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Practical XRD with Confidence

Episode 1 – Powder XRD Fundamentals: How to Identify Phases and Trust Your Data

Wednesday, May 6, 2026, at 1 pm CDT

Presenter: Ekaterina Vinogradova, PhD

Host: Tom Concolino, PhD

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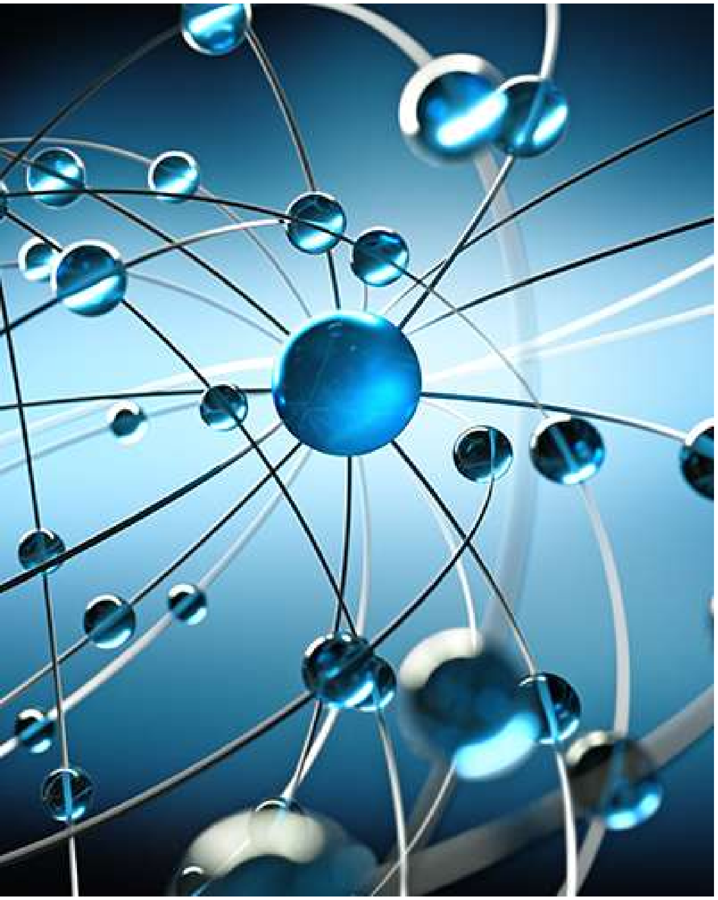
- *You will be muted during the workshop*
- *You can ask questions using the Q&A tool.*
- *You should hear music if your sound is working*

Presenter:
Ekaterina Vinogradova
Applications Scientist

Host:
Tom Concolino
Sales Manager, XRD

You can ask questions during the presentation. Please use the Q&A to ask questions.

Recording will be available tomorrow.

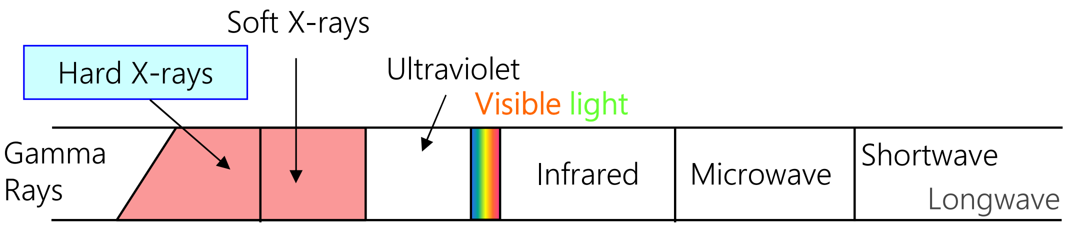


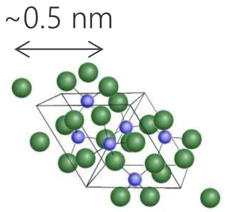
Agenda

- The physics behind the peaks
- Phase identification foundations
- Basics of sample preparation
- Data collection quality
- Practical example

Basics of X-rays

- Discovered by Wilhelm Roentgen (1895)
- Electromagnetic waves with very short wavelength (~0.01–10nm)





- Interaction with crystals occurs via elastic (Thomson) scattering from atomic planes
- Wavelength is comparable to interatomic distances in crystals (0.1–0.5 nm)

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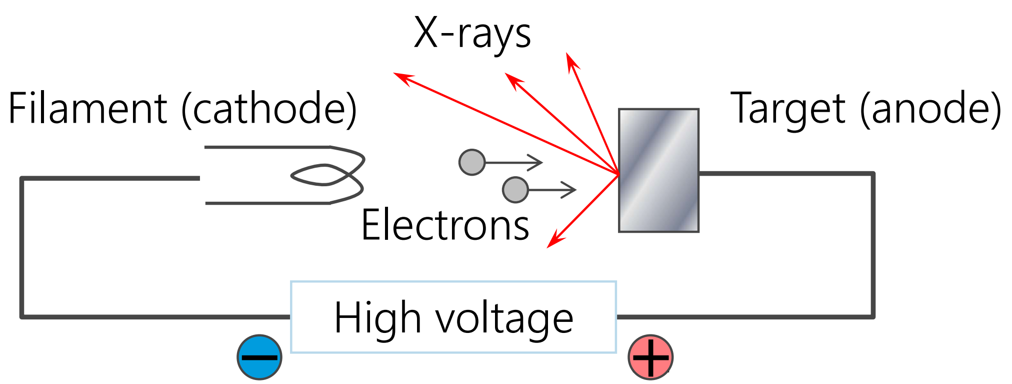
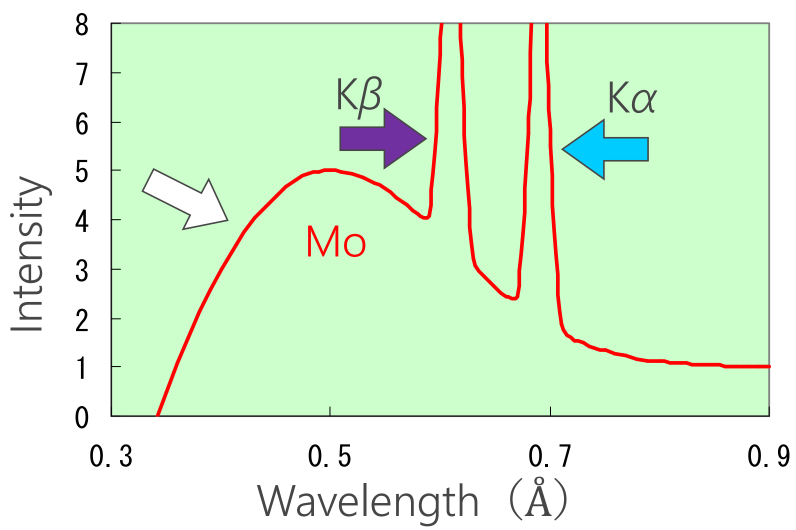
Generation of X-rays

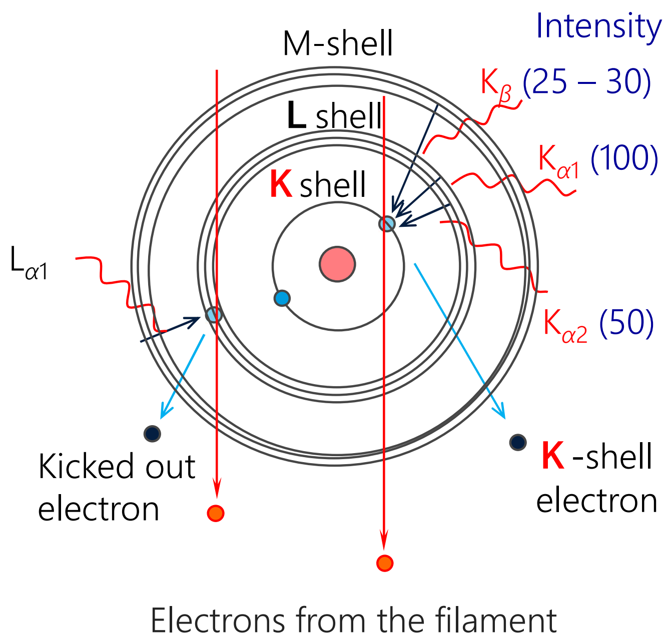


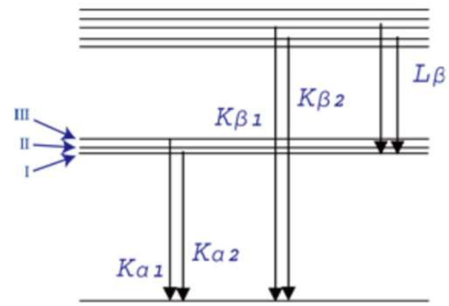
Generation efficiency $\varepsilon = 1.1 \times 10^{-9}$ Z V （Cu 0.1%）
Z：atomic number of a target material V：applied voltage（V）



- The X-ray tube voltage is optimized to suppress bremsstrahlung and maximize characteristic X-ray emission.
- Biggest source of intensity fluctuations: cooling water t° (1°C change ~ 10% variation).

