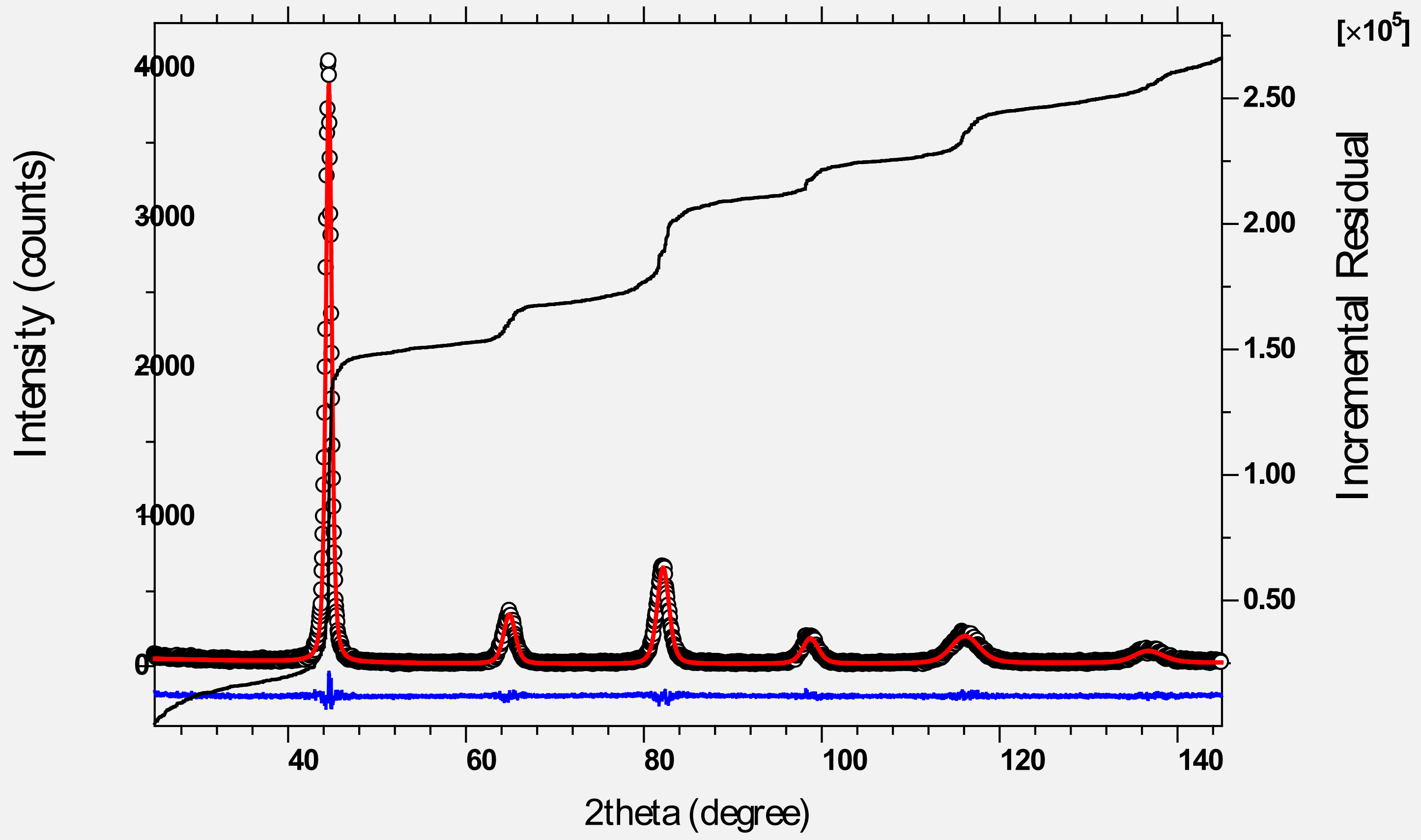
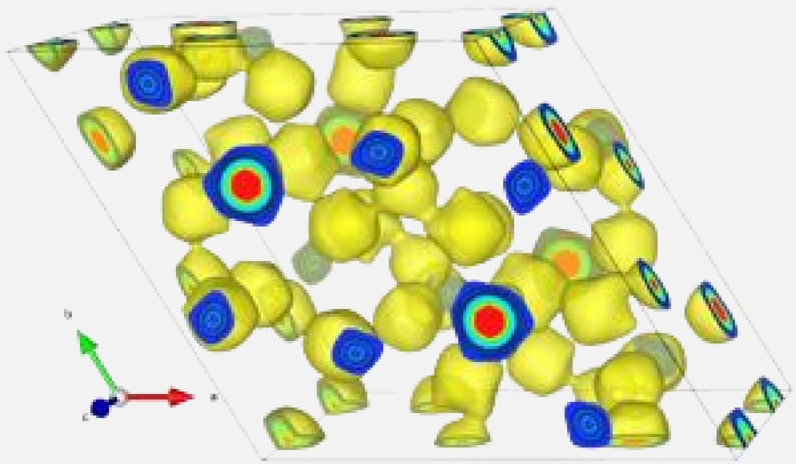
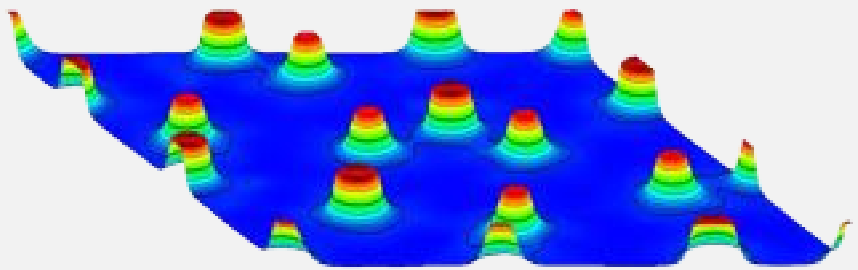






X-ray Diffraction Analysis for Advanced Research (Lec-02)

Dr. Maykel T. E. Manawan // LECTURER & RESEARCHER

Course Materials

- Crystallography
  - Symmetry & crystal system
  - Bravais lattices
  - Point group & space group
  - Miller indices
- Diffraction
  - Diffraction by atom and plane of atoms
  - Miller indices and diffraction
  - Structure Factor
  - Instrumentation
  - X-ray generation
  - Beam path (optics)
    - X-ray monochromatizating
  - Instrument configuration
    - Optimum setup

Brief overview

- Sample Preparation
  - Particle/Crystallite Size
  - Homogeneity
  - Texture/Preferred Orientation
- Data Acquisition
  - Angular Range
  - Step Size
  - Counting Time
- Qualitative Analysis (Identification)
  - Data base
  - Phase Identification
- Quantitative Analysis (Rietveld Analysis)
  - Intensity Equation
  - Profile Function
    - Instrumental function
    - Sample physical function
  - R-indices (How good is good enough)
- Qualitative Analysis
  - Practices with QualX (Sieve+, Match!, JADE, EVA, HSP, PDXL)
    - Sample : mixture of Al_2O_3, CaF_2, Zincite (Quantitative Analysis Round Robin (QARR) sample from International Union for Crystallography (IUCr))
- Quantitative Analysis
  - Practices with MAUD, GSAS, Fullprof, Profex (JADE, TOPAS, HSP, PDXL)
    - Sample : mixture of Al_2O_3, CaF_2, Zincite (QARR sample from IUCR)
- Advanced Analysis (optional)
  - Practices with PM2K (Whole Powder Pattern Modelling) => Microstructure
    - Sample : CeO_2 (Size Strain Round Robin (SSRR) sample from IUCR)
  - Practices with Rietan-FP/GSAS/Fullprof/Z-Rietveld
    - Sample : CeO_2/Fa-apatite - electron density
    - Sample : Ca_3LiRuO_6 - magnetic phase and nuclear density

XRD user wanna be

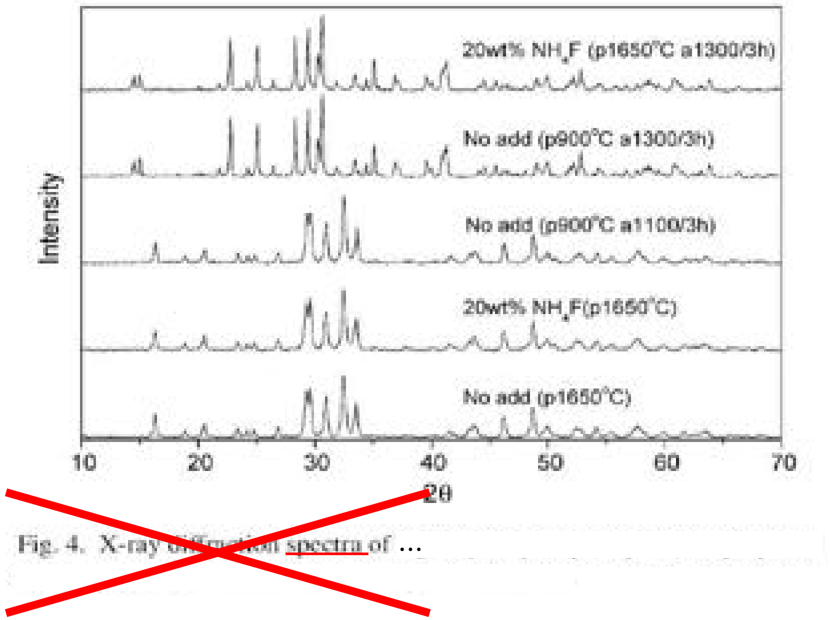
What is diffraction?

- The type of diffraction we will talk about is more properly called ***Bragg diffraction***.
- It occurs when waves of a suitable wavelength interact with periodically ordered matter.
- In the chemical context “periodically ordered matter” usually means crystalline substances, but it may also be ordered arrangements of clusters or pores.

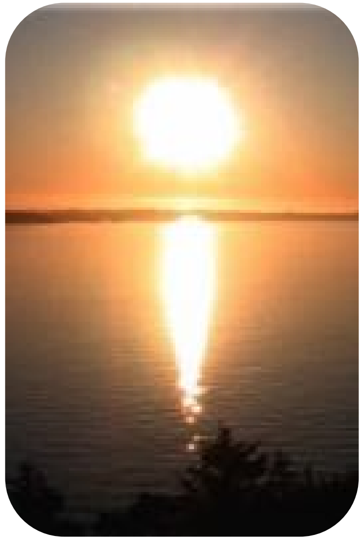
Note: Diffraction is not spectroscopy!

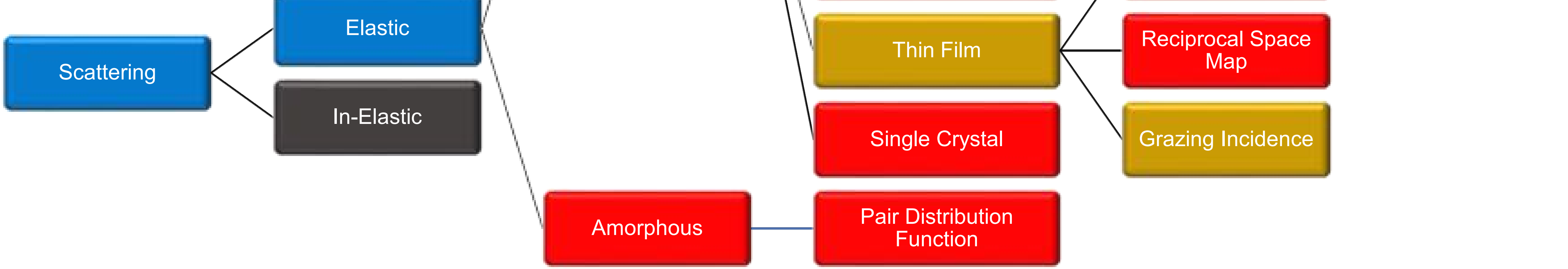
i.e. it is not
- “X-ray spectrum” (that’s a different method!),
- “XRD spectrum”,
- or “X-ray diffraction spectrum”,

but
- ***XRD pattern***,
- ***X-ray diffraction pattern***,
- or ***diffractogram***!



Scattering Techniques





X-Ray Scattering Techniques

Small angle

- Small Angle X-Ray Scattering (SAXS)
- X-Ray Reflectivity (XRR)
- Gracing Incidence X-Ray Scattering (GI-SAXS)

Wide angle

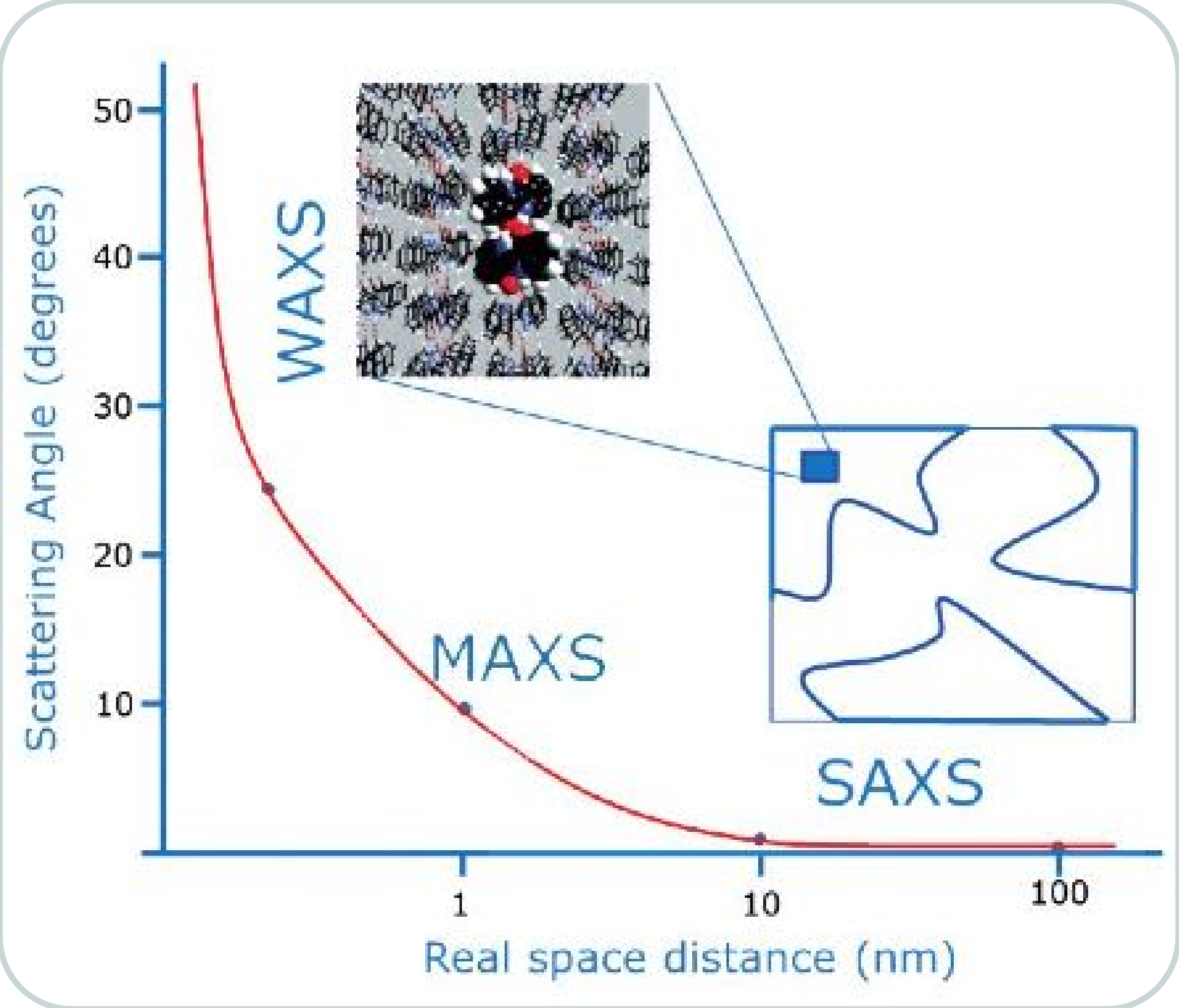
- X-Ray Diffraction (XRD)
- Gracing Incidence X-Ray Diffraction (GI-XRD)
- In-Plane X-Ray Diffraction (IP-XRD)
- Pair Distribution Function (PDF)
- Single Crystal

Selection Angle

- Reciprocal Space Map (RSM)
- Texture (Pole Figure, ODF)
- Residual Stress

In-situ

- Non ambient (time, temperature, pressure, etc)
- Battery (charge – discharge)

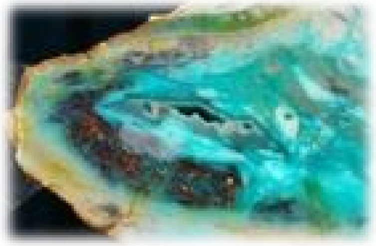
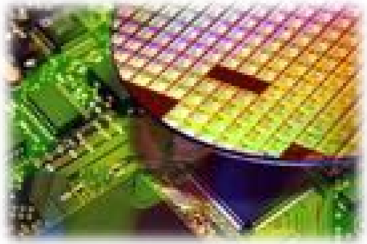
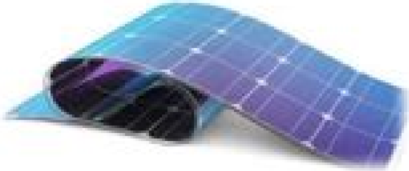
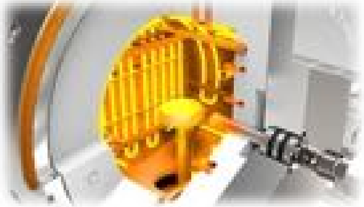


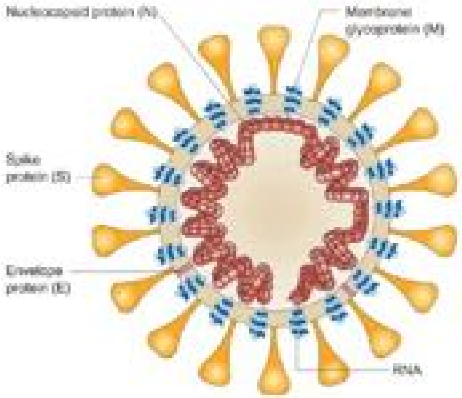
Sample Types

- **POWDER**
    - Common powder (PXRD, SAXS)
- **Bulk/Chunk**
    - Single/multi phase homogen (XRD, PF, Psi)
    - Multiphase non-homogen (micro-XRD, Psi)
- **Small quantity**
    - Precious material (XRD)
    - Nano material (XRD, SAXS, PDF)
- **Thin Film**
    - Epitaxy (XRR, HRXRD, RC)
    - Polycrystalline (XRR, XRPD, GI, Psi)
    - Textured (PF, Psi, RC, HRXRD, XRPD, XRR, GIXRD, IP-GI)
    - Amorphous (XRR)
- **Non-Ambient**
    - Material formation / structure changes
    - Heat treatment & annealing
    - Calcination & Sintering
    - (De)Hydration processes
    - Material changes during operation
- **Single Crystal (SCD)**
- **Amorphous / Short Range Order (PDF)**





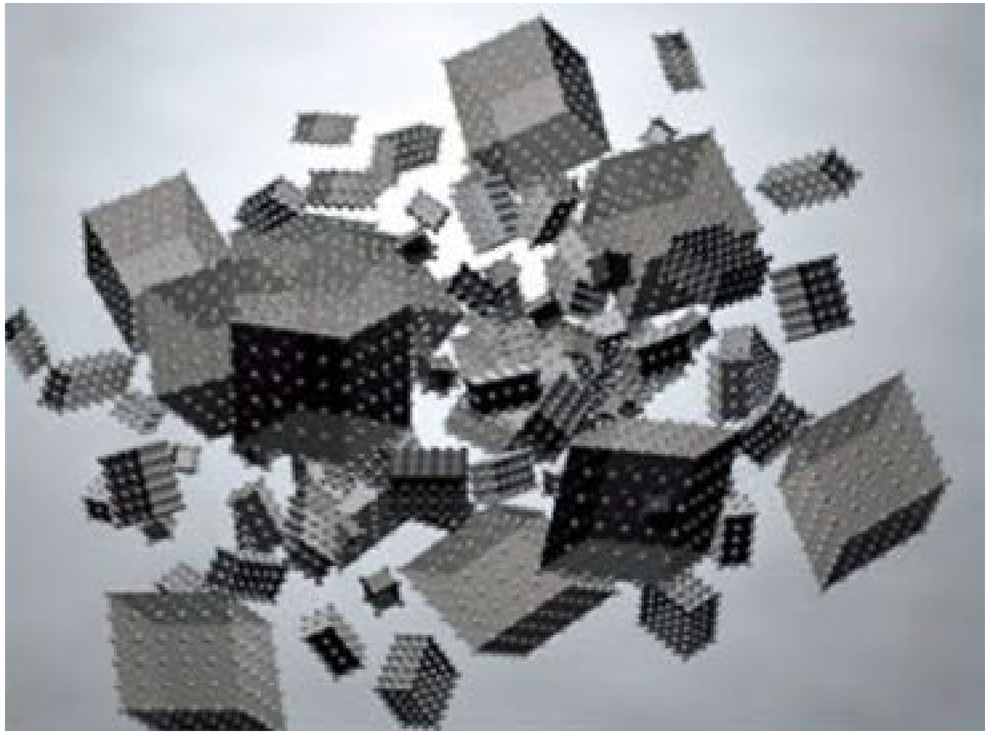








- What is Powder
- X-ray generation
- Data Acquisition
- Peak Profiles
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- Miscellaneous

Powder XRD

- What is a powder?



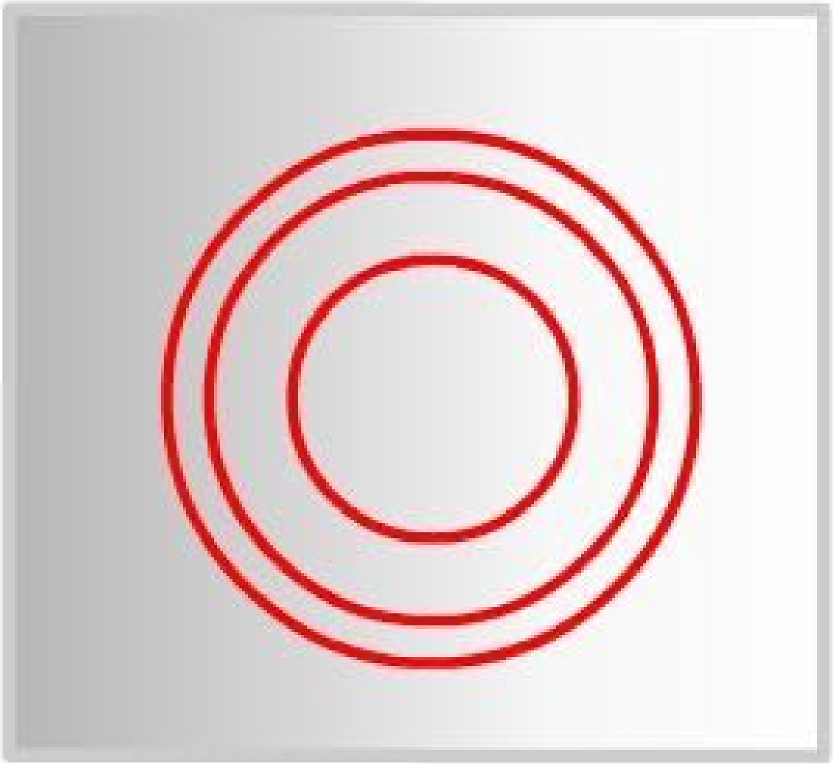
A collection of many, many very small crystals (also called “crystallites”)

A powder in XRD terms may not always be ‘powdered’…

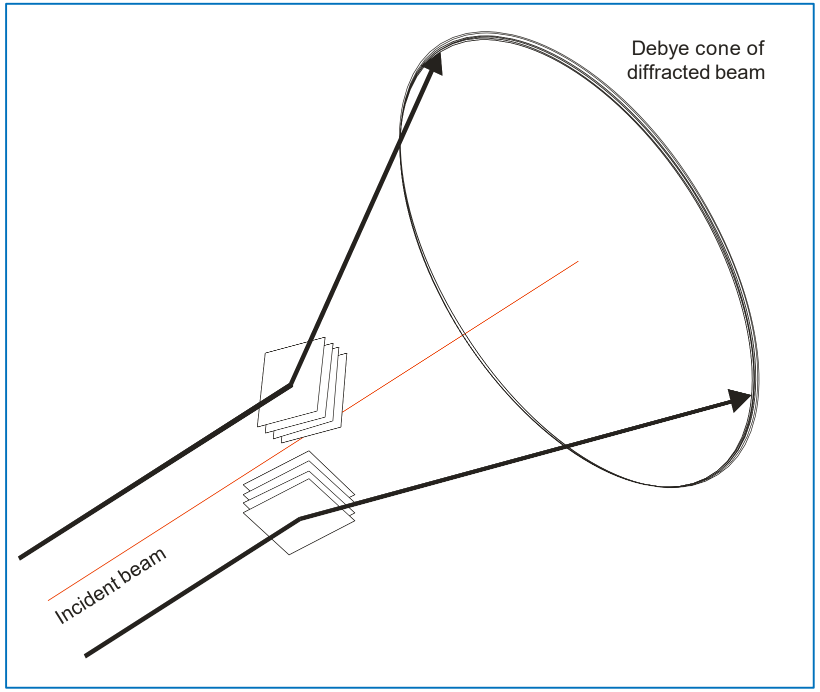
- Drug tablets (pressed into tablet form)
- Metals (crystallites are held together by grain boundaries)
- Ceramics
- Other multi-crystalline materials

“Powder” = Polycrystalline material

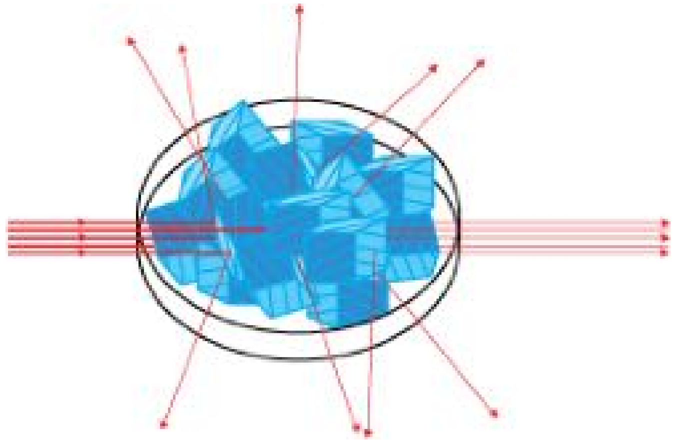
Powder XRD

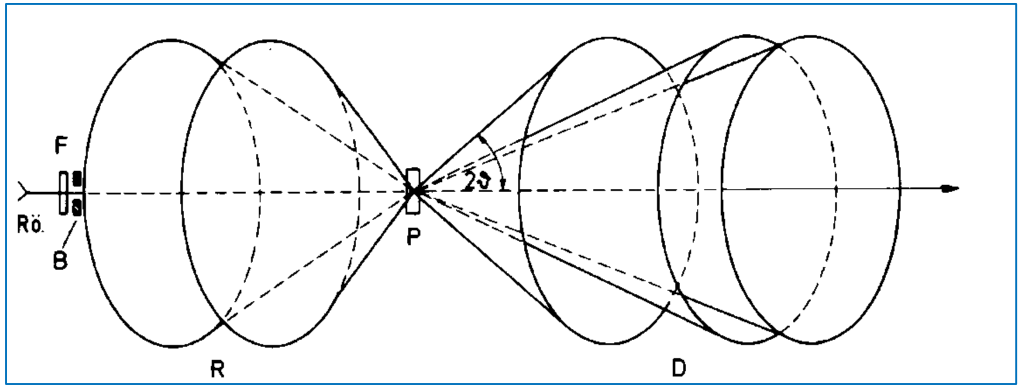


Powder XRD – Debye Cone



- Instead of giving spots, diffraction from a powder results in Debye cones or Debye rings
- Ideally, one needs 10^8-10^{10} crystallites in a completely random orientation for good statistics





Powder XRD – Particle Statistic

- Issue of graininess relates to particle statistics
- Particle statistics is what makes a powder a true powder!

Comparison of the particle statistics for samples with different crystallite sizes

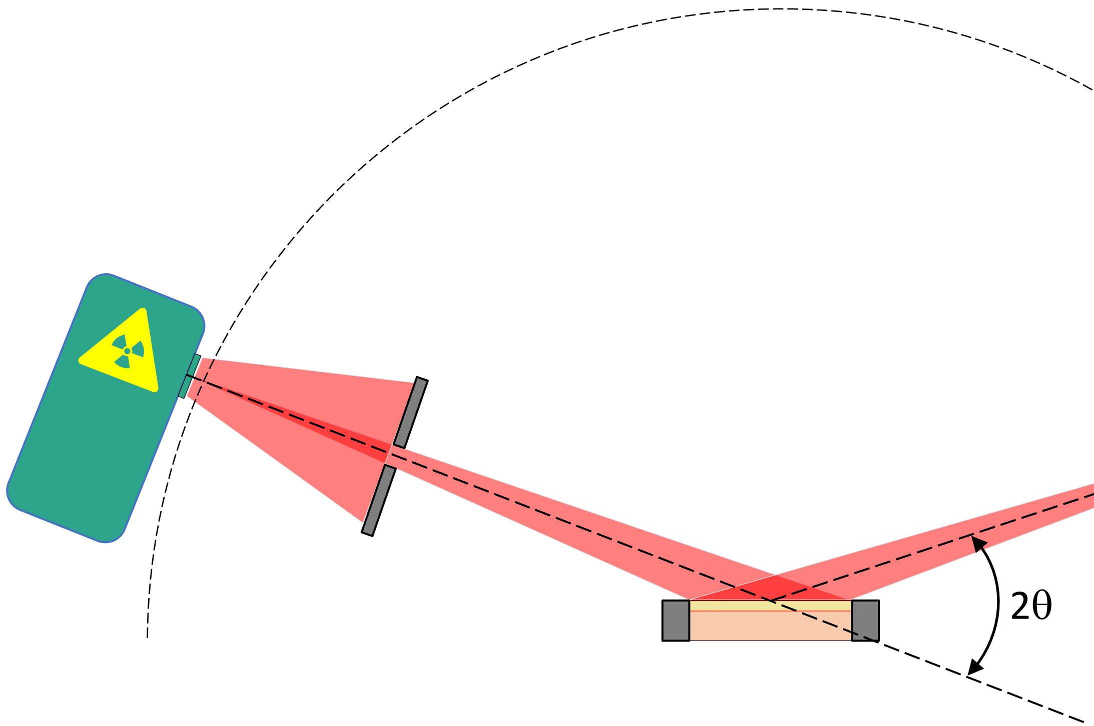
Diameter	40μm	10μm	1μm
Crystallites / 20mm³	5.97×10^5	3.82×10^7	3.82×10^{10}
No. of diffracting crystallites	12	760	38000

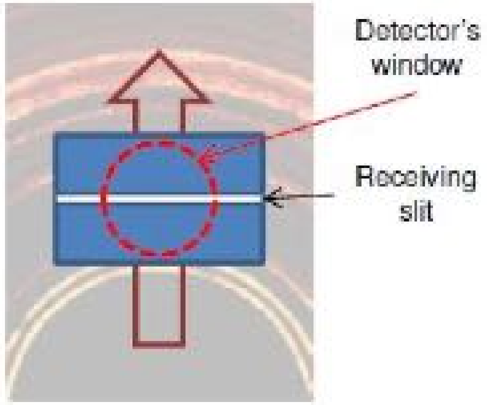
Particle size range	15-20μm	5-50μm	5-15μm	<5μm
Intensity reproducibility	18.2%	10.1%	2.1%	1.2%

Reproducibility of the intensity of the quartz (113) reflection with different crystallite sizes

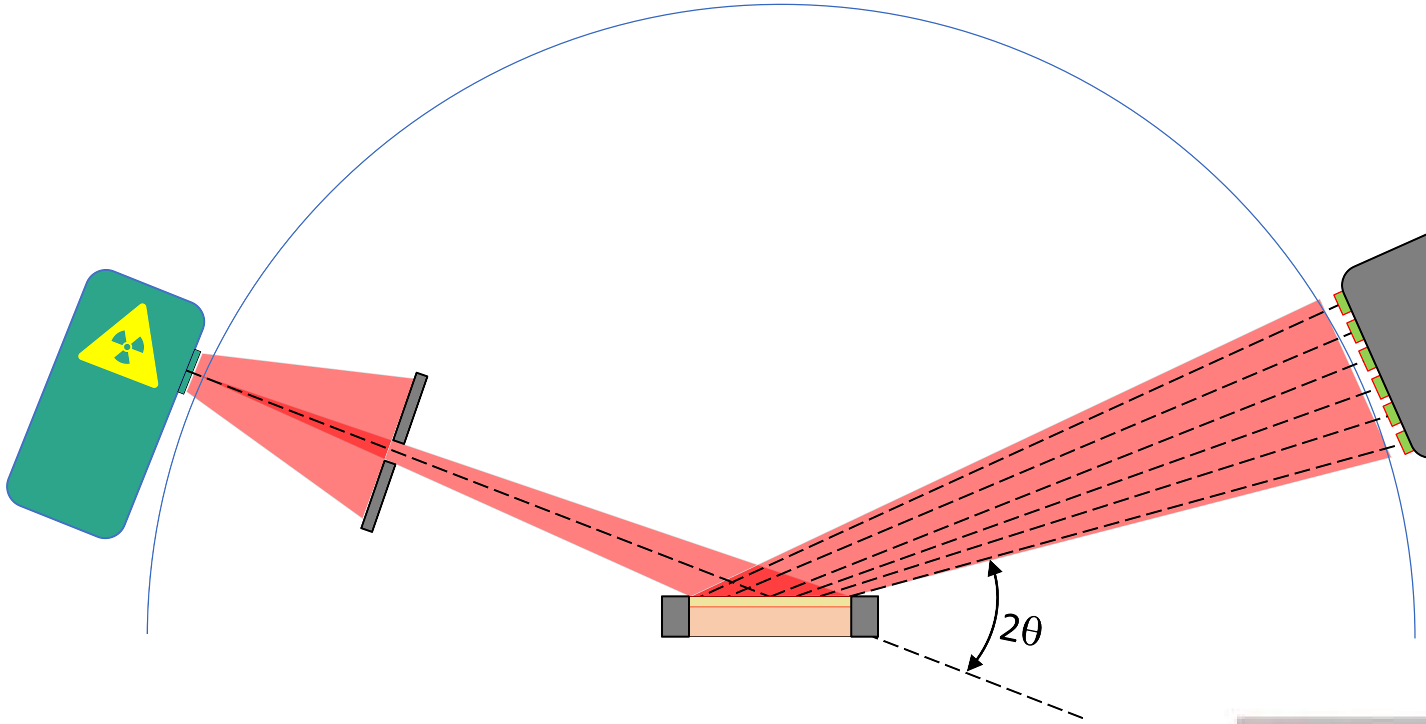
Courtesy of Dean Smith (1992)

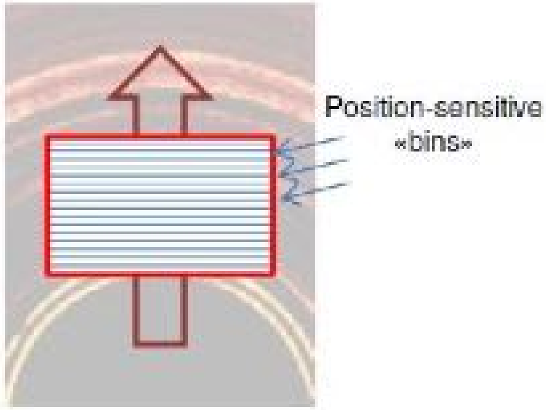
Detector – Scintillation (0D)



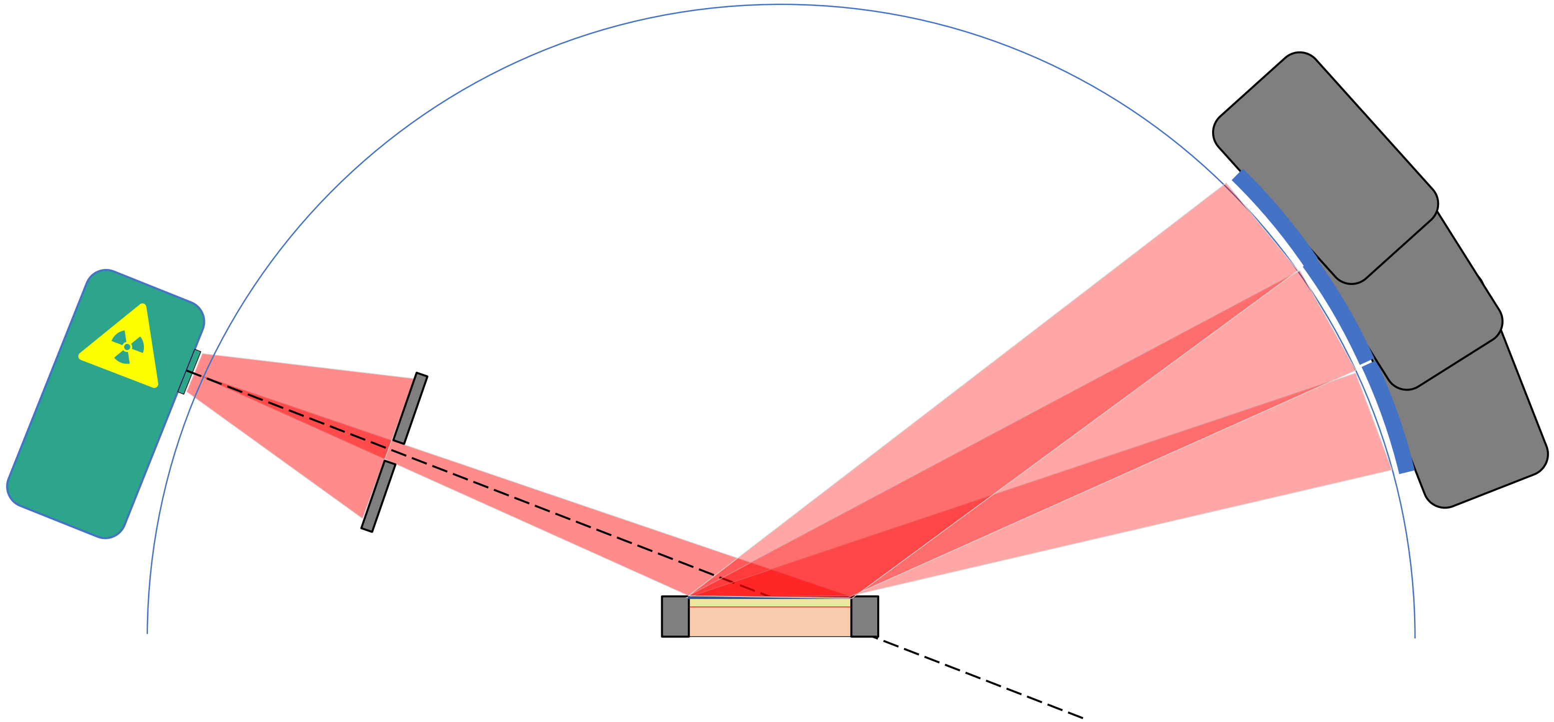


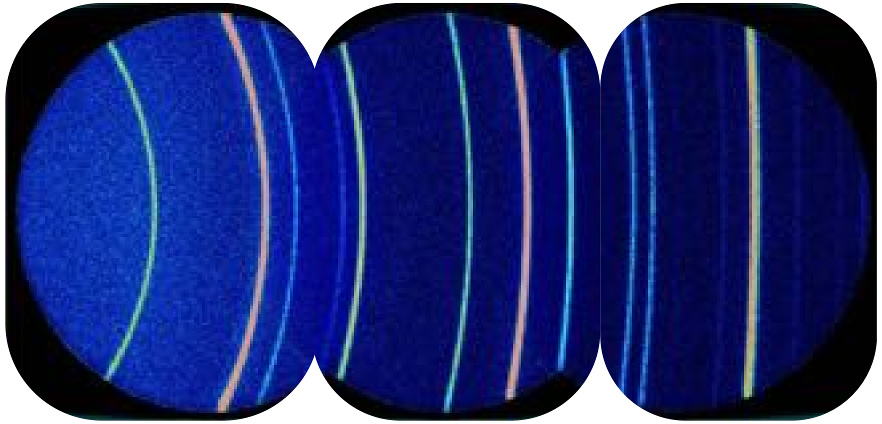
Detector – Position Sensitive Detector (1D)



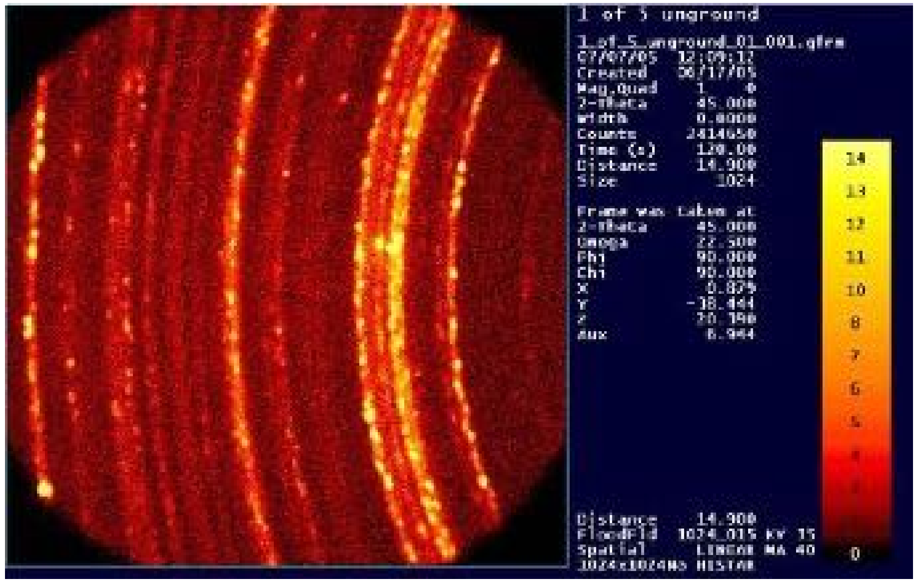


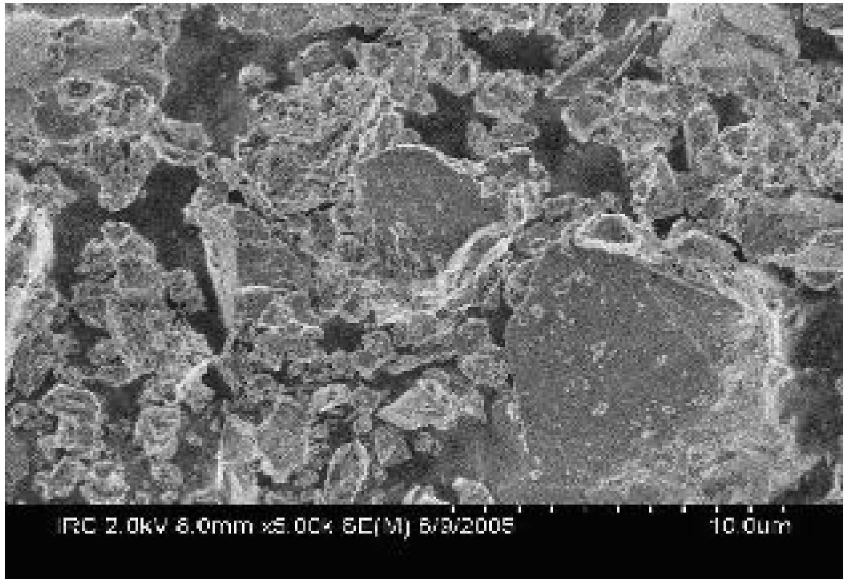
Detector – Position Sensitive Detector (2D)

