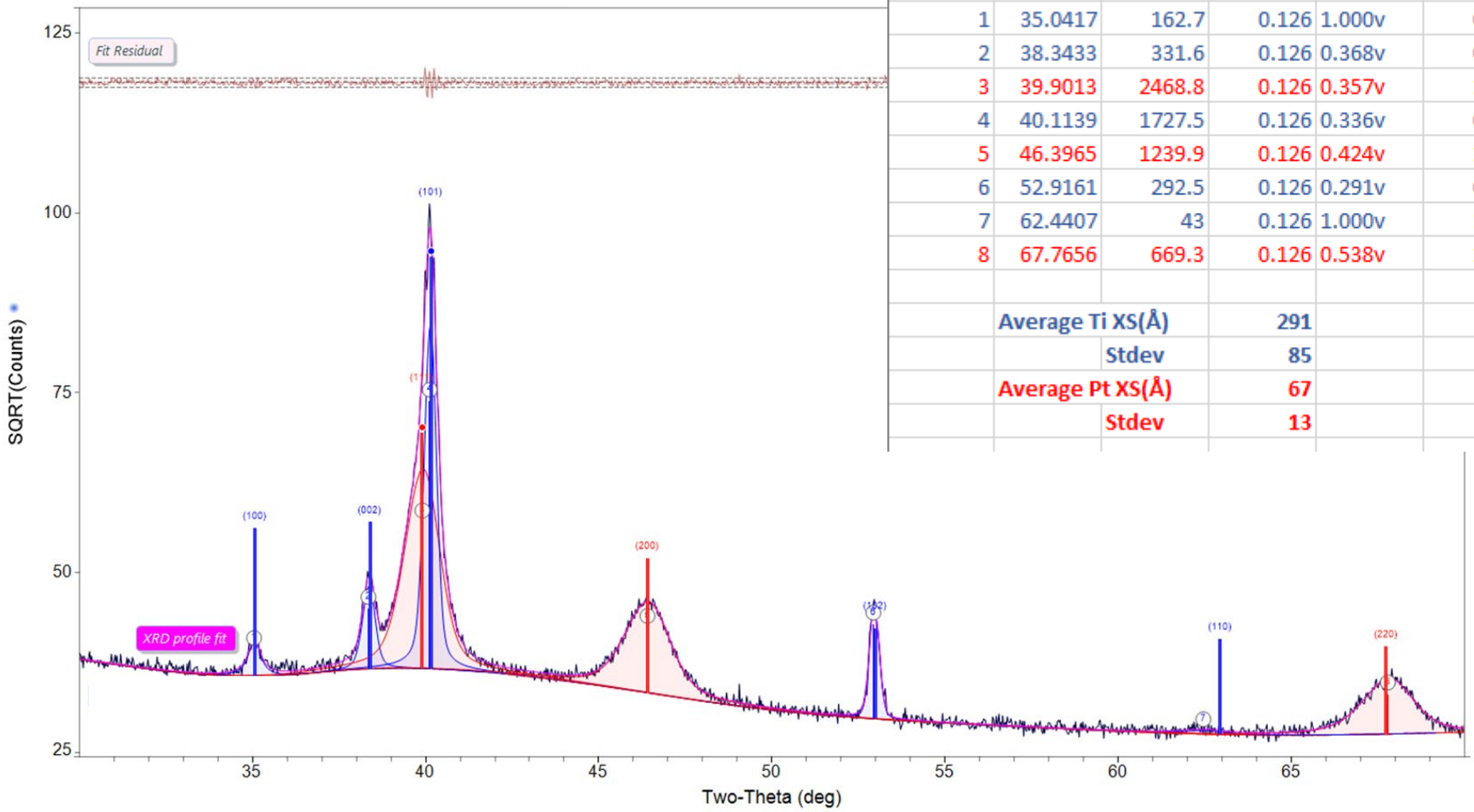



# Non-ambient Diffraction of Sodium Bismuth Titanate - Barium Titanate (NBT-BT)




Non-ambient XRD can create large datasets where one needs to be able to display the data in various ways to highlight certain features. Scans can have a vertical offset applied to better visualize changes. Zoomed views create focus.

# Profile Fitting Tools – Pt film on Ti Substrate




| # | Angle(°) | Area(α1) | Skew¹ | Shape | FWHM(°) | XS(Å) | ( h k l) | Phase ID |
|---|---|---|---|---|---|---|---|---|
| 1 | 35.0417 | 162.7 | 0.126 | 1.000v | 0.386 | 237 | ( 1 0 0) | Titanium ● Ti |
| 2 | 38.3433 | 331.6 | 0.126 | 0.368v | 0.394 | 234 | ( 0 0 2) | Titanium ● Ti |
| 3 | 39.9013 | 2468.8 | 0.126 | 0.357v | 1.056 | 82 | ( 1 1 1) | Pt ● Platinum |
| 4 | 40.1139 | 1727.5 | 0.126 | 0.336v | 0.343 | 276 | ( 1 0 1) | Titanium ● Ti |
| 5 | 46.3965 | 1239.9 | 0.126 | 0.424v | 1.446 | 60 | ( 2 0 0) | Pt ● Platinum |
| 6 | 52.9161 | 292.5 | 0.126 | 0.291v | 0.257 | 415 | ( 1 0 2) | Titanium ● Ti |
| 7 | 62.4407 | 43 | 0.126 | 1.000v | 1.444 | 65 | ( 1 1 0) | Titanium ● Ti |
| 8 | 67.7656 | 669.3 | 0.126 | 0.538v | 1.625 | 59 | ( 2 2 0) | Pt ● Platinum |
| | | | | | | | | |
| | Average Ti XS(Å) | | 291 | | | | | |
| | | Stdev | 85 | | | | | |
| | Average Pt XS(Å) | | 67 | | | | | |
| | | Stdev | 13 | | | | | |

Estimated Crystallite size

# Questions?

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