Generation of characteristic X-rays

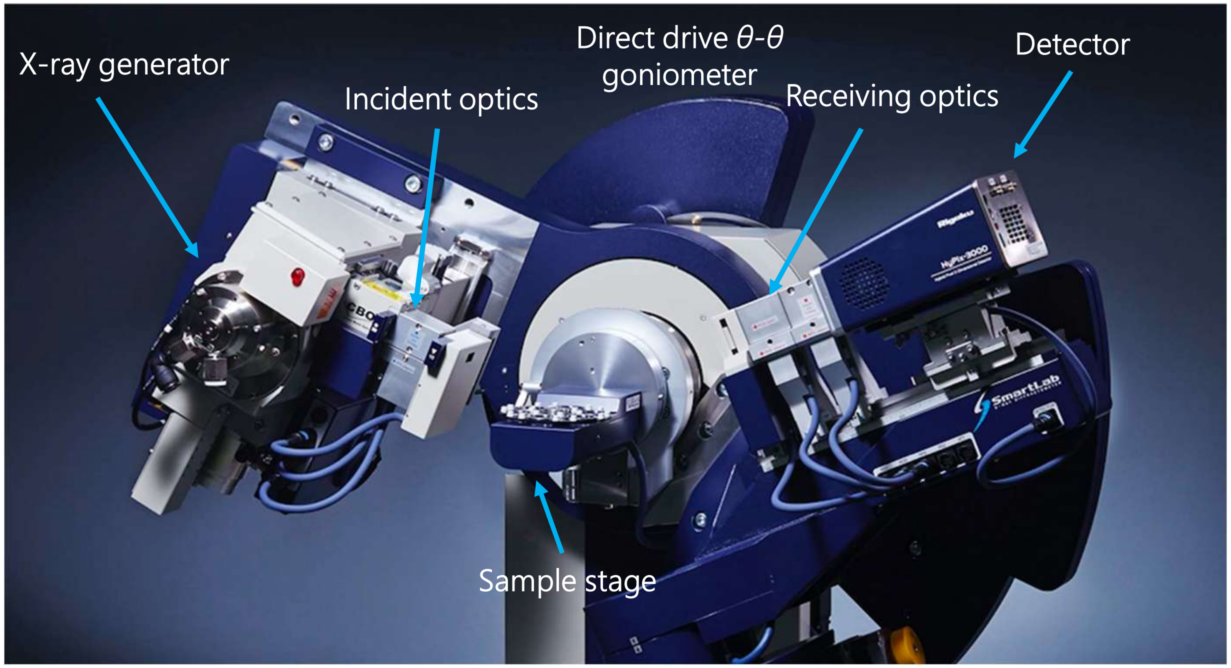




Wavelength

	Target	Kα1, Å	Kα2, Å	Kα2/Kα1, Å	Kα, Å	Kβ, Å
✓	Cu	1.540593	1.544414	0.497000	1.541862	1.392246
	Co	1.789001	1.792886	0.497000	1.790291	1.620823
	Mo	0.709317	0.713607	0.497000	0.710741	0.632303
	Cr	2.289746	2.293652	0.500000	2.291048	2.084912
	Fe	1.936081	1.940019	0.500000	1.937394	1.756645
	V	2.503610	2.507430	0.500000	2.504883	2.284446

X-ray diffractometer



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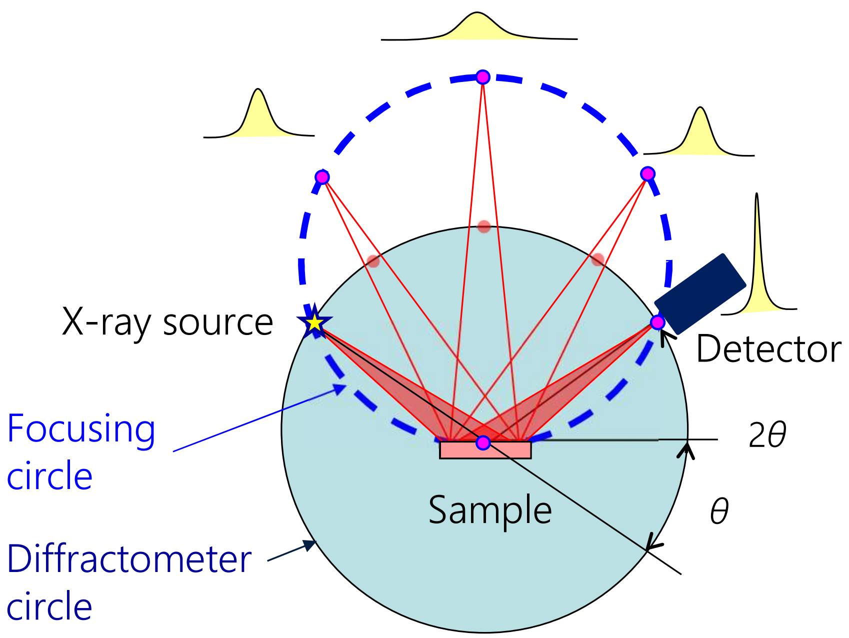
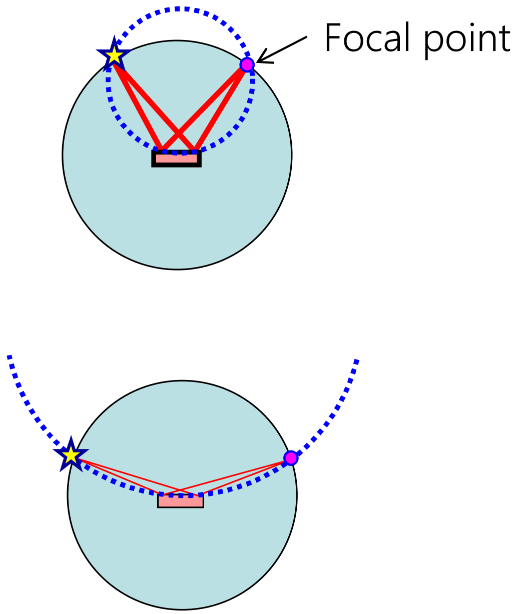
Bragg-Brentano para-focusing geometry

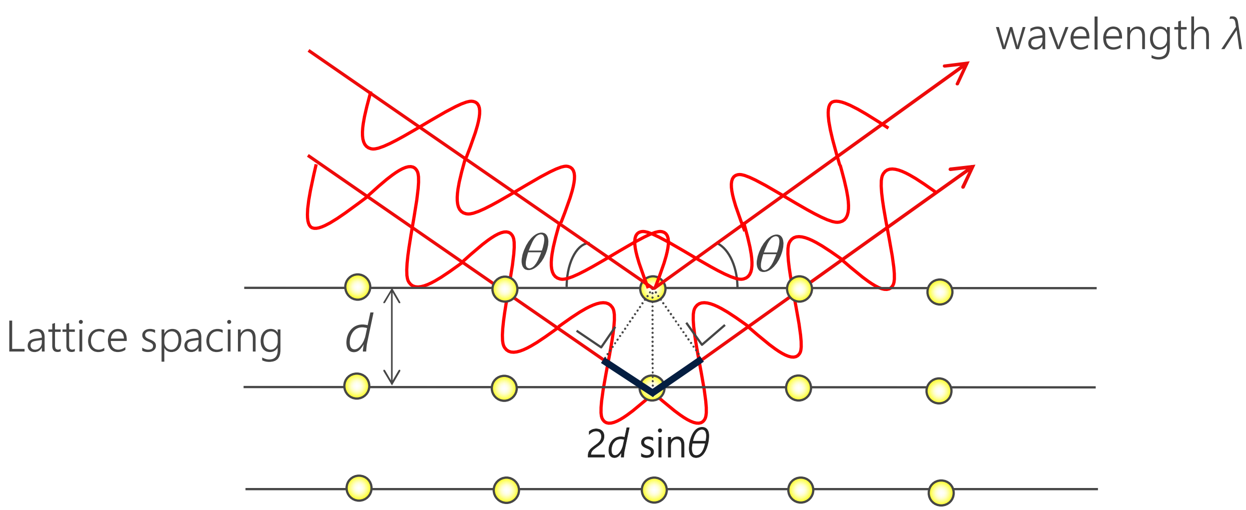




The radius of focusing circle changes depending on the diffraction angle

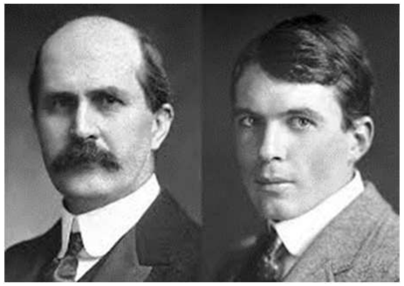
Bragg's Law

Diffraction peaks occur when the path difference between waves scattered by planes spaced *d* equals an integer multiple of λ, fixing the observed 2θ angles.



The Bragg's Equation: $n\lambda = 2d_{hkl} \sin\theta$

n: integer



Sir William Henry Bragg and William Lawrence Bragg (Nobel Prize in 1915)