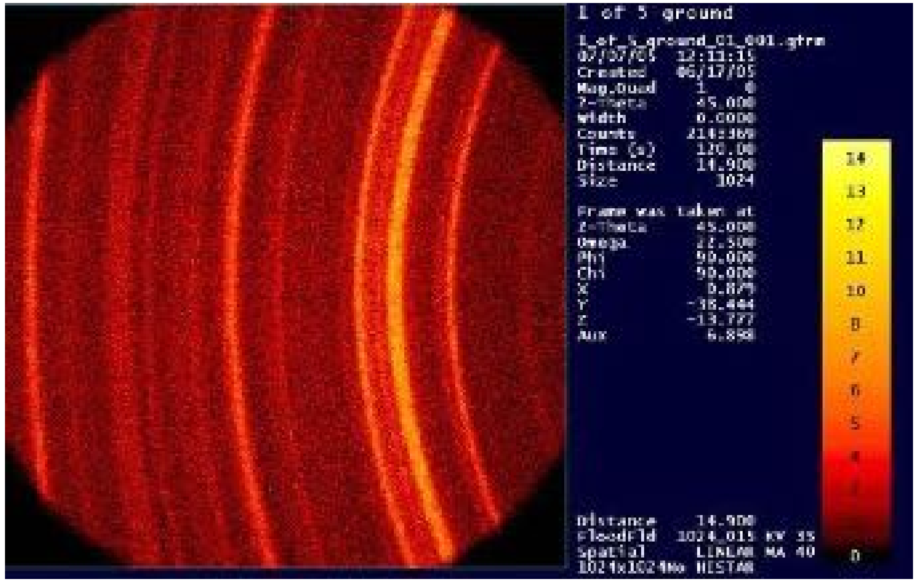


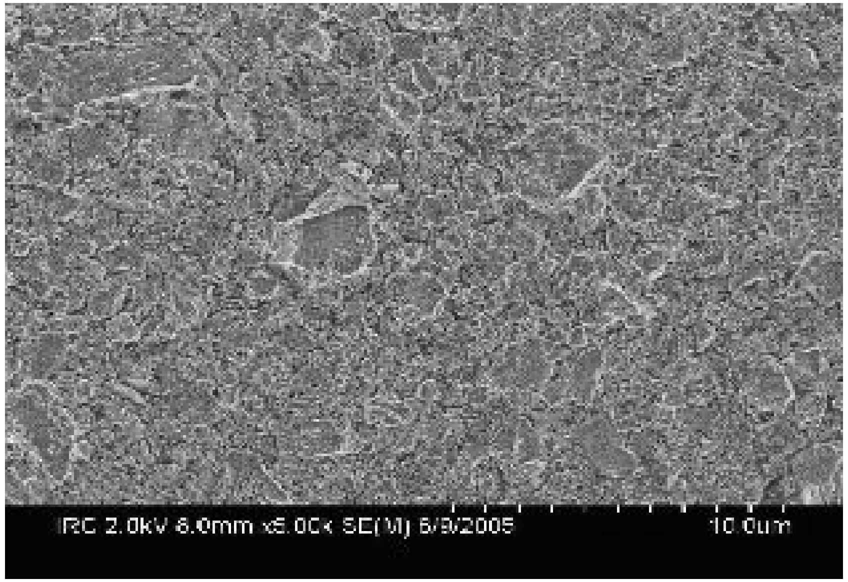


“Seeing” Particles Statistic

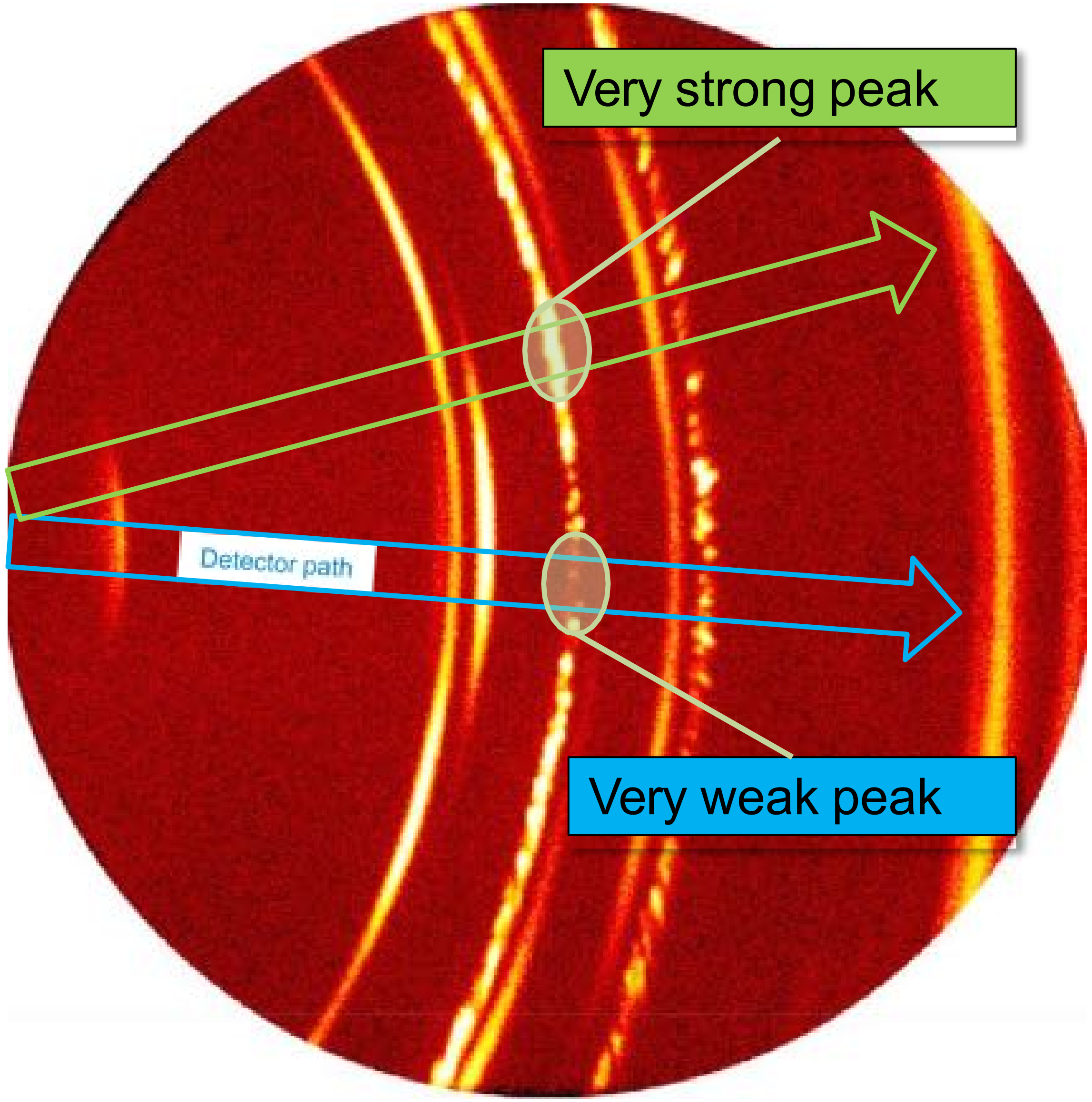


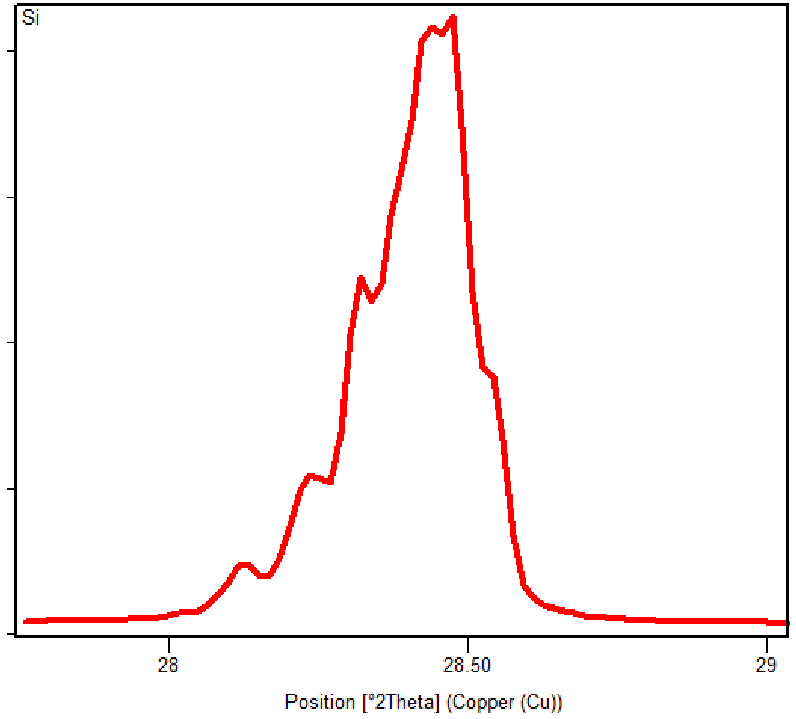






Graininess Effect

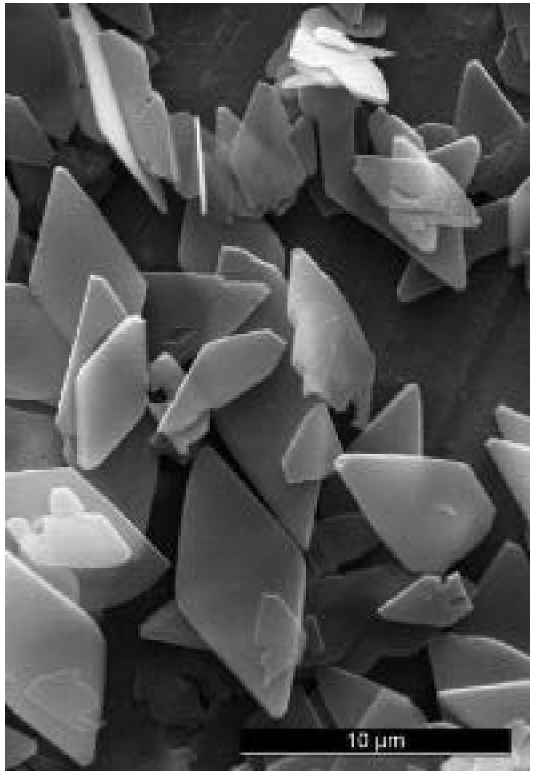


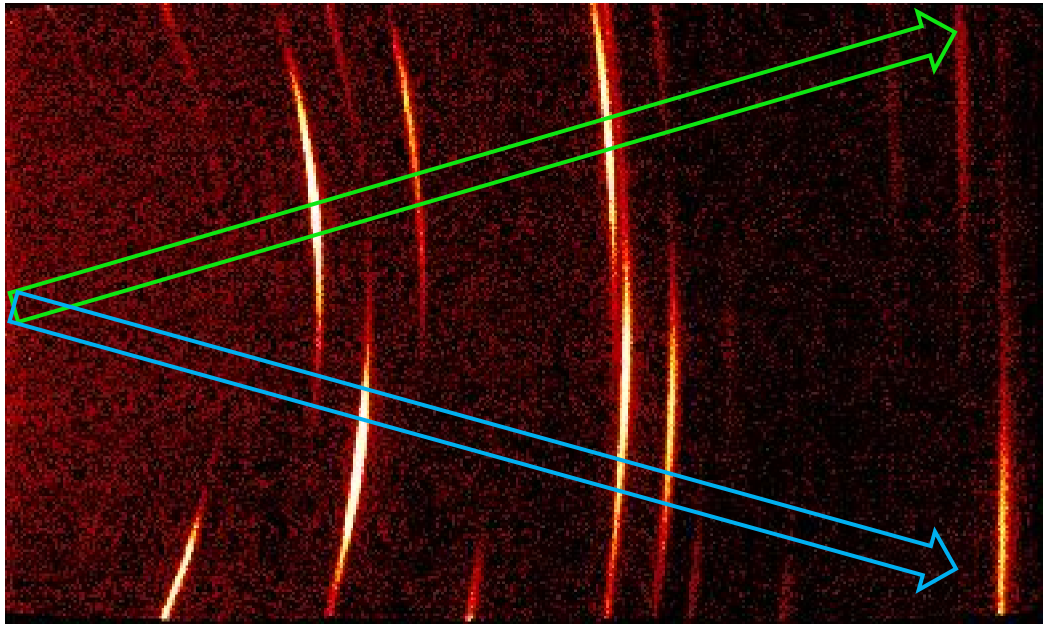


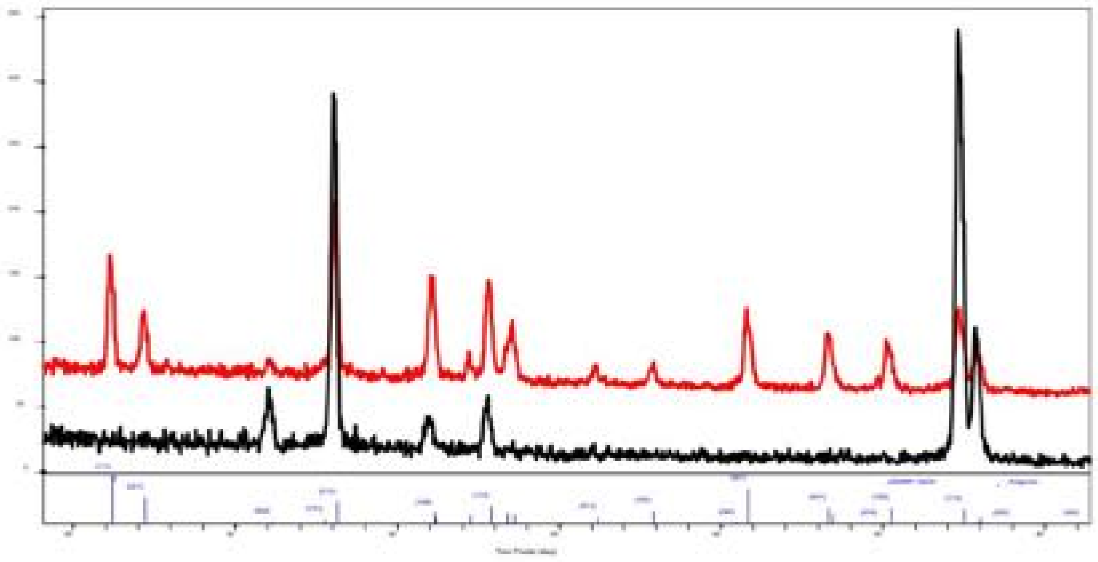
Grainy samples:

- **Non-reproducible** intensities
- No data treatment tricks can save this data

Preferred Orientation (Texture in Bulk)





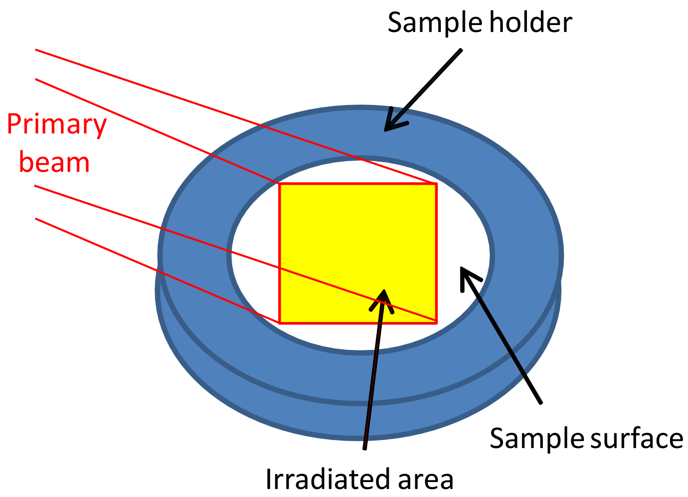


The grains have **preferred crystallographic direction** normal to the **surface of the sample** during crystallization and growth, processing, or sample preparation.

Improve Particle Statistics

There are several potential ways to improve particle statistics

- Reduce particle size
- Increase the illuminated are
  - Wider divergence angle
  - Watch for beam overspill at low angles
- Rotate samples
  - but not replacement for proper sample prep!



Mortar and pestle = bad

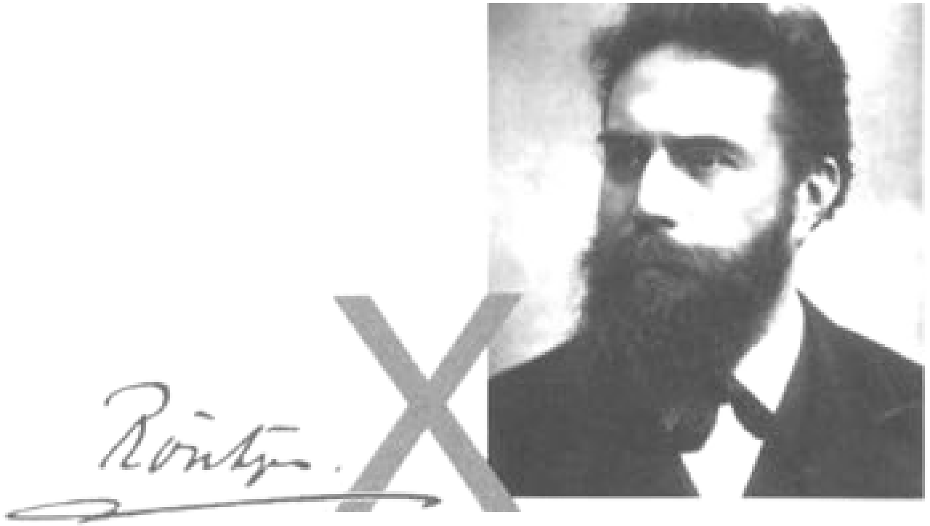
Professional mill = good

The Perfect PXRD Sample

The perfect sample for Bragg-Brentano diffractometers:

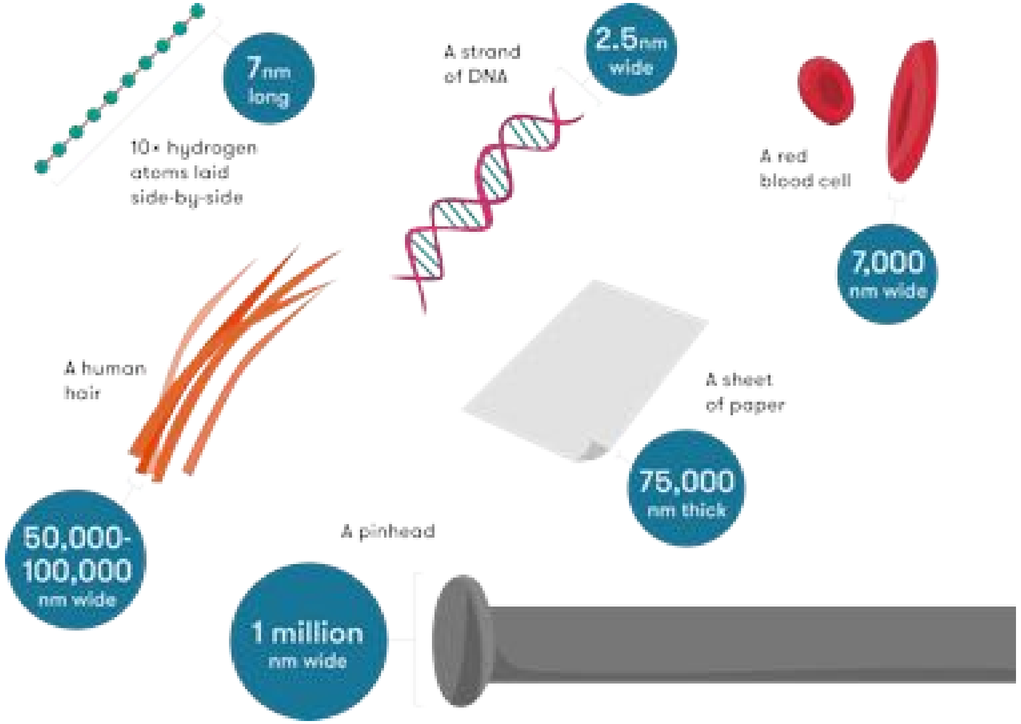
- ✓ Crystallites and particles of 1-5 μm size
- ✓ Perfectly random orientation
- ✓ Perfectly flat surface
- ✓ Surface precisely centered in the goniometer
- ✓ Not transparent (absorb all primary radiation)

- What is Powder
- **X-ray generation**
- Data Acquisition
- Peak Profiles
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- Miscellaneous



Wilhelm Conrad Röntgen,1895
Noble prize in physics,1901

Why Use X-Rays?



energy — Planck constant — speed of light — wavelength

$$E = \frac{hc}{\lambda}$$

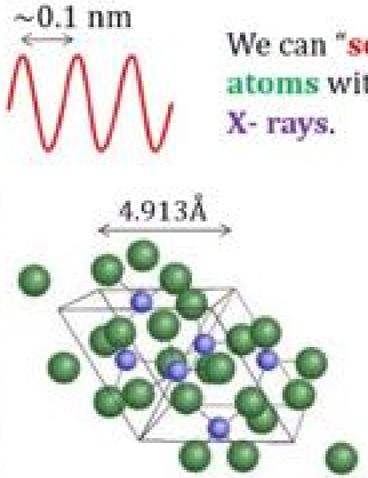
Voltage — Currents — time

$$E = VIt$$

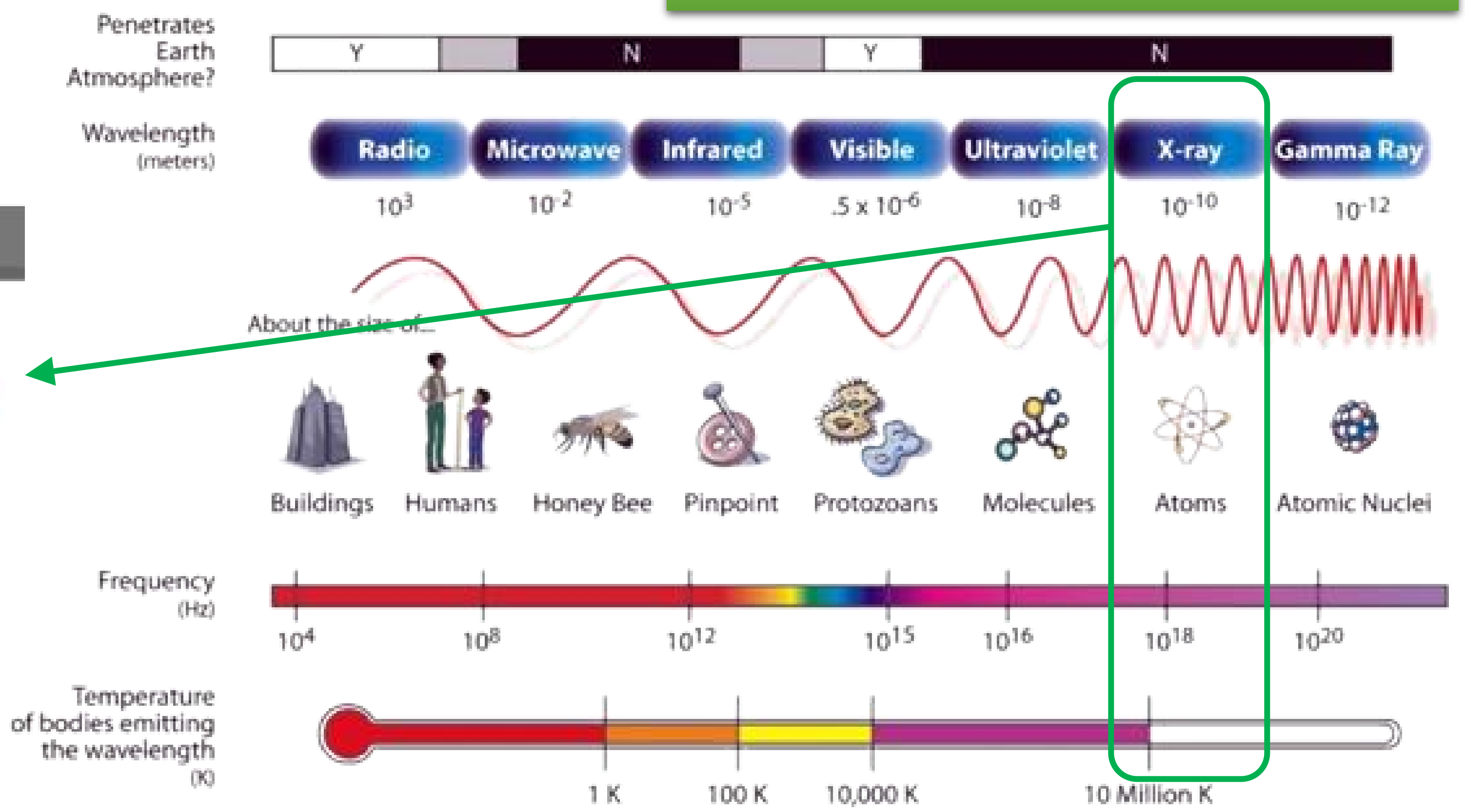
*PSI Synchrotron 10 MW, ~ USD 200 million

Wavelength 380 – 720 nm

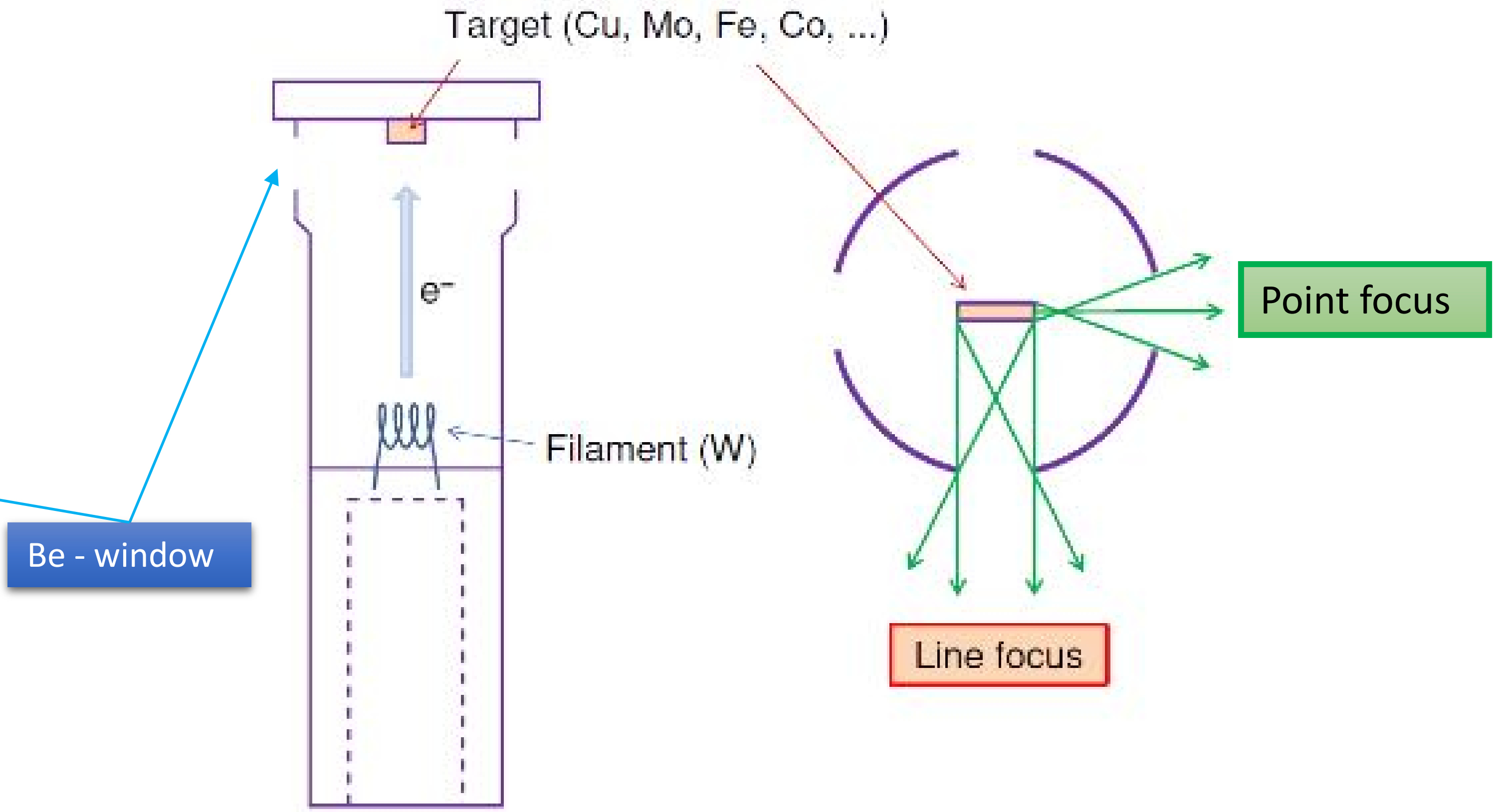
With the **naked eye**, we can see colors and shapes of objects.



We can "**see**" **atoms** with **X- rays.**



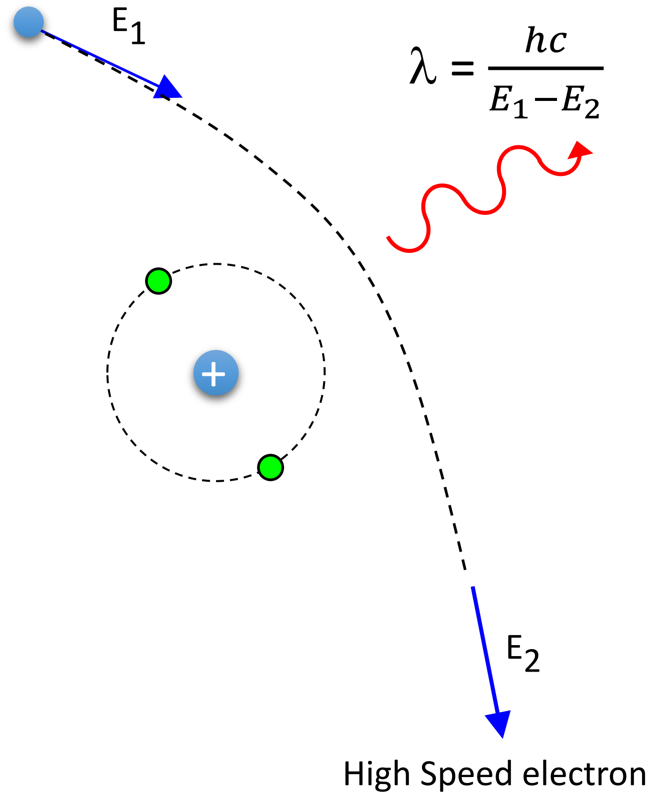
X-Ray Tube



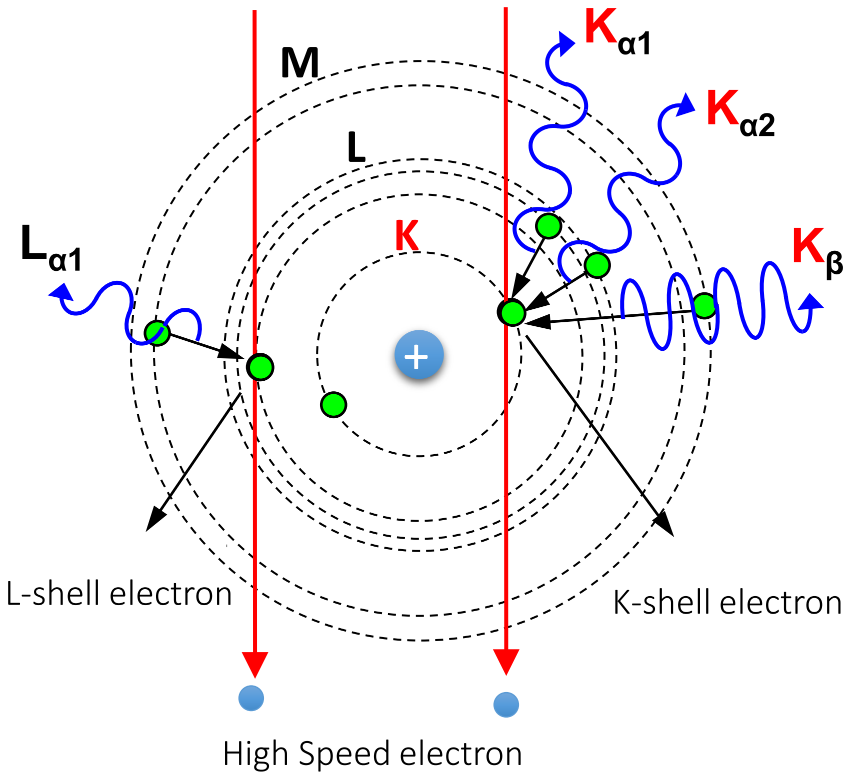
Type	Acronym	Dimension [mm x mm]
Fine focus	FF	0.04 x 8
Long fine focus	LFF	0.04 x 12

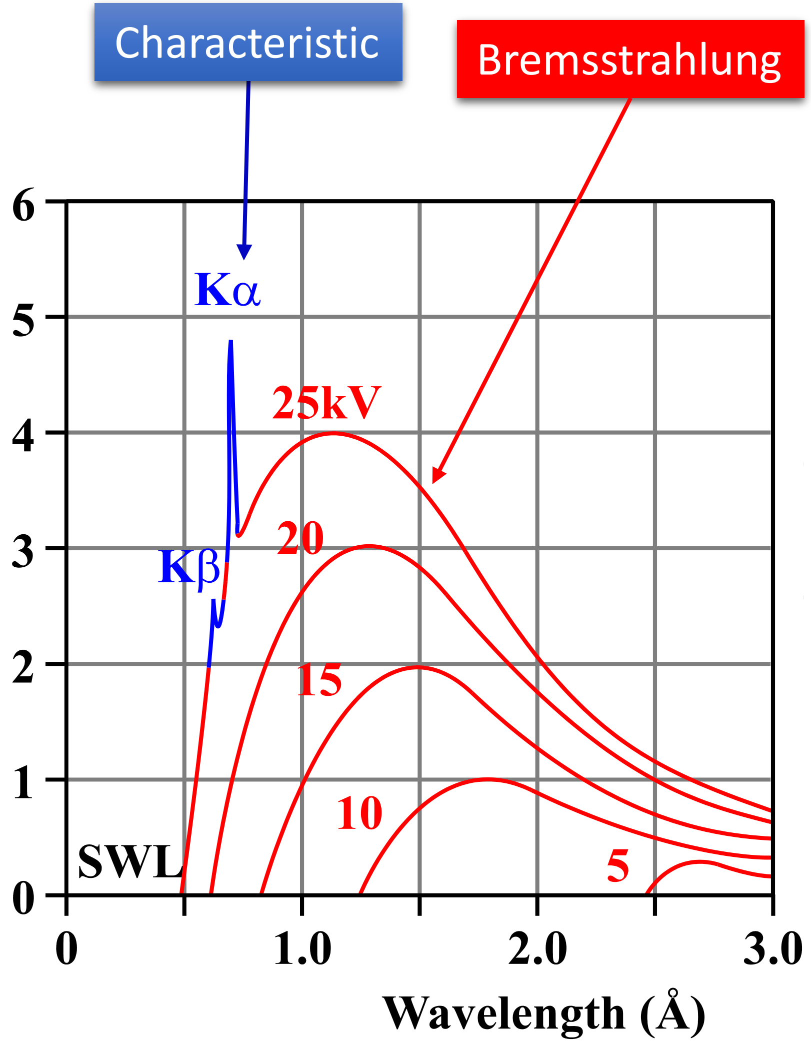
Bremsstrahlung vs Characteristic

deceleration of the free electron when passing the nuclei

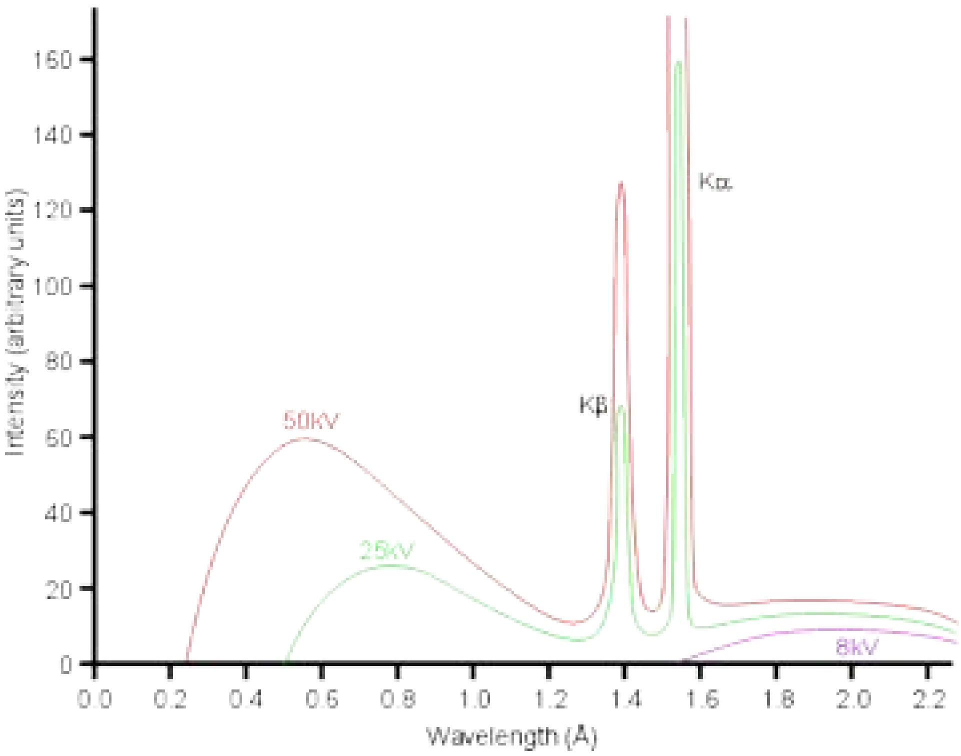


electron is knocked out of its orbit and takes its place by other electron from a different shell



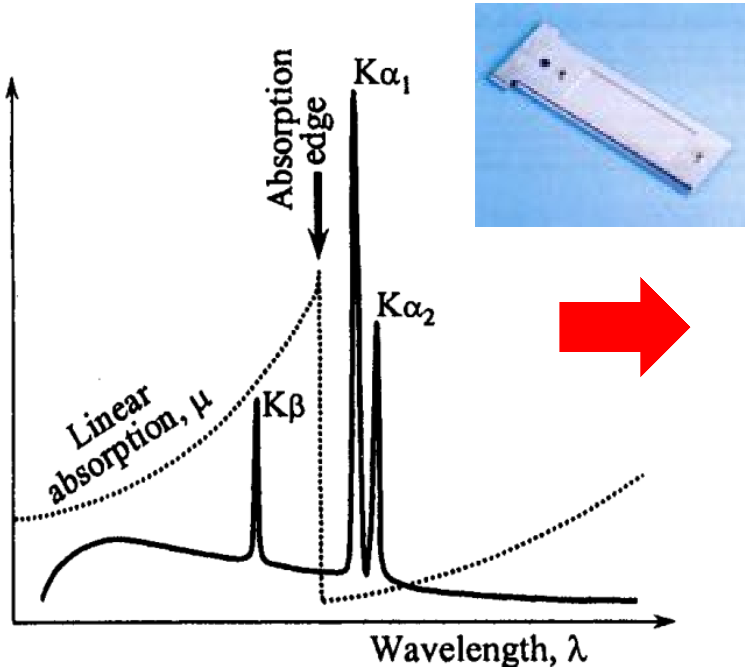


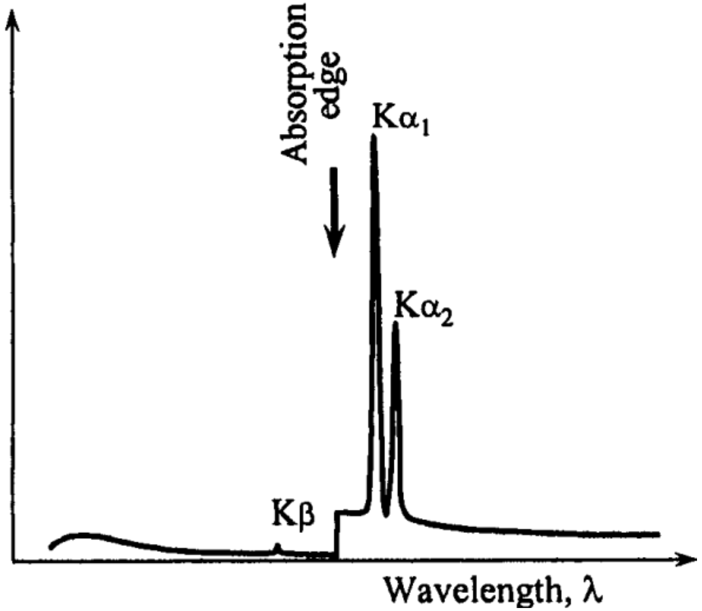
Cu-anode and Monochromatization



R.E. Dinnebier and S.J.L. Billinge in *Powder Diffraction* (2008)

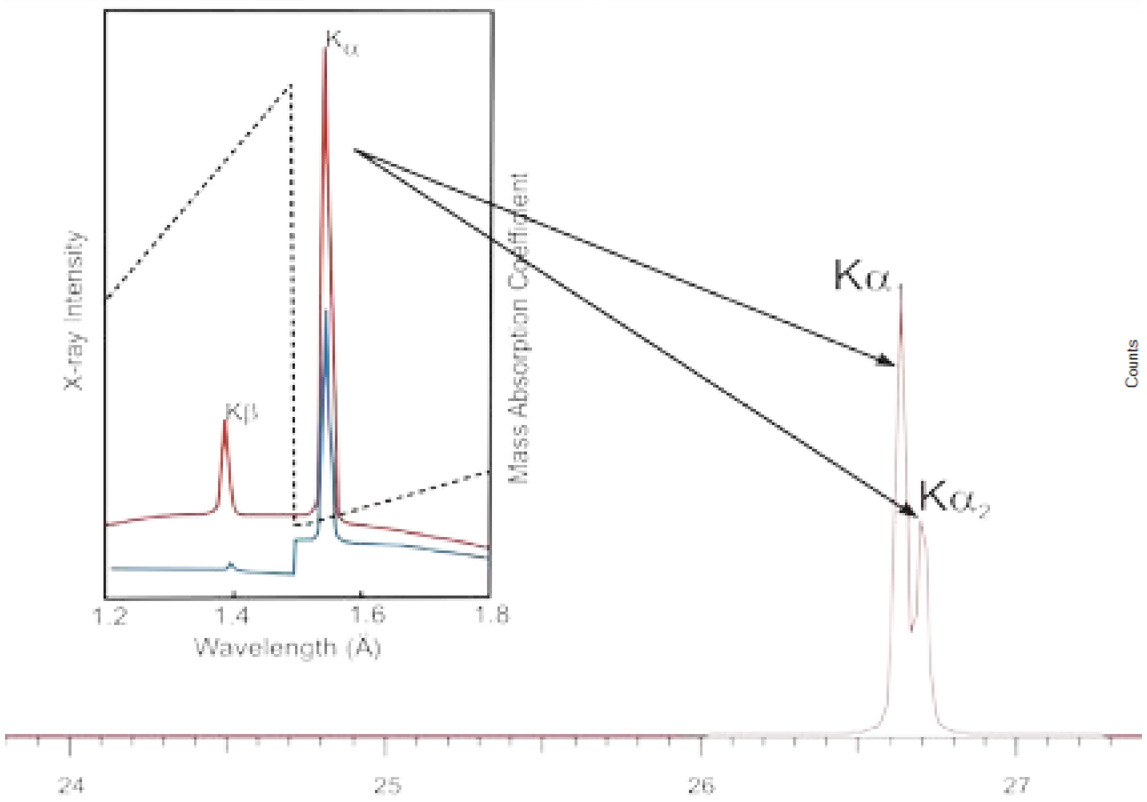
$$I_t = I_0 \exp(-\mu x)$$

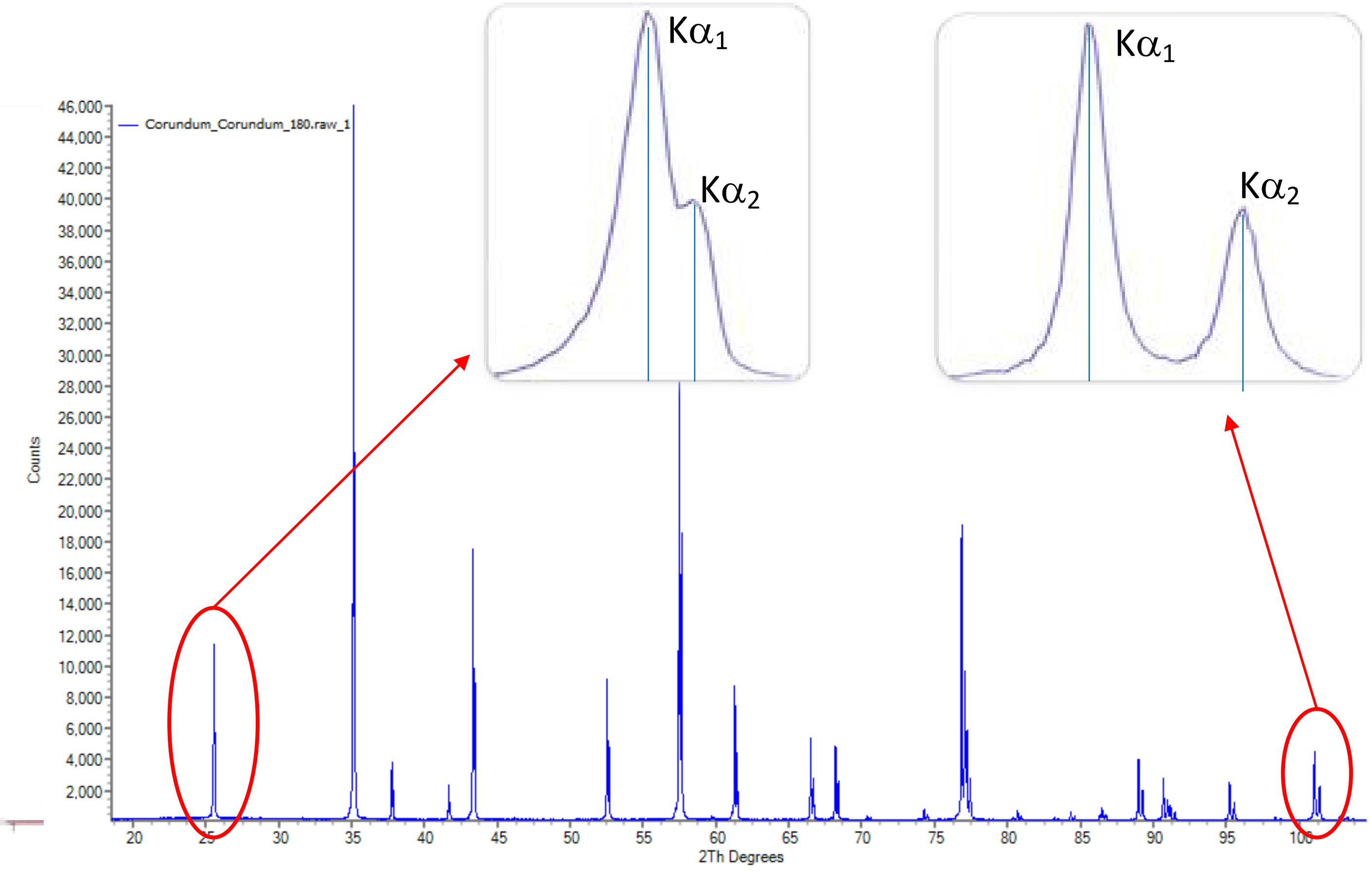




Tube anode material	Beta-filter material	Thickness (μm)	Kβ intensity reduction (%)	Kα intensity reduction (%)
Mo	Zr	75	97	54
Cu	Ni	20	99	58
Co	Fe	16	99	51
Cr	V	13	98	45

Kß-Filter and Kα_{1+2} effect





- Kβ_1 filter reduce 58-70%
- Left the absorption edge artifact

Majority of monochromators used in laboratory XRD unable to separate Kα_1 and Kα_2 radiation.

X-Ray Tube Efficiency

Production of X-rays

~99% Heat

~1% X-Rays

~90% Bremsstrahlung

~10% Characteristics

~1.5% Reach Be-window

~90% Passthrough Be-window

~35% Absorb in Air

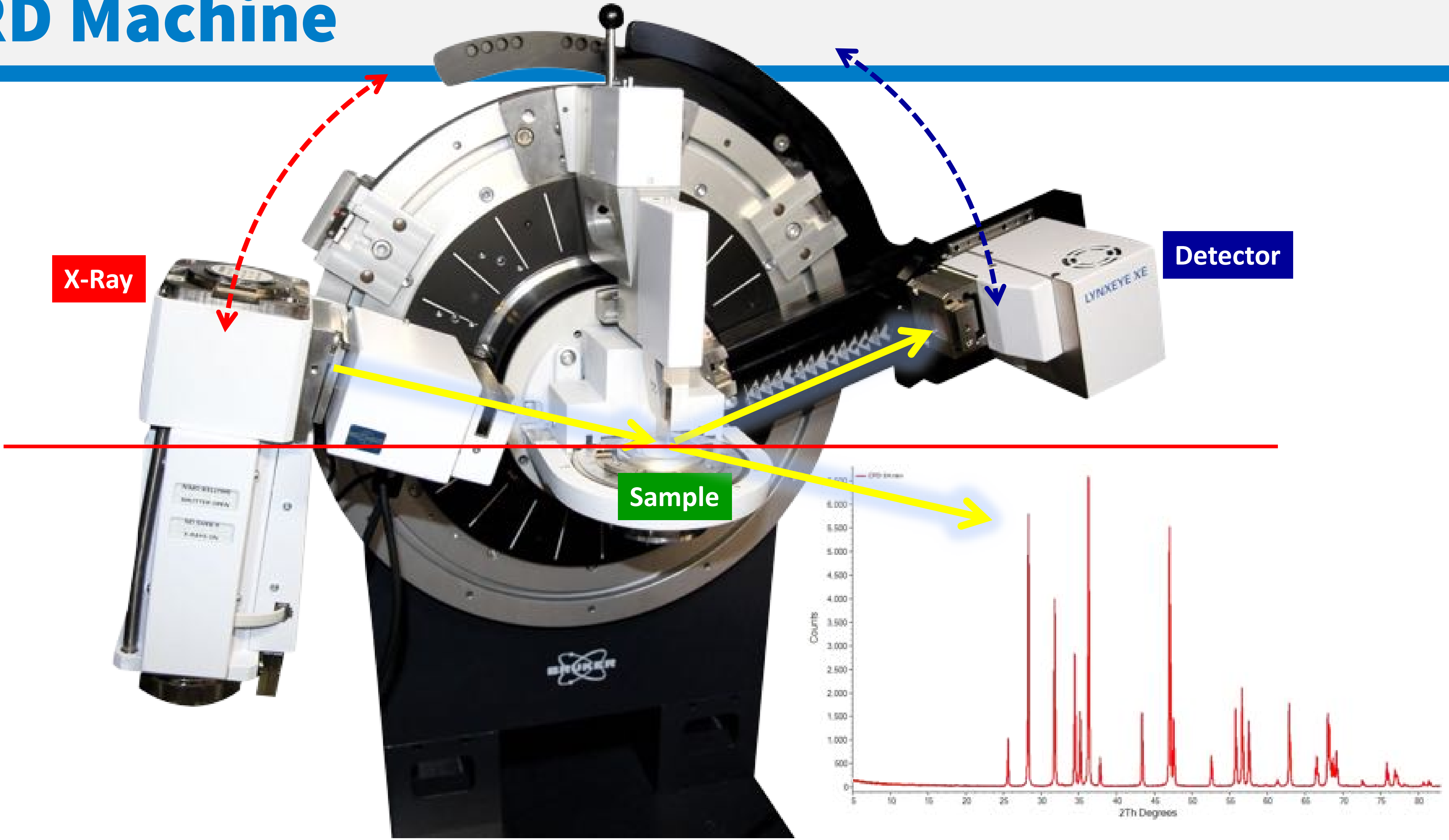
Conclusion:

1% x 10% x 1.5% x 90% x 65% = 0.00088%

reaches the sample

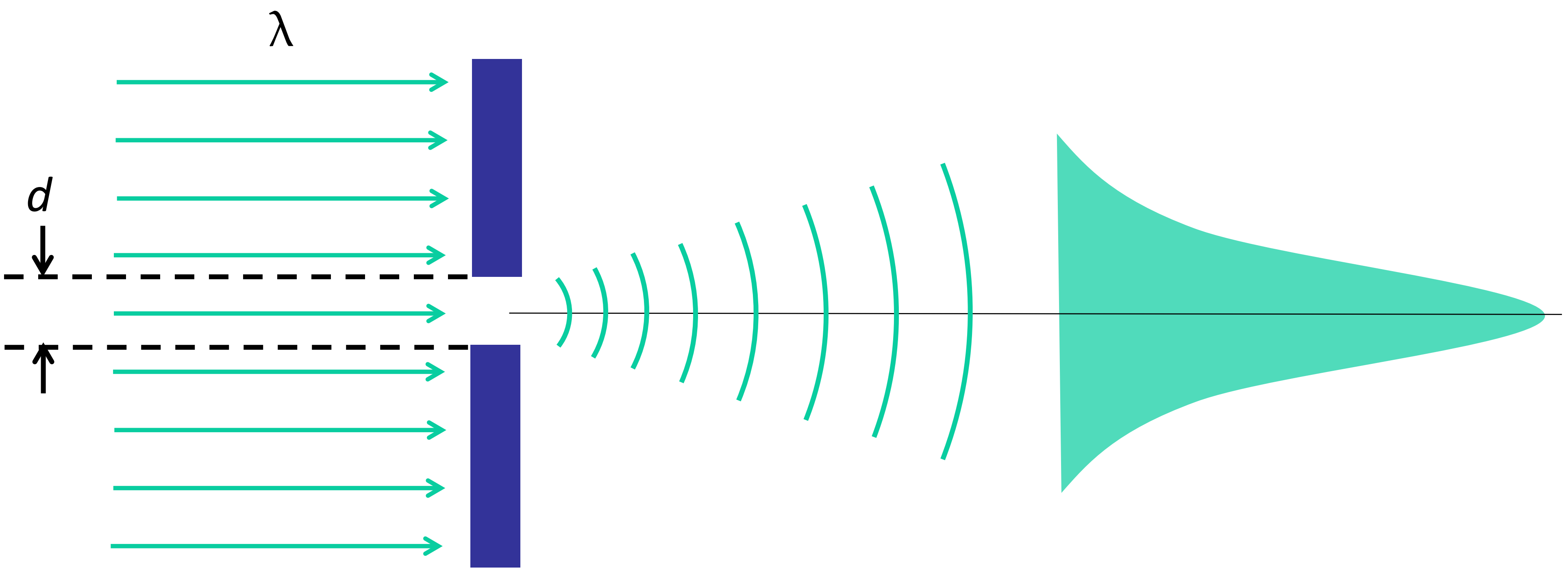
Stefan B & Uwe O. (2017), Ebel H. (1999), Mars S. & Carroll Q. (1982), warren W. N. (1930)

XRD Machine

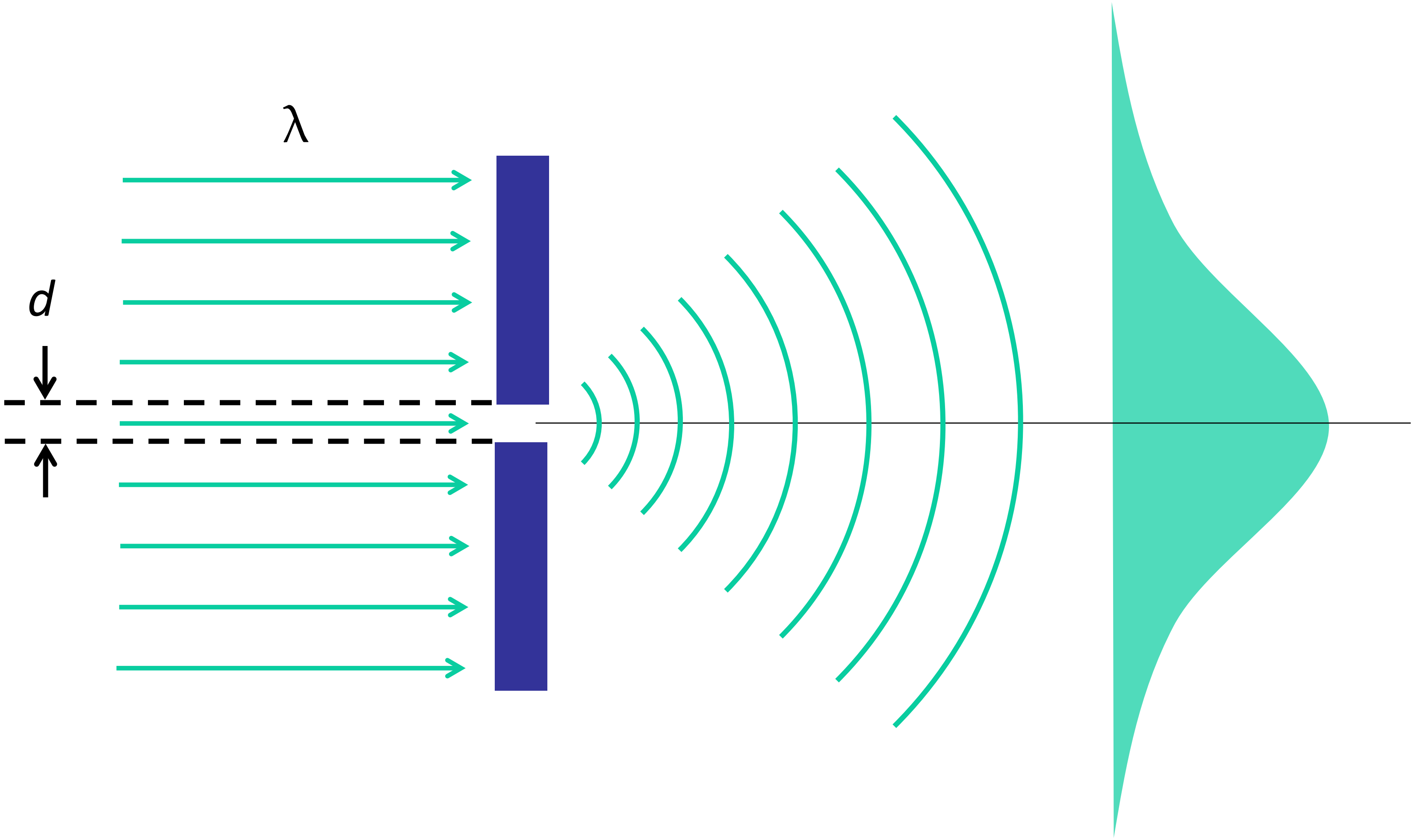


Single Slit Diffraction

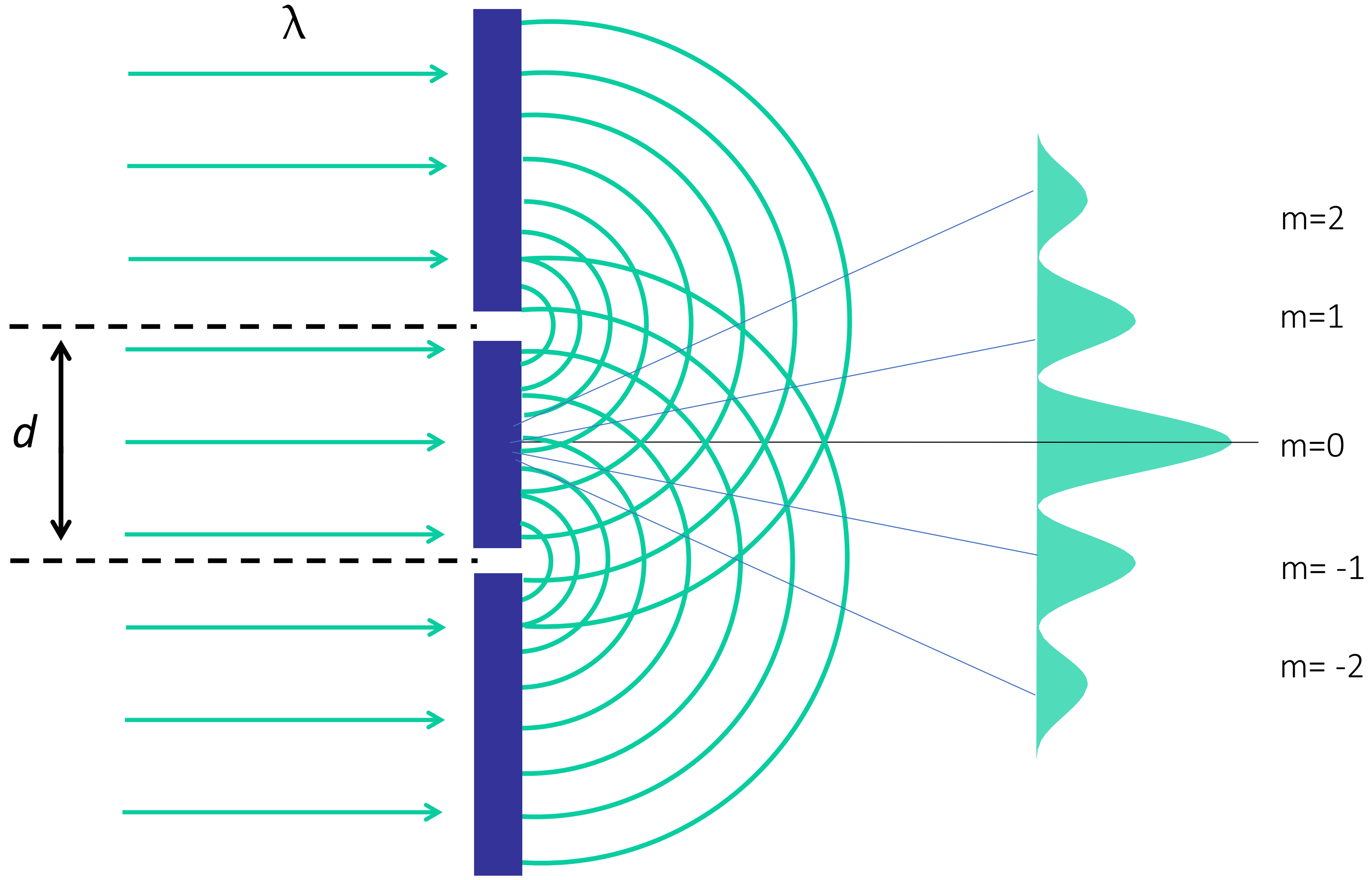
The ‘slit’ scatters light and become a point source



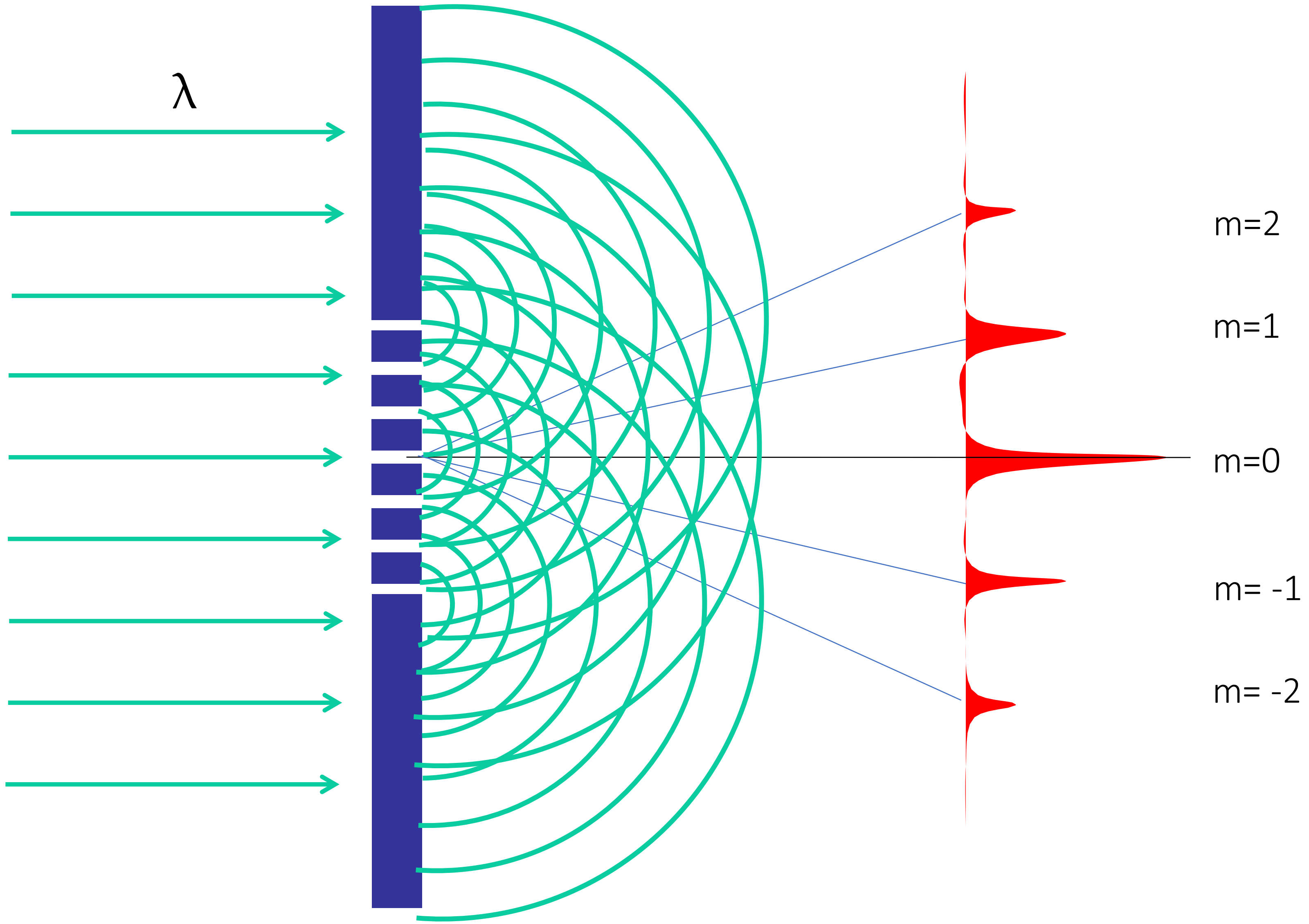
Single Slit Diffraction



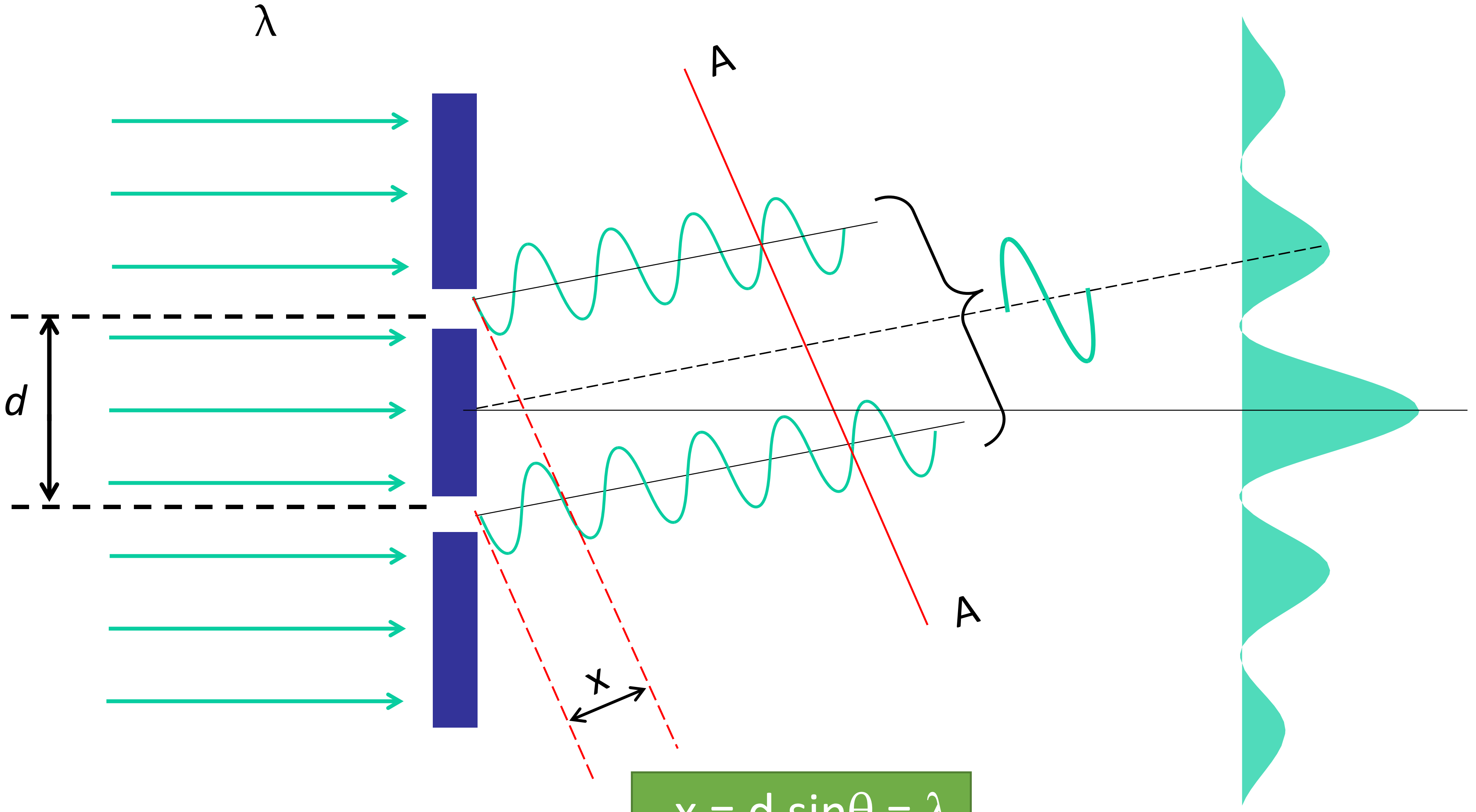
Double Slit Diffraction



Multi Slit Diffraction

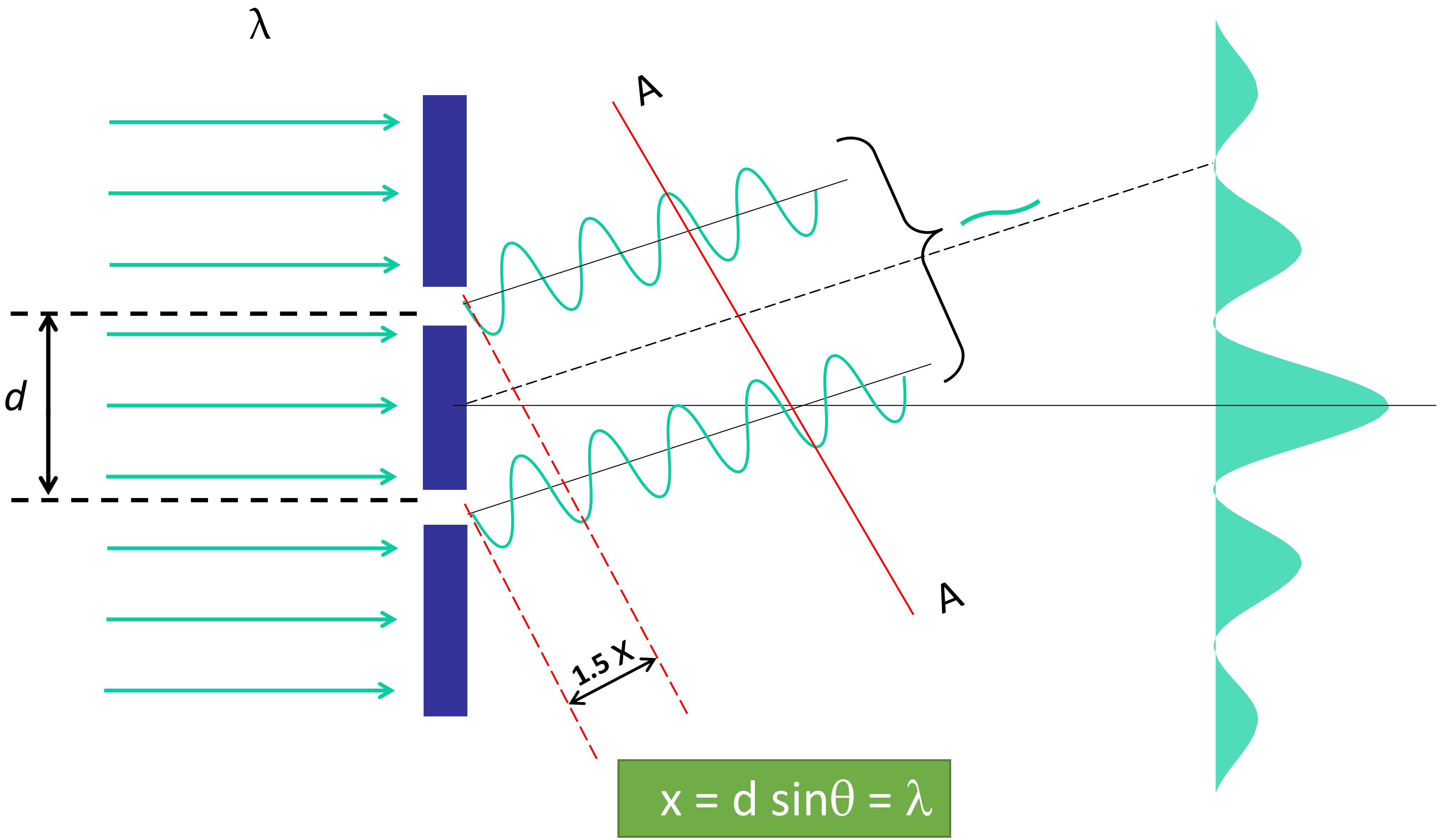


Constructive Interference

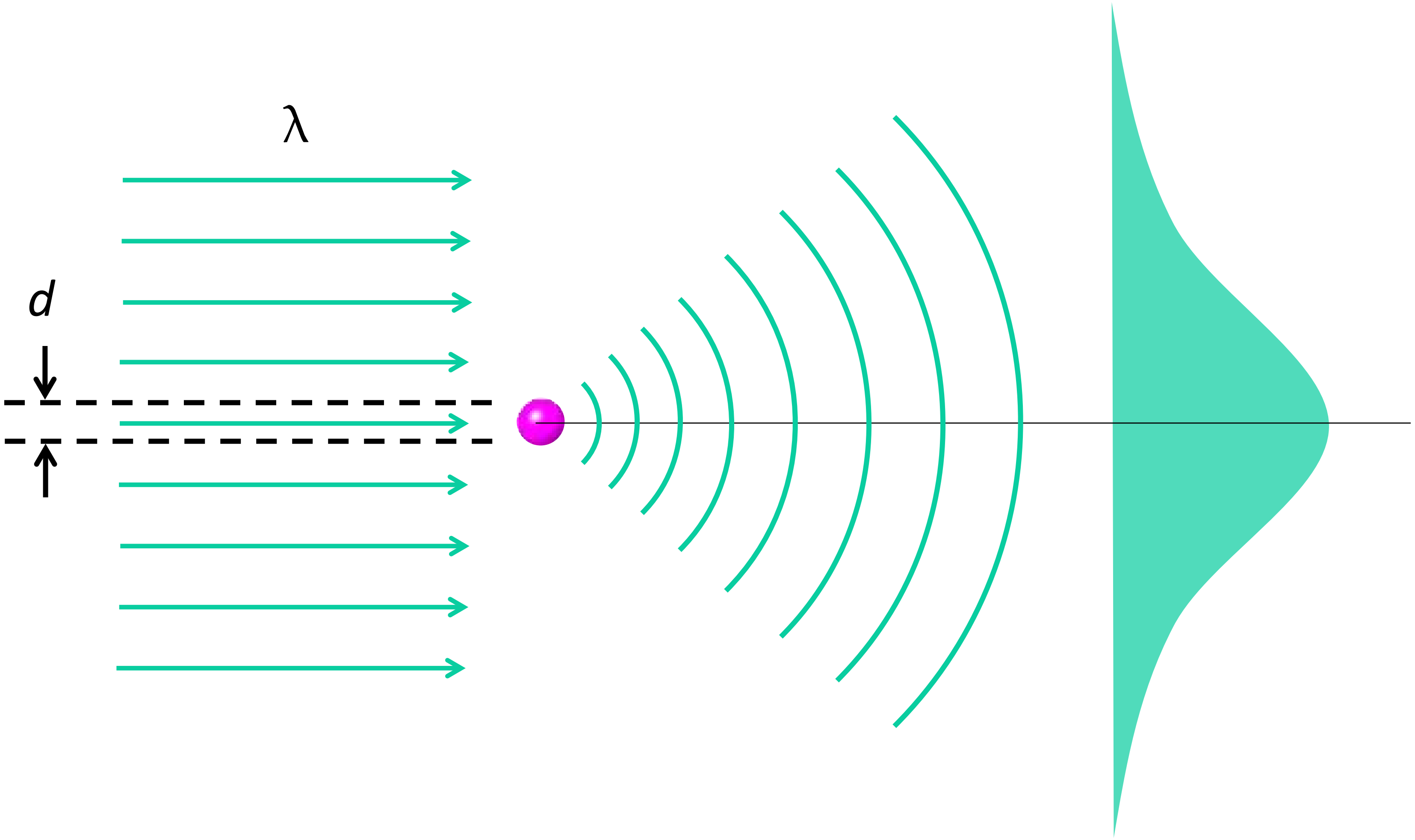


$x = d \sin\theta = \lambda$

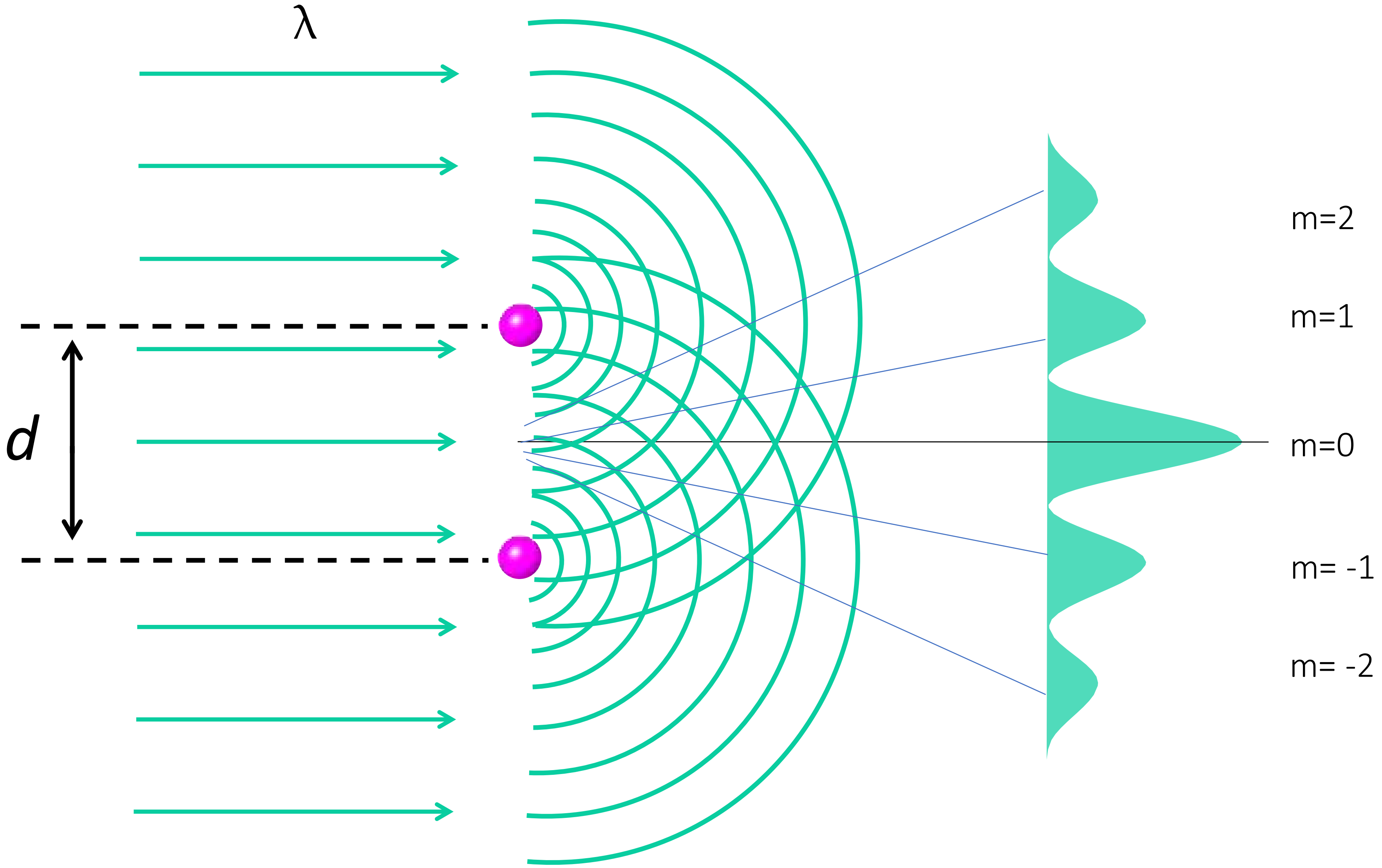
Destructive Interference



Diffraction by One Atom



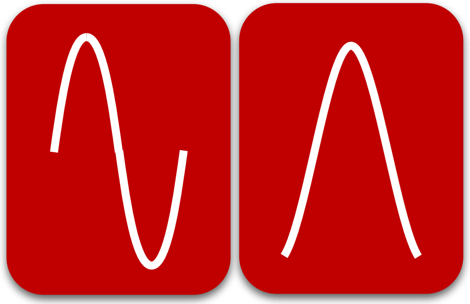
Diffraction by Two Atom



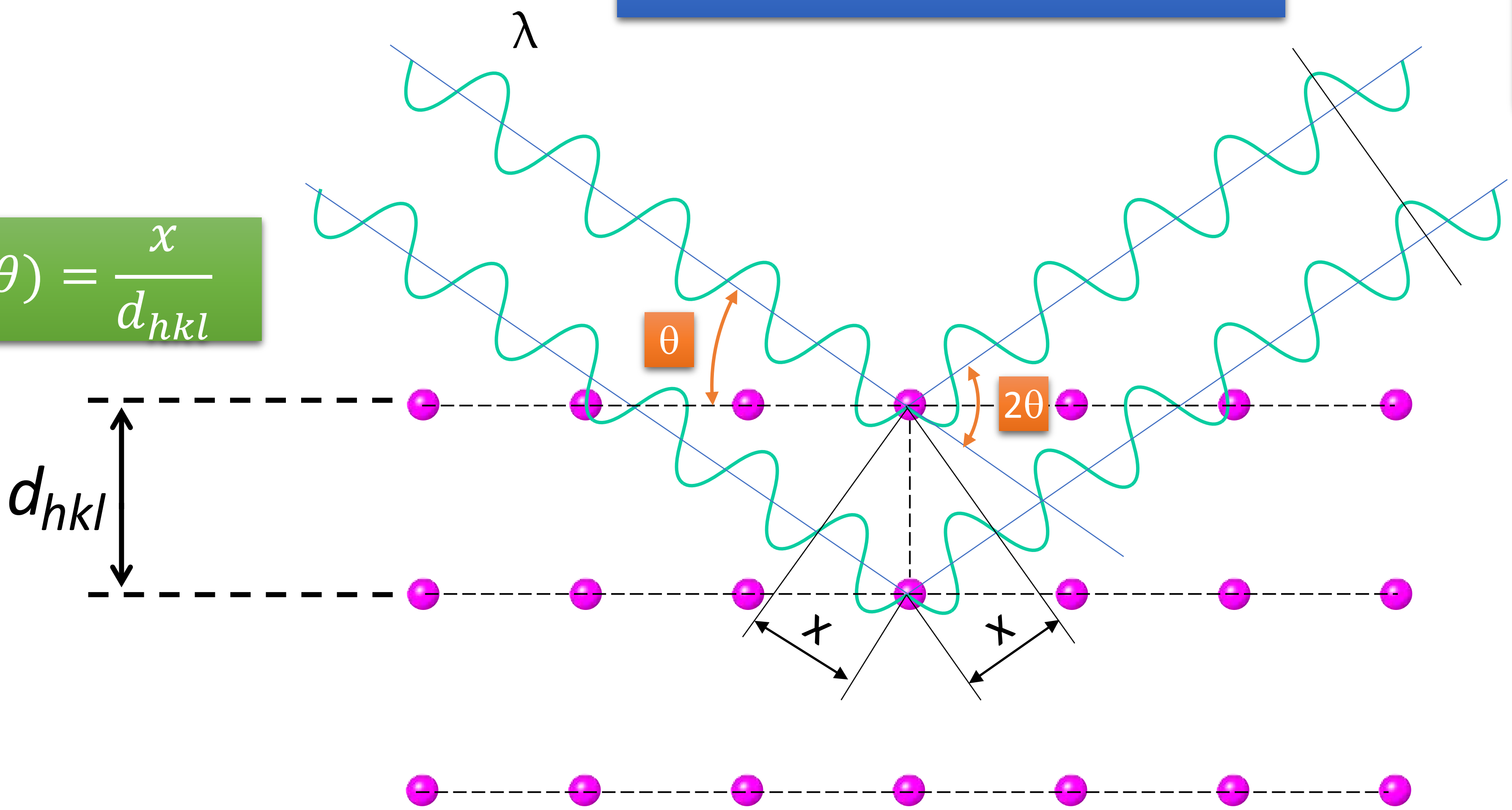
Diffraction by Planes of Atom

- Path difference Δ = 2x => phase shift
- Constructive interference if Δ = nλ
- Criterion for constructive interference:

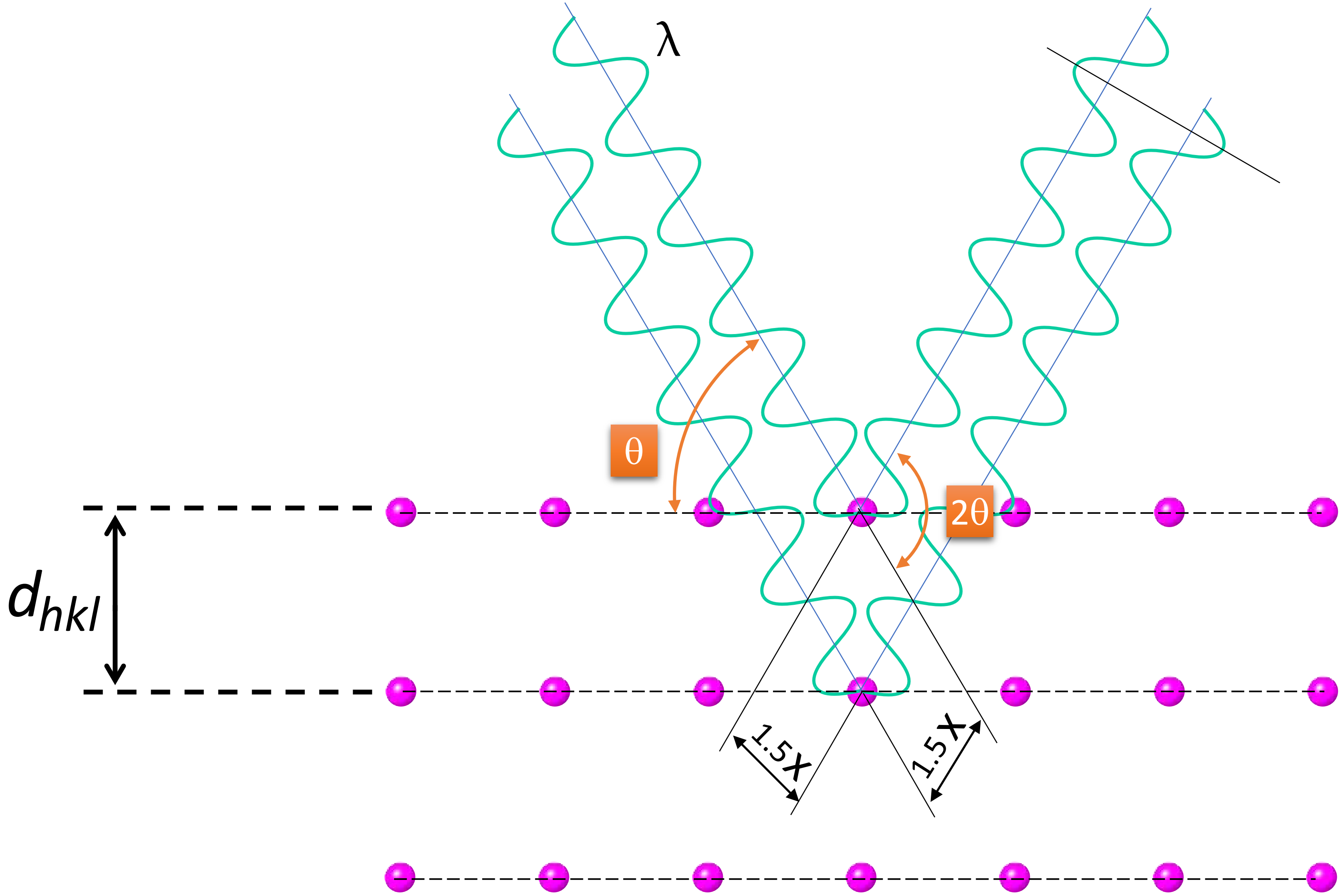
$$\Delta = 2d_{hkl}\sin(\theta) = n\lambda$$

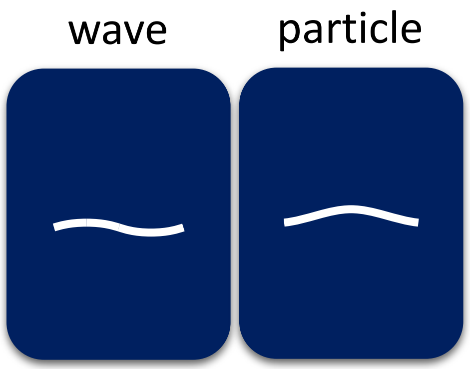


$$\sin(\theta) = \frac{x}{d_{hkl}}$$

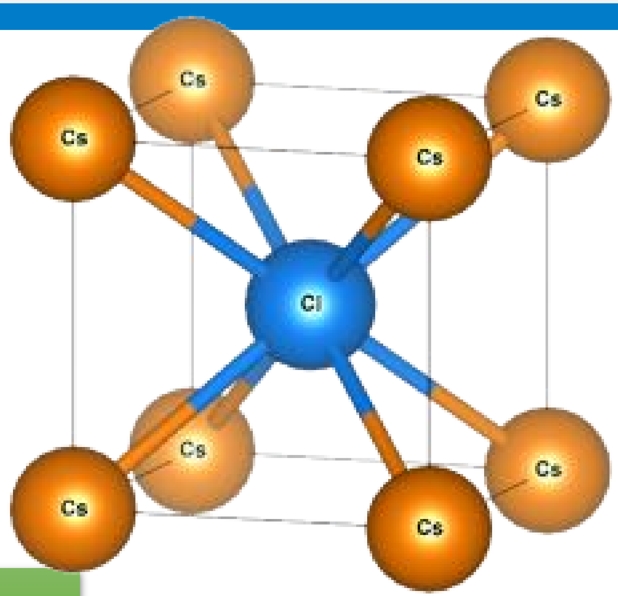


Diffraction by Planes of Atom





Diffraction Intensities – Structure factor



$$F_{hkl} = \sum_{1}^{N} f_n \, e^{2\pi i(hx_n + ky_n + lz_n)}$$

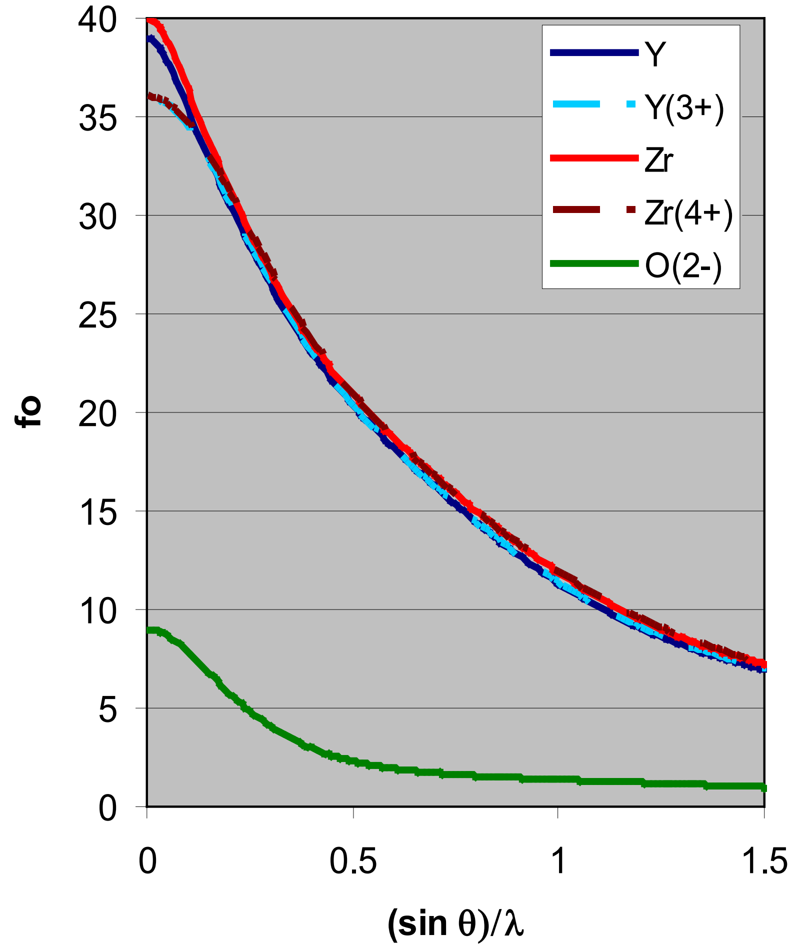
$$F_{hkl} = \sum_{1}^{N} f_n \left[\cos 2\pi(hx_n + ky_n + lz_n) + i \sin 2\pi(hx_n + ky_n + lz_n)\right]$$

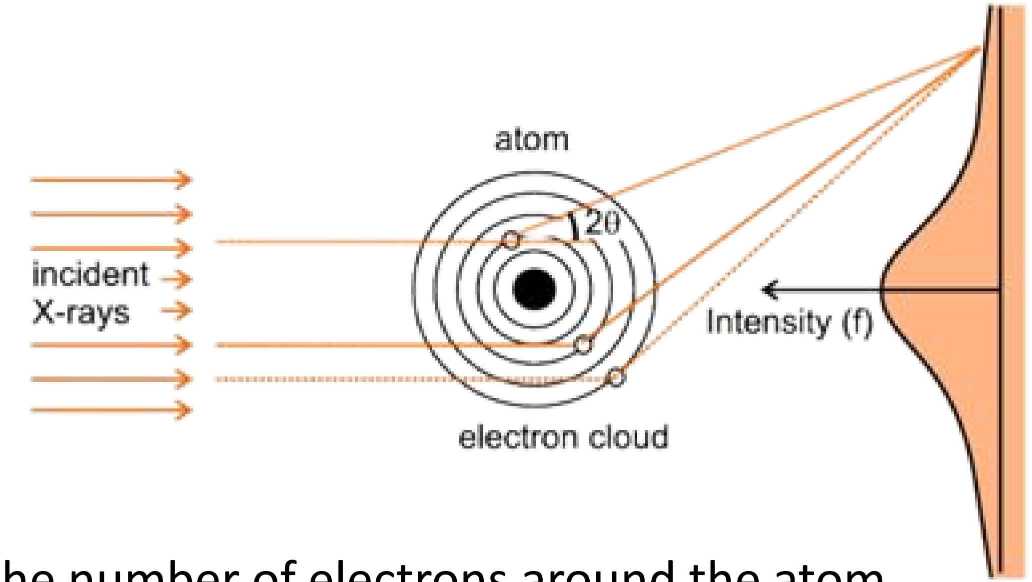
$$F^2 = [f_1 \cos 2\pi(hx_1 + ky_1 + lz_1) + f_2 \cos 2\pi(hx_2 + ky_2 + lz_2) + \cdots]^2 + [f_1 \sin 2\pi(hx_1 + ky_1 + lz_1) + f_2 \sin 2\pi(hx_2 + ky_2 + lz_2) + \cdots]^2$$