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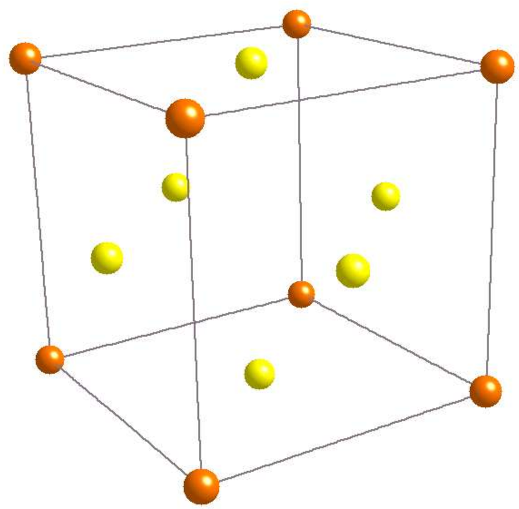
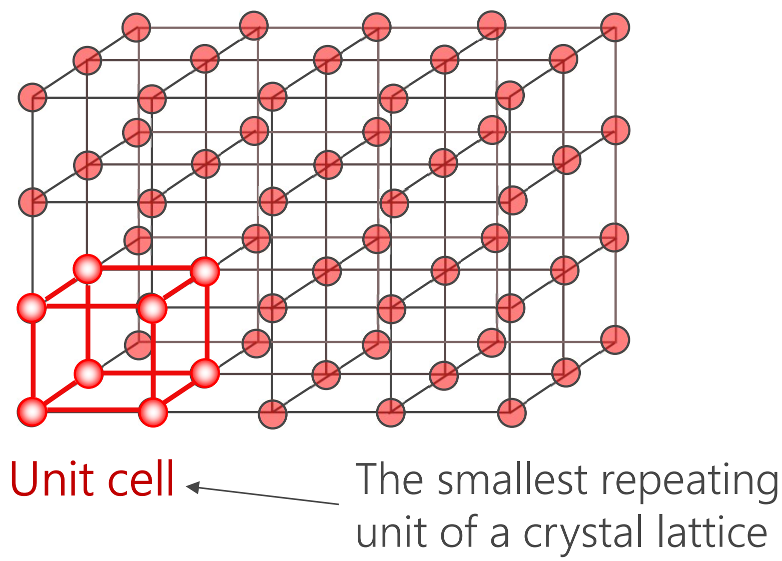
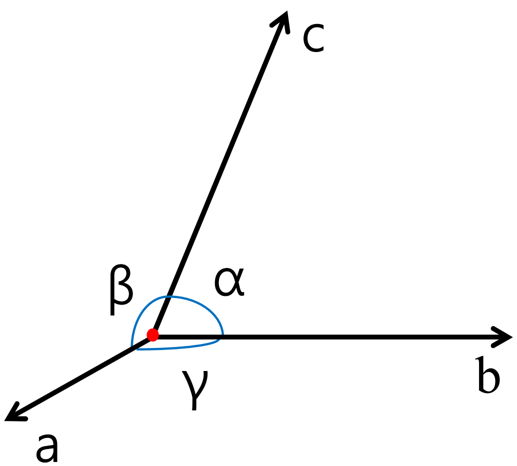
What is a crystal?

A crystal is a solid in which atoms are arranged with long-range order, producing a repeating pattern.

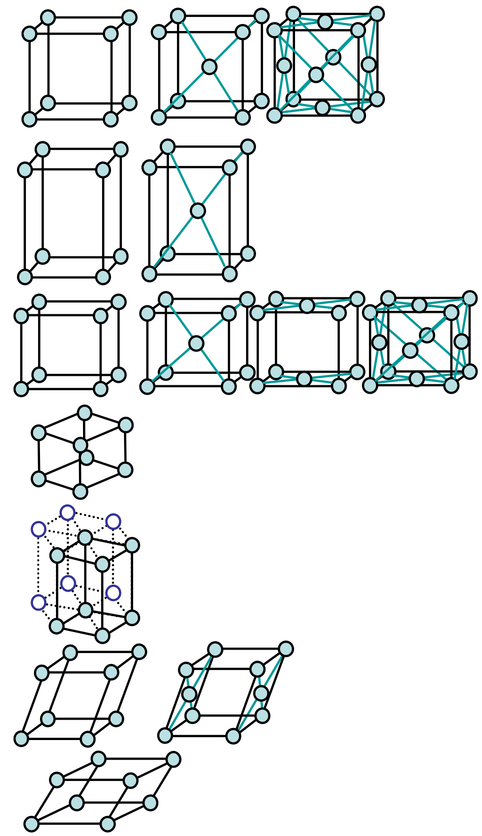




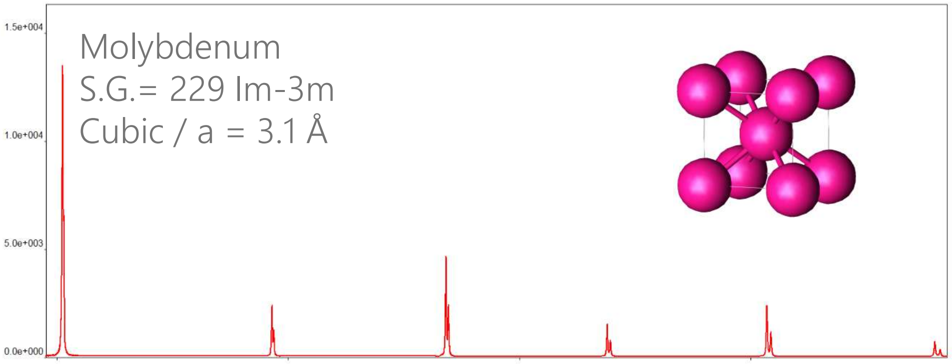
Crystal systems

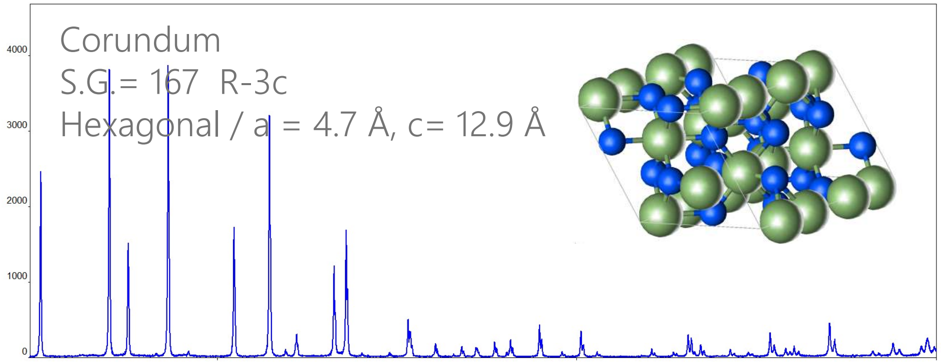


Cubic	$a=b=c$ $\alpha=\beta=\gamma=90°$	
Tetragonal	$a=b\neq c$ $\alpha=\beta=\gamma\ =90°$	
Orthorhombic	$a\neq b\neq c$ $\alpha=\beta=\gamma=90°$	
Rhombohedral	$a=b=c$	
Trigonal and Hexagonal	$\alpha=\beta=\gamma\neq 90°$ $a=b\neq c$ $\alpha=\beta=90°\quad \gamma=120°$	
Monoclinic	$a\neq b\neq c$ $\alpha=\beta=90°\quad \neq\gamma$	
Triclinic	$a\neq b\neq c$ $\alpha\neq\beta\neq\gamma\ \neq 90°$	



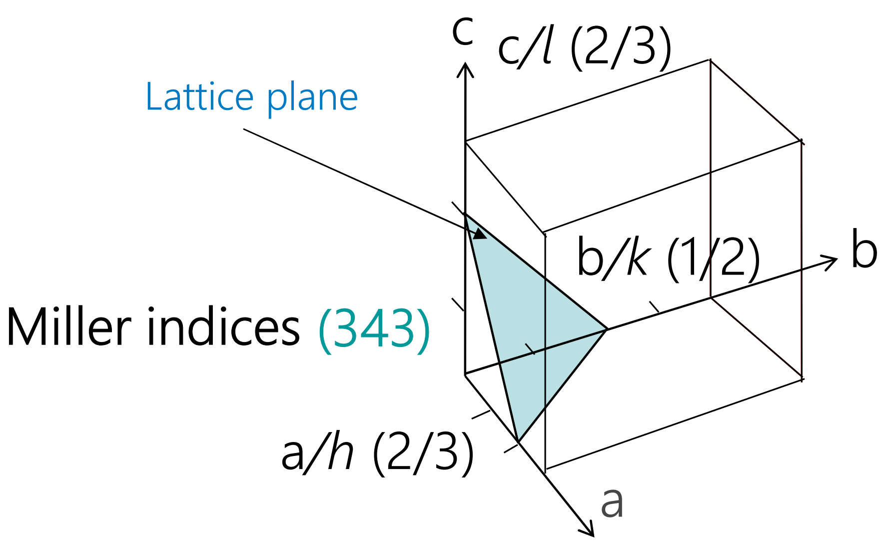
High and low symmetries





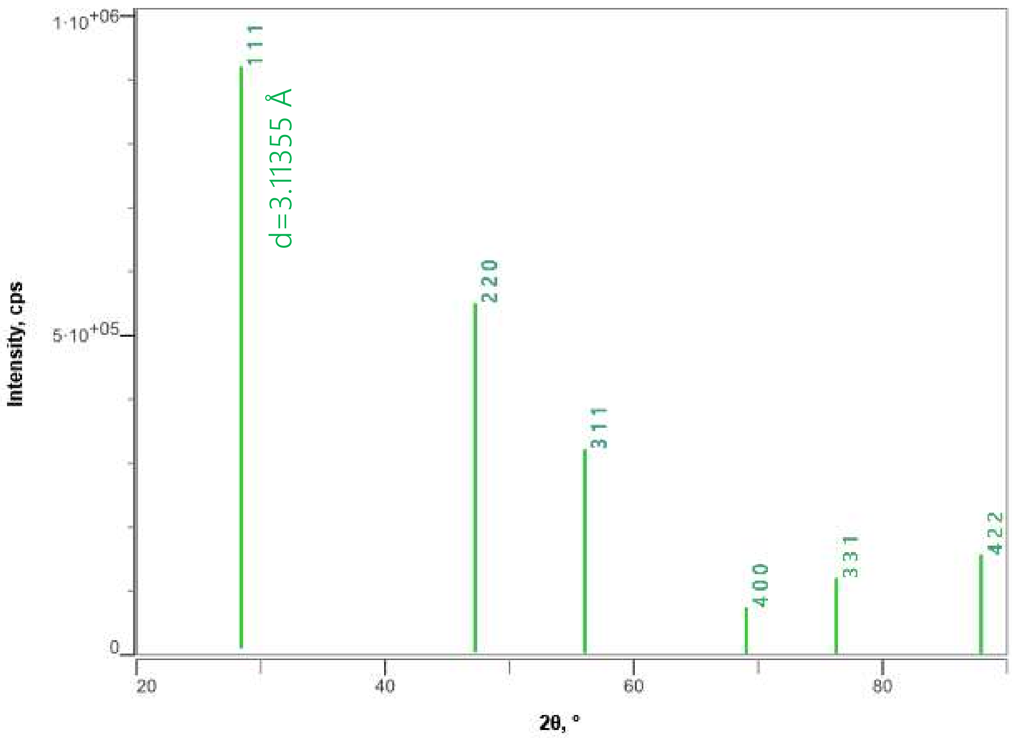
Miller indices

- The Miller indices (h k l) specify the orientation of crystal planes relative to the unit cell.
- Obtained by taking the reciprocals of the plane's axis intercepts and reducing to the smallest integers.



Database powder diffraction pattern from Si

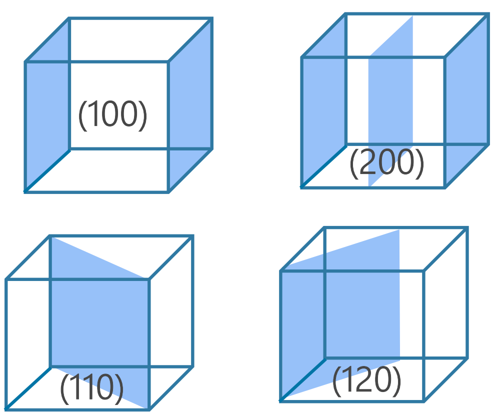
$2d \sin\theta = n\lambda$



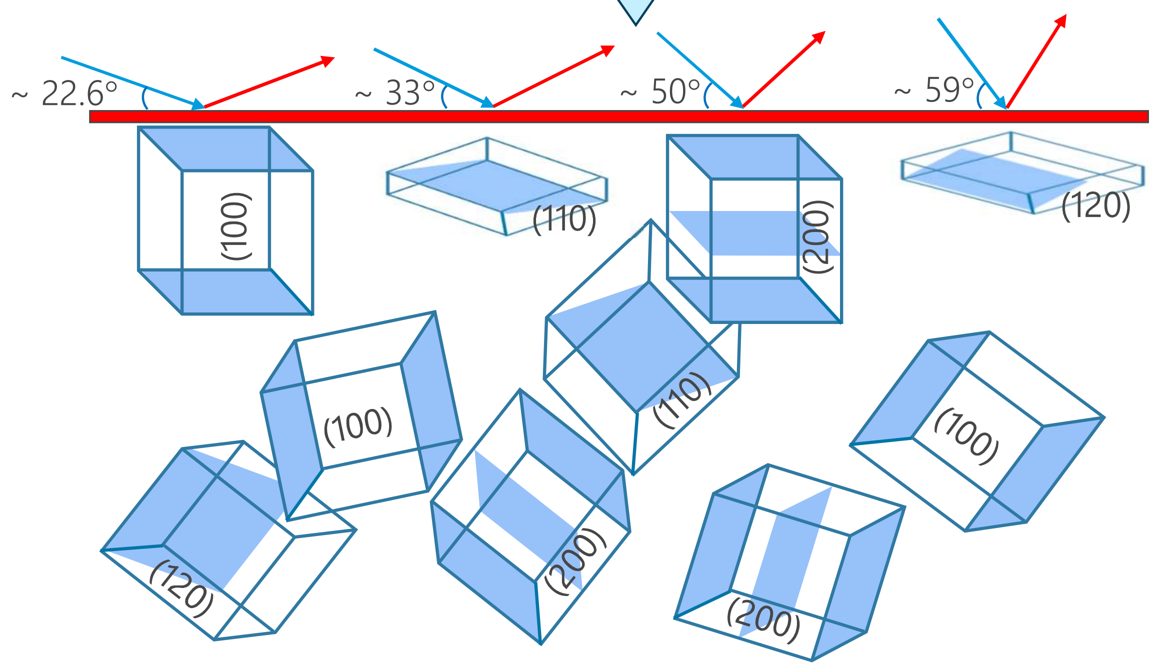
Visualizing diffraction peaks

a = 2Å; λ = 1.54Å

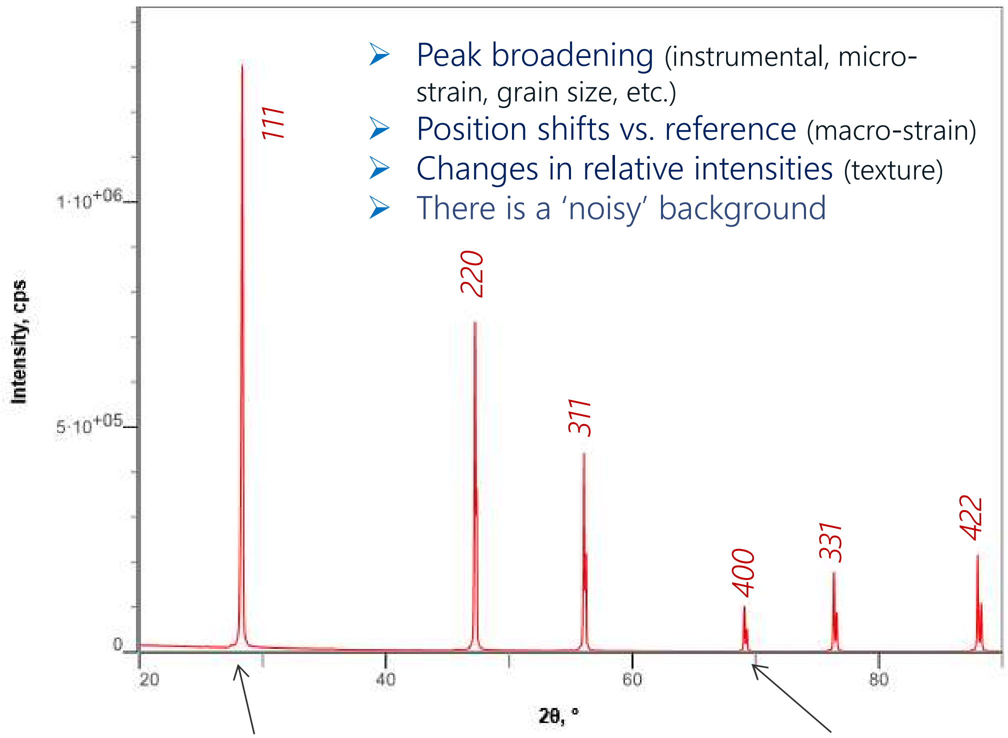
hkl	d	Sin(θ)	θ
100	2.00	0.38	22.64
110	1.41	0.54	33.00
200	1.00	0.77	50.36
120	0.89	0.86	59.40



As 2θ increases, lattice planes with appropriate d-spacing satisfy Bragg's law and diffract from favorably oriented crystallites.



Experimental powder diffraction pattern from Si



Low 2θ: the Kα_1/Kα_2 doublet is unresolved

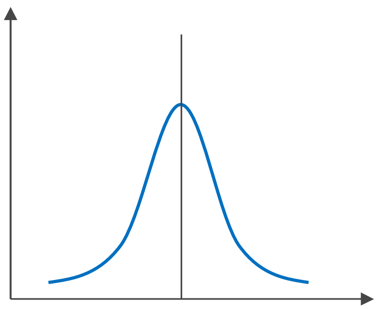
Kα1-Kα_2 splitting is resolved at high angles

Peak position

- Lattice parameter
- Wavelength

$$2d\sin\theta = n\lambda, (n = 1,2,3\cdots)$$
$$d = f(\mathrm{a}, \mathrm{b}, \mathrm{c}, \alpha, \beta, \gamma)$$

- Instrument related systematic errors
  - Flat sample
  - Sample displacement
  - Absorption
  - Axial divergence
  - Misalignment



Peak width

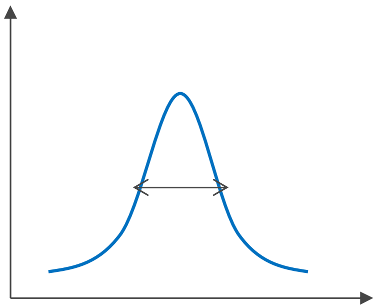
- Crystallite size

$$D = \frac{K\lambda}{\beta \cos\theta}$$

- Lattice distortion

$$e = \frac{\beta}{4 \tan\theta}$$

- Instrument related broadening
  - Resolution
  - Flat sample
  - Absorption
  - Axial divergence
  - Misalignment



Peak intensity

- Crystal structure

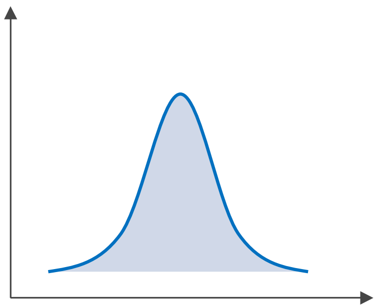
$$I_{hkl} \propto |F_{hkl}|^2 K_{\mathrm{LP}}$$

$$F_{hkl} = \sum_{j=1}^{N} f_j e^{-B_j(\sin\theta_{hkl}/\lambda)^2} e^{2\pi i(hu_j+kv_j+lw_j)}$$