- The structure factor quantifies the amplitude of X-rays scattered by a crystal
- F_{hkl} sums the result of scattering from all of the atoms in the unit cell to form a diffraction peak from the (*hkl*) planes of atoms
- The amplitude of scattered light is determined by:
  - where the atoms are on the (*hkl*) planes
    - ➢ this is expressed by the fractional coordinates x_j y_j z_j
  - what atoms are on the atomic planes
    - ➢ the scattering factor f_j quantifies the relative efficiency of scattering at any angle by the group of electrons in each atom

Diffraction Intensities - Atomic scattering factor

$$|f|^2 = \left(f_0 \exp\left[-\frac{B\sin^2\theta}{\lambda^2}\right] + (\Delta f')^2\right)^2 + (\Delta f")^2$$

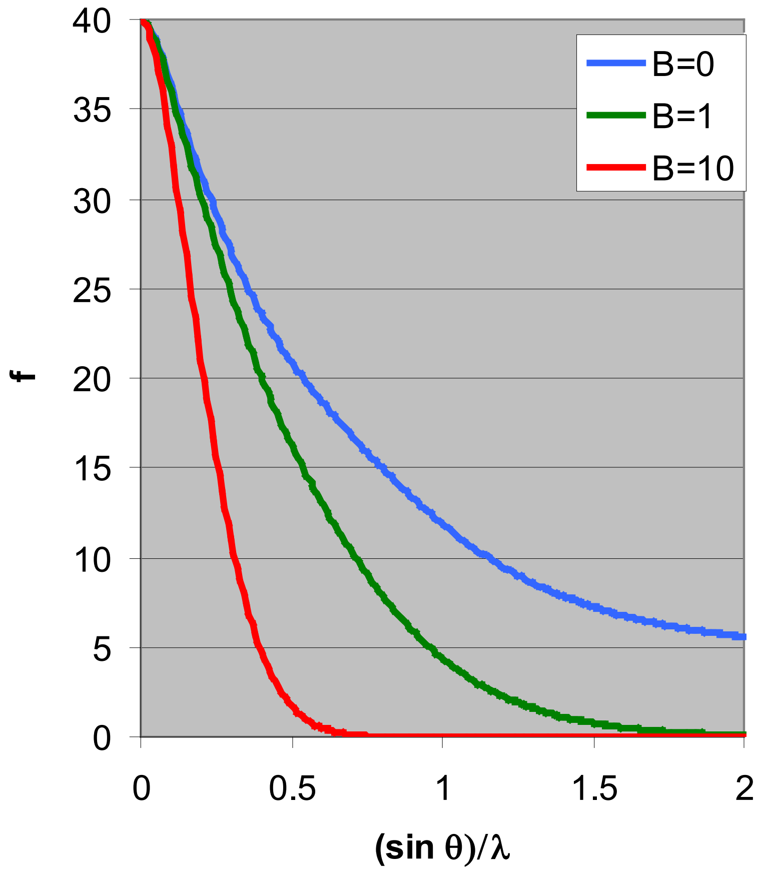


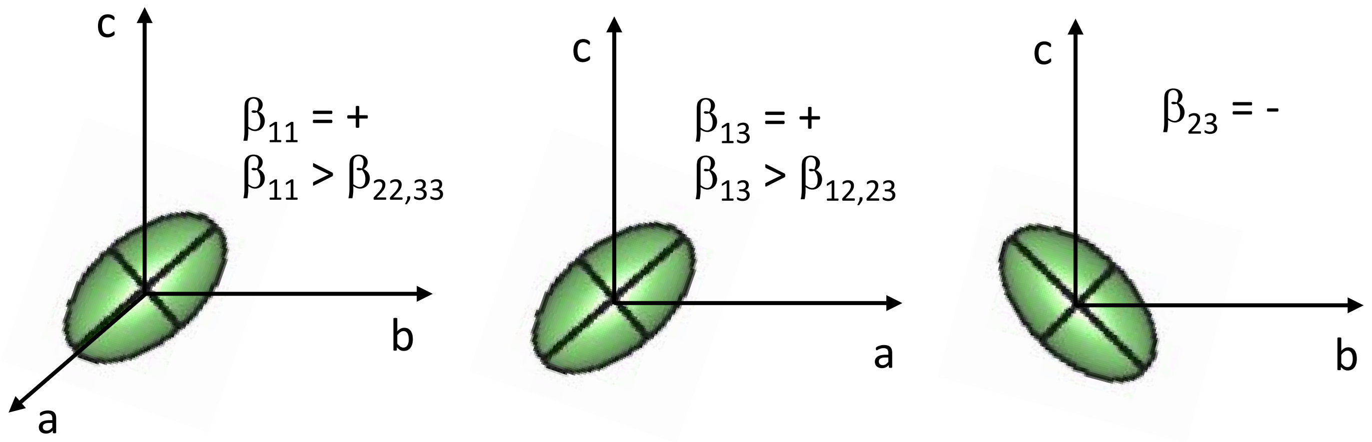


- f_0 at 0° is equal to the number of electrons around the atom
  - Y and Zr are similar, but slightly different, at 0°
  - Zr and Zr^{4+} are slightly different at 0°
  - Y^{3+} and Zr^{4+} are identical at 0°
- The variation with (sin θ)/λ depends on size of atom
  - smaller atoms drop off quicker
  - at higher angles, the difference between Y^{3+} and Zr^{4+} is more readily discerned
  - at higher angles, the difference between different oxidation states (eg Zr and Zr^{4+}) is less prominent

Diffraction Intensities – Temperature Factor

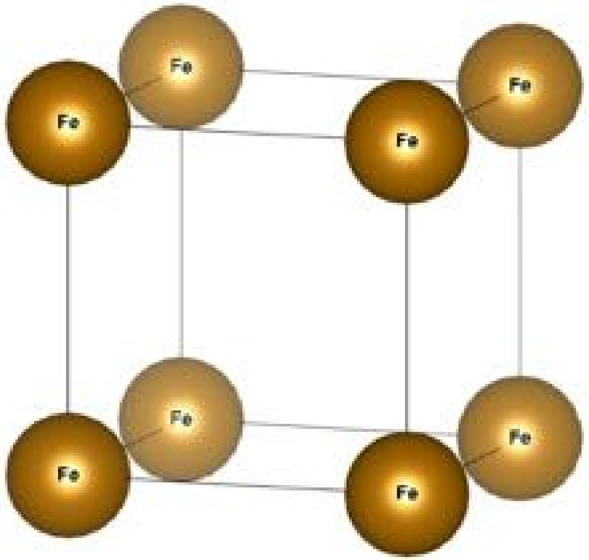
$$f = f_0 \exp\left[-\frac{B \sin^2\theta}{\lambda^2}\right]$$





- Efficiency of scattering by an atom is reduced because the atom and its electrons are not stationary - atom is vibrating about its equilibrium lattice site
- The amount of vibration is quantified by the Debye-Waller temperature factor:
  - $B=8\pi^2U^2$, U^2 is the mean-square amplitude of the vibration
  - this is for isotropic vibration: sometimes B is broken down into six Bij anisotropic terms if the amplitude of vibration is not the same in all directions.
  - aka temperature factor, displacement factor, thermal displacement parameter

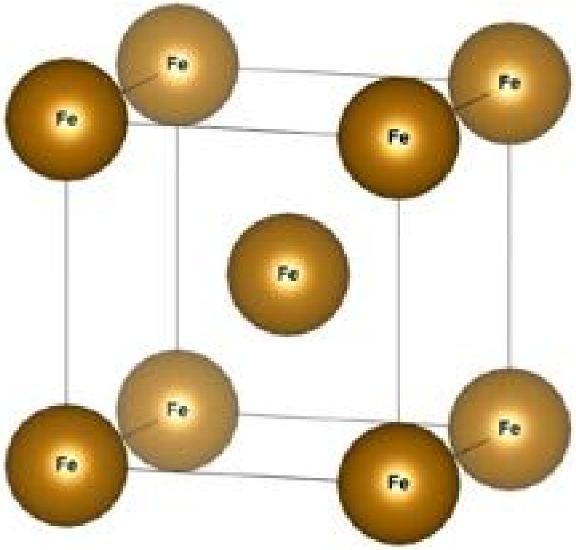
Exp. Structure Factor Calculations



The simplest case of a unit cell containing only one atom at the origin, i.e., having fractional coordinates 0 0 0. Its structure factor is

$$F = fe^{2\pi i(0)} ;$$
$$F^2 = f^2$$

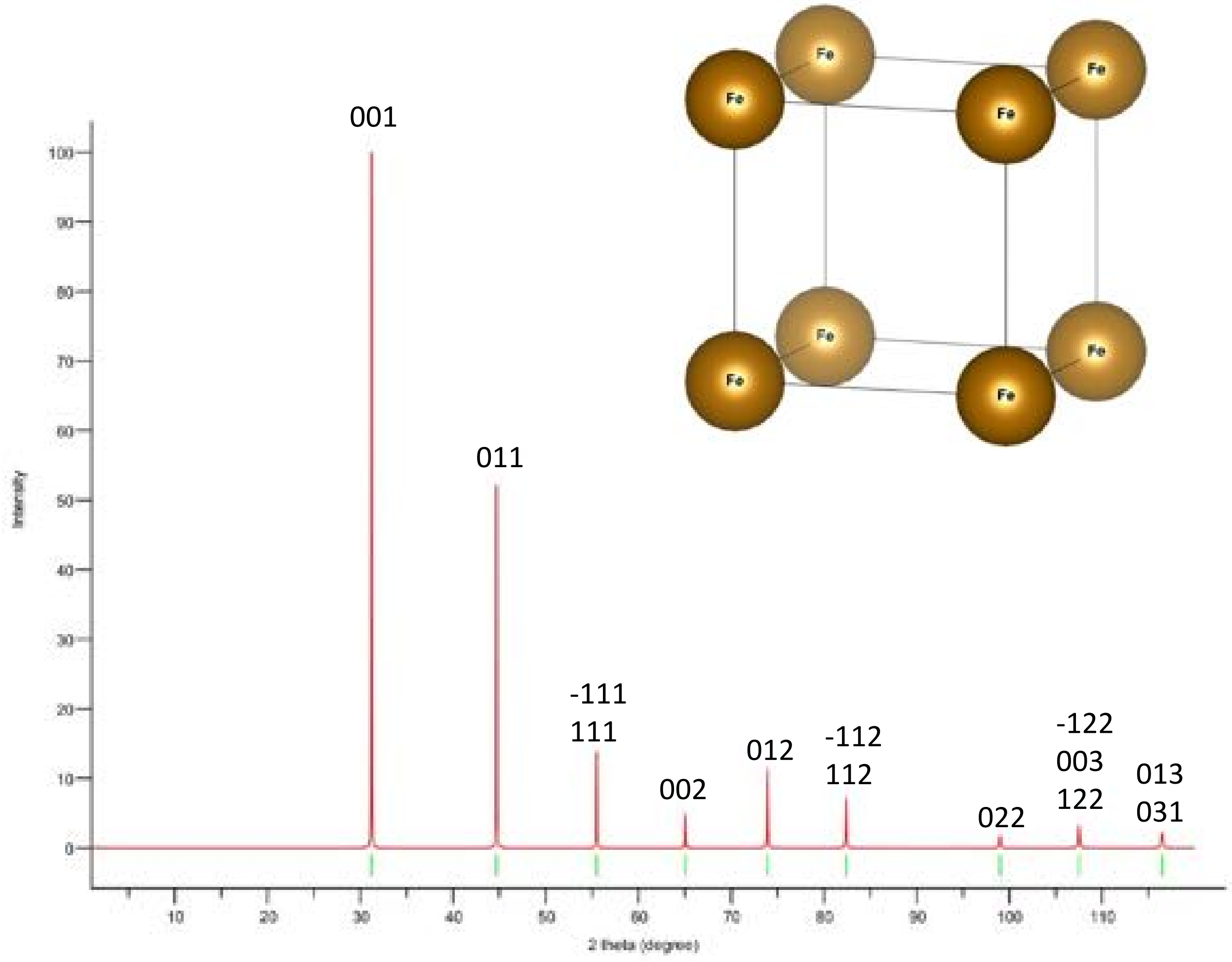
F^2 is thus independent of *h*, *k*, and *l* and is the same for all reflections.

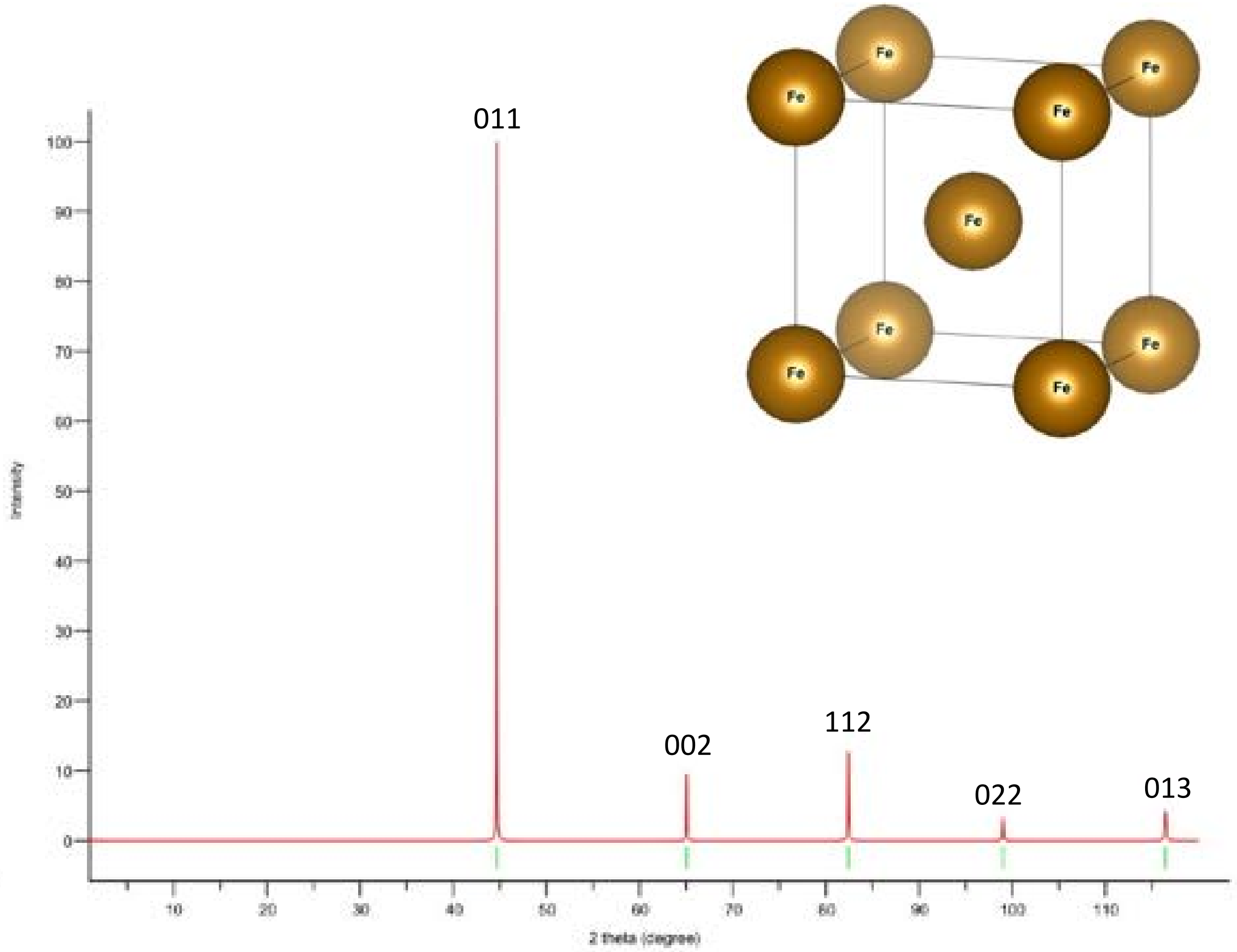


Consider the base-centered cell with two atoms of the same kind per unit cell located at 0 0 0 and ½ ½ ½.

$$F = fe^{2\pi i(0)} + fe^{2\pi i\left(\frac{h}{2}+\frac{k}{2}+\frac{l}{2}\right)}$$
$$= f\left[1 + e^{2\pi i(h+k+l)}\right]$$
$$F = 2f \quad \text{when } (h+k+l) \text{ is even; } F^2 = 4f^2$$
$$F = 0 \quad \text{when } (h+k+l) \text{ is odd; } F^2 = 0$$

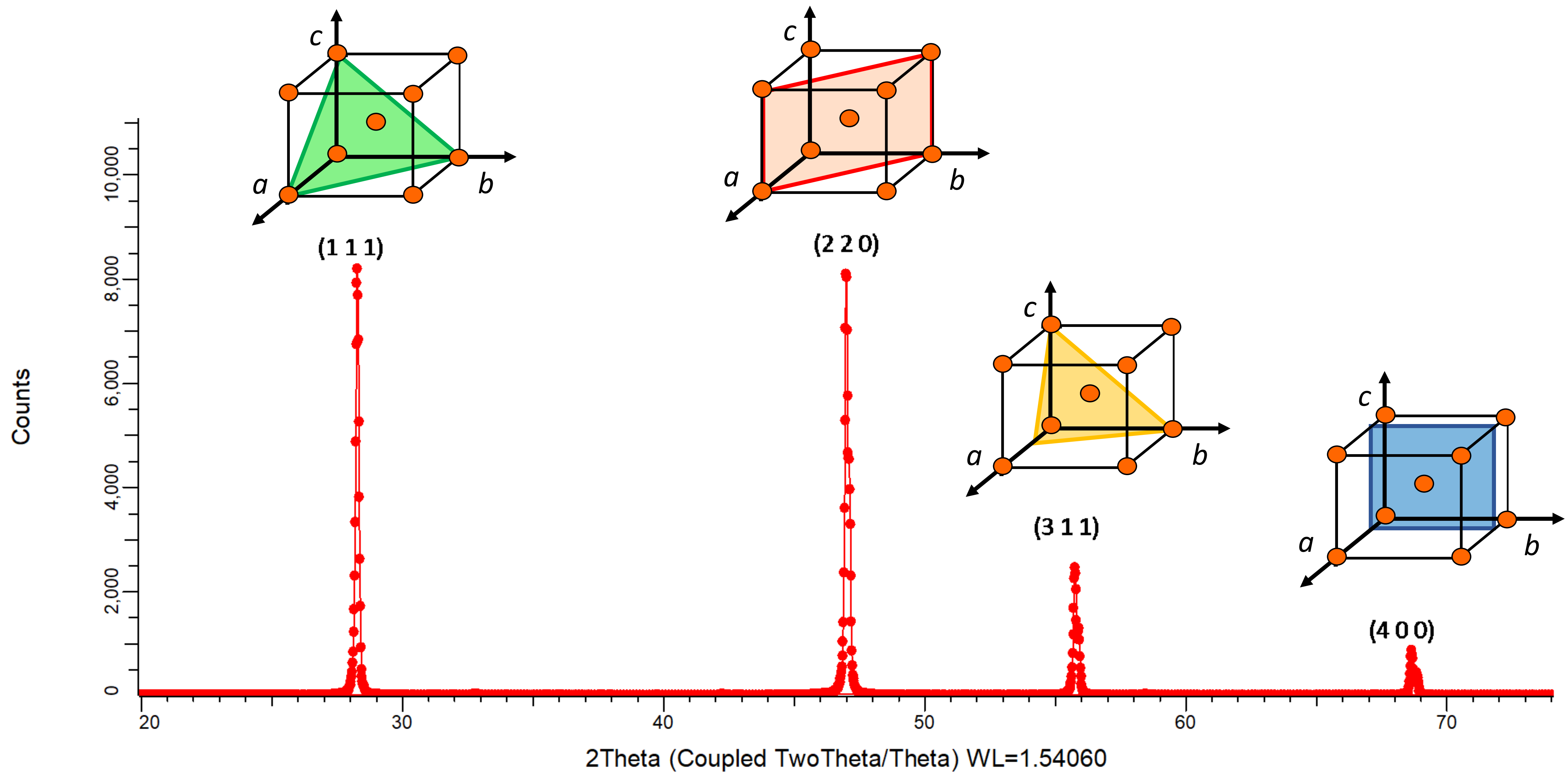
Exp. Structure Factor Simulation



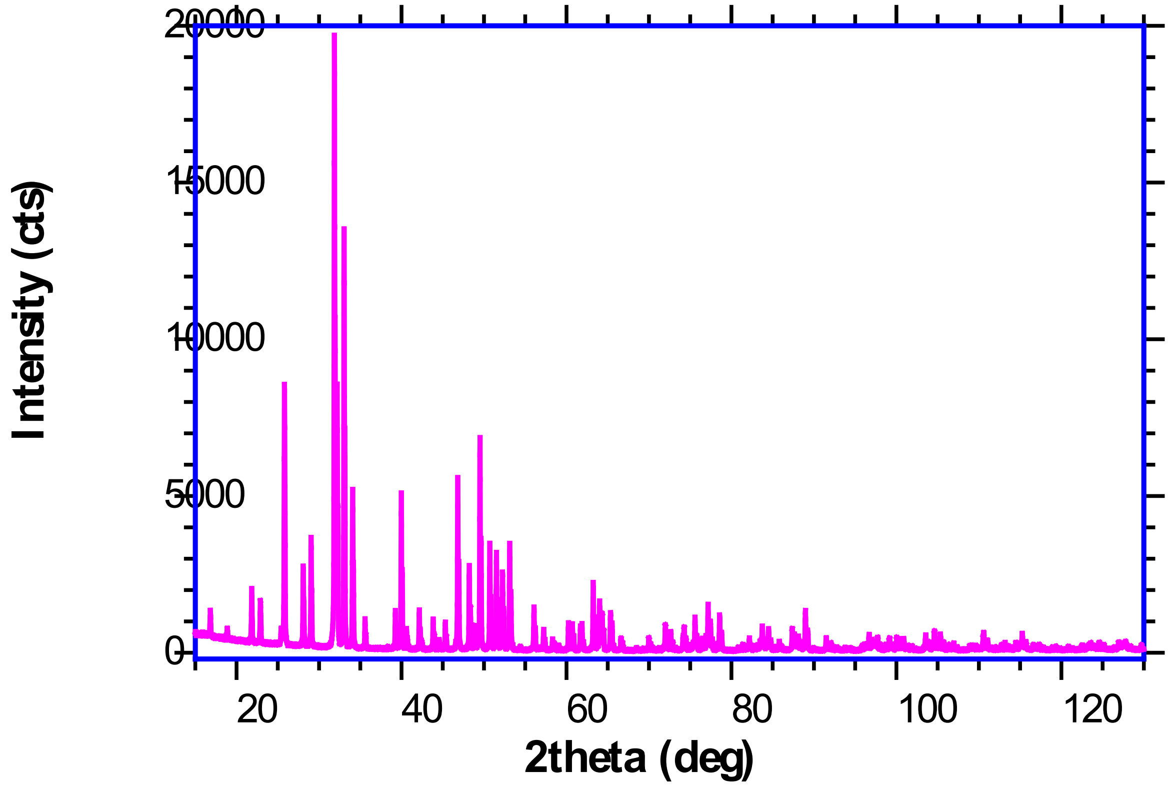


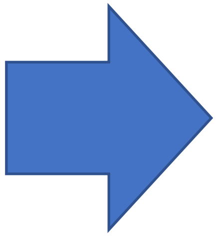
No.	h	k	l	d (Å)	F(real)	F(imag)	|F|	2θ	I	M
1	1	1	0	2.02600	33.6292	6.20768	34.1973	44.6928	100.0000	12
2	2	0	0	1.43260	26.7047	6.05816	27.3832	65.0532	13.7590	6
3	2	1	1	1.16971	22.1339	5.91224	22.9099	82.3764	24.8582	24
4	2	2	0	1.01300	18.875	5.76983	19.7372	99.0007	8.0713	12
5	3	1	0	0.90606	16.4507	5.63086	17.3877	116.459	14.4626	24

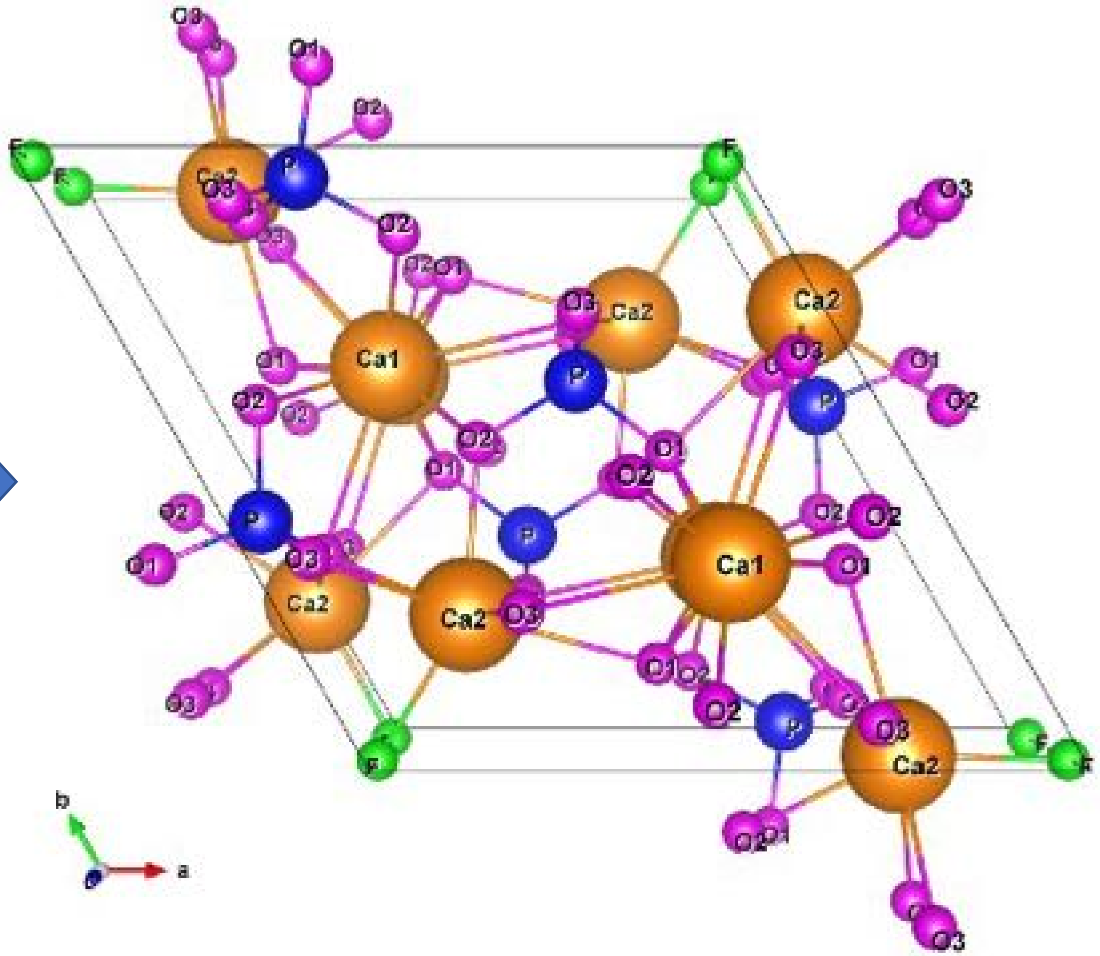
Powder Diffraction Pattern



How does PXRD work?

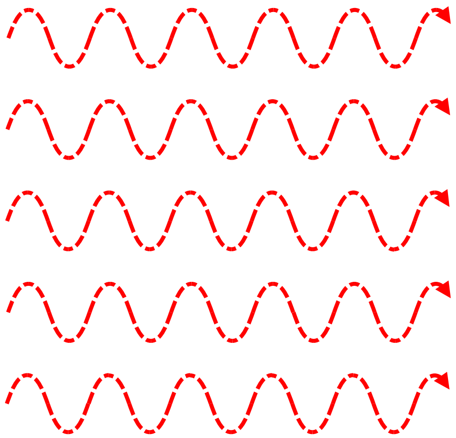




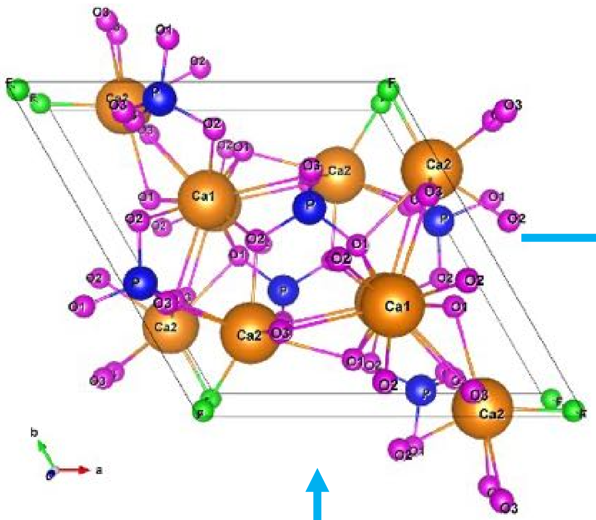


The Structure & Fourier Transform

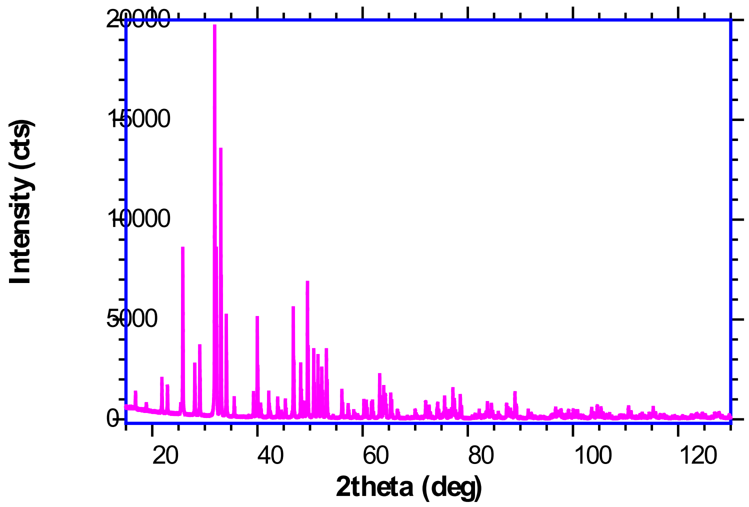
X-Ray



Crystal Space (3D)



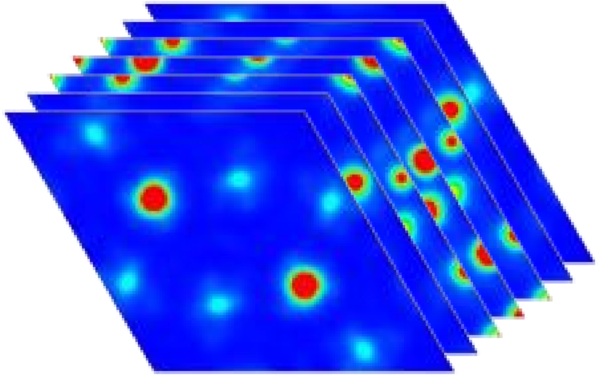
Reciprocal Space (1D)



$I \sim F^2$

$$F_{hkl} = \sum_{1}^{N} f_n \, e^{2\pi i (h x_n + k y_n + l z_n)}$$

Fourier Synthesis

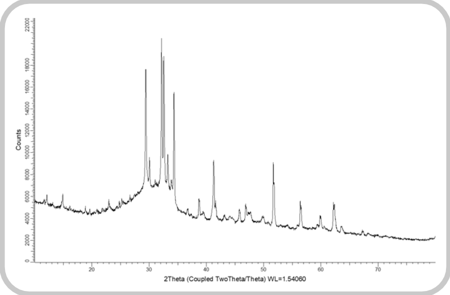


Electron/nuclear density map

Phase Analysis Flow Chart

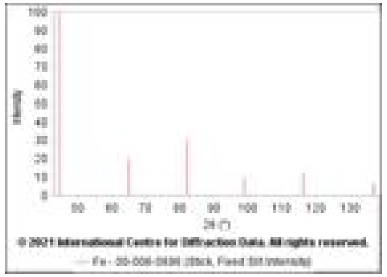
XRD (Emission, Radius, Optics, Detector)

RAW data (.brml, .raw, .xy)



EVA

Qualitative Analysis (Search and Match)



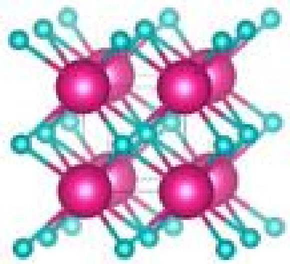
Stick pattern

ICDD-PDF4 (Database)

*.CIF, *.STR

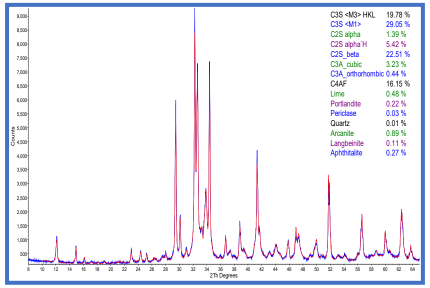
TOPAS

Quantitative Analysis (Rietveld analysis)

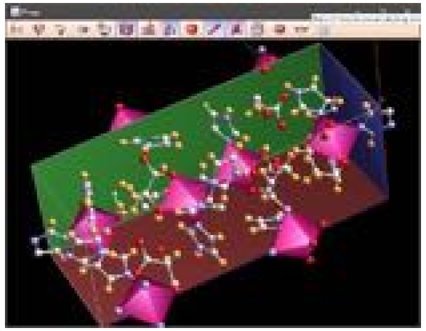


VESTA

Refinement report
- Diffractogram
- R-factors
- Structure
- QPA
- Crystallite Size
- Preferred Orientation
- etc.



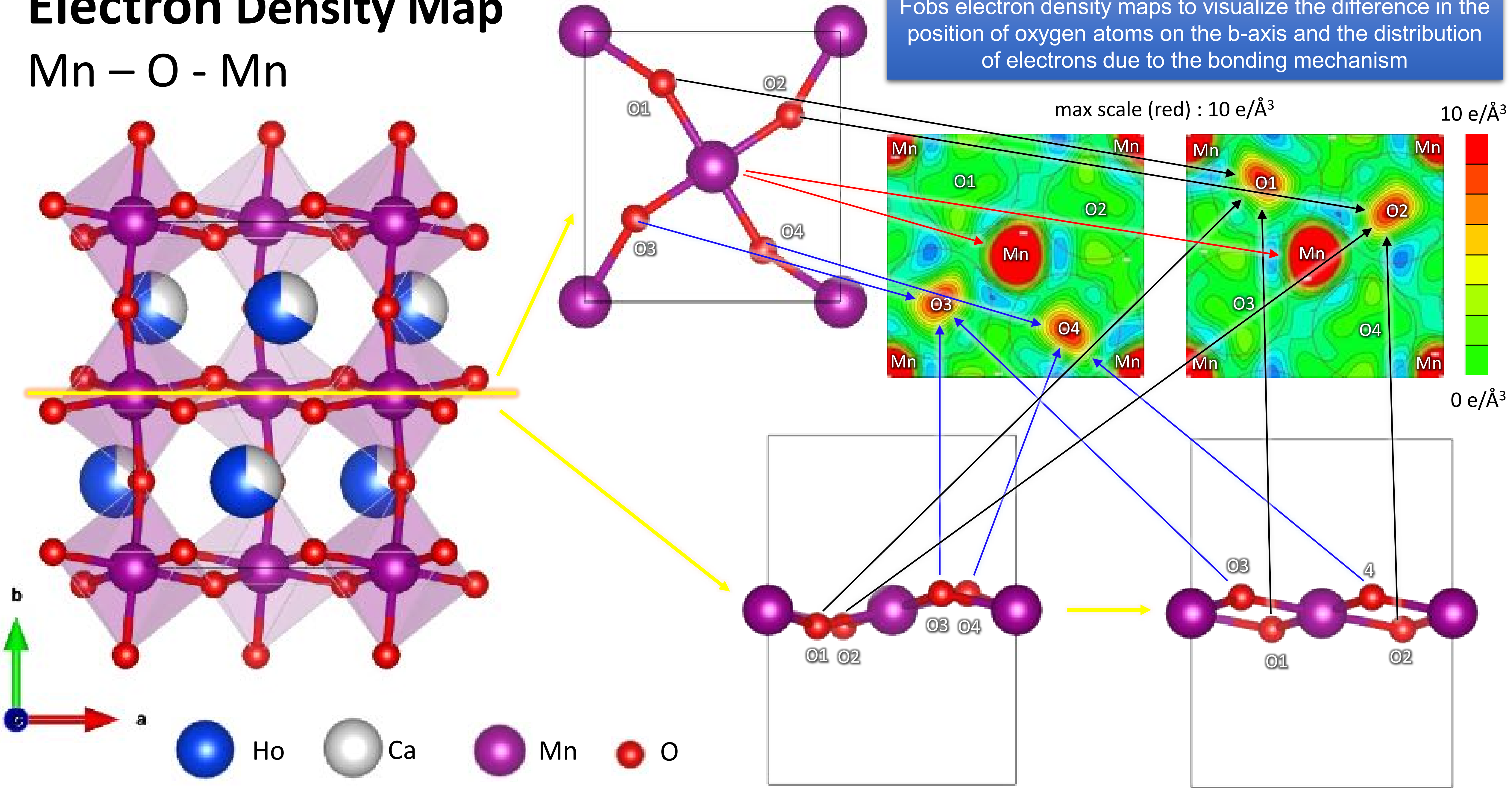
Refined Structure files
- *.CIF
- *.STR



Electron Density Map

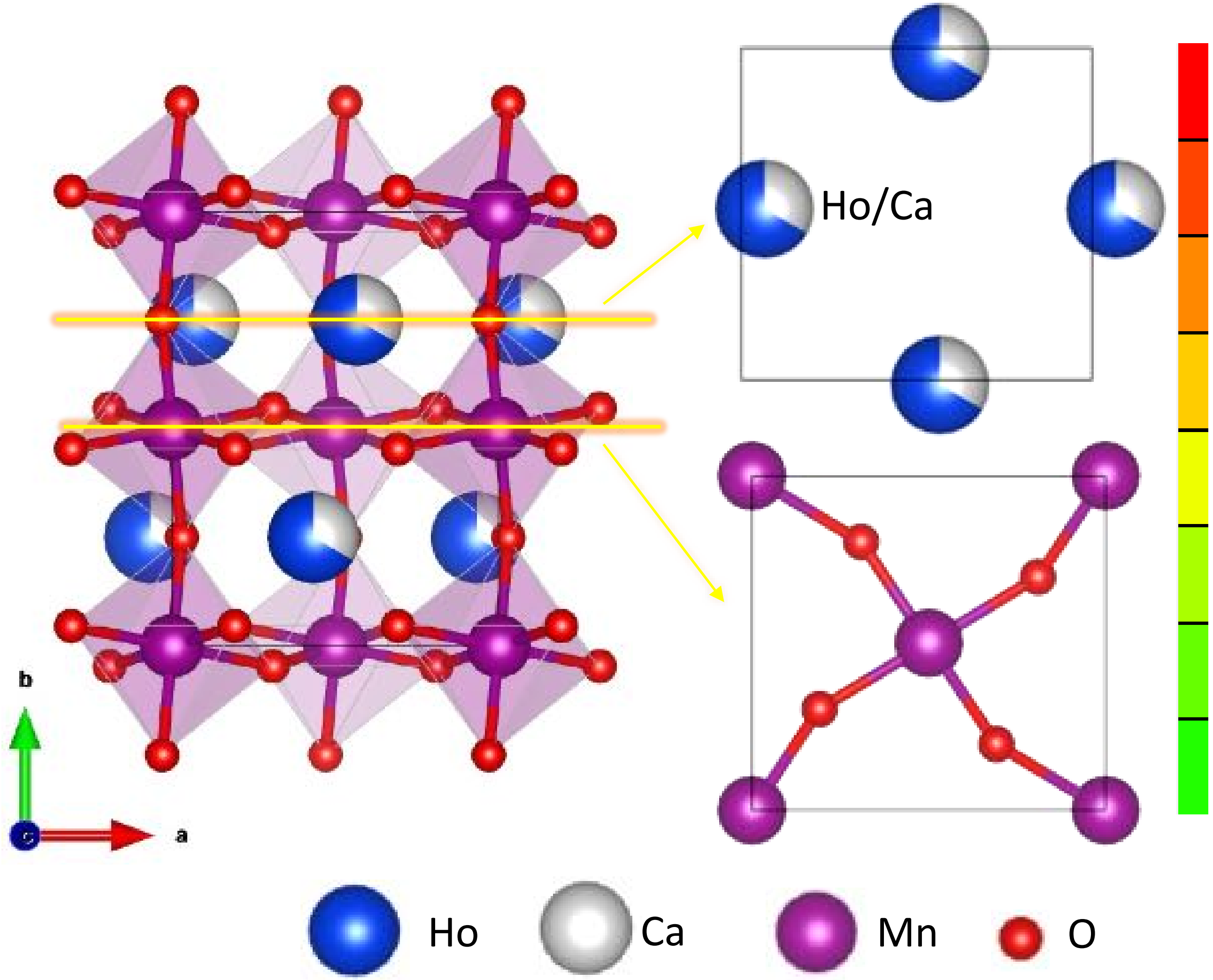
Mn – O - Mn

Fobs electron density maps to visualize the difference in the position of oxygen atoms on the b-axis and the distribution of electrons due to the bonding mechanism



Electron Density Map

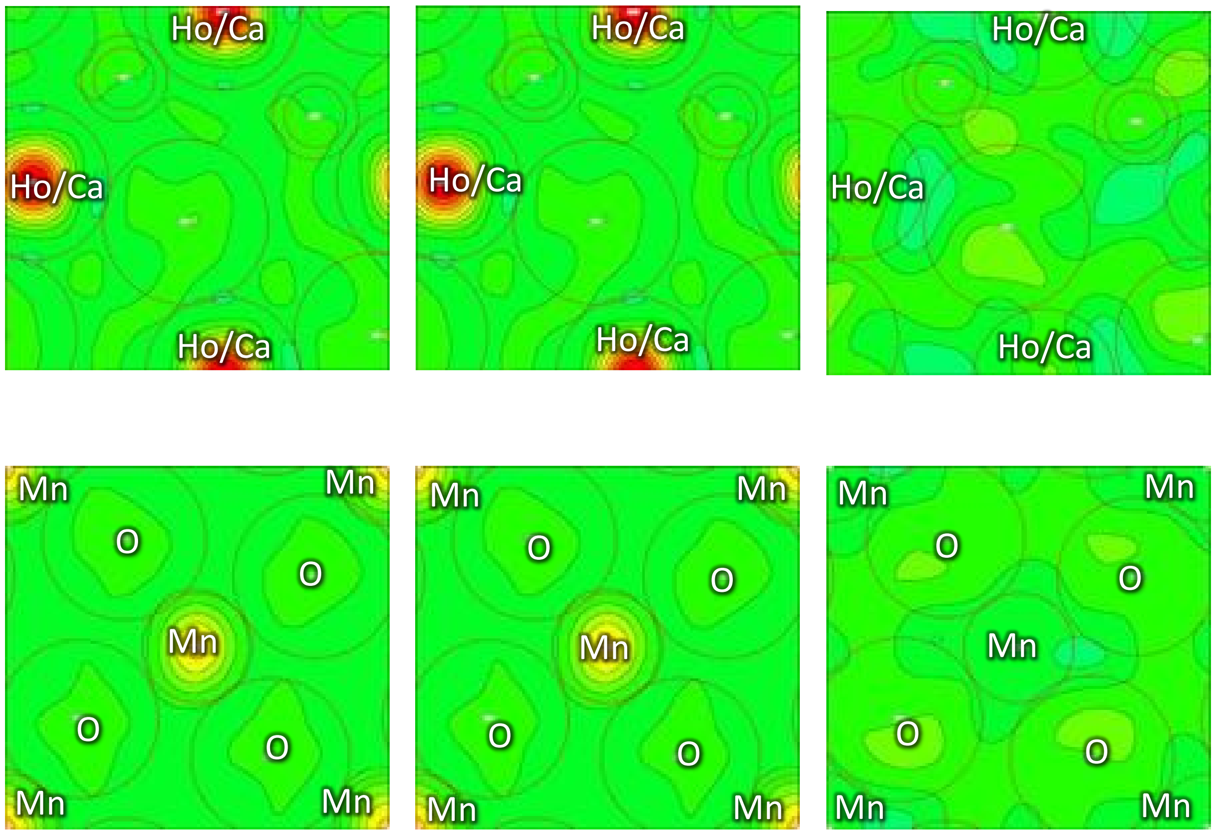
(Ho0.669 Ca0.331 MnO3)



Fobs
max scale (red): 75.6 e/Å^3

Fcalc
max scale (red): 75.4 e/Å^3

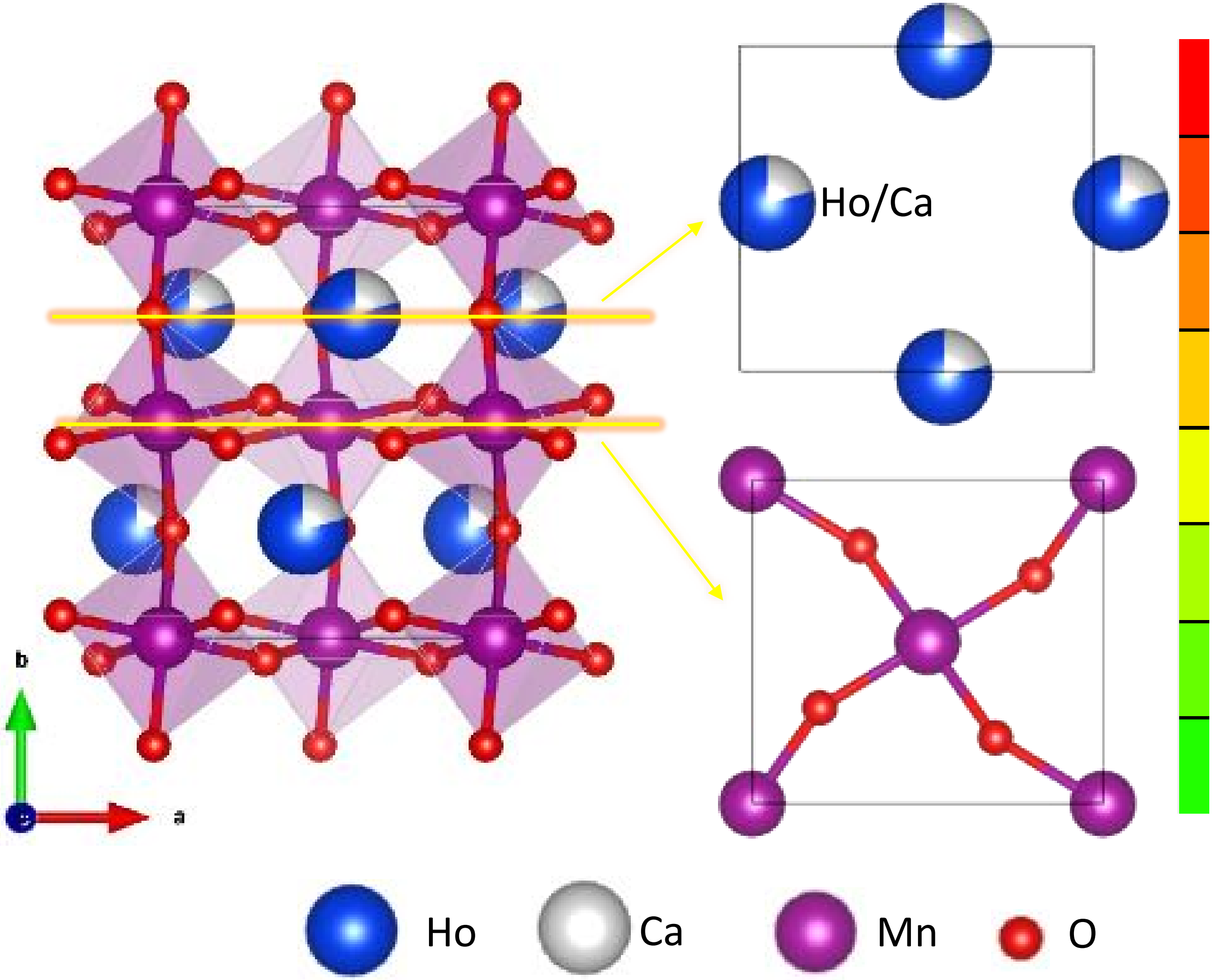
(Fobs – Fcalc)
max scale (red): 2 e/Å^3

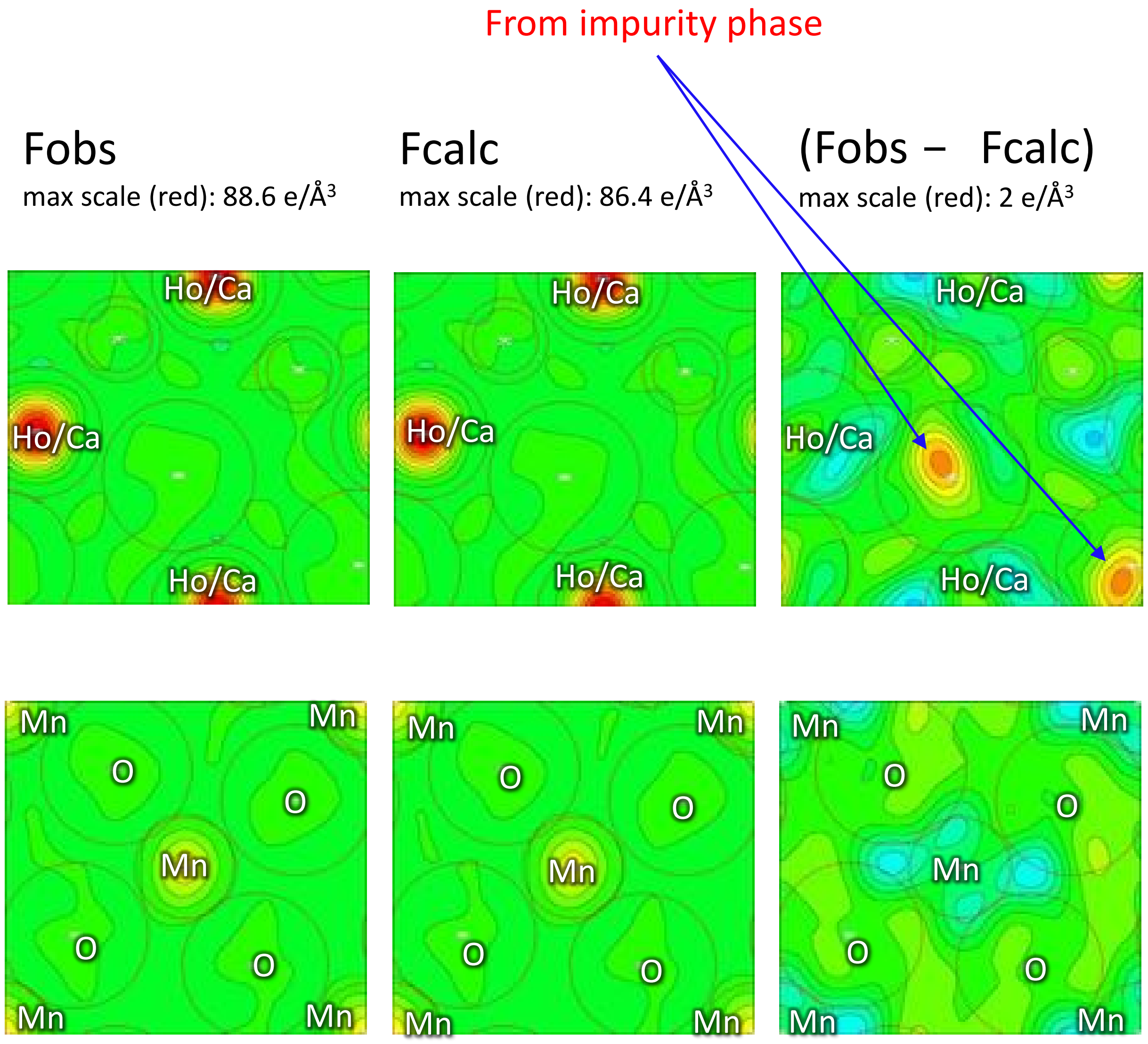


Fobs	:	Fourier electron density map from Observed data
Fcalc	:	Fourier electron density map from Calculated structure
Fobs-Fcalc	:	Fourier electron density difference (indicate if calculated structure are match with observed data)

Electron Density Map

($Ho_{0.799}$ $Ca_{0.201}$ MnO_3)



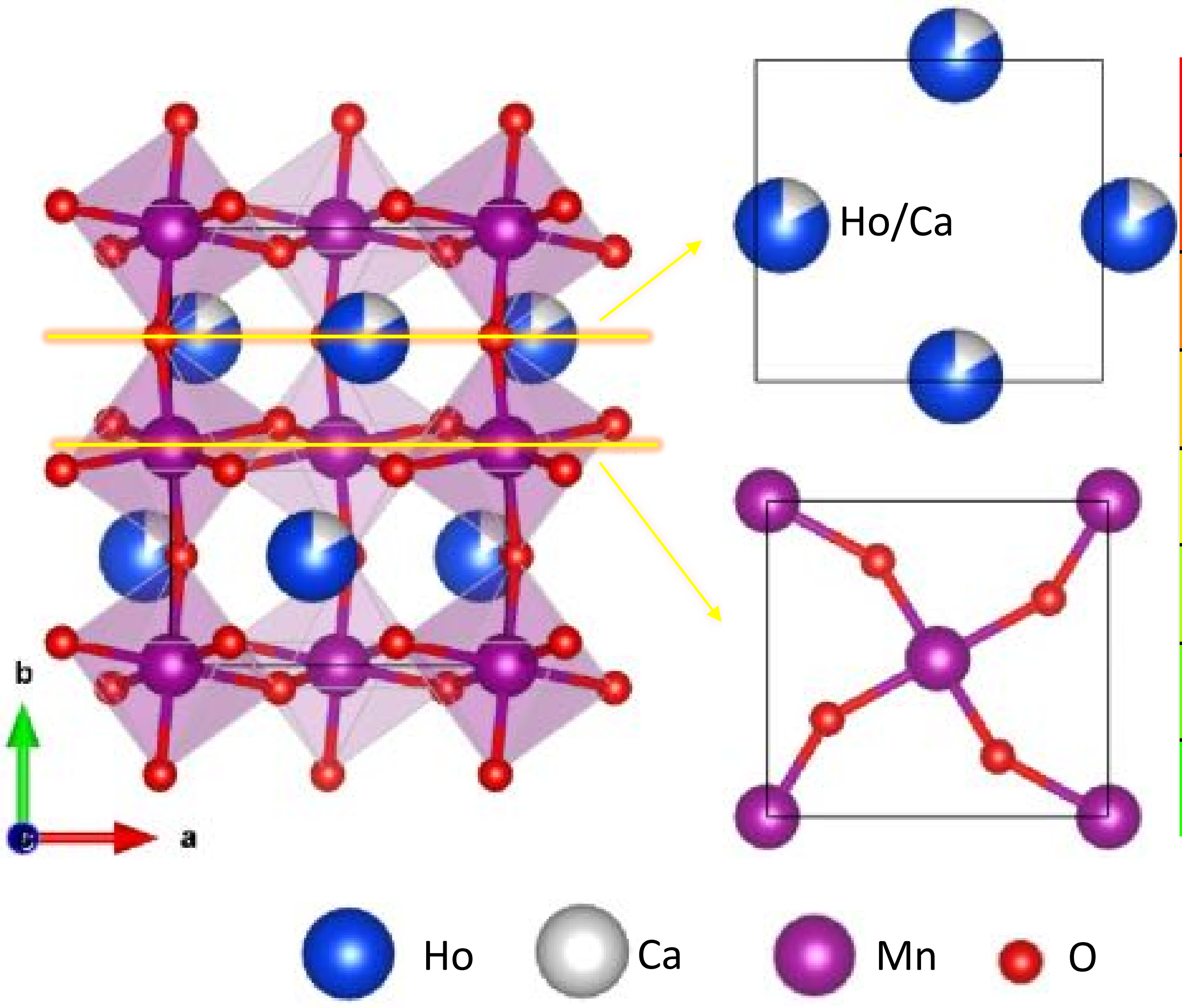


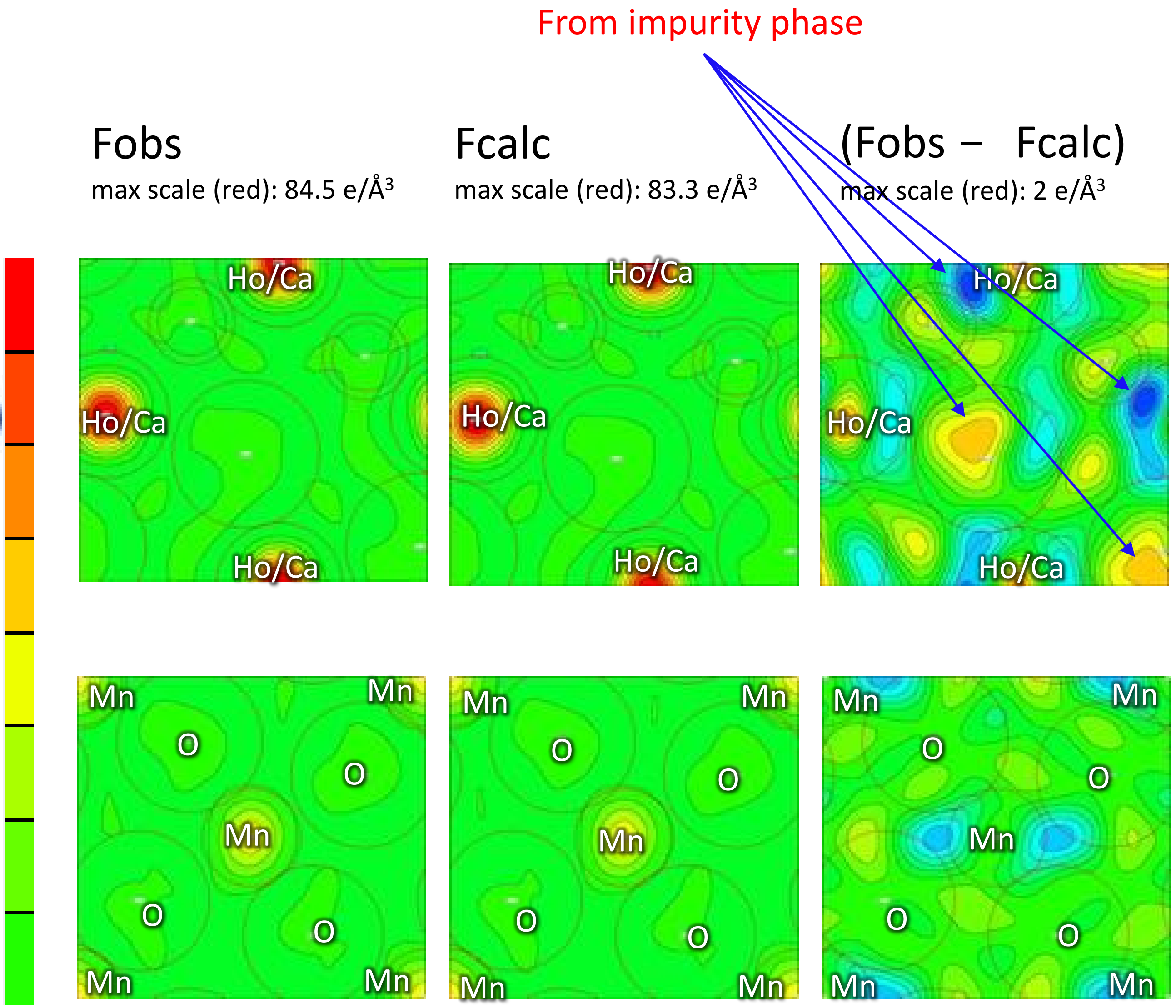
Fobs : Fourier electron density map from Observed data

Fcalc : Fourier electron density map from Calculated structure

Fobs-Fcalc : Fourier electron density difference (indicate if calculated structure are match with observed data)

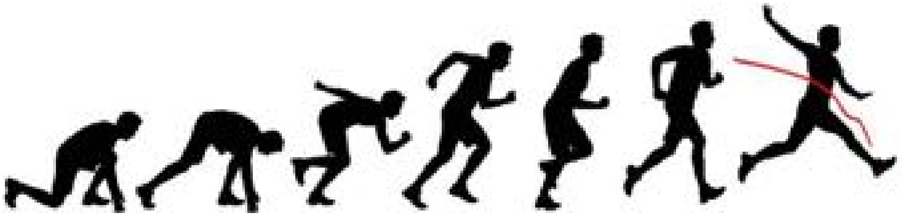
Electron Density Map
(Ho0.812 Ca0.188 MnO3)



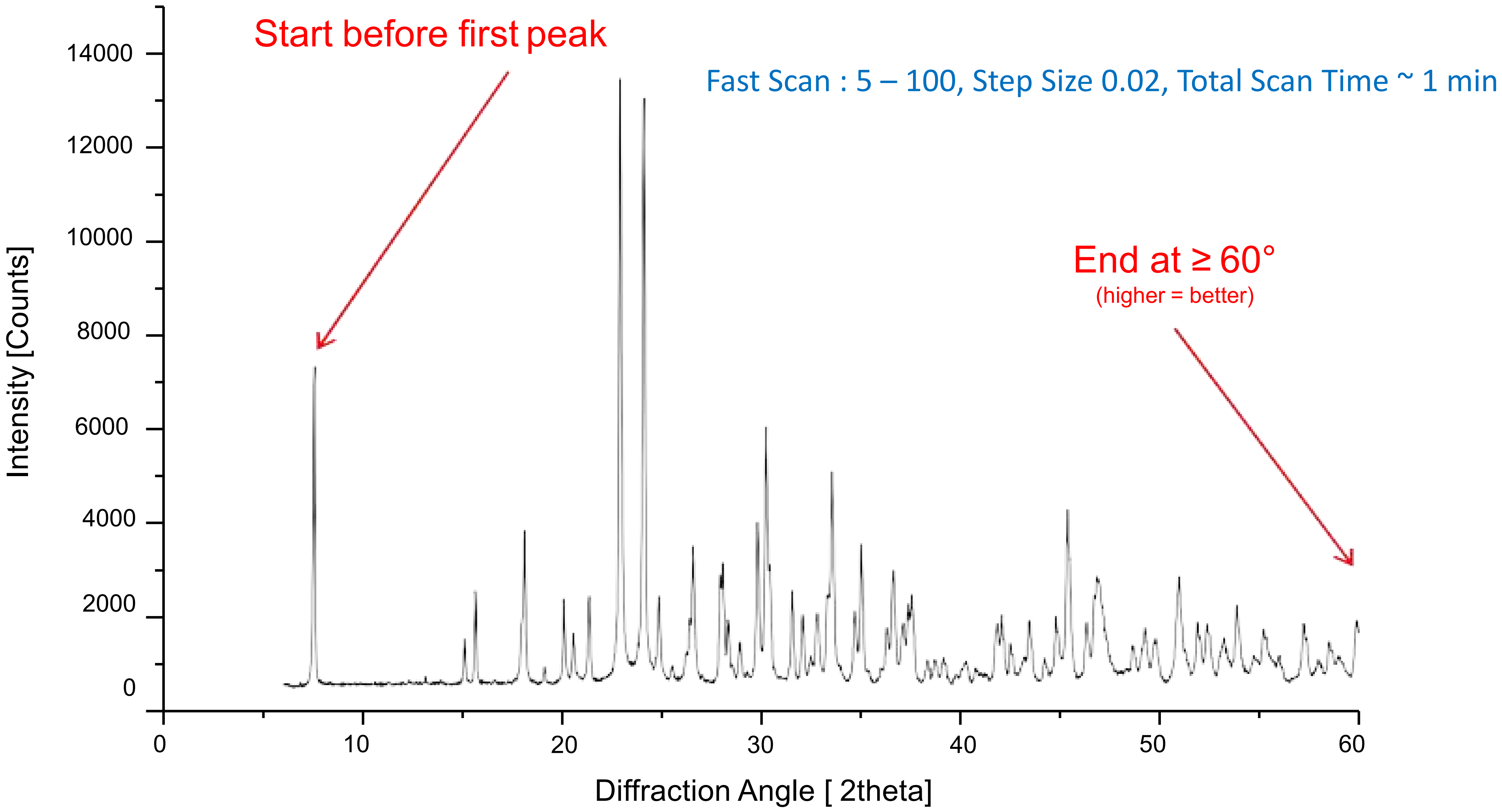


Fobs	:	Fourier electron density map from Observed data
Fcalc	:	Fourier electron density map from Calculated structure
Fobs-Fcalc	:	Fourier electron density difference (indicate if calculated structure are match with observed data)

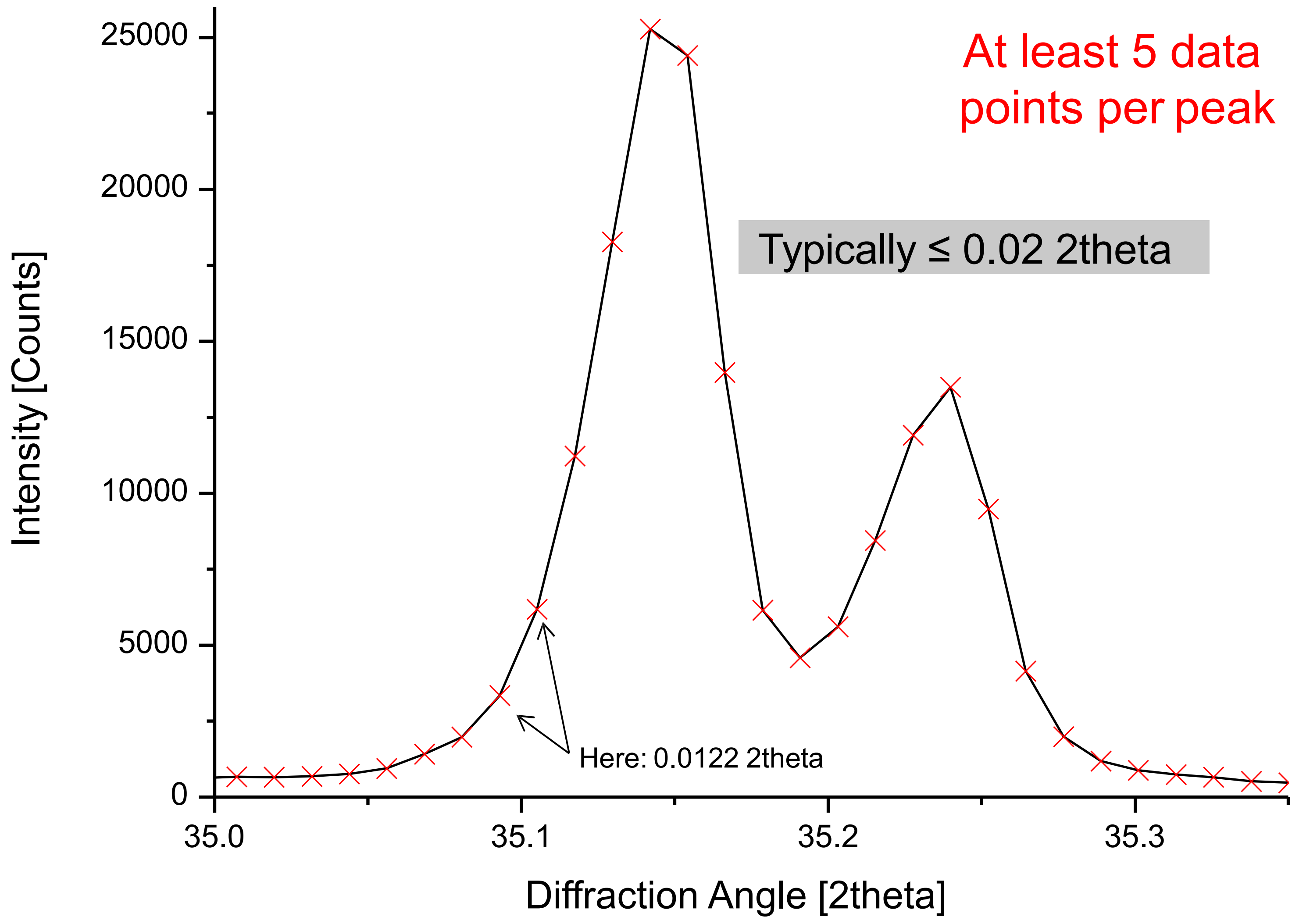
- What is Powder
- X-ray generation
- Data Acquisition
- Peak Profiles
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- Miscellaneous



Angular Range



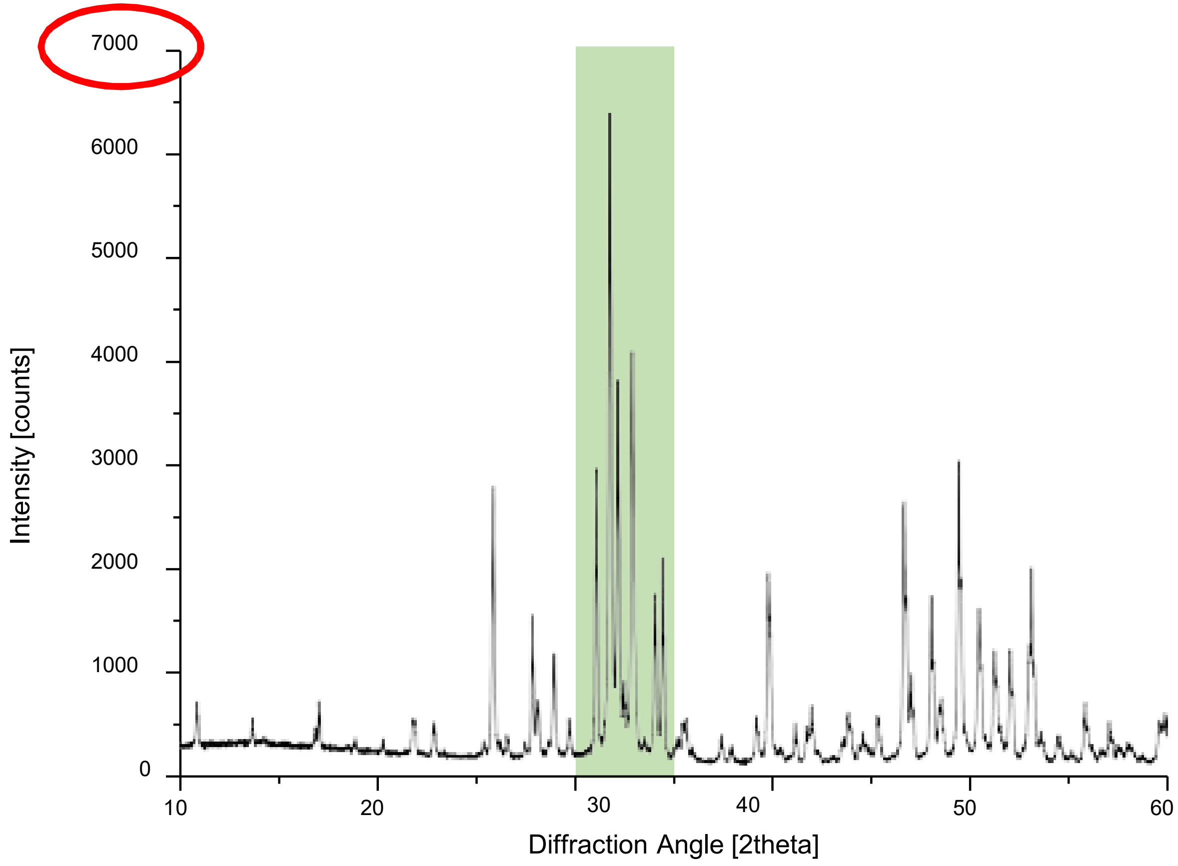
Step Size



At least 5 data points per peak
Typically ≤ 0.02 2theta
Here: 0.0122 2theta
Intensity [Counts]
25000
20000
15000
10000
5000
0
35.0
35.1
35.2
35.3
Diffraction Angle [2theta]

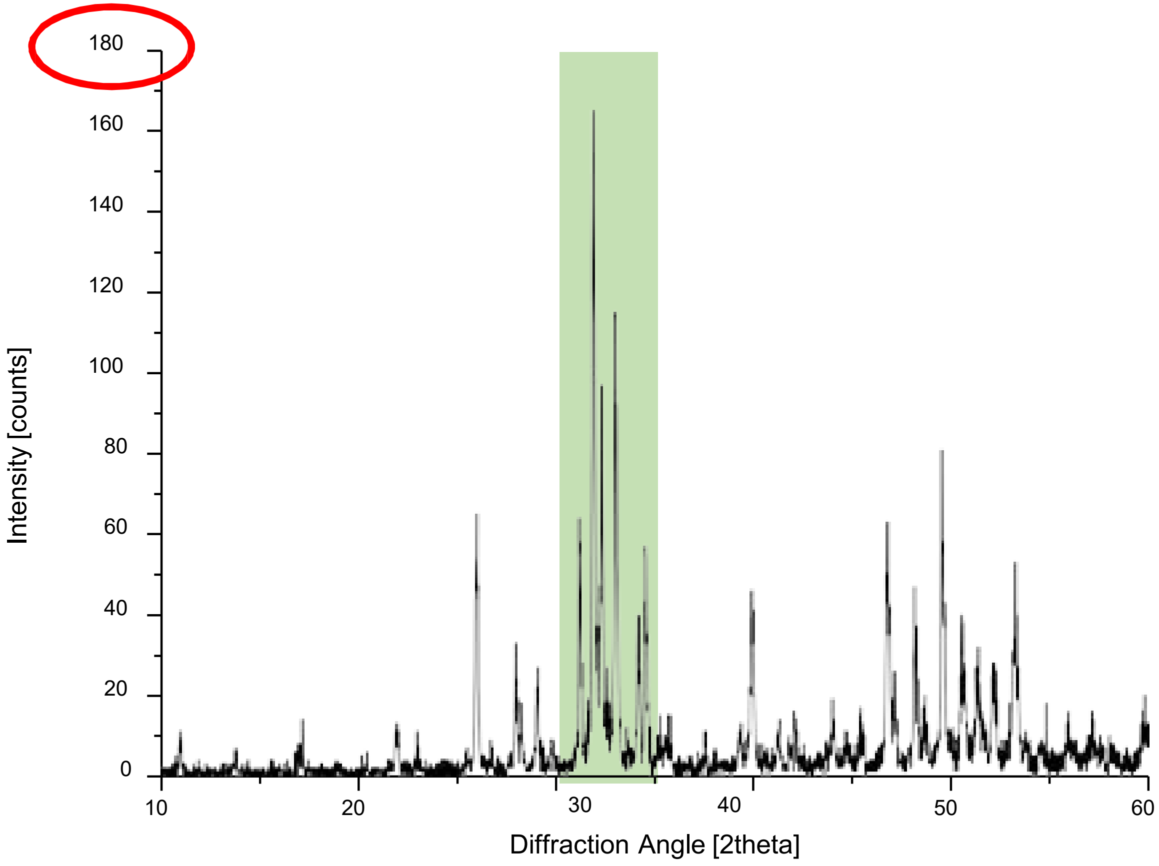
Scan Time

1D Energy dispersive Detector



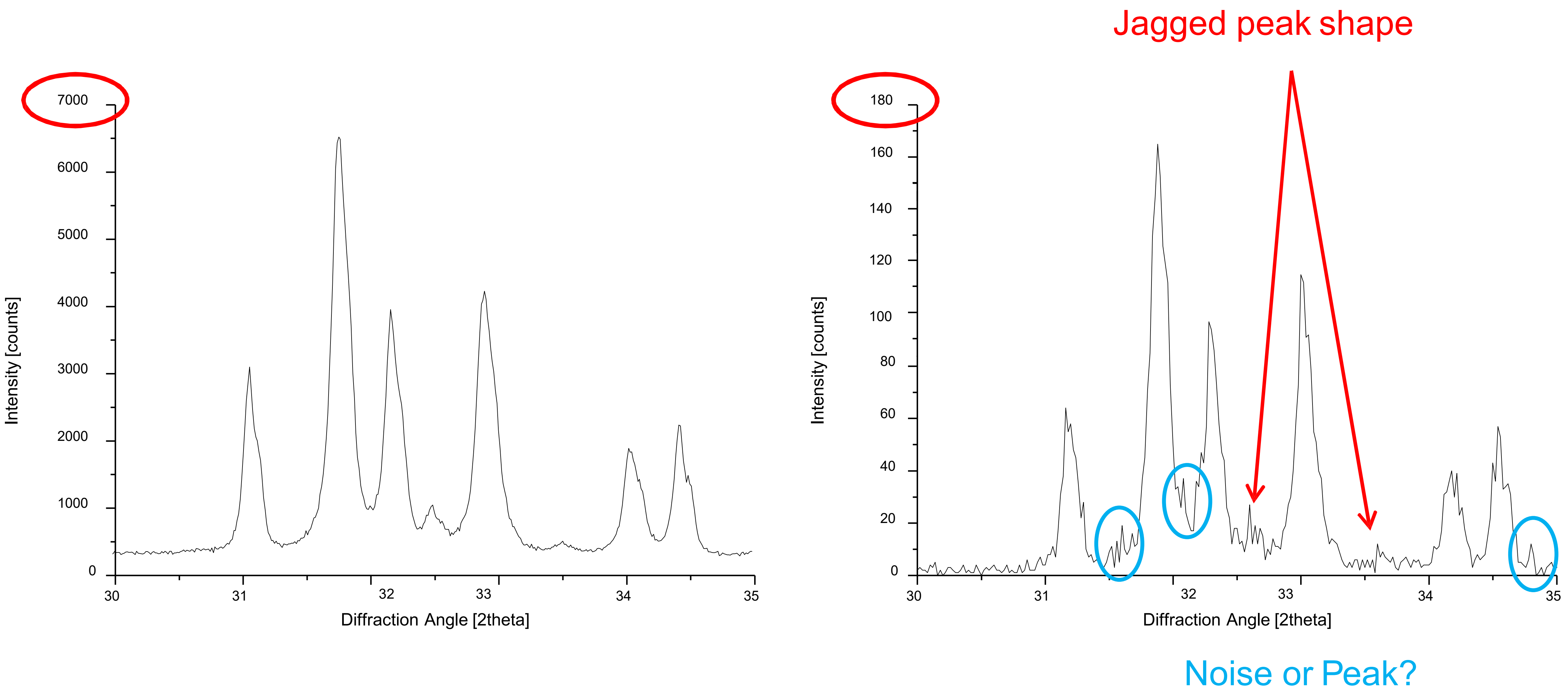
4.2 min

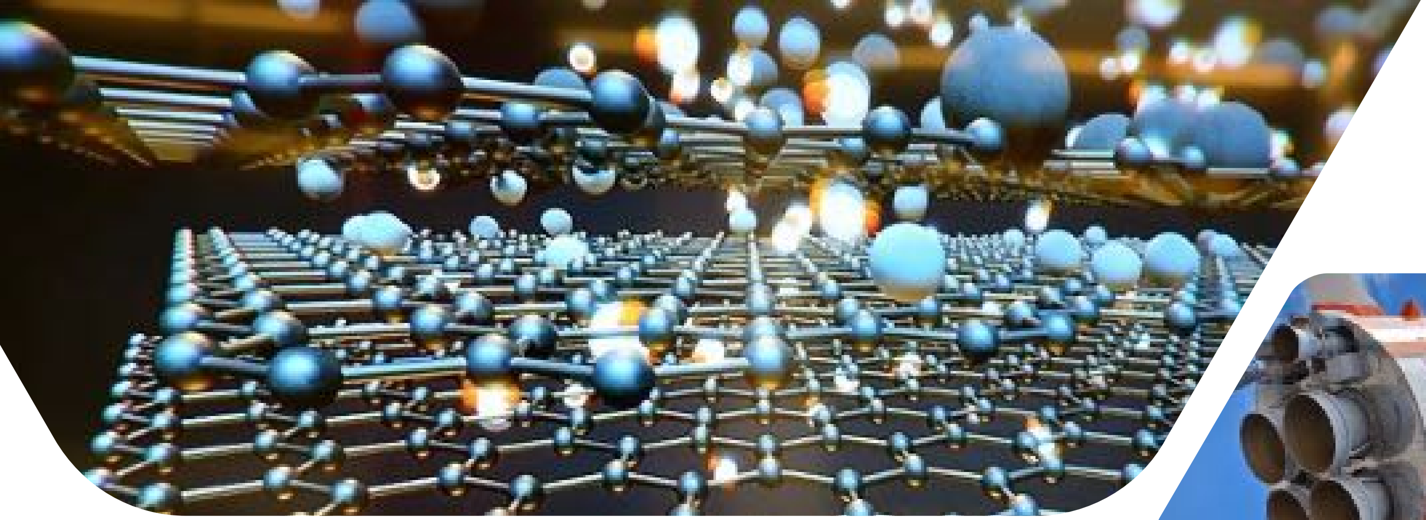
0D Detector



4.2 min

Scan Time

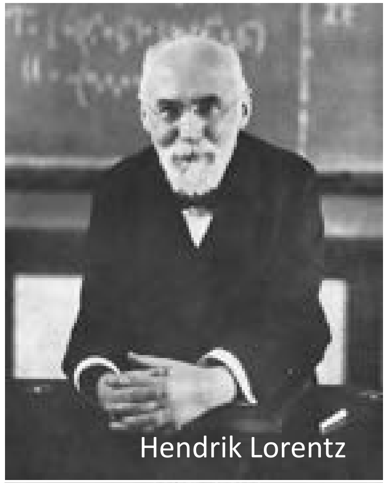

Jagged peak shape
7000
6000
5000
4000
3000
2000
1000
0
Intensity [counts]
30
31
32
33
34
35
Diffraction Angle [2theta]
180
160
140
120
100
80
60
40
20
0
Intensity [counts]
30
31
32
33
34
35
Diffraction Angle [2theta]
Noise or Peak?



END
Session 3

- What is Powder
- X-ray generation
- Data Acquisition
- **Peak Profiles**
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- Miscellaneous

Carl Friedrich Gauss

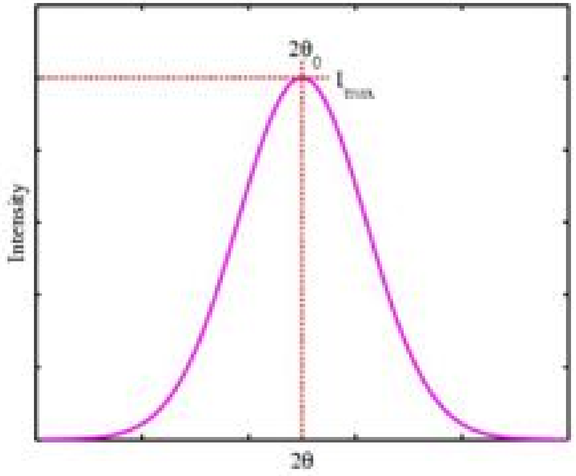


Hendrik Lorentz

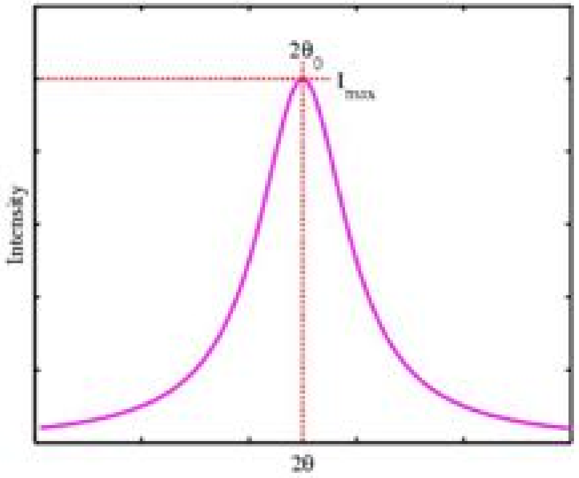
Augustin-Louis Cauchy

Woldemar Voigt

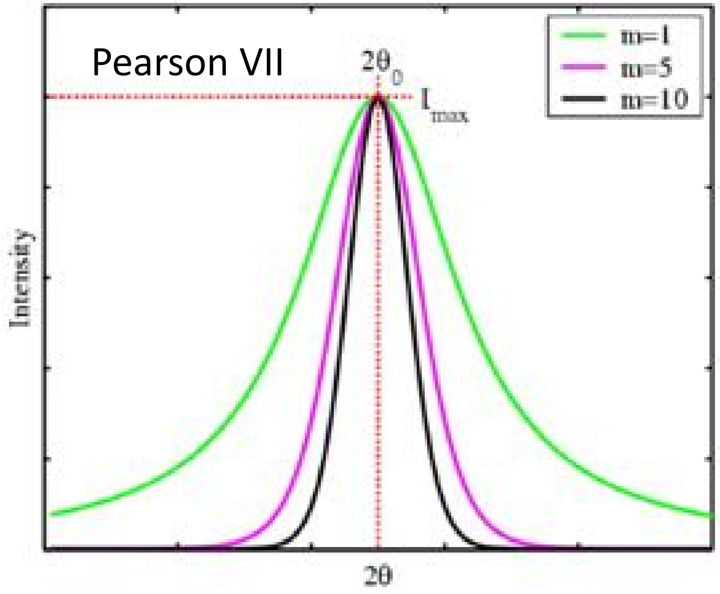
Origin of Profile Shapes

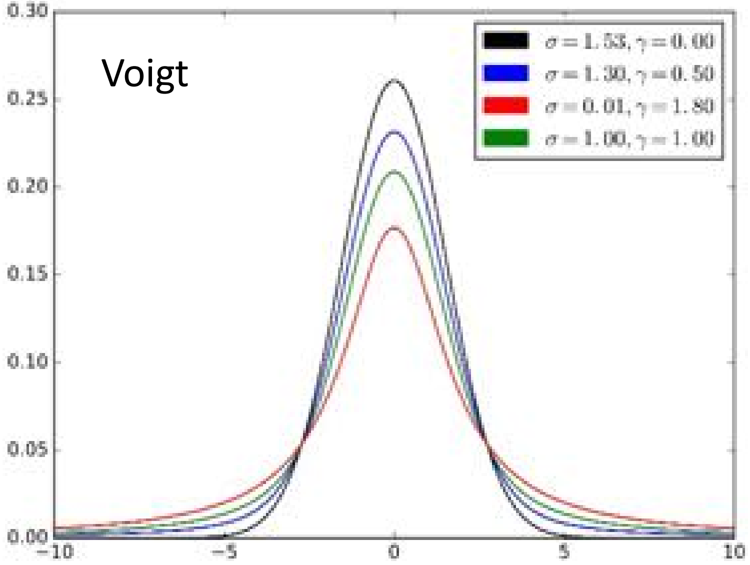


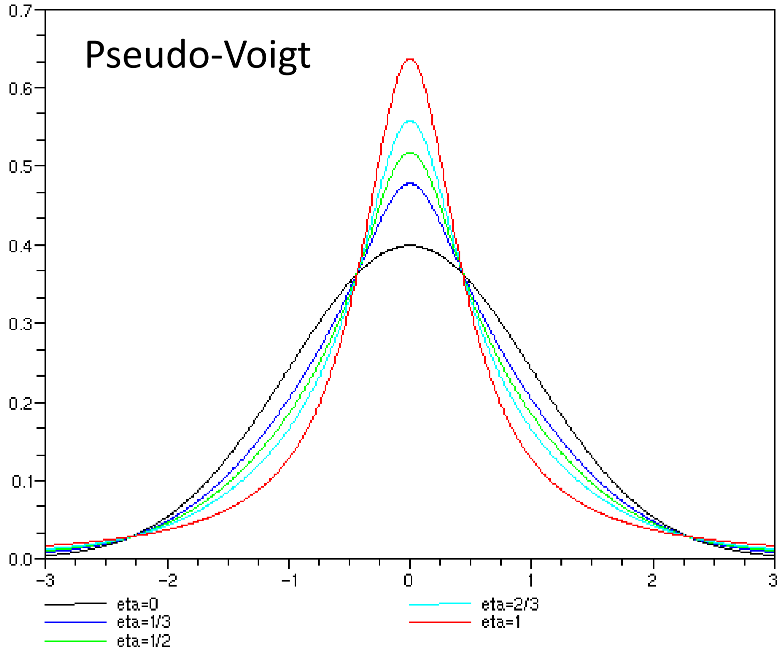
Gaussian Function



Lorentzian/ Cauchy Function

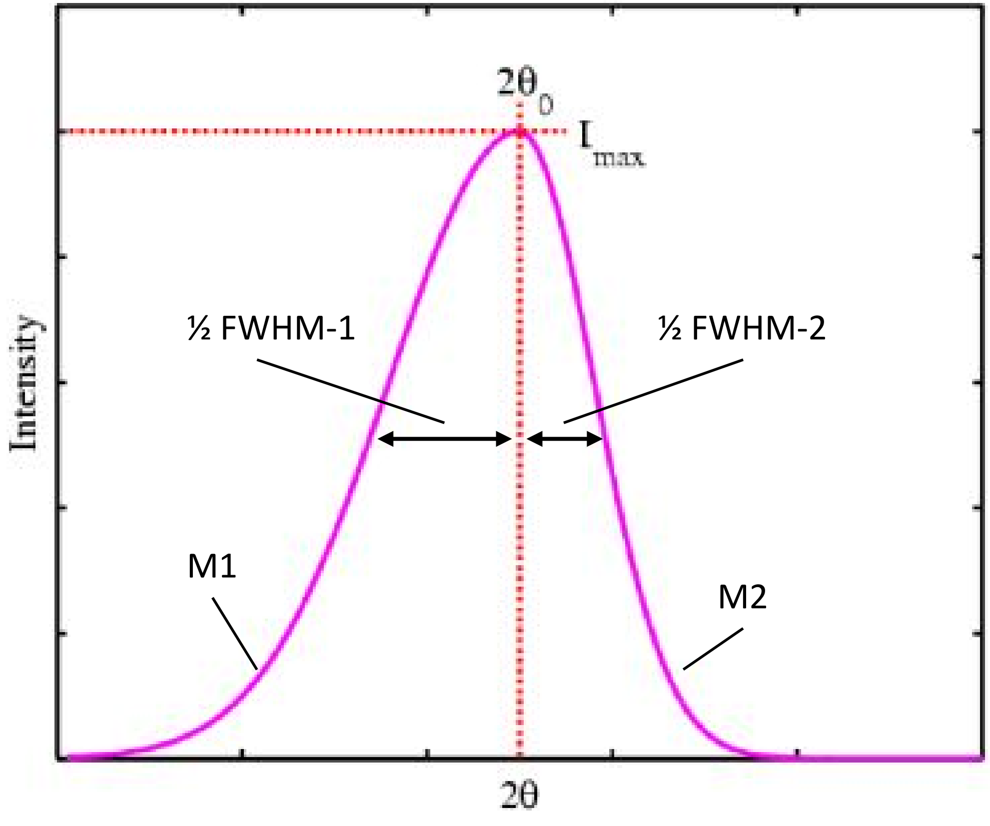






Real Peak Shape

- Split the peaks in halves to fit peak asymmetry
- Split functions model the low angle and high angle side of a peak separately, and thus can better describe asymmetric peaks

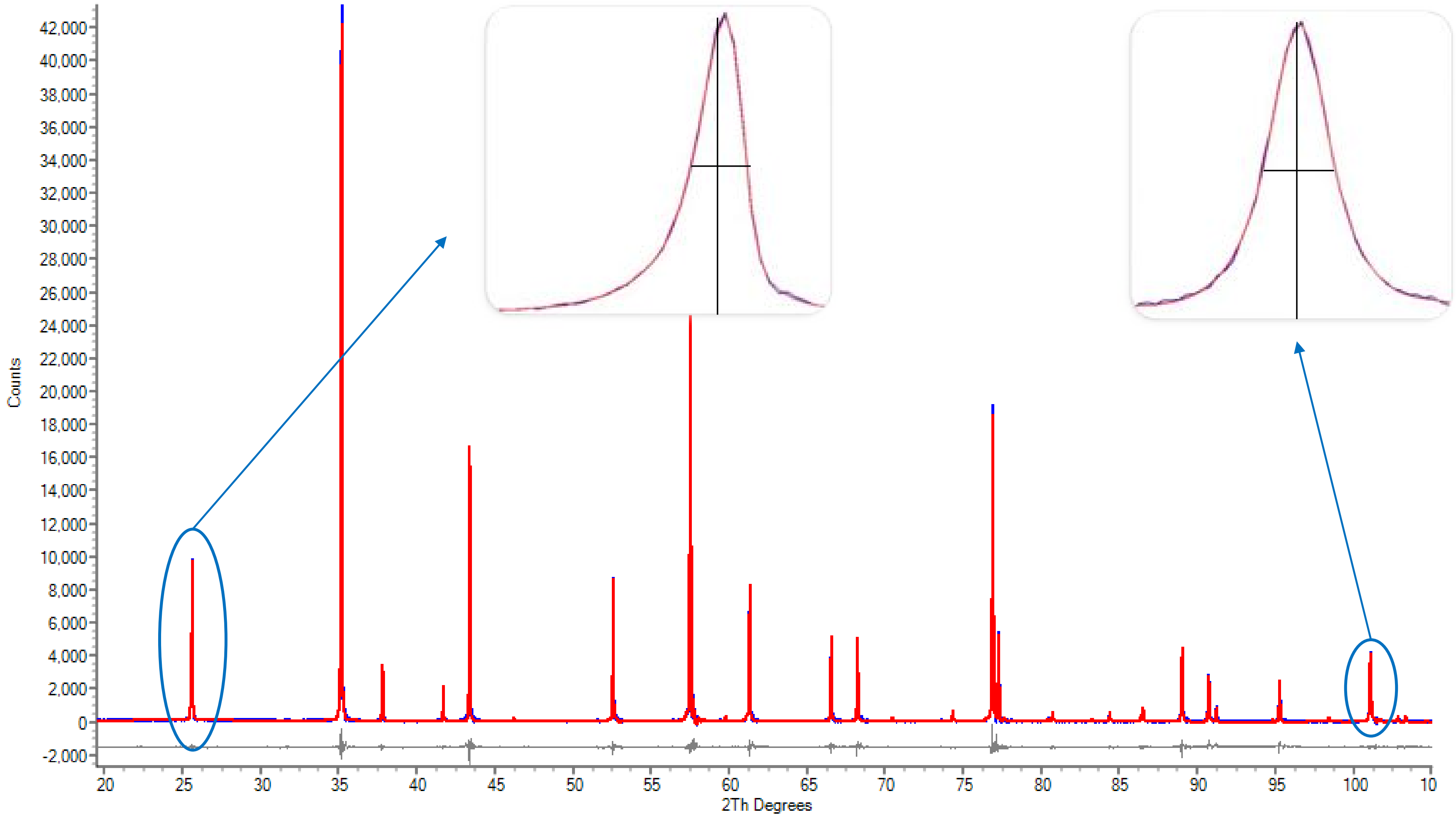


Split Pearson-VII model (A. Kern, 1992)

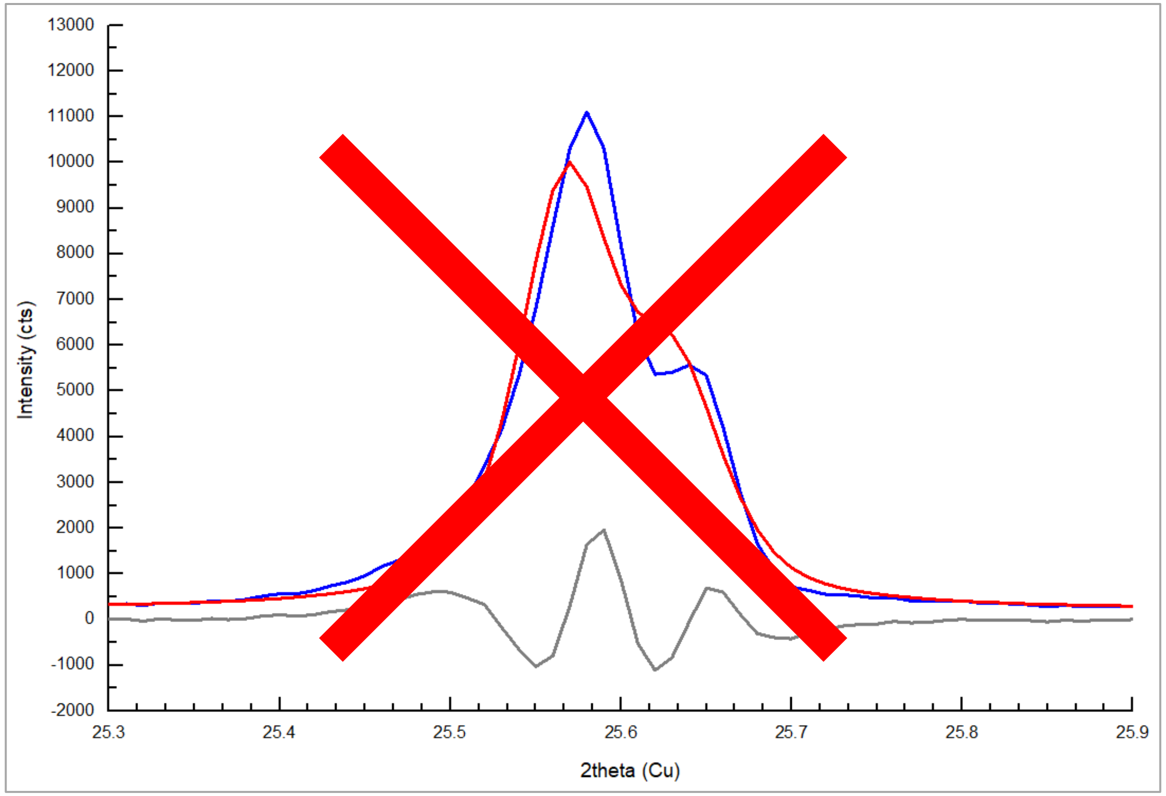
6 refinable parameters!