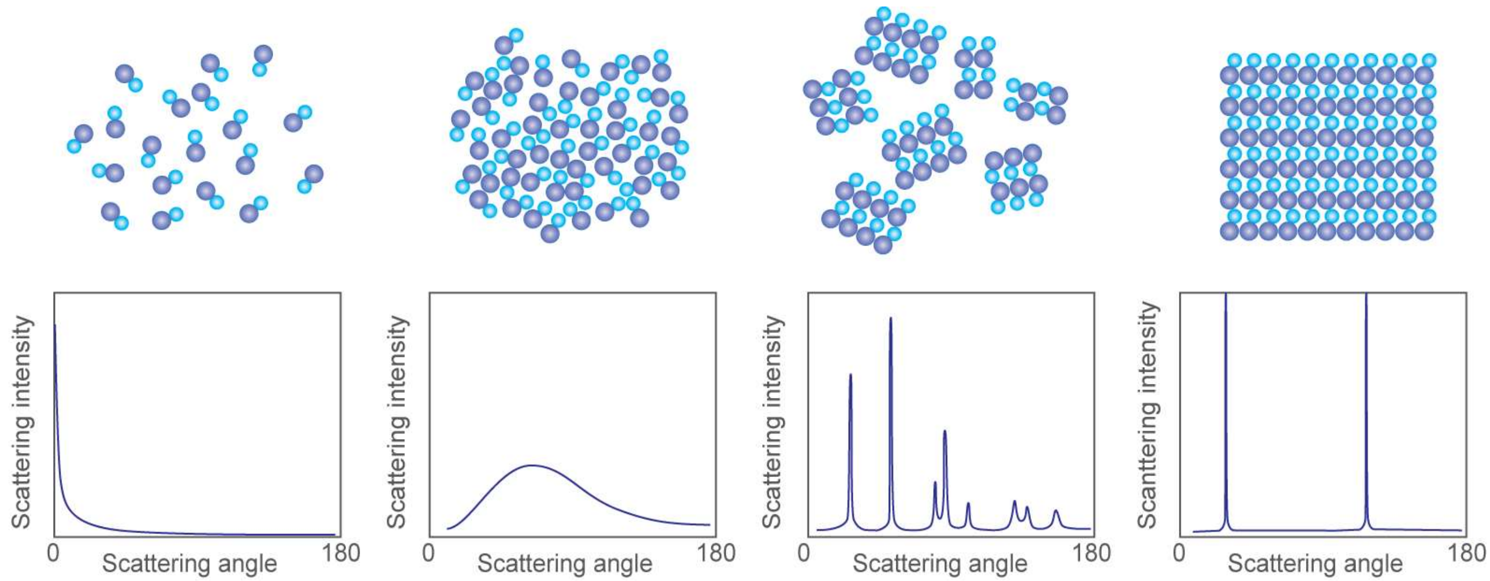
- Preferred orientation (March-Dollase)

$$P_{hkl} = \frac{1}{m_{hkl}} \sum_{j=1}^{m_{hkl}} \left(r^2 \cos^2 \alpha_j + r^{-1} \sin^2 \alpha_j\right)^{-3/2}$$

- Particle statistics



Types of diffraction patterns



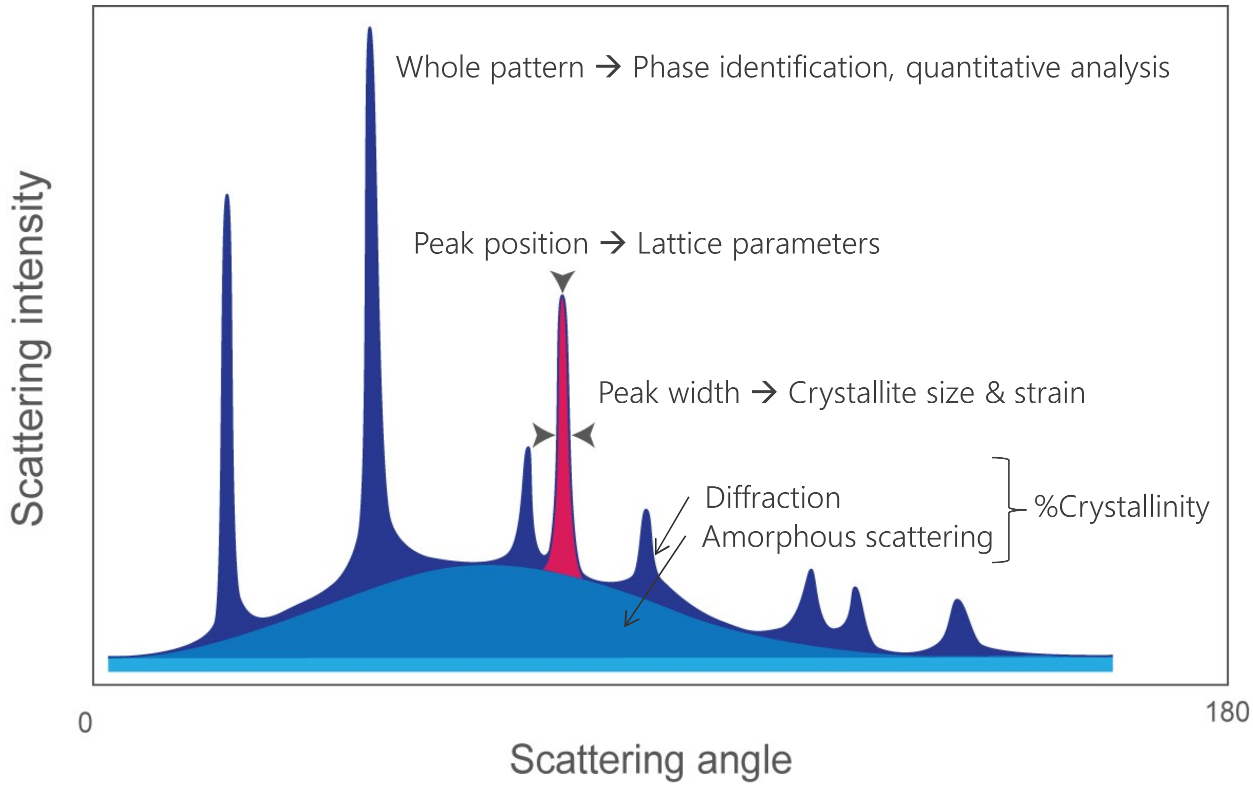
Gas

Liquid / amorphous

Powder / polycrystalline

Single crystal

XRD pattern / Sample information

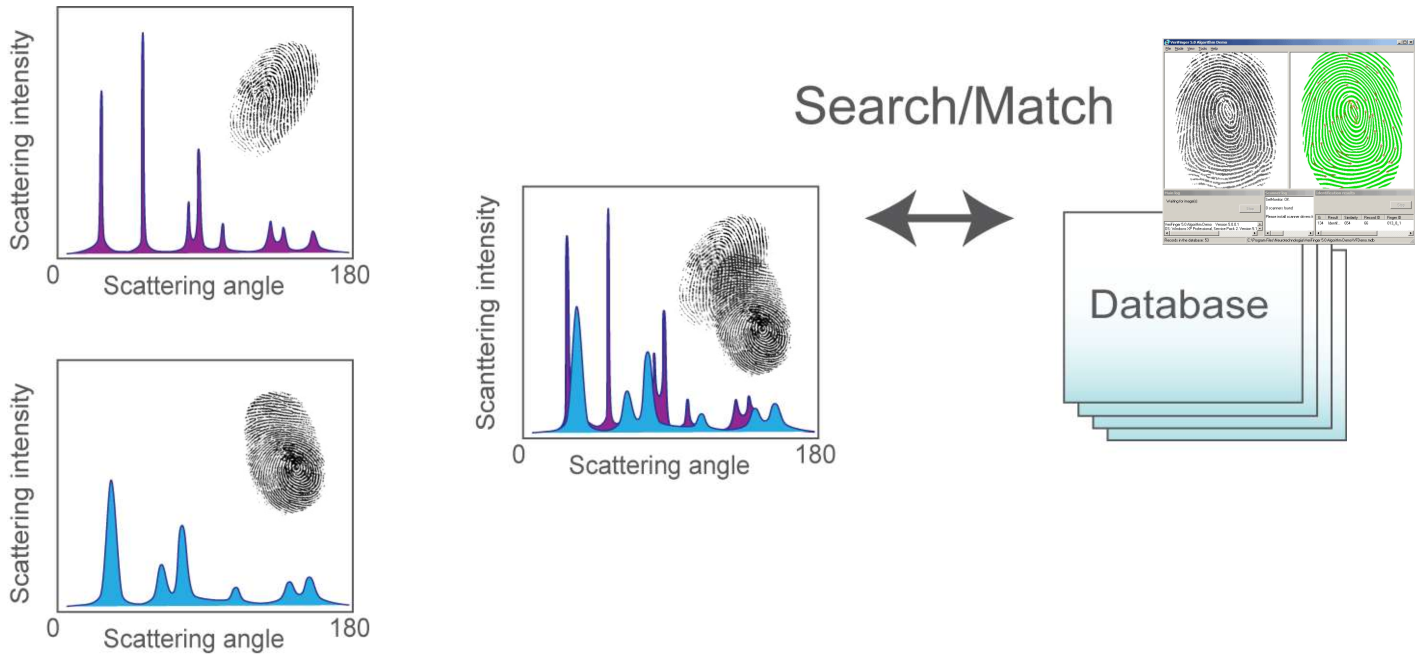


Phase identification – Search/Match

Powder XRD identifies crystalline phases from the **positions** and relative **intensities** of diffraction peaks.

- ✓ Database of reference patterns
- ✓ Experimental diffraction data
- ✓ Search/Match software

Phase identification



Crystallographic databases

- ICDD（International Centre for Diffraction Data）:
  - ✓ PDF-5+: 1,126,200+ entries (1-year license)
  - ✓ PDF-4/Axiom: 114,000+ entries (3-year license)
  - ✓ PDF-2: 369,200+ entries (5-year license)

- ICSD（Inorganic Crystal Structure Data）

FIZ Karlsruhe – Leibniz Institute for Information Infrastructure

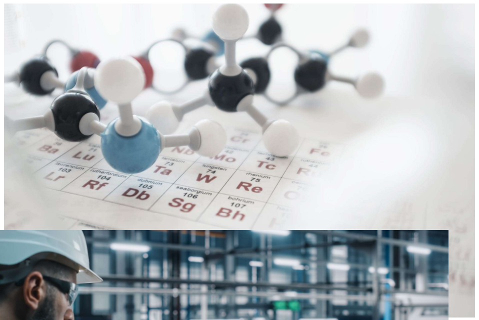
Structure Database of Inorganic Crystal Compounds

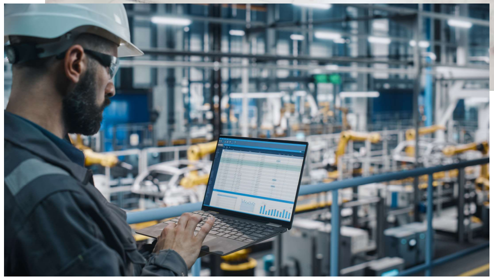
- ✓ 318,000+ entries

- Crystal Open Database（COD）: open-access

Administrated by Journal of Applied Crystallography

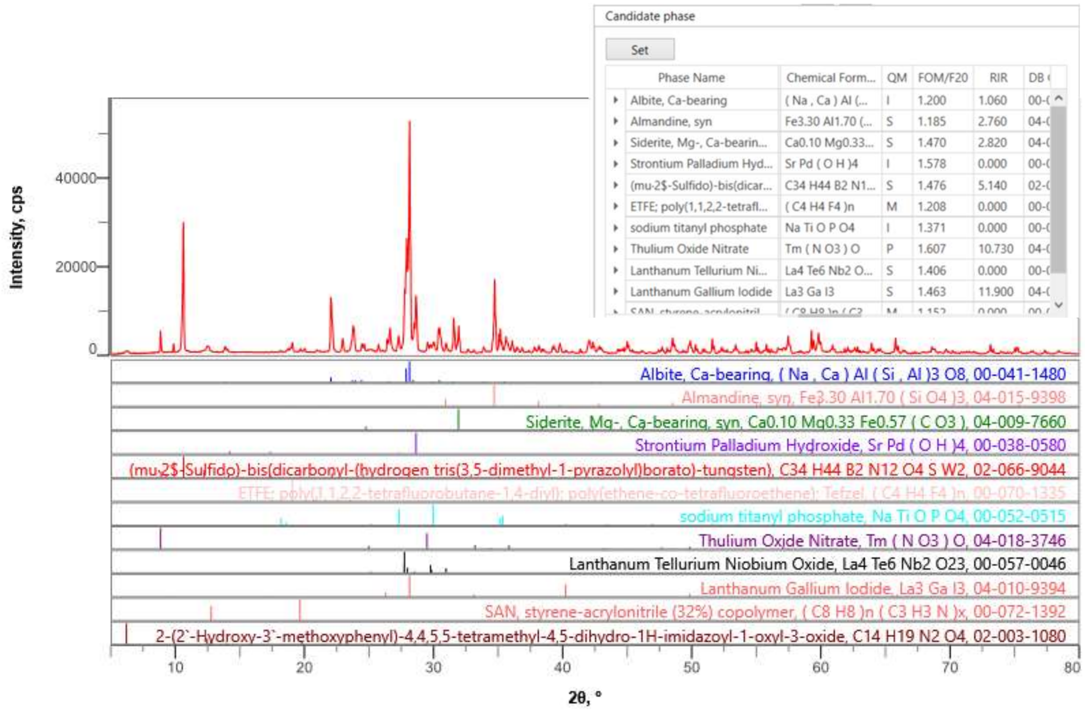
- ✓ 530,000+ entries

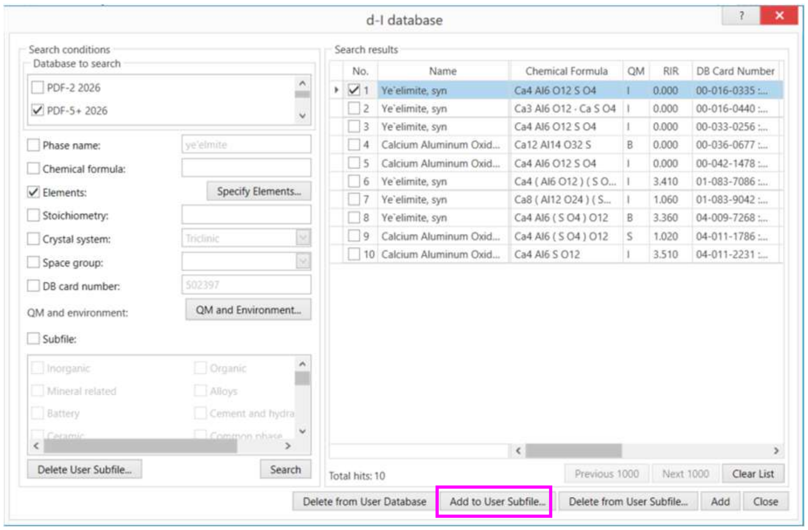




User subfiles

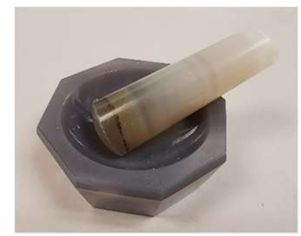
Search/Match returns too many phases – how do we narrow to the likely ones

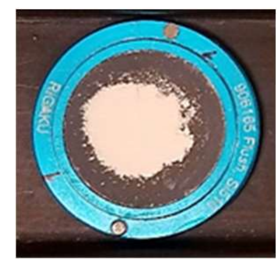




Sample preparation for powder XRD

- Use finely ground powder (< 45µm / 325 mesh) to produce a flat surface and allow X-rays penetration.
- Ensure the sample surface is level with the holder rim.
- The sample holder cavity should be completely filled, unless using a low background holder).

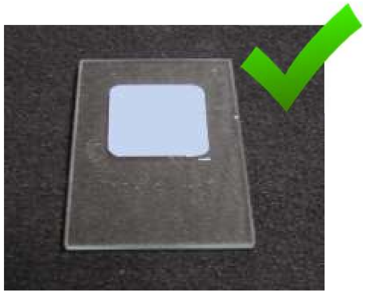




Sample preparation

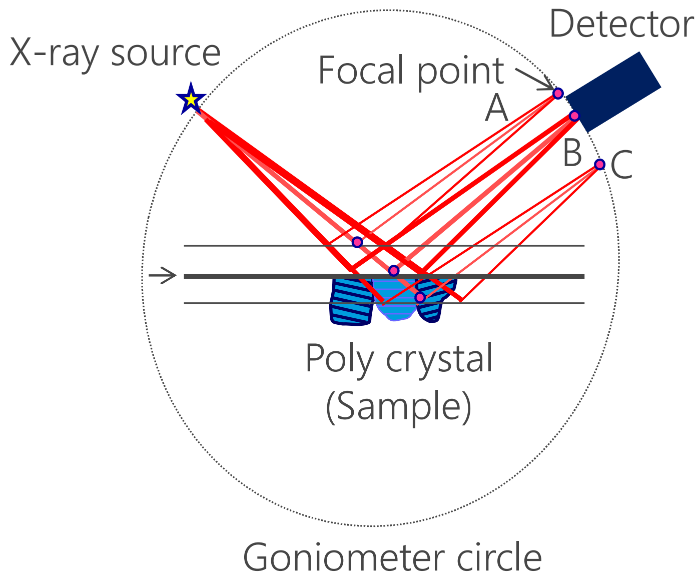
Common factors and errors

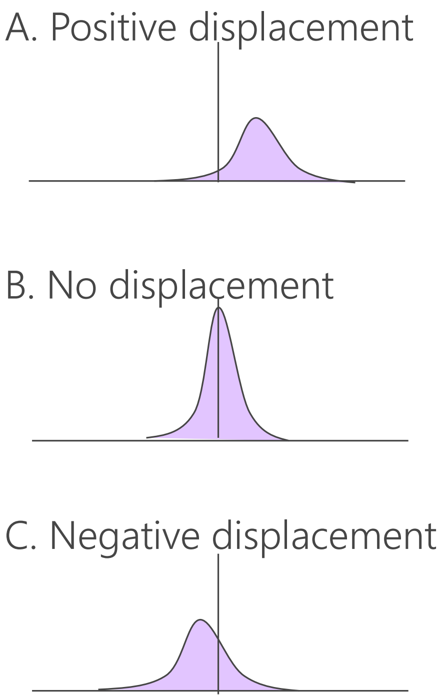
- Sample height displacement
- Sample flatness
- Sample particle size
- Preferred orientation
- Sample transparency



Sample displacement

- The biggest source of error
- Peak position shifts ~ 0.02° for every 30 µm
- $\Delta 2\theta = 2h\cos\frac{\theta}{R},$ where R – goniometer radius.