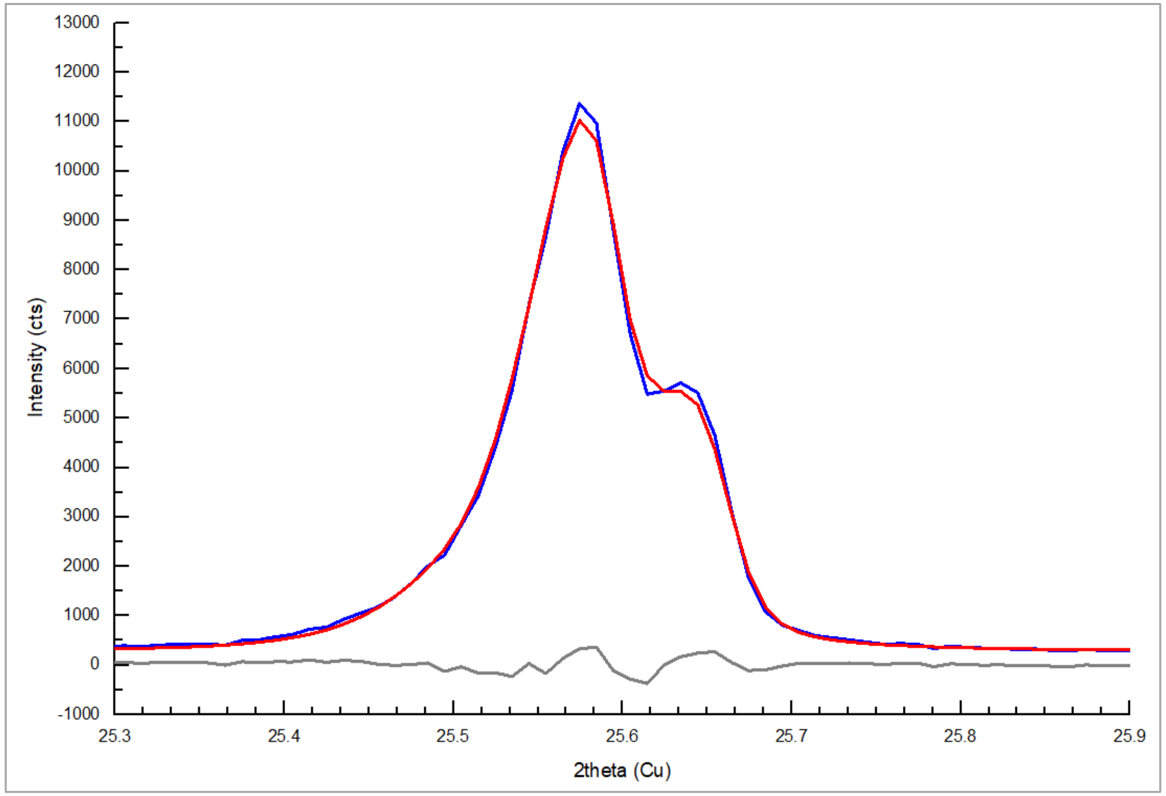
$$I(2\theta) = I_{max} \frac{w^{2m}}{[\, w^2 + (2^{1/m} - 1)\,(2\theta - 2\theta_0)^2 \,]^{\,m}}$$

Real Diffraction Data ($K\alpha_2$ subtracted)



Good Fitting





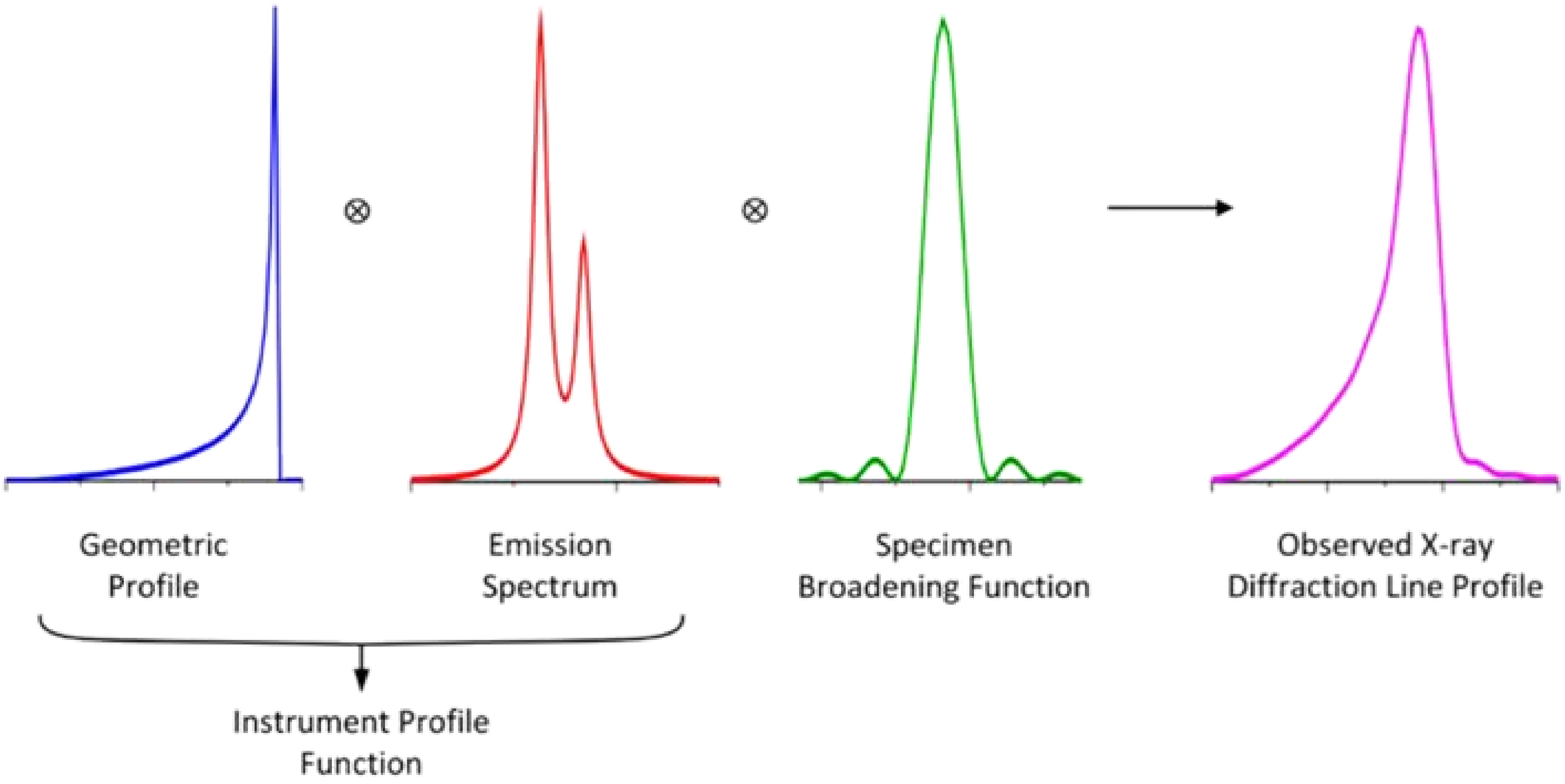
Good fitting is necessary to extract accurate:

- Peak position
- FWHM/Integrated intensity
- Profile coefficients

Note the numbers refined is from the calculated peak (red) not the measured peak (blue)

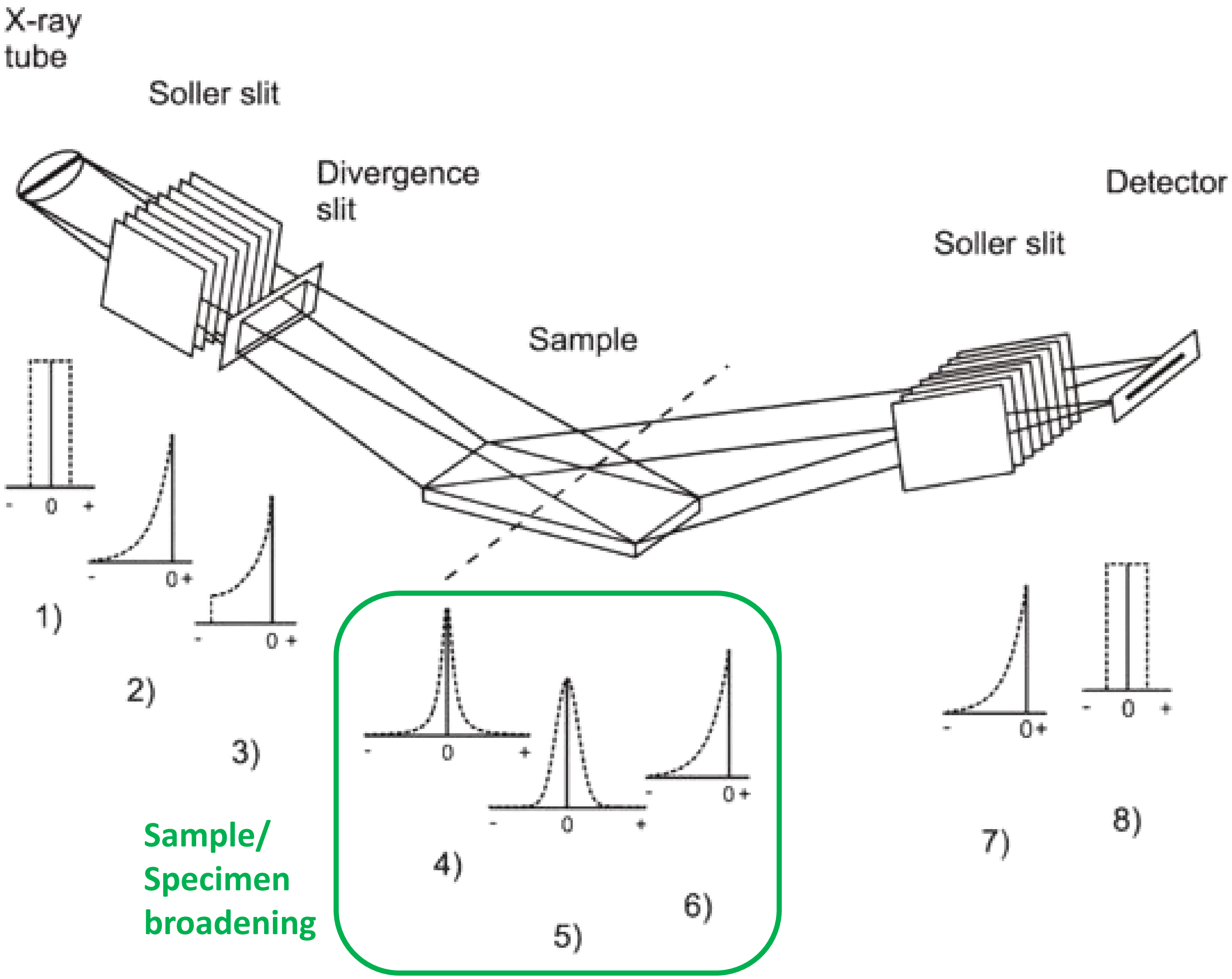
Fundamental Parameter Approach

$$Y(2\theta) = (W \otimes G) \otimes S$$



J. Res. Natl. Inst. Stand. Technol. 109 (2004) & 120 (2015)

Fundamental Parameter Approach

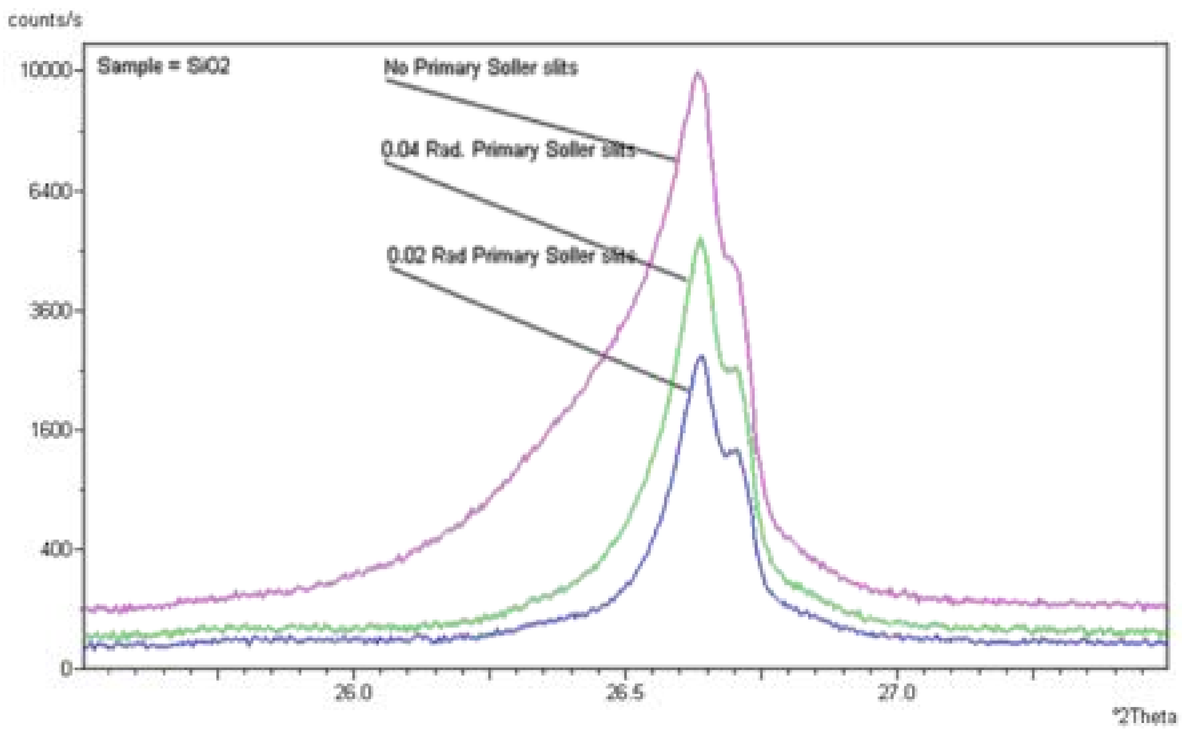


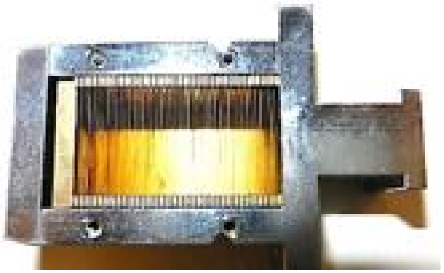
0) Emission Spectrum
1) Finite width of X-ray source
2) Primary axial divergence
3) Equatorial divergence
4) Crystallite size
5) Macrostrain
6) Specimen transparency
7) Secondary axial divergence
8) Receiving slit width

Topas 6 - Technical Reference (2017)

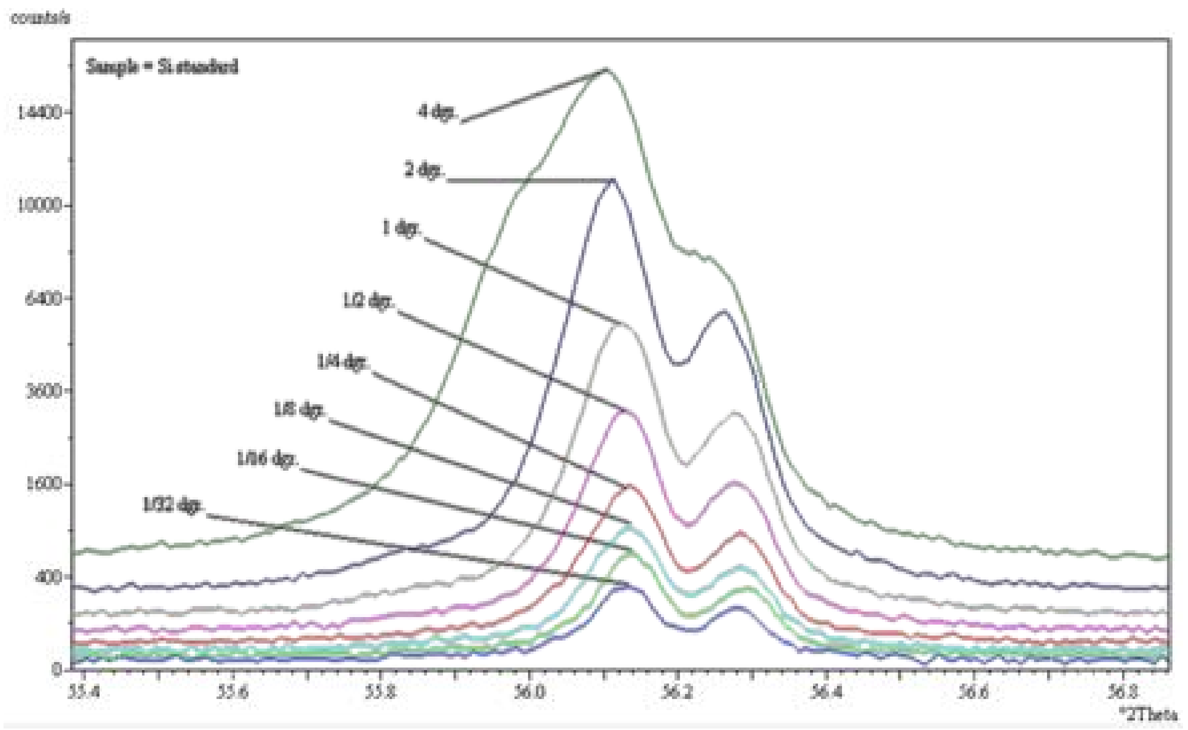
Instrumental Broadening

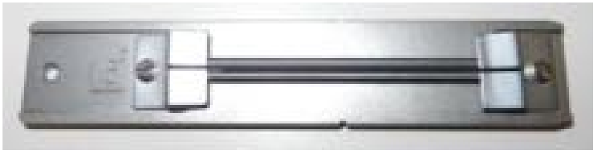
Soller Slit



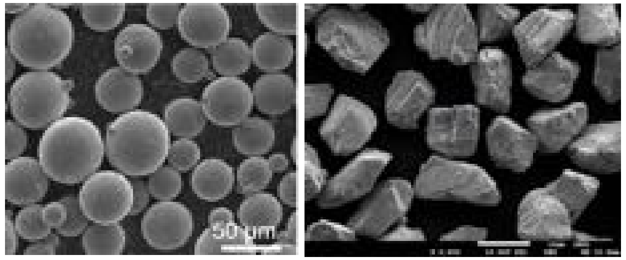


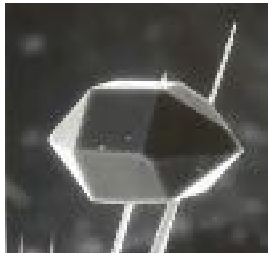
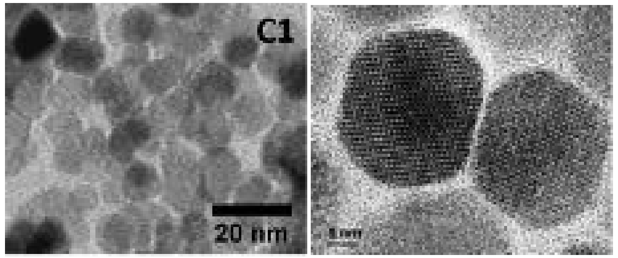
Divergence Slit

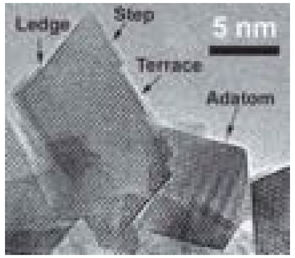


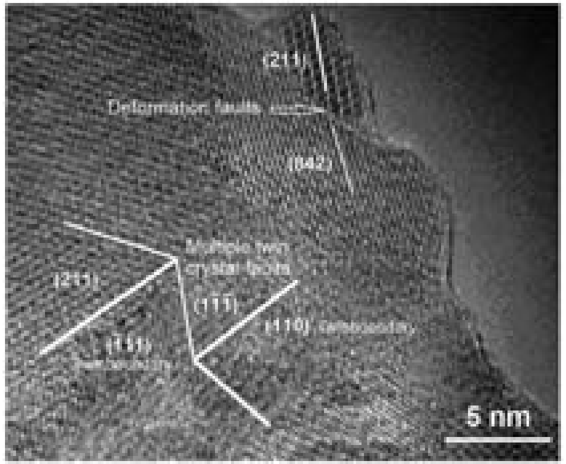


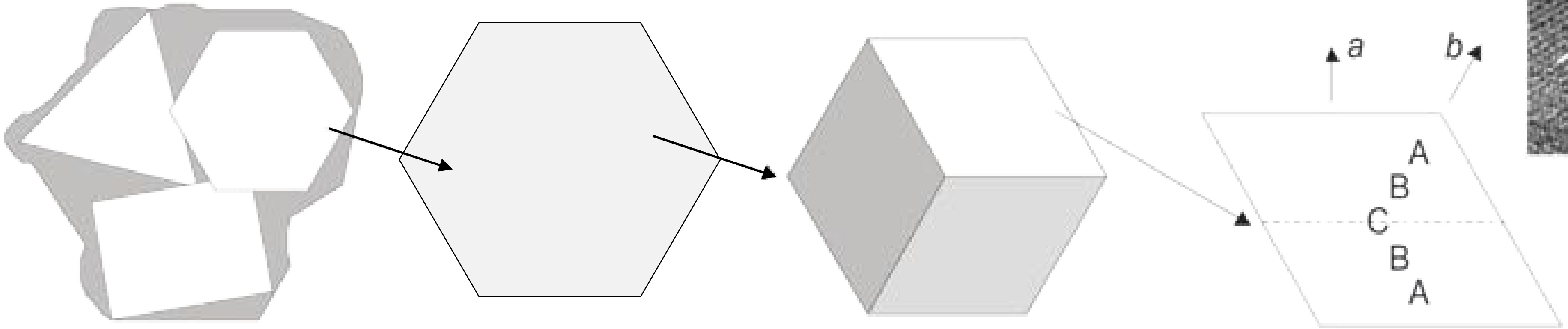
Sampel Broadening – Crystallite Size

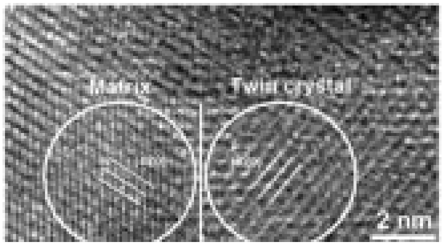


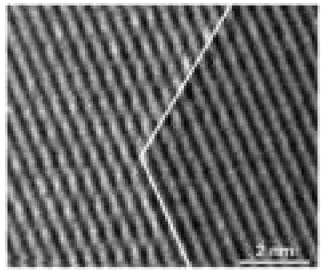













Particle

- Consists of several, separated crystals

Crystal

- Infinite, 3D periodic lattice
- Surface → 2D defect

Crystallite

- Small crystals
- Possibly held together through defective boundaries

Indirectly determined by PXRD

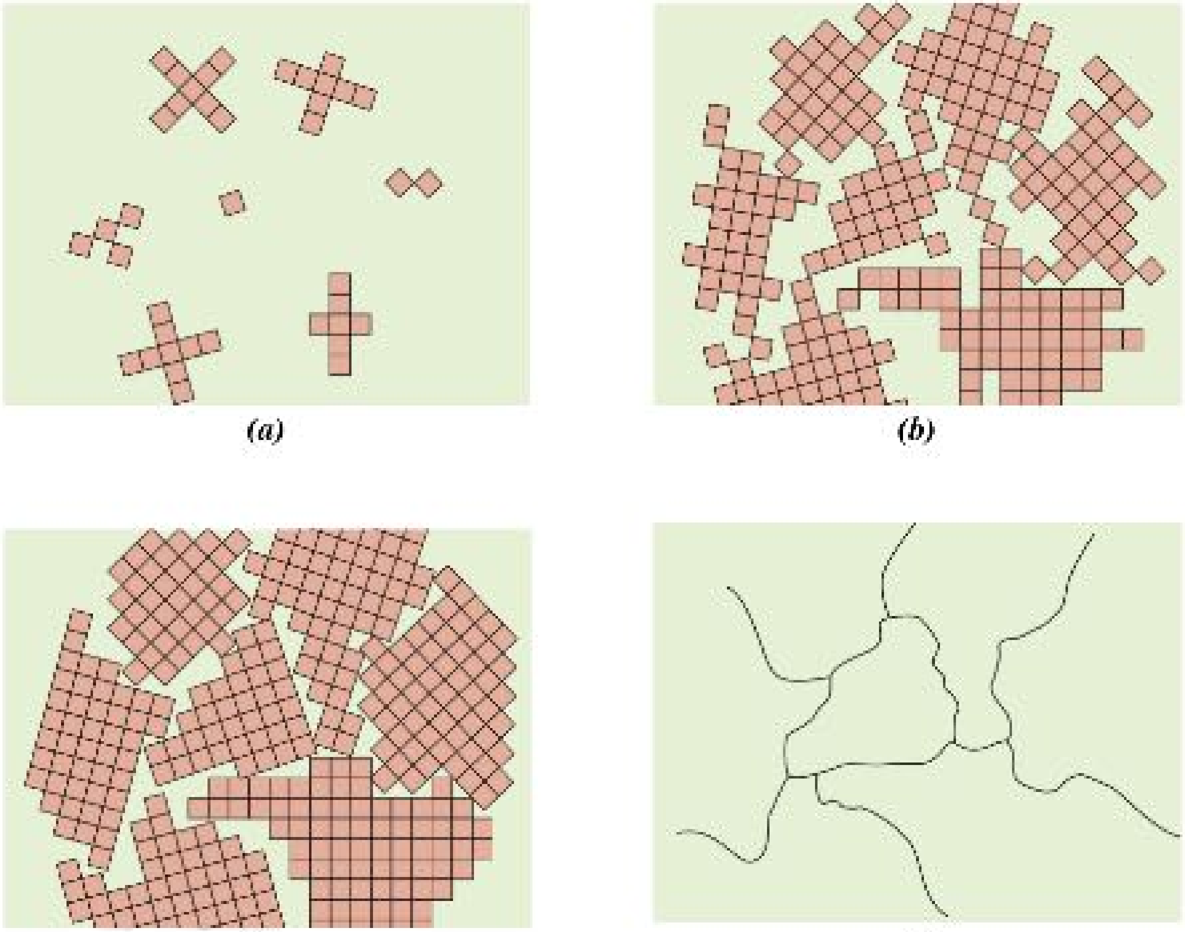
Domains

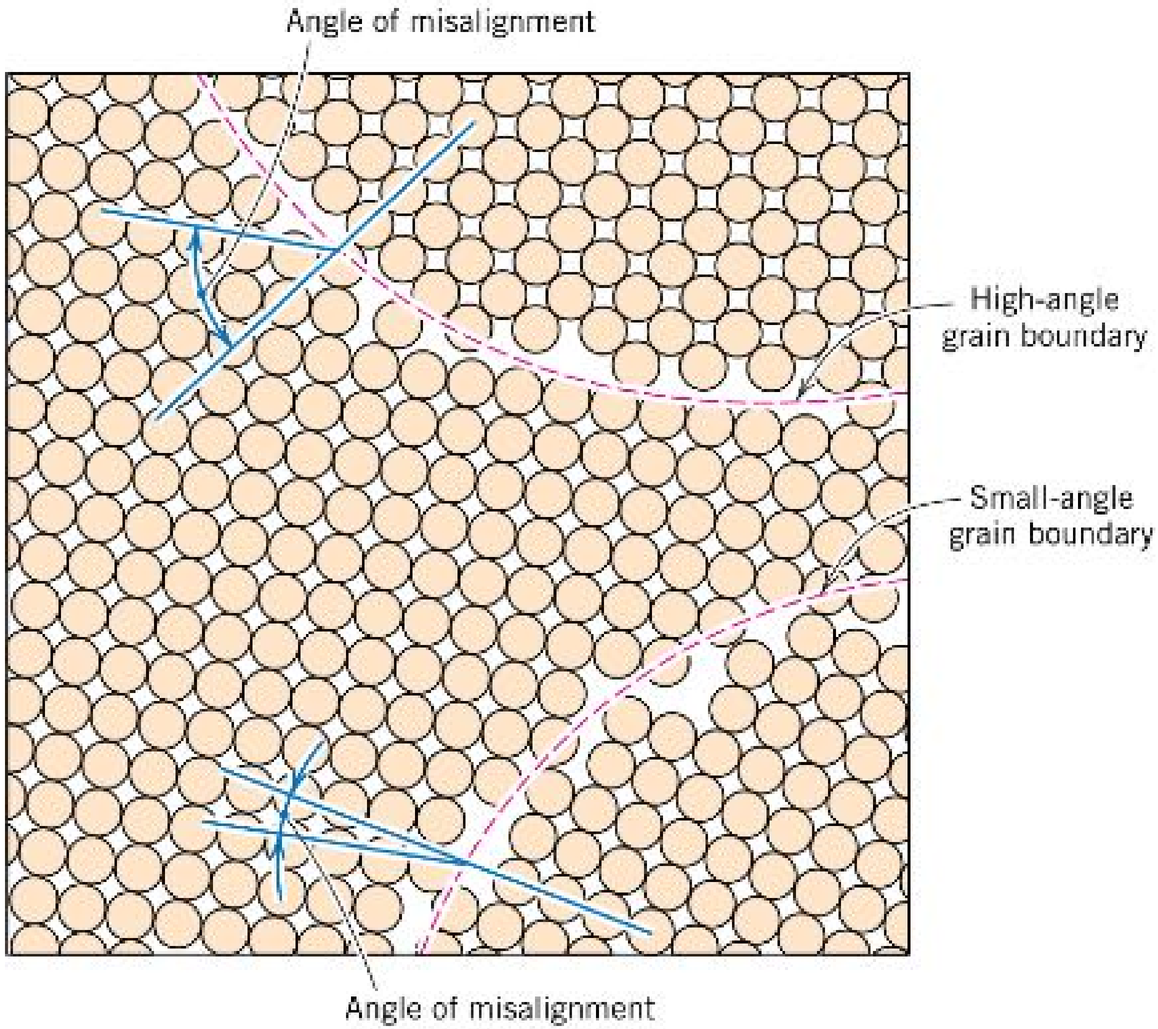
- Coherently diffracting volumes without 2D defects

Underlying source of size broadening

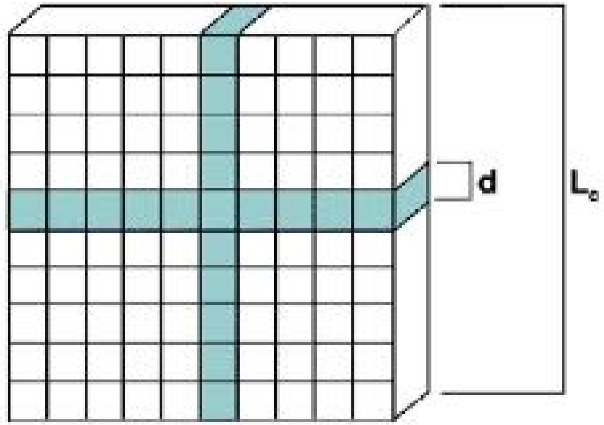
Topas 6 - Technical Reference (2017)

Size Brodening - Mechanism

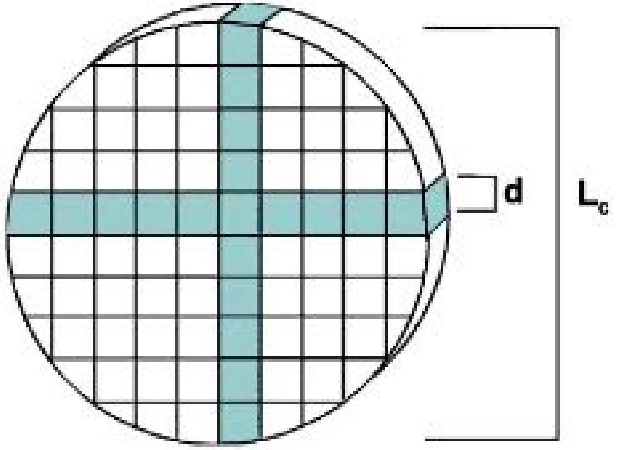

(a)
(b)
(c)
(d)



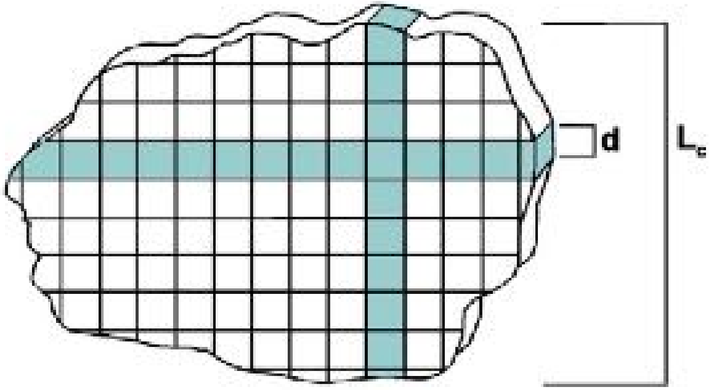
Size Broadening – Column Length



For a cube and only for *h00* reflections the column height L_{Vol} is identical with the edge-length L_0 of the cube (i.e. the crystallite size for *h00*)

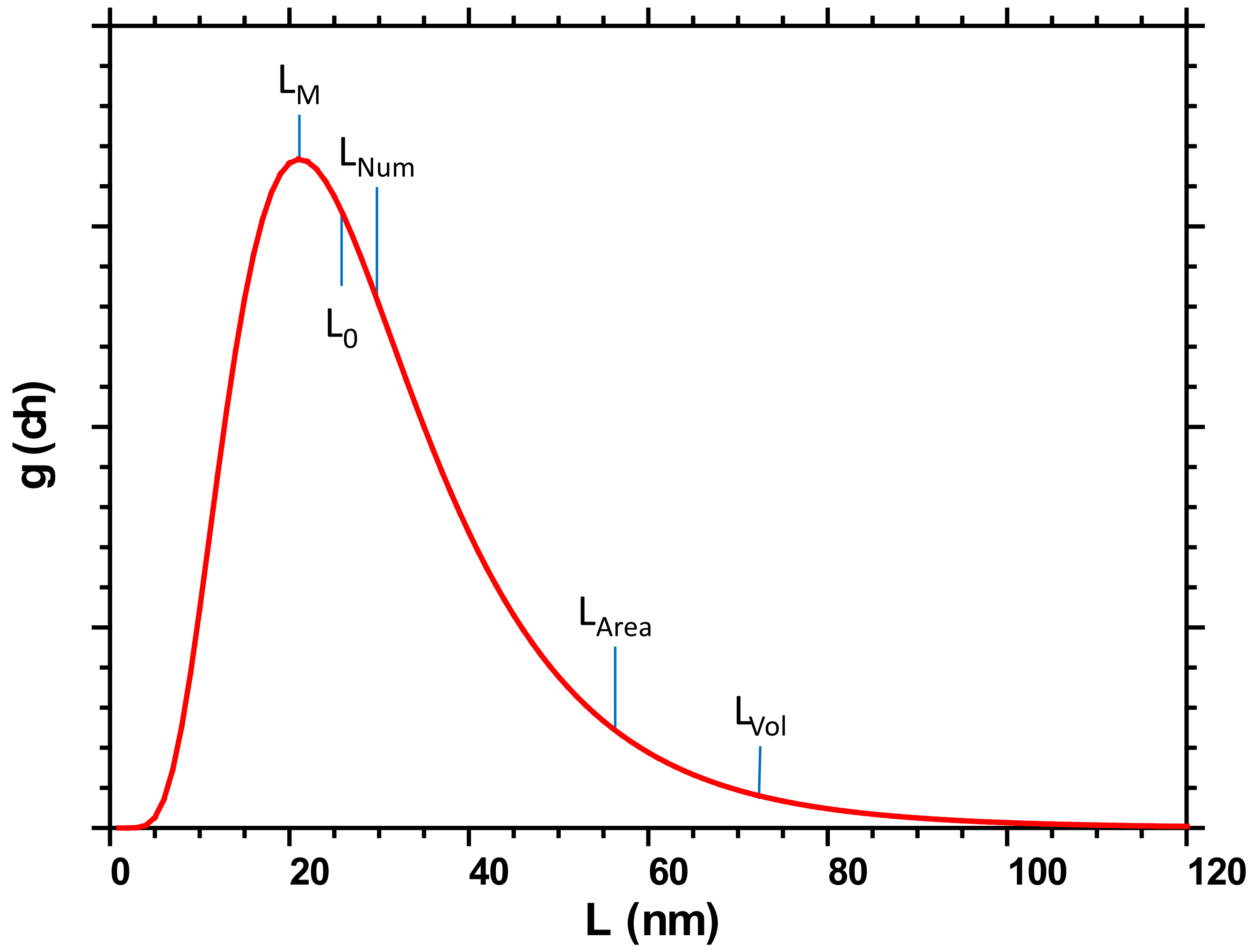


For a sphere a column height distribution is observed for any given *hkl*. L_{Vol} is not identical with the diameter of the sphere, but can be derived from the known crystallite shape: $L_0 = 4\ L_{Vol}/3$ (Wilson, A.J.C. X-ray Optics, 1949)



In most polycrystal systems the real crystallite shape is unknown, thus only L_{Vol} can be reported

Size Broadening – Column Length ???



- Schematic representation of column height distribution g(ch) showing the following characteristic points:
  - L_M : mode
  - L_0 : median
  - L_{Num} : number weighted mean
  - L_{Area} : area weighted mean
  - L_{Vol} : volume weighted mean

- The interpretation of size values is difficult, as different methods of analysis may report different quantities.
- Results obtained from model dependent and independent approaches are usually not comparable due to different parameter definitions (e.g. Klug & Alexander, 1974; Balzar, 1999)
  - Warren-Averbach analysis - area weighted column heights
  - Profile fitting methods - volume weighted column heights
- More complicated in any direct imaging techniques for crystallite size analysis (e.g. SEM, TEM, AFM) provide number weighted column heights!

Topas 6 - Technical Reference (2017)

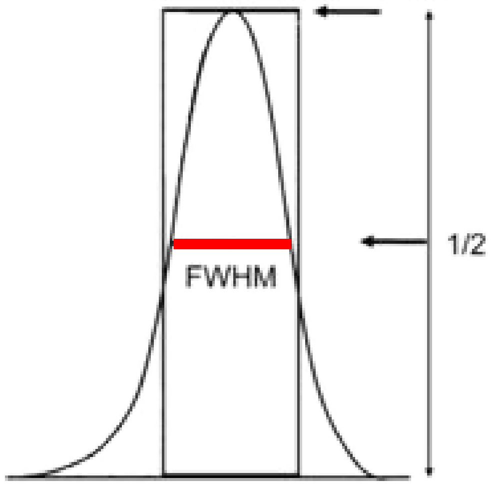
Size Broadening – Scherrer Equation

- Scherrer (1918): Line profile width is inversely proportional to crystallite size.

$$L_{vol} = \frac{K\lambda}{\beta_{FWHM_s} \cdot \cos\theta}$$

volume weighted column length

peak broadening



- K: Scherrer constant (typically 0.87 – 1)
  - Depends on the how the width is determined, the shape of the crystal, and the size distribution. Usually not known
  - Commonly (and indiscriminately!) used K values:
    - K = 0.89 for spherical crystallites
    - K = 0.94 for cubic crystallites
    - …
- Only true if other microstructural effects are negligible!

Langford, J. I. & Wilson, A. J. C. (1978). *J. Appl. Crystallogr.* **11**, 102–113.