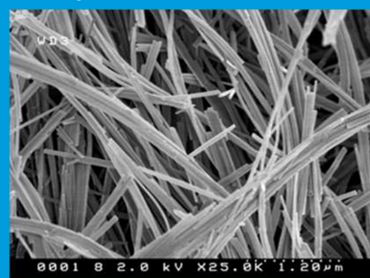




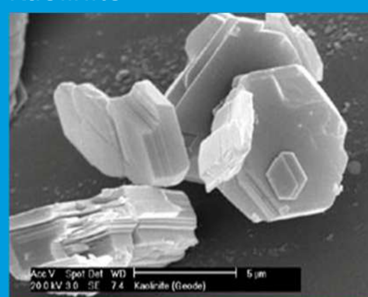
Preferred orientation

- Common in samples with plate/needle/fiber morphologies
- Alters relative peak intensities
- Can compromise phase ID
- Certain sample preparation methods can increase the effect

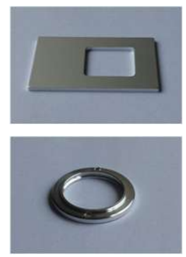
Halloysite



Kaolinite

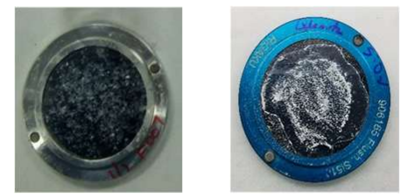


Back loading



Side loading

- Randomized mounting
- Slurry mounting with acetone

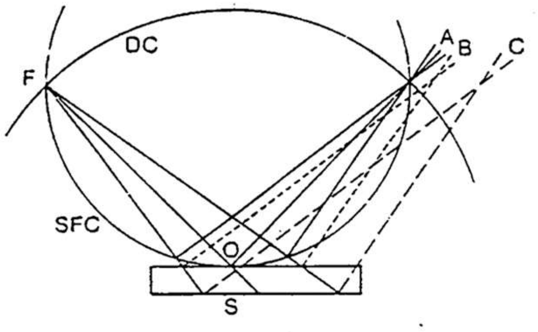


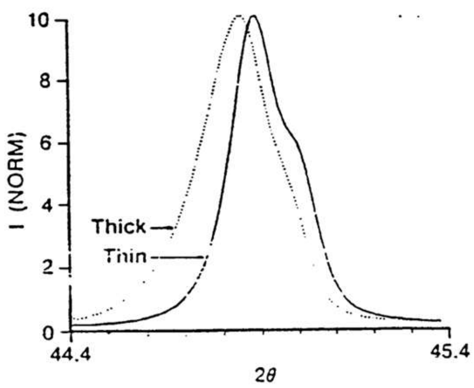
Spinning

Transmission geometry

Sample transparency

- Diffracted intensity arises form multiple depths
- Common in low-attenuation (low-μ) materials: organics, polymers, low-Z compounds
- Shifts peaks (to lower 2θ) and makes them asymmetric



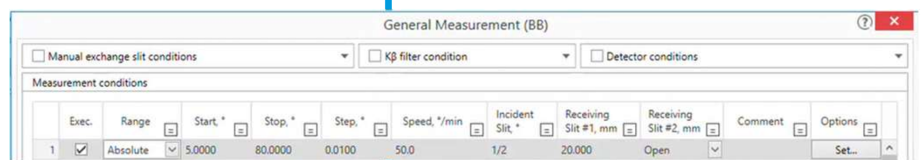


Solutions:

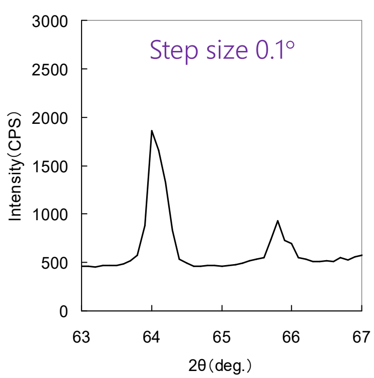
- ➢ Thin sample layer
- ➢ Transmission geometry

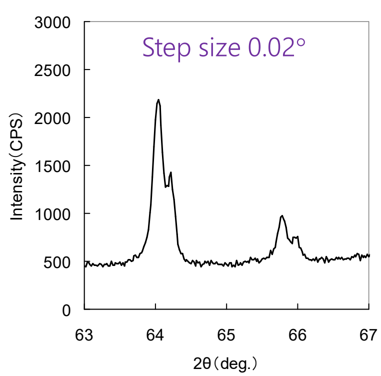
Data collection strategies

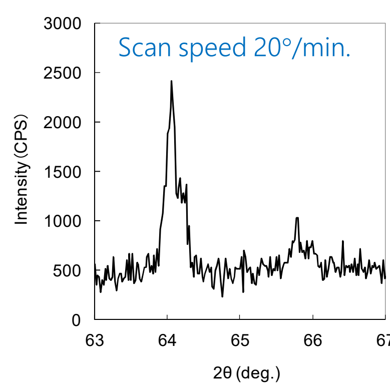
- ✓ Pre-scan your sample (5-80° 2θ, **0.02°** step, 50°/min).
- ✓ Adjust the scan range, step size and speed.
  - Step size: FWHM x 1/10
  - Adjust the scan speed to have high diffraction intensities and decrease statistical fluctuation.

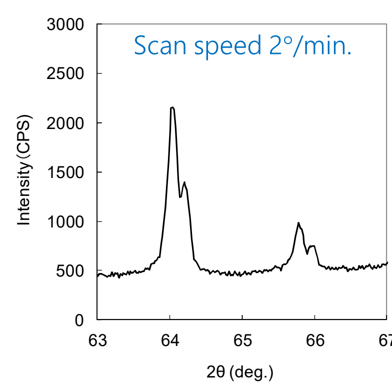


Scan duration: 2 min.







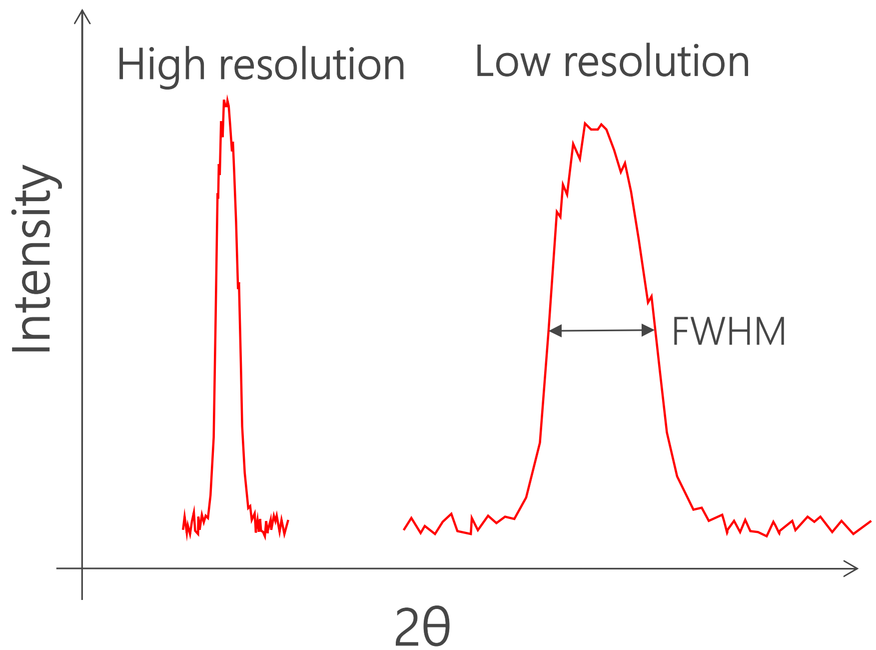


Data quality

- Resolution (peak width and shape)
- Peak-to-background ratio
- Signal-to-noise ratio

Peak resolution

- = Angular resolution
- Diffraction maps $d \rightarrow 2\theta$



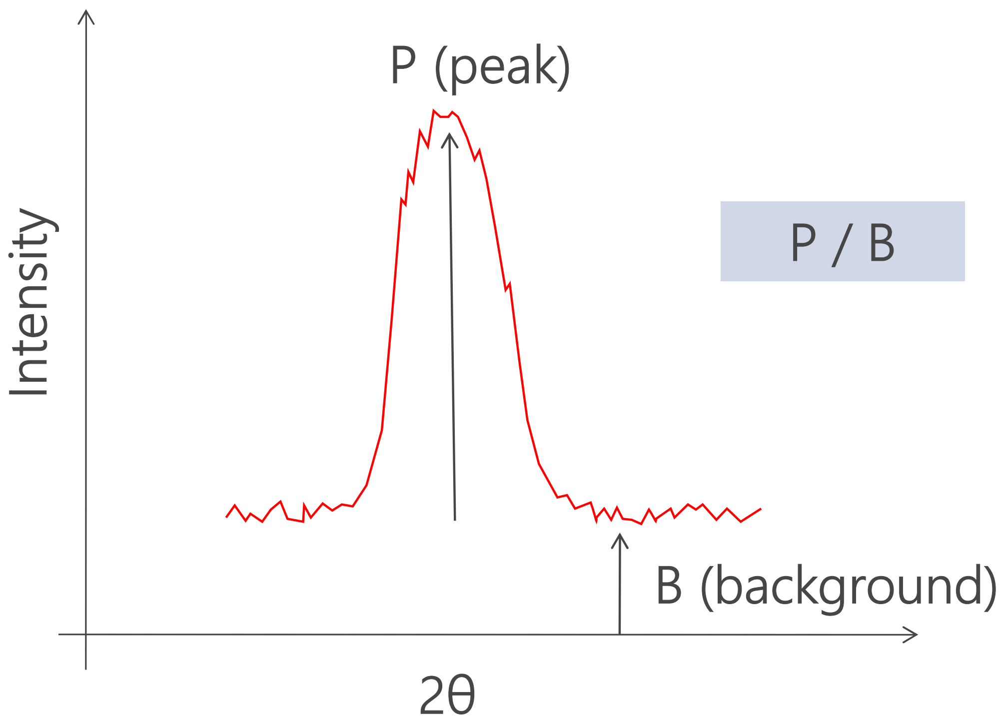
Peak List

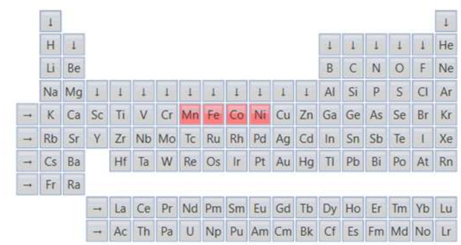
Only displayed in chart   Phase name:

No.	2θ, °	d, Å	Height, cps	FWHM, °
1	25.244(5)	3.5250(6)	21862(191)	0.153(4)
2	27.372(3)	3.2556(4)	8049(123)	0.111(3)
3	36.019(3)	2.49139(18)	3845(90)	0.100(3)

Peak-to-background ratio

Contrast of a peak against the baseline

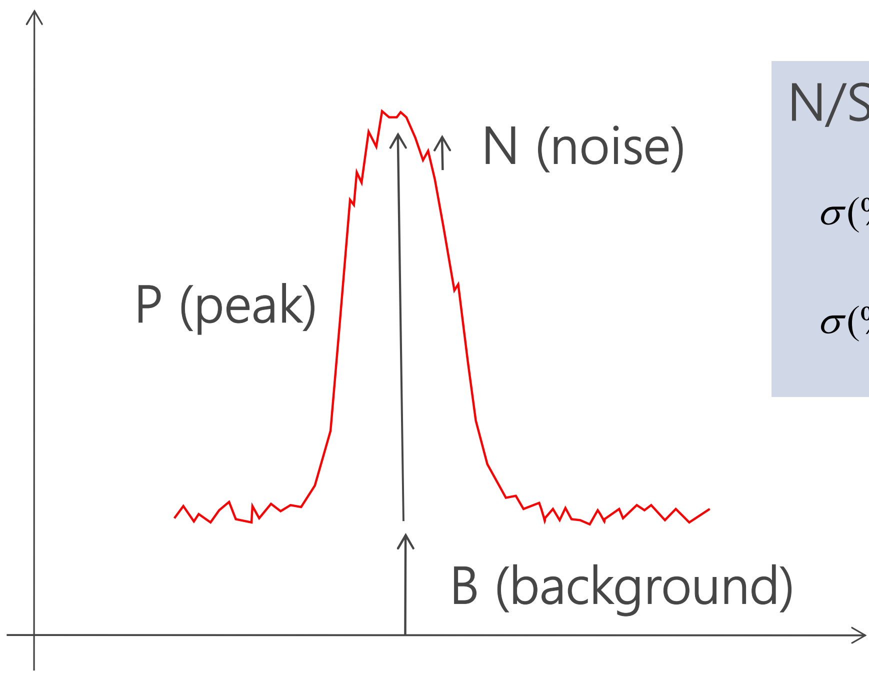




Use P/B to compare optics choices and background reduction

Signal-to-noise ratio

Statistical confidence of the peak

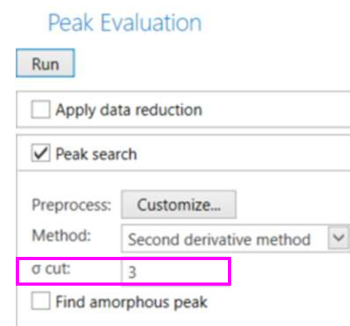


Use SNR to decide if a weak peak is truly detected

N/S: σ %

$$\sigma(\%) = \frac{1}{\sqrt{P}}\sqrt{\frac{P+2B}{P}} \times 100$$

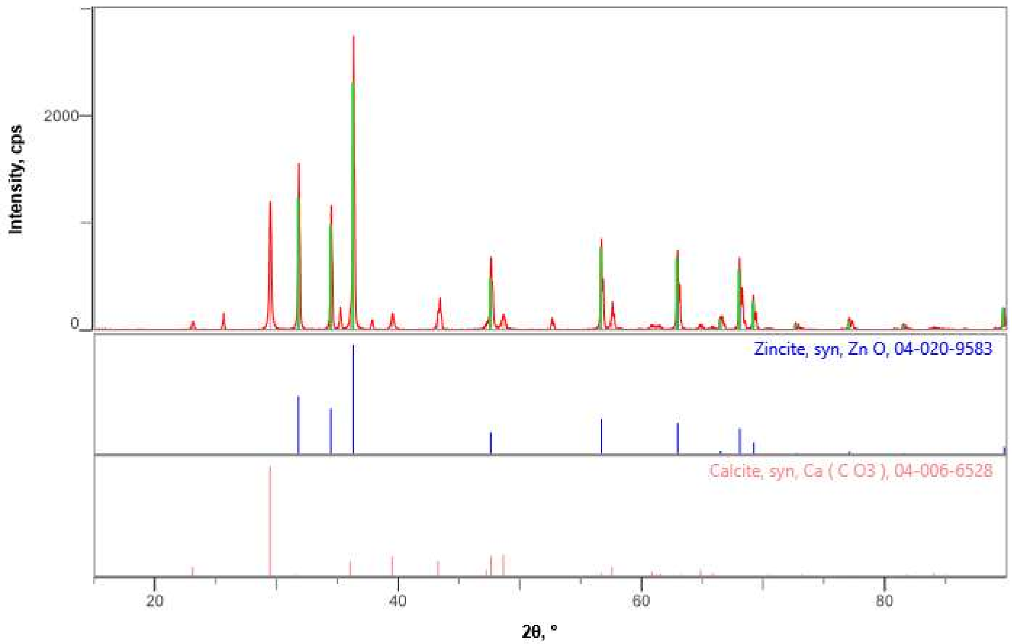
$$\sigma(\%) = \frac{1}{\sqrt{P}} \times 100 \quad \text{(B is negligible.)}$$



- Count longer (SNR ∝ √time)
- Repeat N scans and average (SNR ∝ √N)

Phase identification / Pattern matching

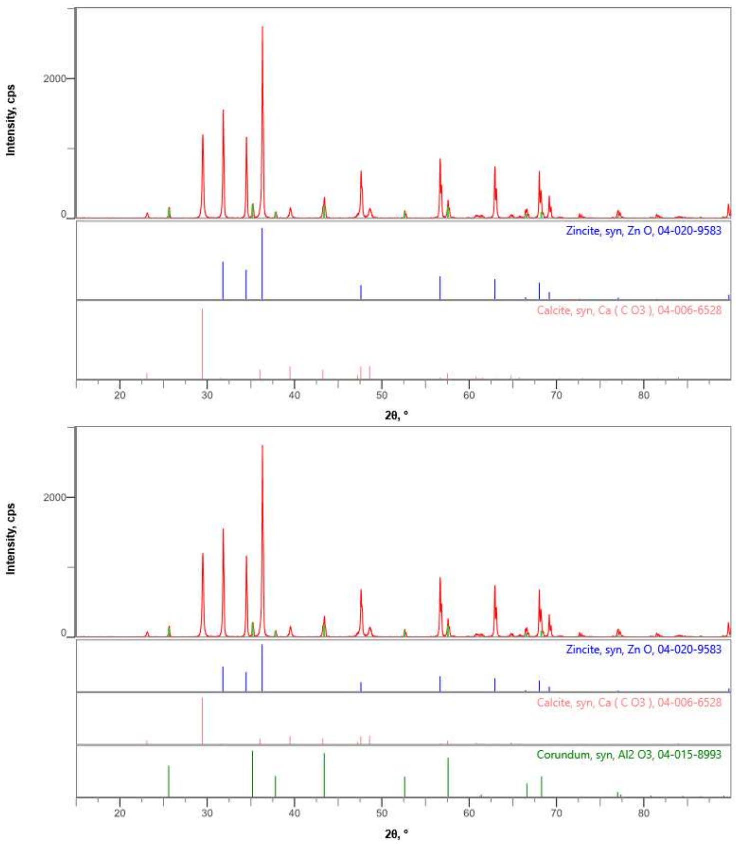
All reference peaks must be present in the data; unmatched peaks indicate no valid match.



Exceptions

- Preferred orientation (check if absences are *hkl*-specific)
- Bad statistics / coarse grains (regrind to reduce size)
- Below detection limit (very weak peaks not visible above background or overlap)

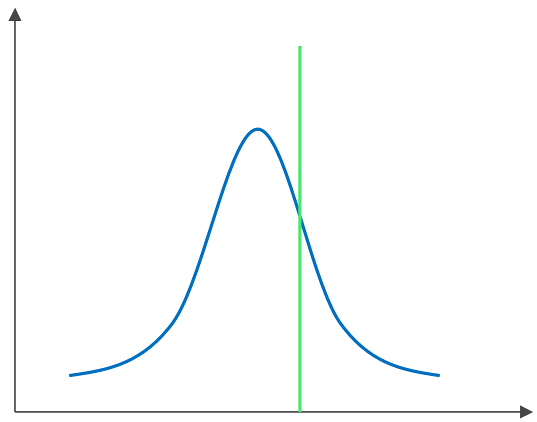
Phase identification / Additional peaks



Possible reasons

- Extra peaks may indicate an impurity in the sample (continue searching for references)
- Very small peaks can be spectral artifacts (e.g. K*β*, W L-lines (verify with the Kα, Kβ, Lα Lines Display cursor)

Phase identification / Peak shift



Diagnosing peak shifts

- Larger at low angles, smaller at high angles ($\propto \cos\theta$)
  - Likely: sample displacement
- Grows with angle ($\propto \tan\theta$)
  - Likely: true lattice-parameter change (thermal expansion/contraction, chemical composition)
  - Direction: expansion $\rightarrow$ lower 2θ; contraction $\rightarrow$ higher 2θ

Questions?

We'll follow up with your questions.

Recording will be available tomorrow.

Register for webinar.

Rigaku

Practical XRD with Confidence

Episode 2 – Quantitative XRD in Practice: From Phase Estimates to Defensible Results

Wednesday, May 20, 2026, at 1 pm CDT

Presenter: Akhilesh Tripathi, PhD

Host: Tom Concolino, PhD