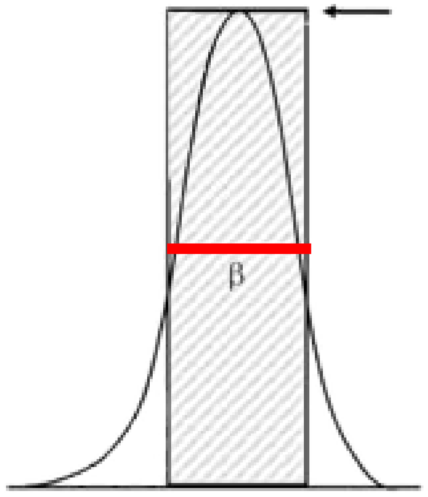
Size Broadening – Integral Bradth

- Laue (1926) realized that using Integral Breadths rather than FWHMs gives an evaluation that is approximately independent of the distribution in size and shape: K can be assumed to be 1
  - **Integral Breadth** (IB): The width of a rectangle with the same height and area as the line profile, obtained from dividing the area under line by the peas height

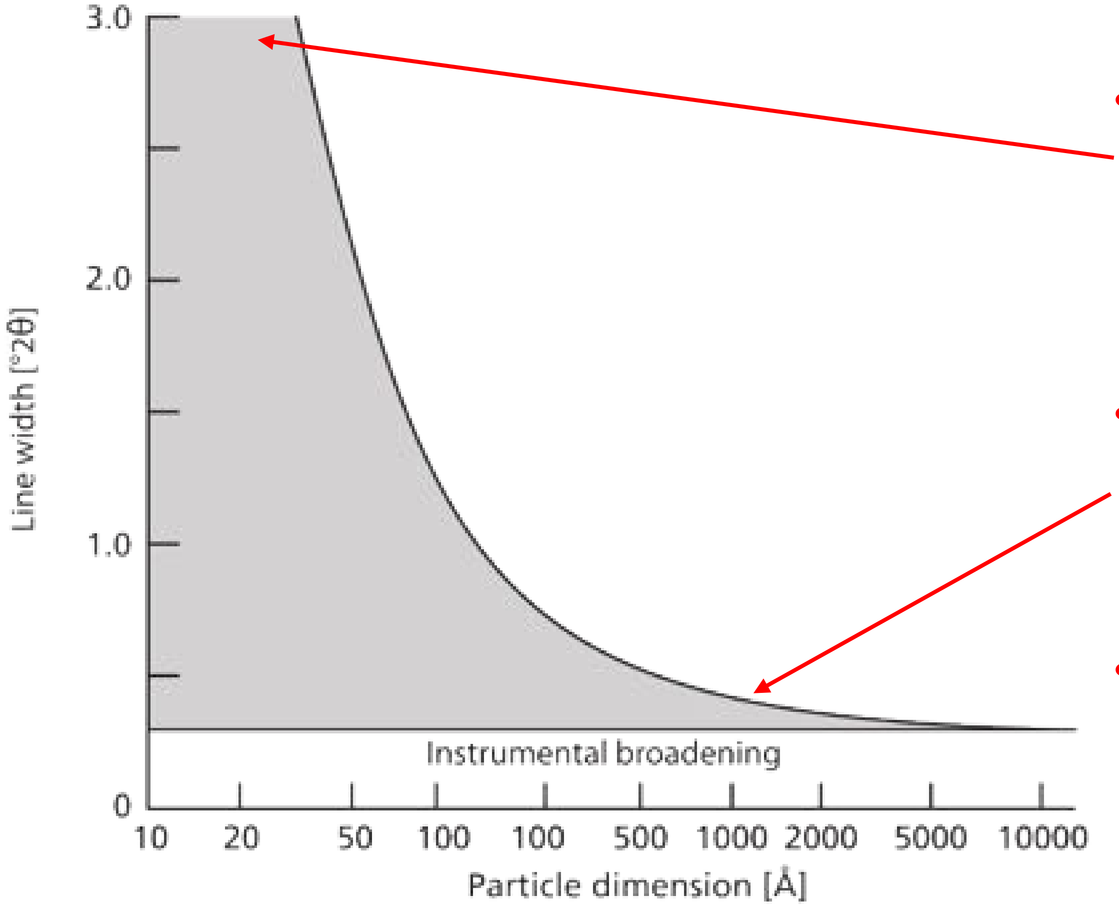
$$L_{vol} = \frac{K\lambda}{\beta_{FWHM_s} \cdot \cos\theta} \Rightarrow L_{vol} = \frac{\lambda}{\beta_{IB_s} \cdot \cos\theta}$$

Volume weighted column length

peak broadening



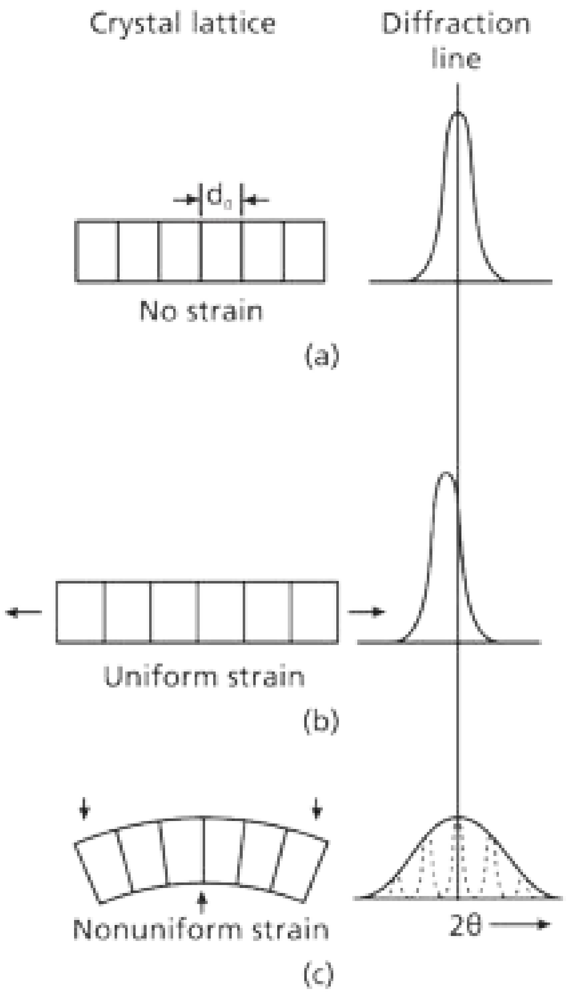
Size Broadening – Limitations



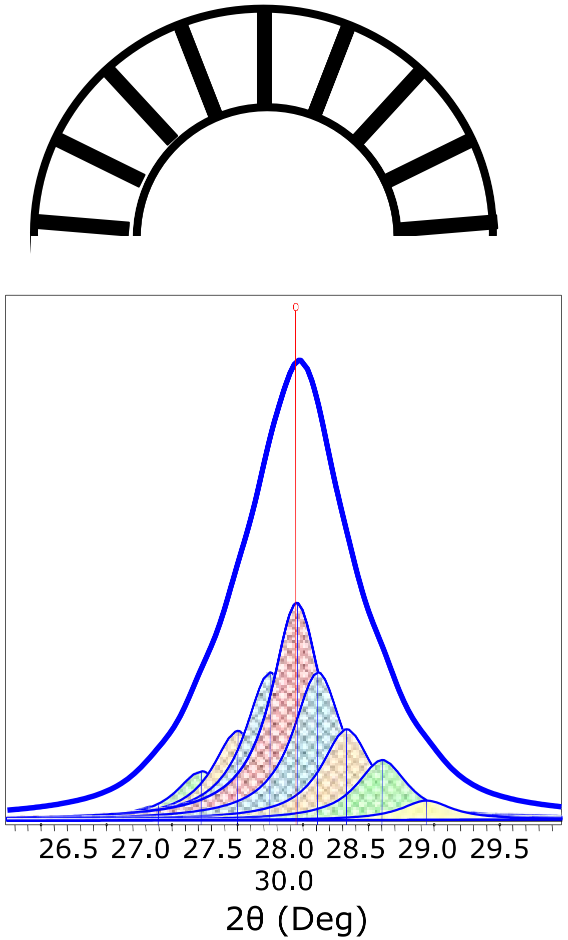
- For crystallites < 30 Å, size broadening so large, that peaks become indistinguishable from background ("XRD amorphous")
- For crystallites > 1000 Å, size broadening is usually smaller than instrumental resolution
- Different methods give different values. Always report details on how crystallite size was calculated.

Jenkins & Snyder, Introduction to X-ray Powder Diffractometry, 1996

Microstrain Broadening

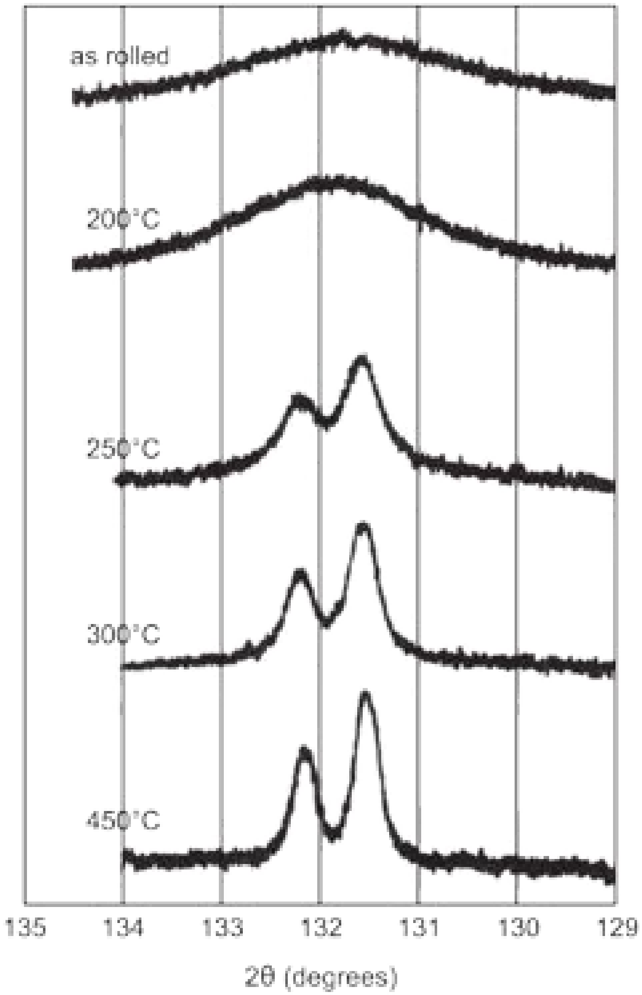


Jenkins & Snyder, 1996



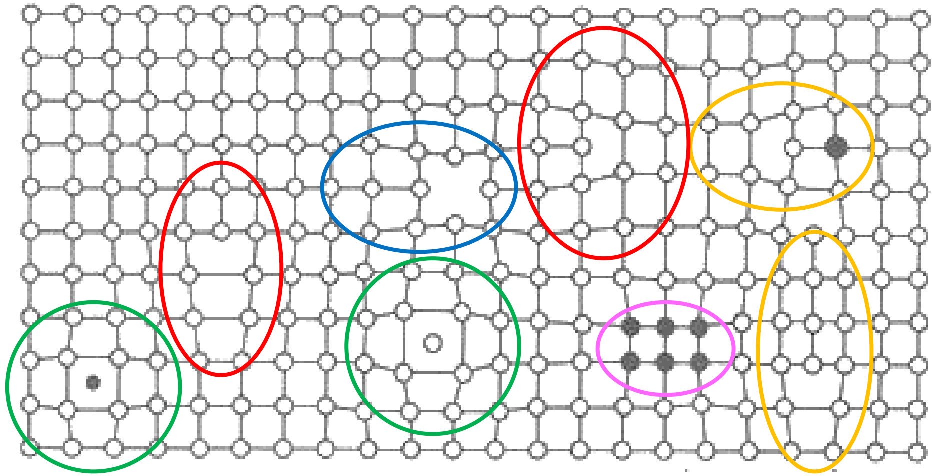
http://prism.mit.edu/xray

Annealed brass



Microstrain Broadening

- **Microstrain** represents displacements of atoms from their ideal positions, produced by any lattice imperfection (Dislocations, vacancies, interstitials, substitutionals, and similar defects)
- Microstrain can be conceived by considering two extreme values of the lattice spacing d, namely d + Δd and d - Δd, where $\varepsilon_0 = \Delta d/d$ represents the "mean" deviation



$$\varepsilon_0 = \frac{\Delta d}{d} = \frac{\beta_{FHM_{str}}}{4\tan\theta}$$

deviating lattice prm

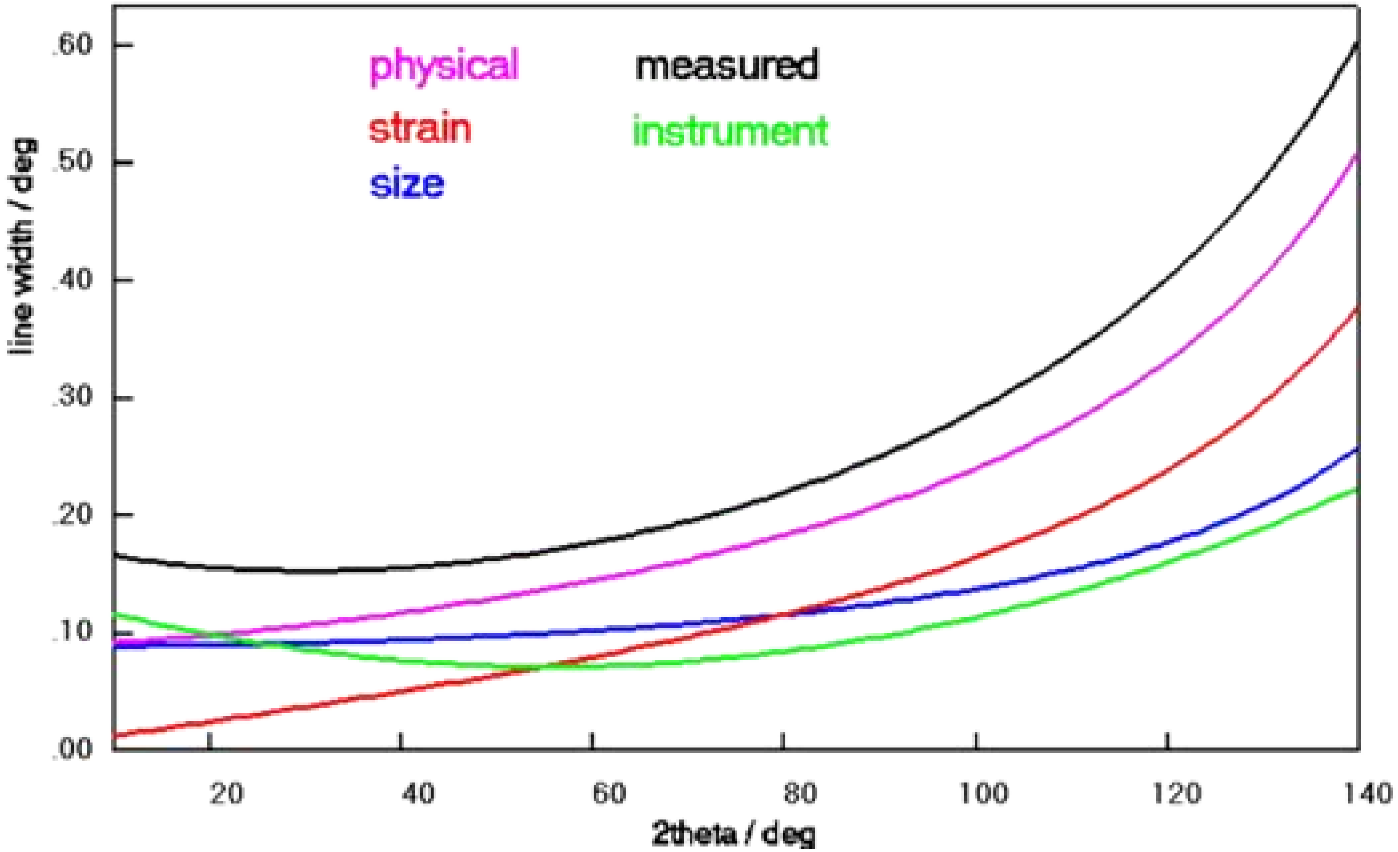
peak broadening

"ideal" lattice prm

- Microstrain leads to d-spacing variation, but d-spacing variation does not necessarily come from microstrain!
  - Mixture of different substitution levels of solid solutions also has lattice parameters variation, this will behave exactly like microstrain!

(Wang, X., Li, J., McDonald, R. G., Van Riessen, A. & Hart, R. D. (2015). *J. Appl. Crystallogr.* **48**, 814–826.)

Size Broadening as f(2θ)



- **Size**
  2θ-dependence is proportional to 1/cos(θ).

- **Microstrain**
  2θ-dependence is proportional to tan(θ).

- Size and strain contributions cannot be determined using a single peak or a few peaks within a small 2θ range.
- Accurate analysis requires measurement of a full pattern; nevertheless parameter correlation and thus errors may be large.

- What is Powder
- X-ray generation
- Data Acquisition
- Peak Profiles
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- Miscellaneous

Lower Limit of Detection

Peak vs. Background

To be able to distinguish a peak from background noise, two conditions have to be fulfilled:

1. the peak intensity has to be significantly higher than the background noise, i.e. typically 2σ
   - The human eye is normally more sensitive and accurate than (automated) software algorithms!
2. a peak has to have a minimum peak width with a typical FWHM ranging between about 0.03 - 0.3° 2θ

$$LOD = 3\sqrt{bkg.}\frac{wt.\%}{Net.\,Peak\,Int.}$$

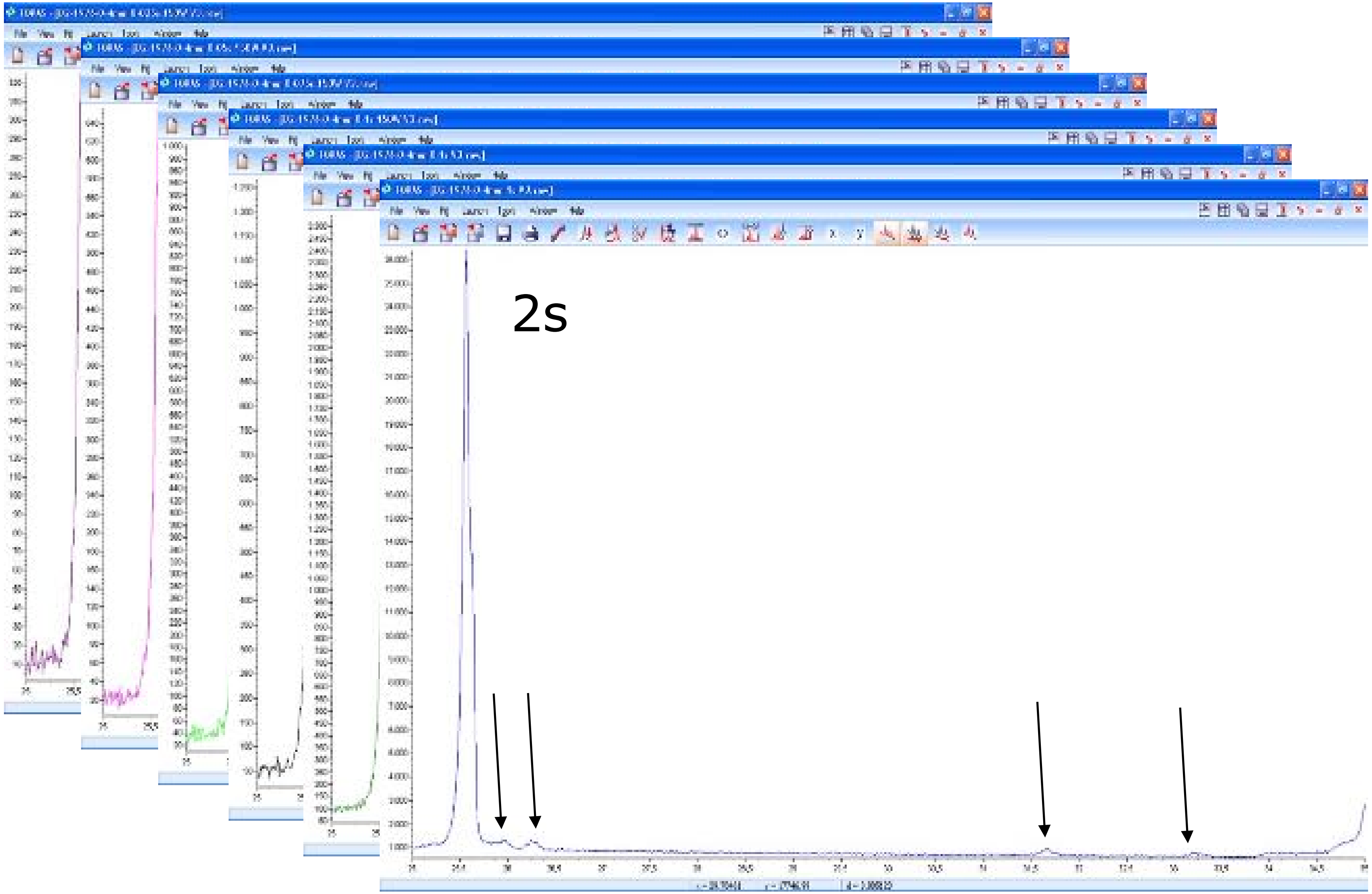
$$LOQ = 9\sqrt{bkg.}\frac{wt.\%}{Net.\,Peak\,Int.}$$

Lowering background is extremely important

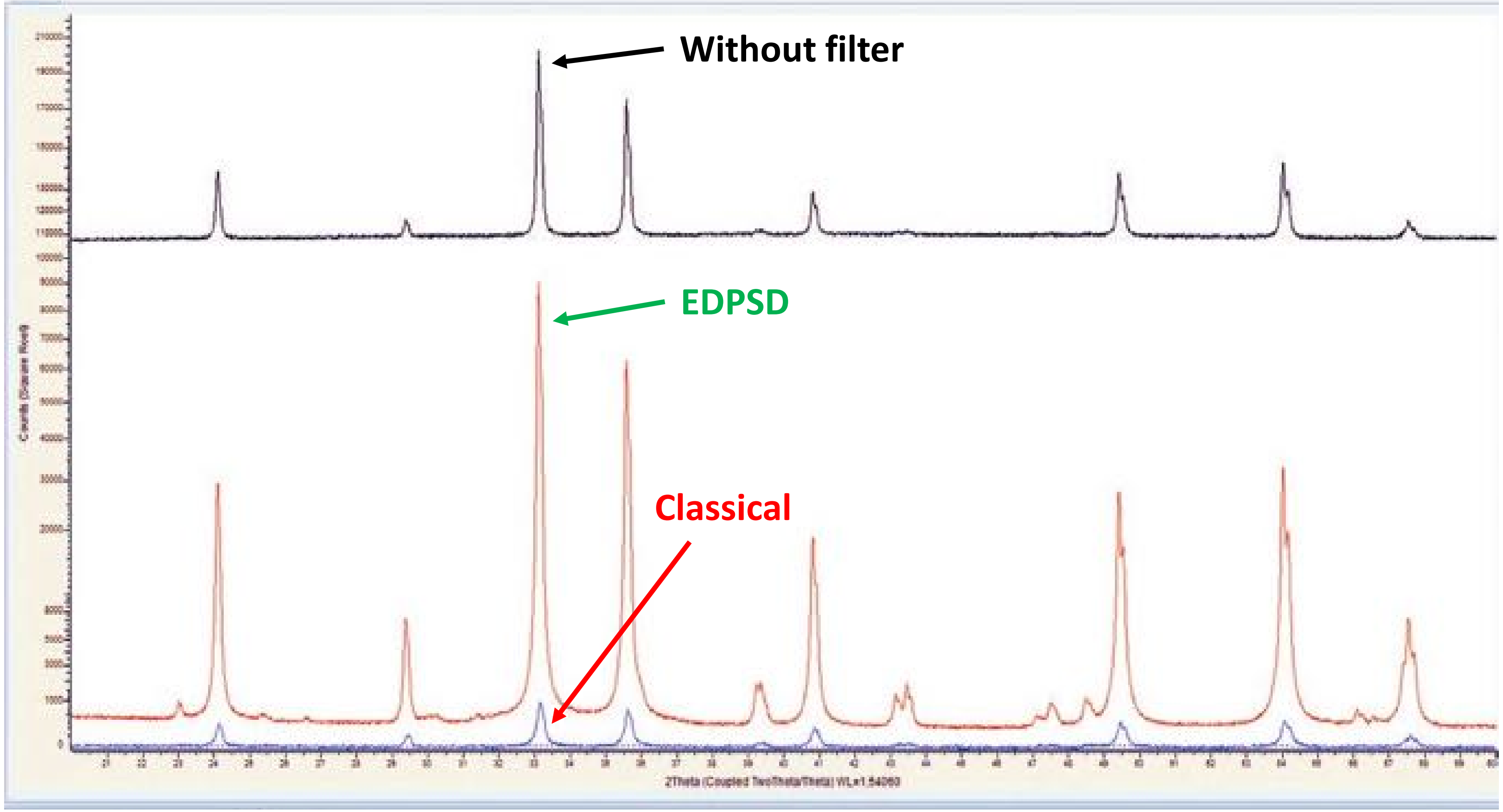
Halving bkg by **EDPSD** has same effect of doubling tube power, which doubles both "Net Peak Int." and "bkg"

EDPSD can easily reduce bkg to its 1/10 level, for samples with fluorescence!

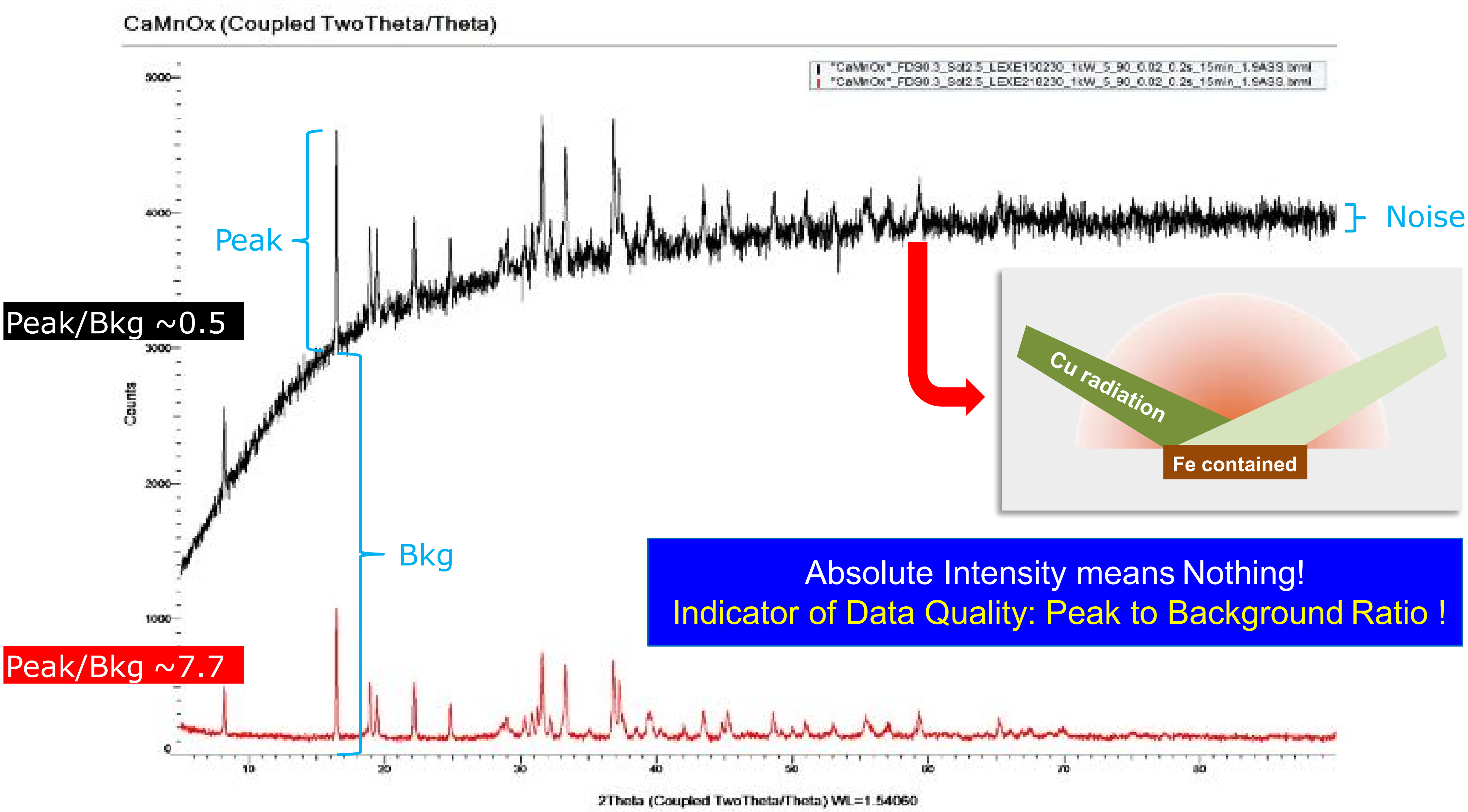
Measurement Time



Energy Discriminative PSD



Quality of PXRD



Absolute Intensity means Nothing!

Indicator of Data Quality: Peak to Background Ratio !

- What is Powder
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gaseous

liquid

solid

Elements & Phases

- **Phase**:

  A physically distinctive form of matter, such as a solid, liquid, gas or plasma. A phase of matter is characterized by having relatively uniform

chemical and physical properties.

H_2O

gaseous

liquid

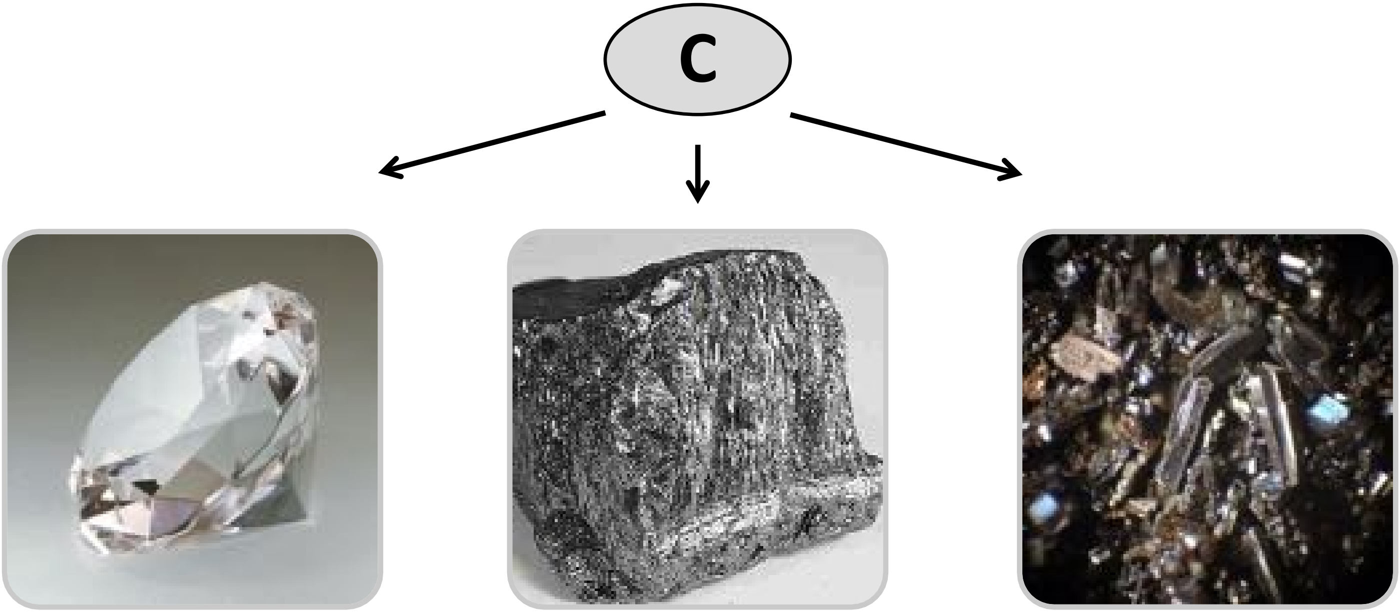
solid

Elements & Phases

- **Phase**:

  A physically distinctive form of matter, such as a solid, liquid, gas or plasma. A phase of matter is characterized by having relatively uniform

chemical and physical properties.



solid solid solid

Elements & Phases

- **Phase**:

  A physically distinctive form of matter, such as a solid, liquid, gas or plasma. A phase of matter is characterized by having relatively uniform

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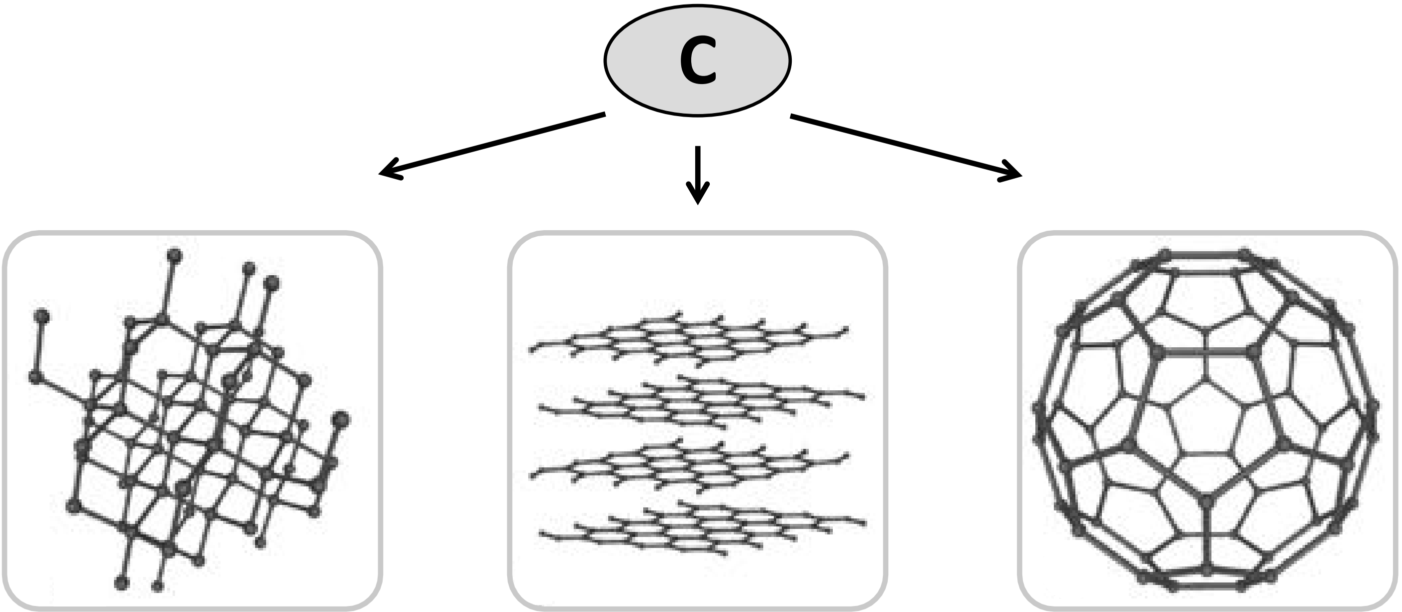
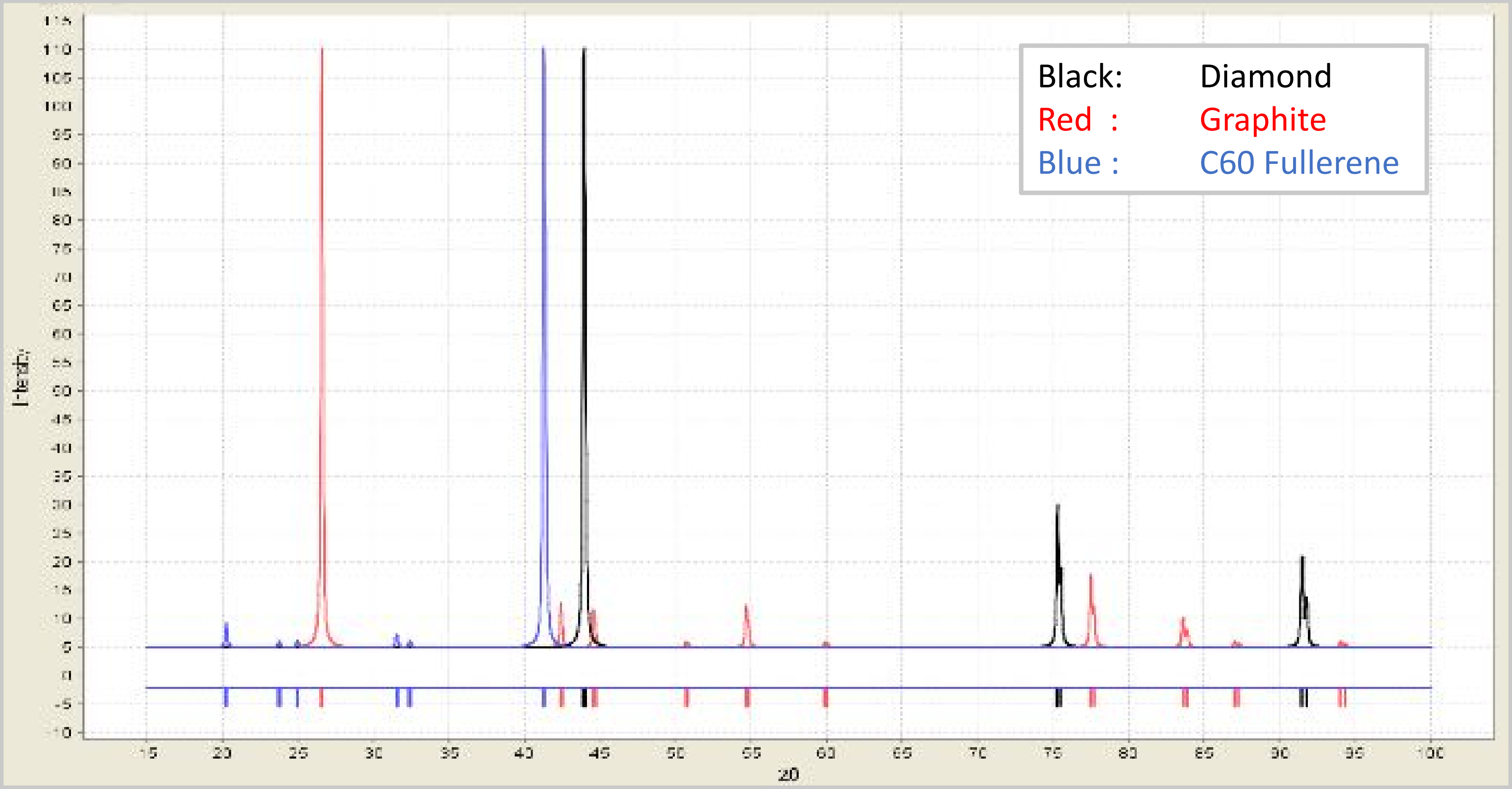


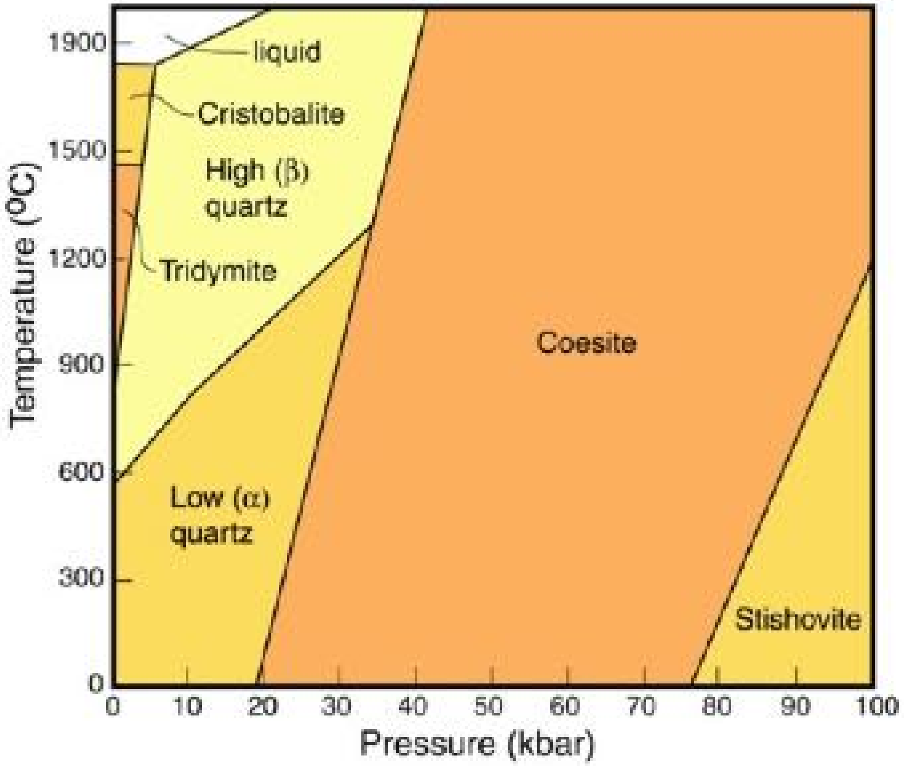
solid solid solid

Diffent Phases – Different XRD Pattern

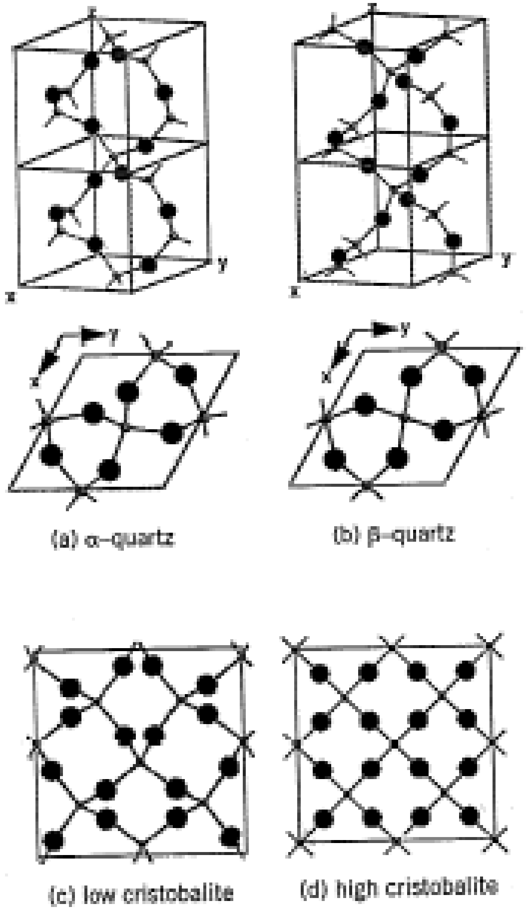


Ideal calculated diffraction patterns from the 3 phases mentioned above

Phase Identification - SiO2

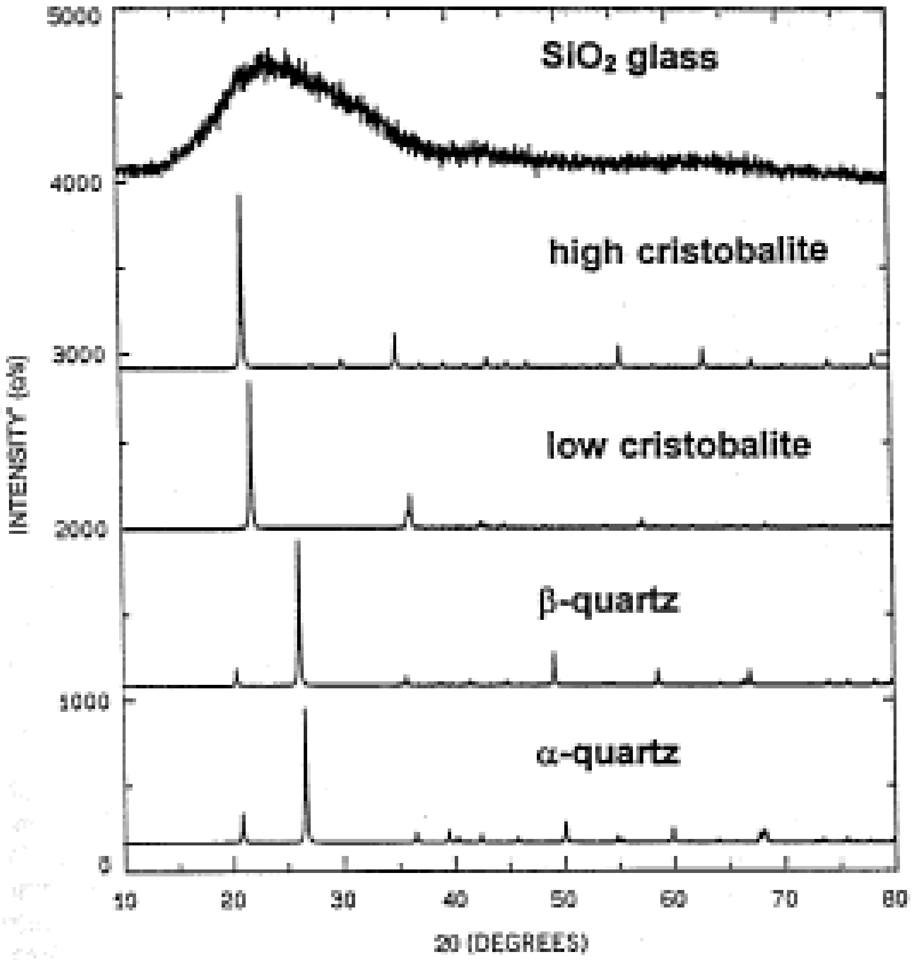


Phase diagram of SiO_2



Crystal Structures

(Jenkins & Snyder 1996)



Powder Diffraction

(Jenkins & Snyder 1996)

PXRD fingerprints

- A particular phase (particular atoms arranged in a particular crystal structure) gives a particular set of diffractions peaks

Crystal structure

Peak intensities

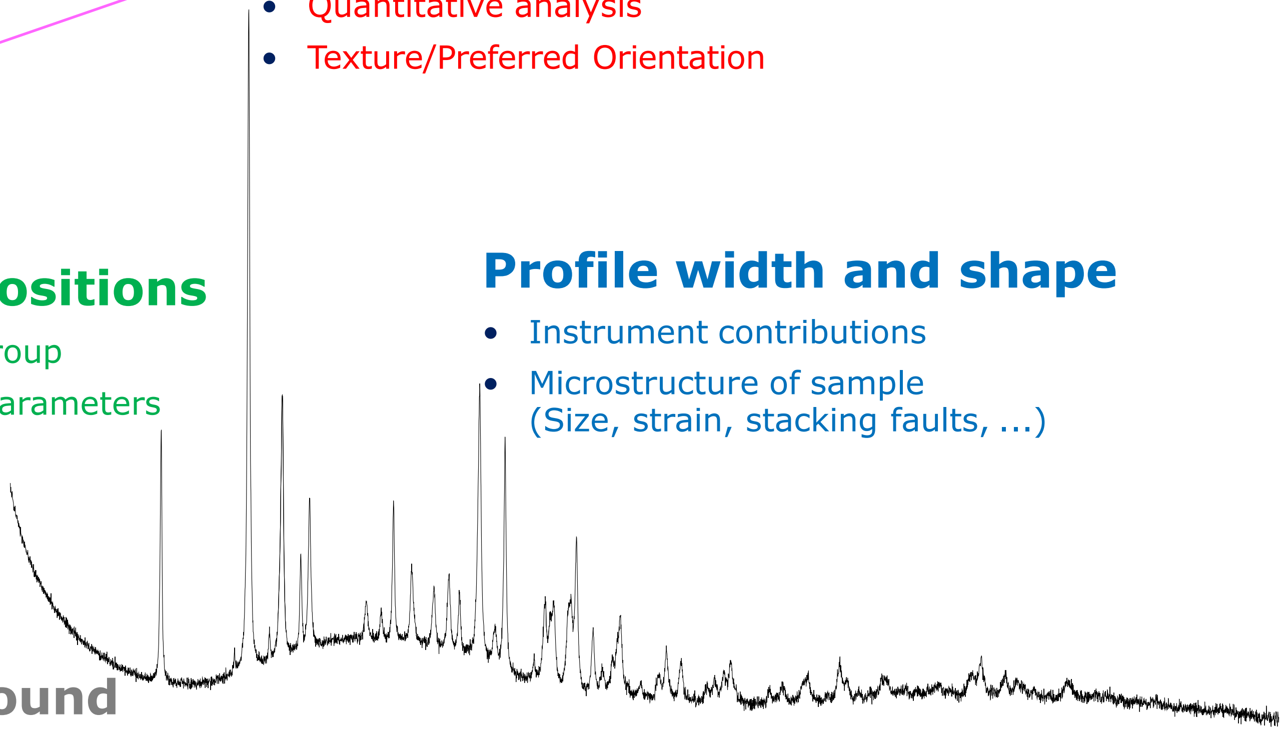
- Atoms on each site
- Quantitative analysis
- Texture/Preferred Orientation

Peak positions

- Space group
- Lattice parameters

Profile width and shape

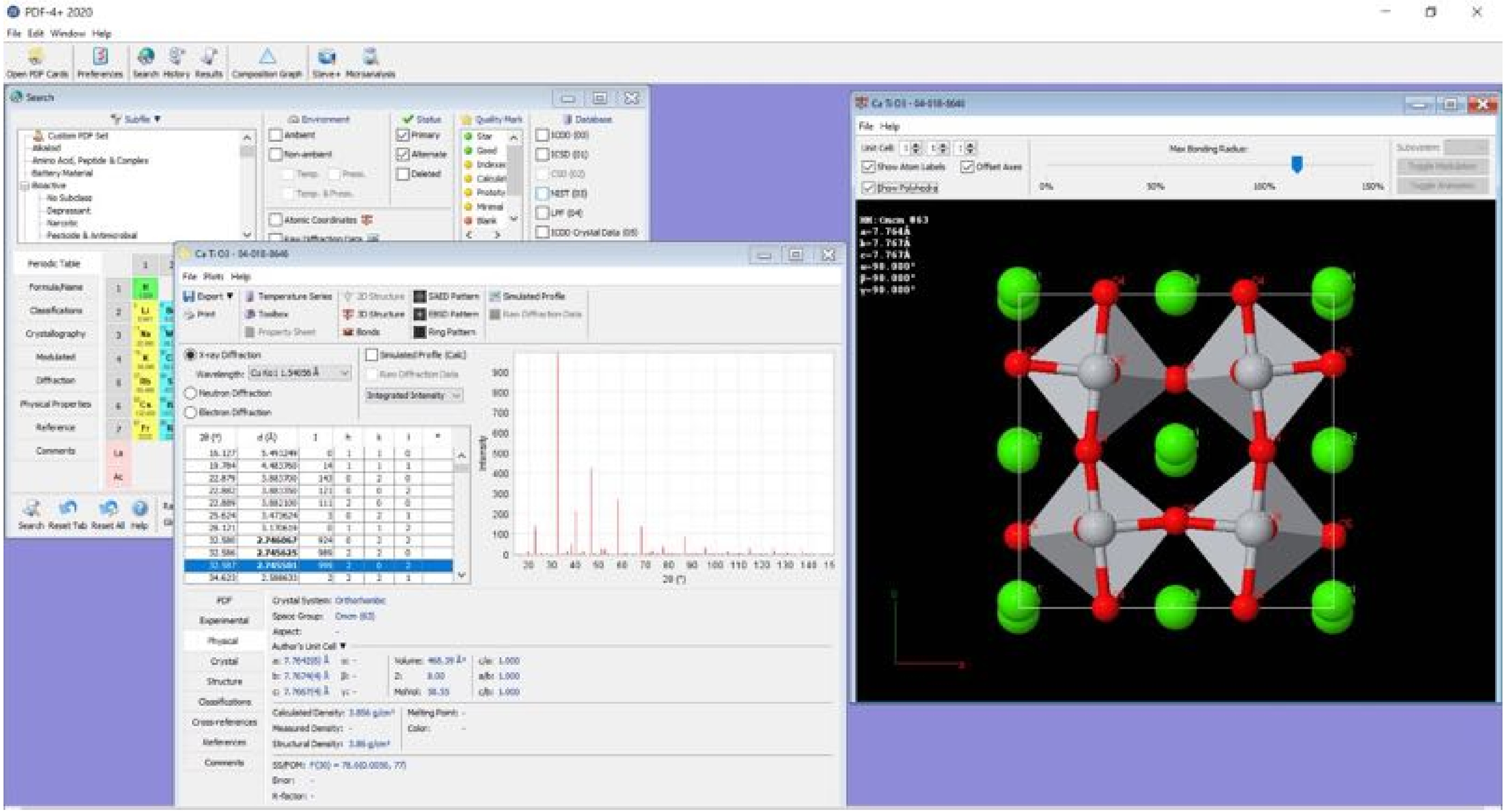
- Instrument contributions
- Microstructure of sample (Size, strain, stacking faults, ...)



Background

- Scattering from sample environment (air, sample holder, ...)
- Local order / disorder
- Amorphous phase amounts, "degree of crystallinity"

ICDD PDF-4+ 2021 Database (444.133)



Qualitative Analysis Round Robin

SMRR-2002 results

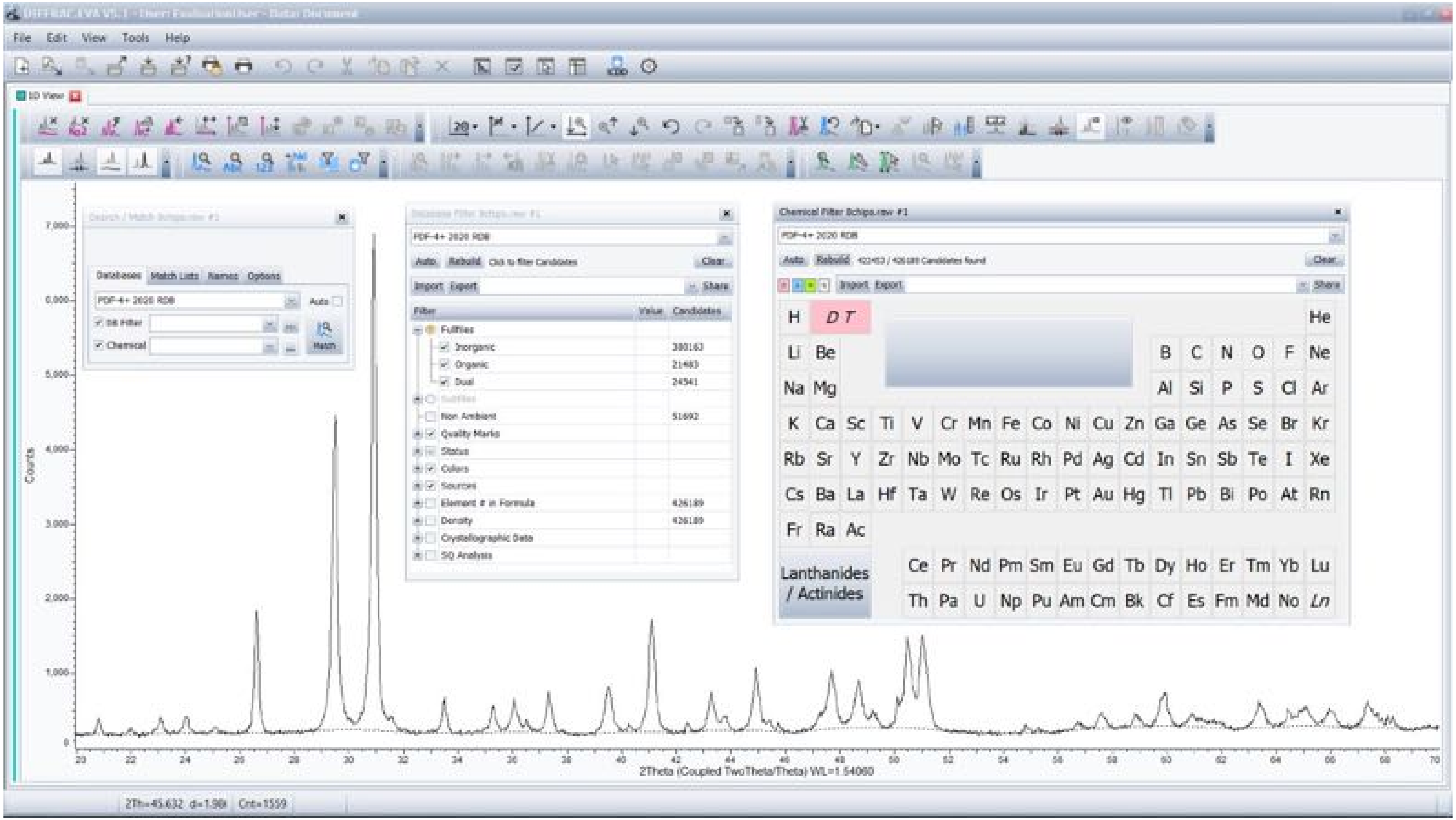
Best seen in 1024x768 resolution or higher
Last update - July 2002
Jean-Marc Le Meins - Lachlan M. D. Cranswick - Armel Le Bail

We have one more software in this Step 2 (Traces) and 5 new participants. The **mean number** of phases identified for each sample is given below (nr = no result provided ; id = incomplete database precluding sample 2 identification), and also the numbers given by the **best performer** (mixing the software versions) and the **worst one**. This table can be compared to the equivalent one above, made at Step 1. An improved score if compare to Step 1 is noted in **blue** (occuring for samples 1 to 4), and a worst score is noted in **dark blue** (occuring for samples 3 and 4).

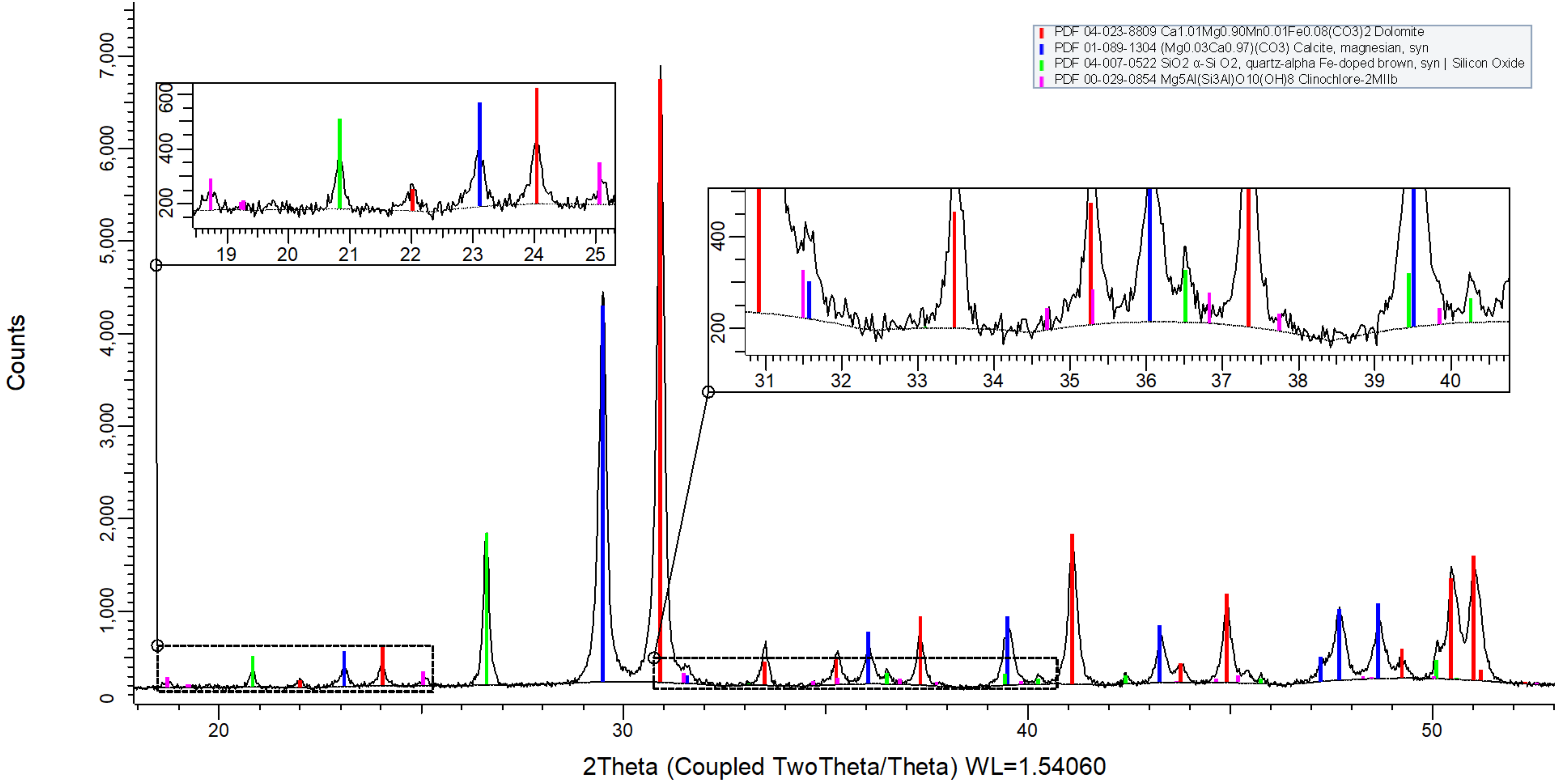
Software	Participants	Sample 1	Sample 2	Sample 3	Sample 4
EVA - Socabim/Bruker	P5, P9, P11, P12, P13, P16, P17, P22, P26, P28	3.6/4 (+0.27)- 4/4 - 3/4	0.50/1 - 1/1 - 0/1	0.7/1 (-0.05)- 1/1 - 0/1	3.3/4 (+0.38)- 4/4 - 3/4
JADE - MDI	P3, P8, P23	4/4 - 4/4 - 4/4	1/1 - 1/1 - 1/1	0.66/1(-0.09) - 1/1 - 0/1	3/4 (-0.25)- 3/4 - 3/4
Highscore - Philips	P4, P30	3.5/4 (+0.5)- 4/4 - 3/4	0.5/1 (+0.5) - 1/1 - 0/1	0.5/1 (+0.5) - 1/1 - 0/1	3/4 (-1) - 4/4 - 2/4
Graphics & Identify - Philips	P7, P24, P29	2.67/4 - 4/4 - 1/4	0.66/1(+0.66)- 1/1 - 0/1	0.33/1 (-0.33)- 1/1 - 0/1	2.67/4(+0.33)- 3/4 - 2/4
FARHAN	P10	3/4	nr	nr	nr
Bede Search/Match	P14	3/4	1/1 (Hanawalt)	0/1	3/4
Yakimov	P19	4/4	1/1 (+1 :set 49)	1/1	4/4
CSM - Oxford Cryosystems	P20	4/4	1/1	1/1 (+1)	3/4
PHAN	P21	4/4 (+1)	id	1/1	3/4
TRACES	P27	4/4	0/1	1/1	3/4

Le Meins et al., 2002 international Search-Match Round Robin. www.cristal.org/smrr

Software – Bruker Eva v.5



Result on RR sample



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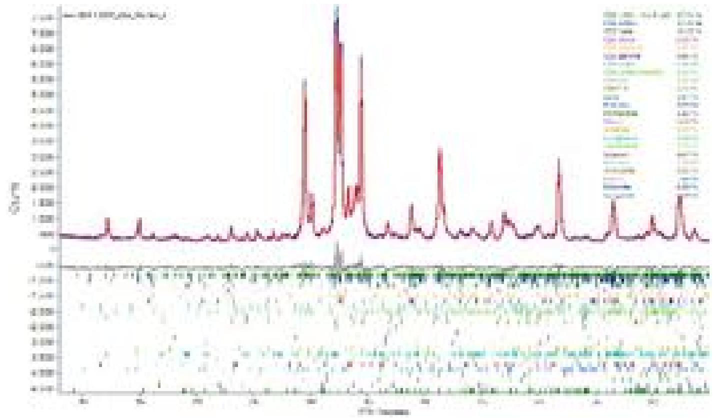
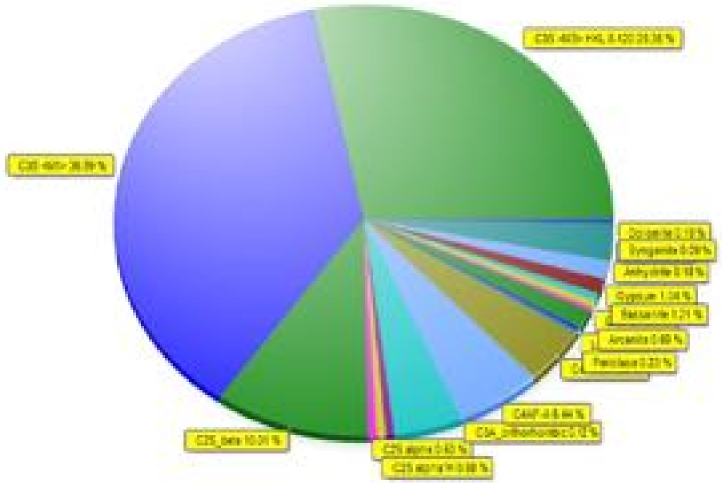
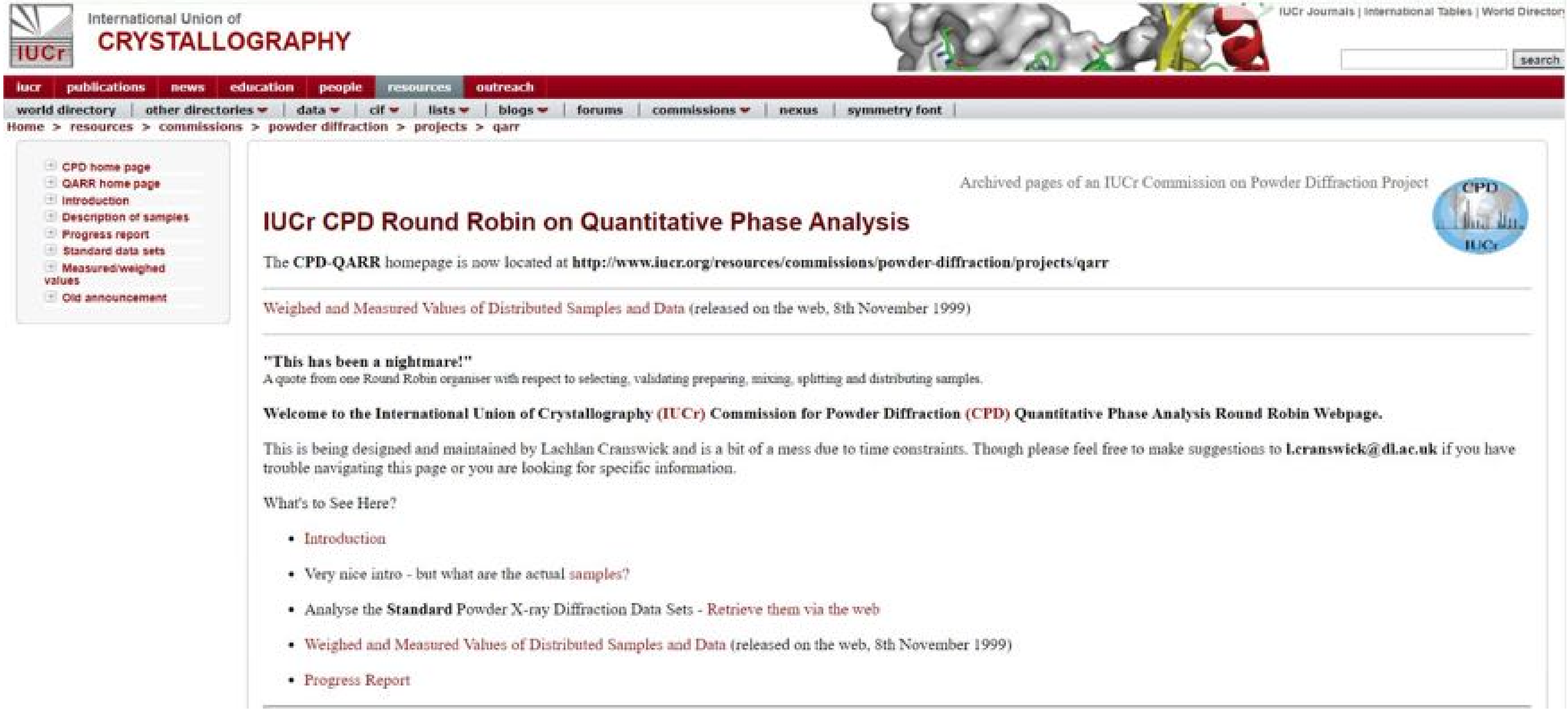




Quantitative Analysis Round Robin



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Home > resources > commissions > powder diffraction > projects > qarr

- CPD home page
- QARR home page
- Introduction
- Description of samples
- Progress report
- Standard data sets
- Measured/weighed values
- Old announcement

Archived pages of an IUCr Commission on Powder Diffraction Project

IUCr CPD Round Robin on Quantitative Phase Analysis

The **CPD-QARR** homepage is now located at **http://www.iucr.org/resources/commissions/powder-diffraction/projects/qarr**

Weighed and Measured Values of Distributed Samples and Data (released on the web, 8th November 1999)

"This has been a nightmare!"
A quote from one Round Robin organiser with respect to selecting, validating preparing, mixing, splitting and distributing samples.

Welcome to the International Union of Crystallography (IUCr) Commission for Powder Diffraction (CPD) Quantitative Phase Analysis Round Robin Webpage.

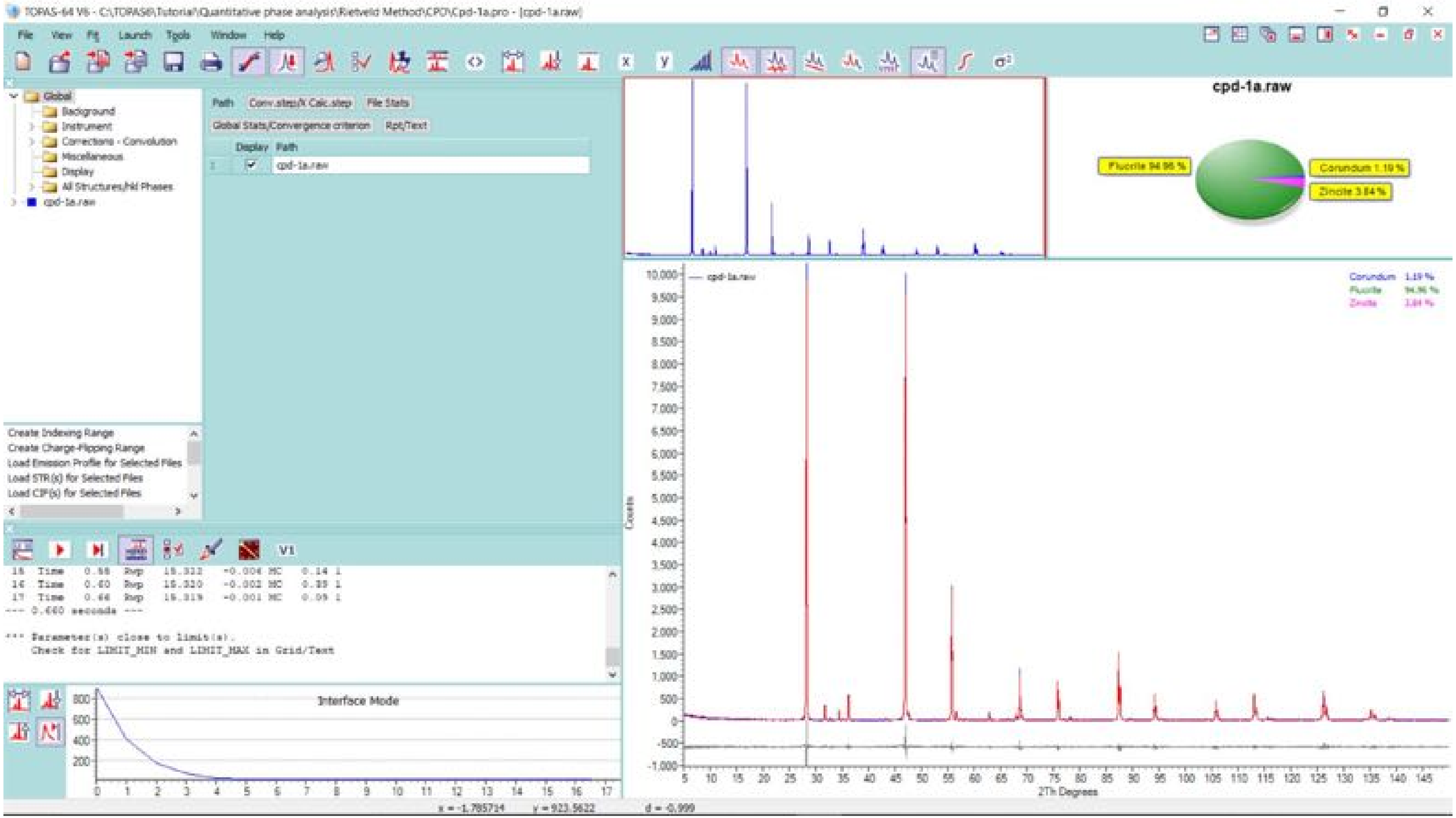
This is being designed and maintained by Lachlan Cranswick and is a bit of a mess due to time constraints. Though please feel free to make suggestions to **l.cranswick@dl.ac.uk** if you have trouble navigating this page or you are looking for specific information.

What's to See Here?

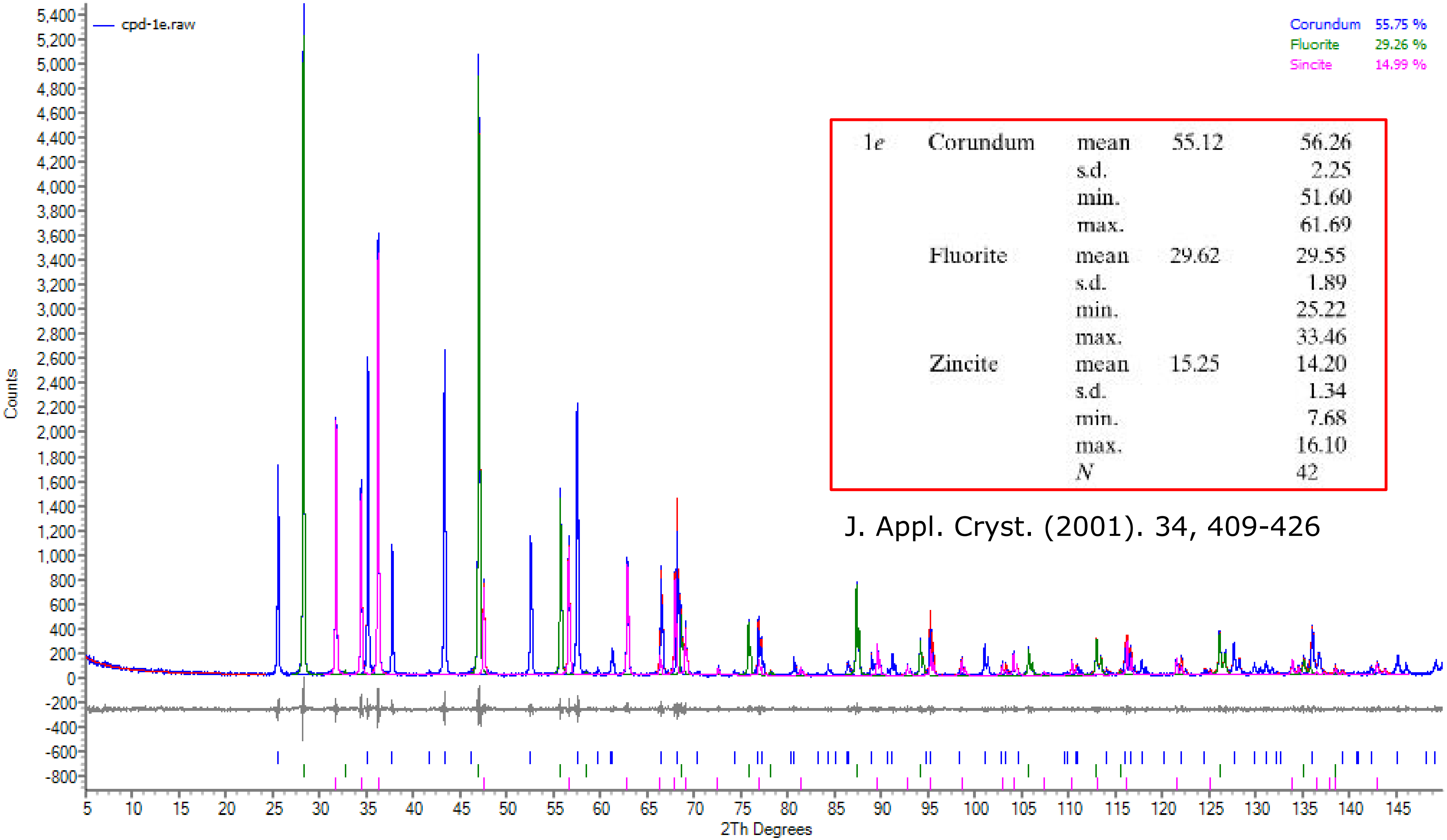
- Introduction
- Very nice intro - but what are the actual samples?
- Analyse the **Standard** Powder X-ray Diffraction Data Sets - Retrieve them via the web
- Weighed and Measured Values of Distributed Samples and Data (released on the web, 8th November 1999)
- Progress Report

- https://www.iucr.org/resources/commissions/powder-diffraction/projects/qarr
- https://www.youtube.com/watch?v=Yyjm_KM0VYI&t=779s

Software – Bruker Topas v.6



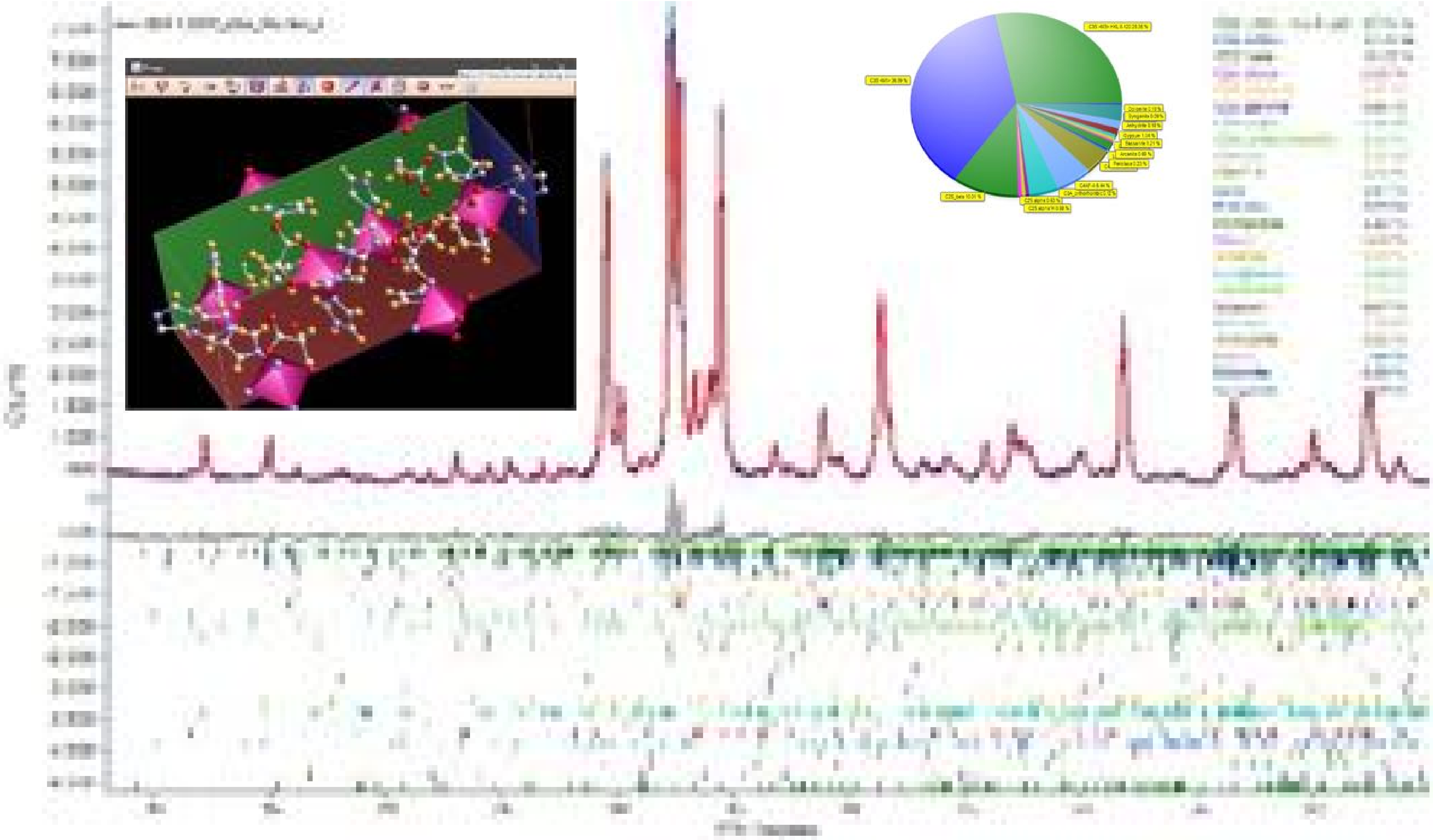
Result on RR sample



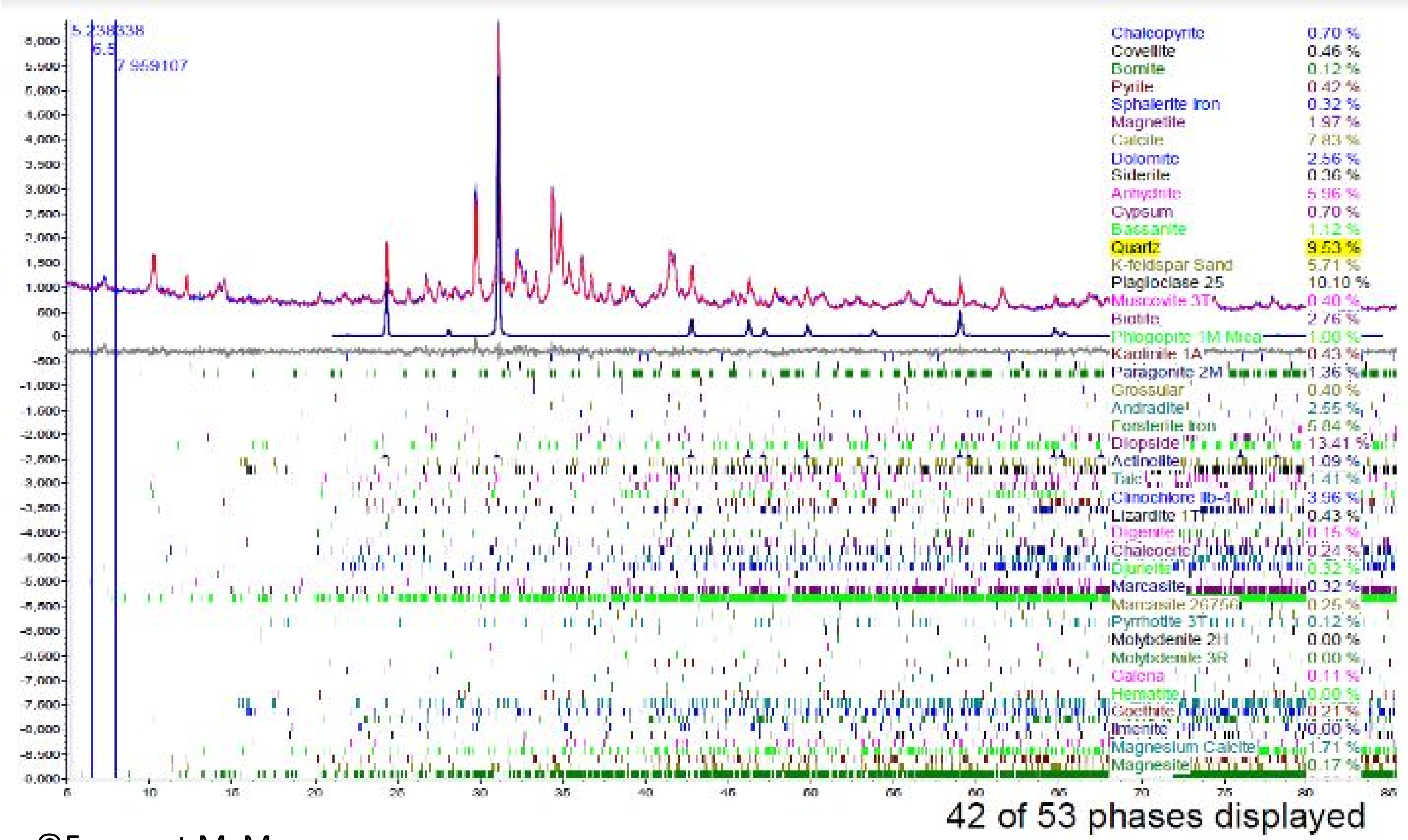
1*e*	Corundum	mean	55.12	56.26
		s.d.		2.25
		min.		51.60
		max.		61.69
	Fluorite	mean	29.62	29.55
		s.d.		1.89
		min.		25.22
		max.		33.46
	Zincite	mean	15.25	14.20
		s.d.		1.34
		min.		7.68
		max.		16.10
		N		42

J. Appl. Cryst. (2001). 34, 409-426

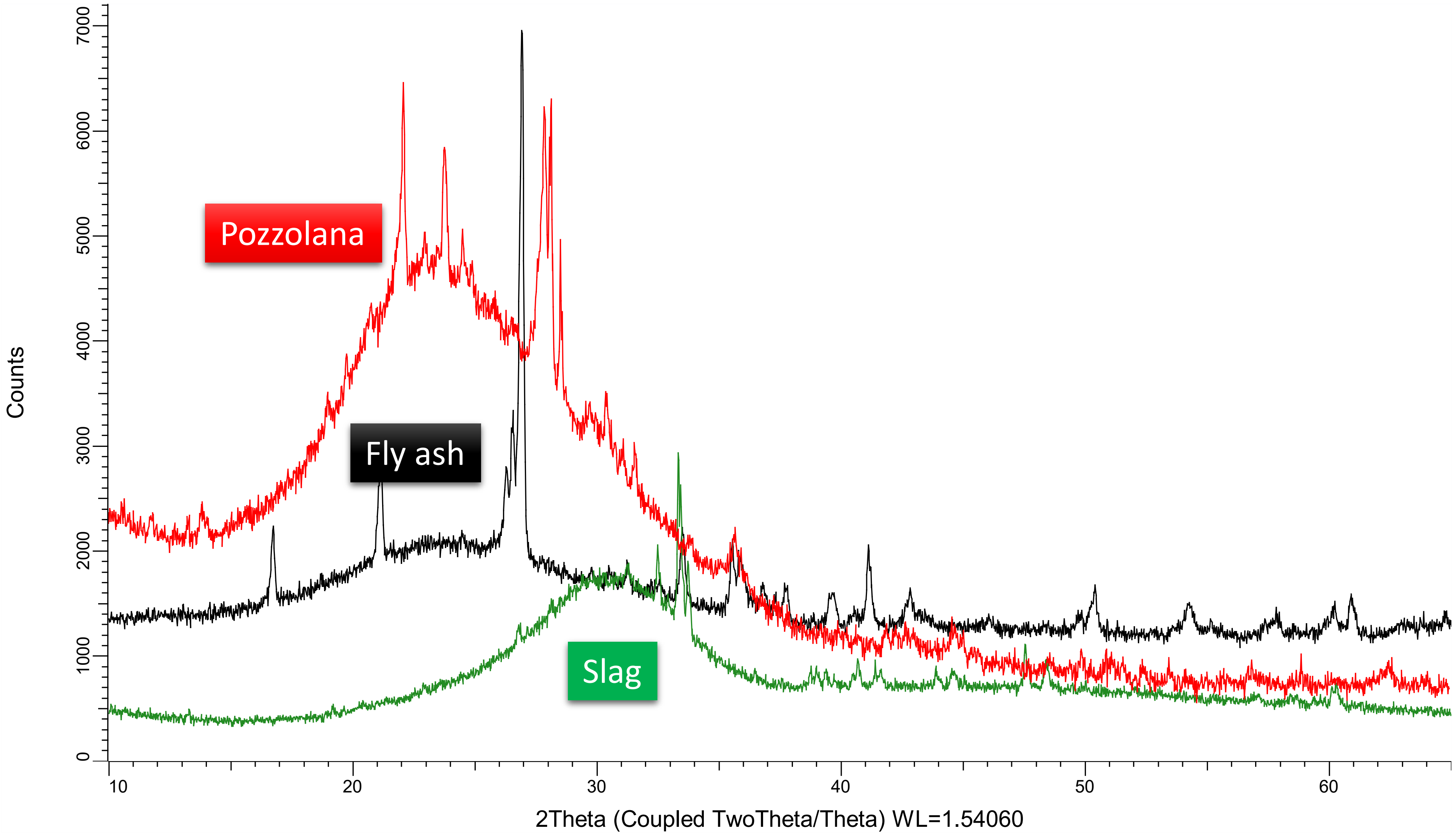
Example on Cement Industries



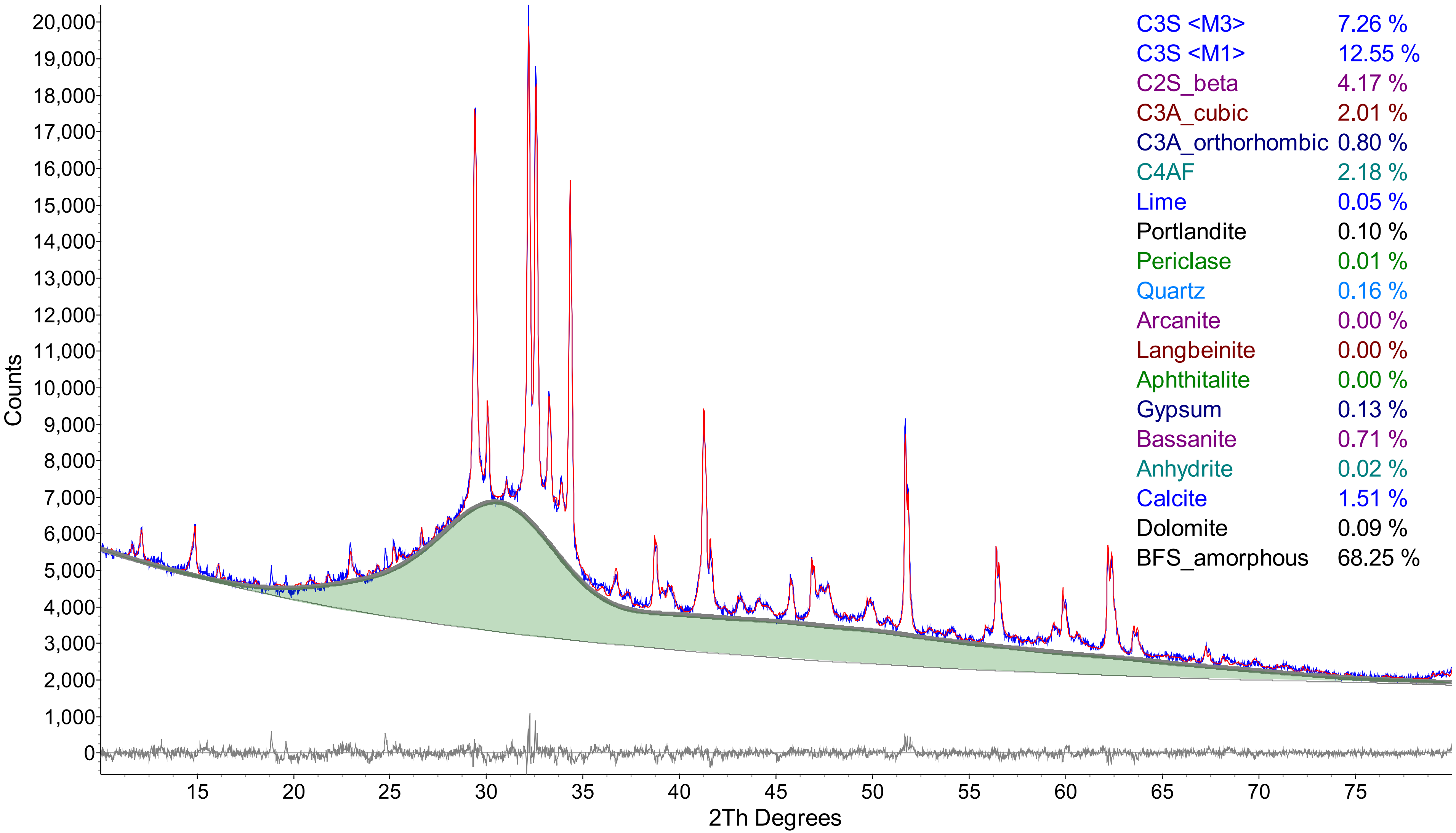
Example on Mining Industries



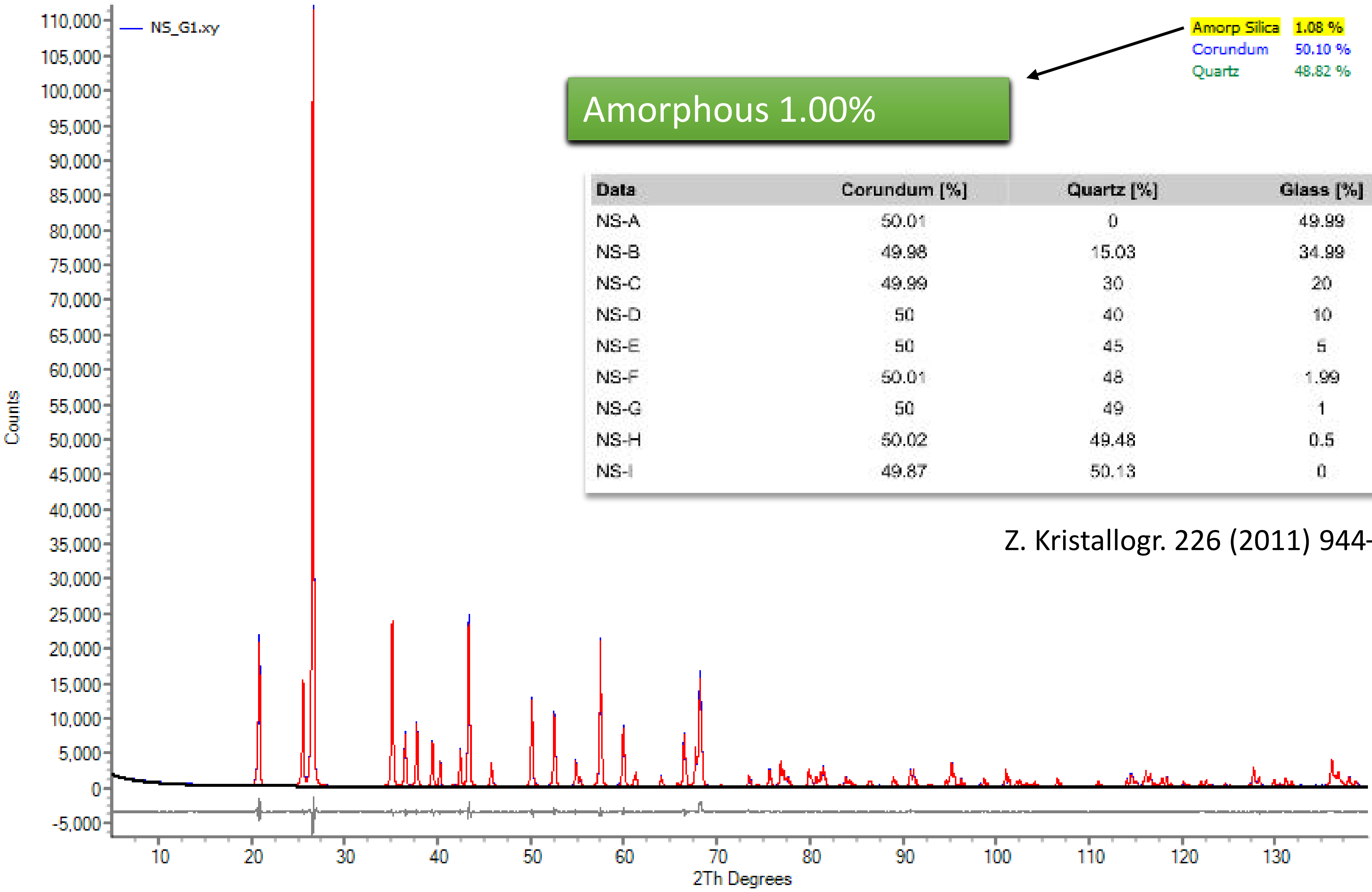
Amorphous Additives in Cement



Amorphous Additives in Cement



Example on Amorphous RR

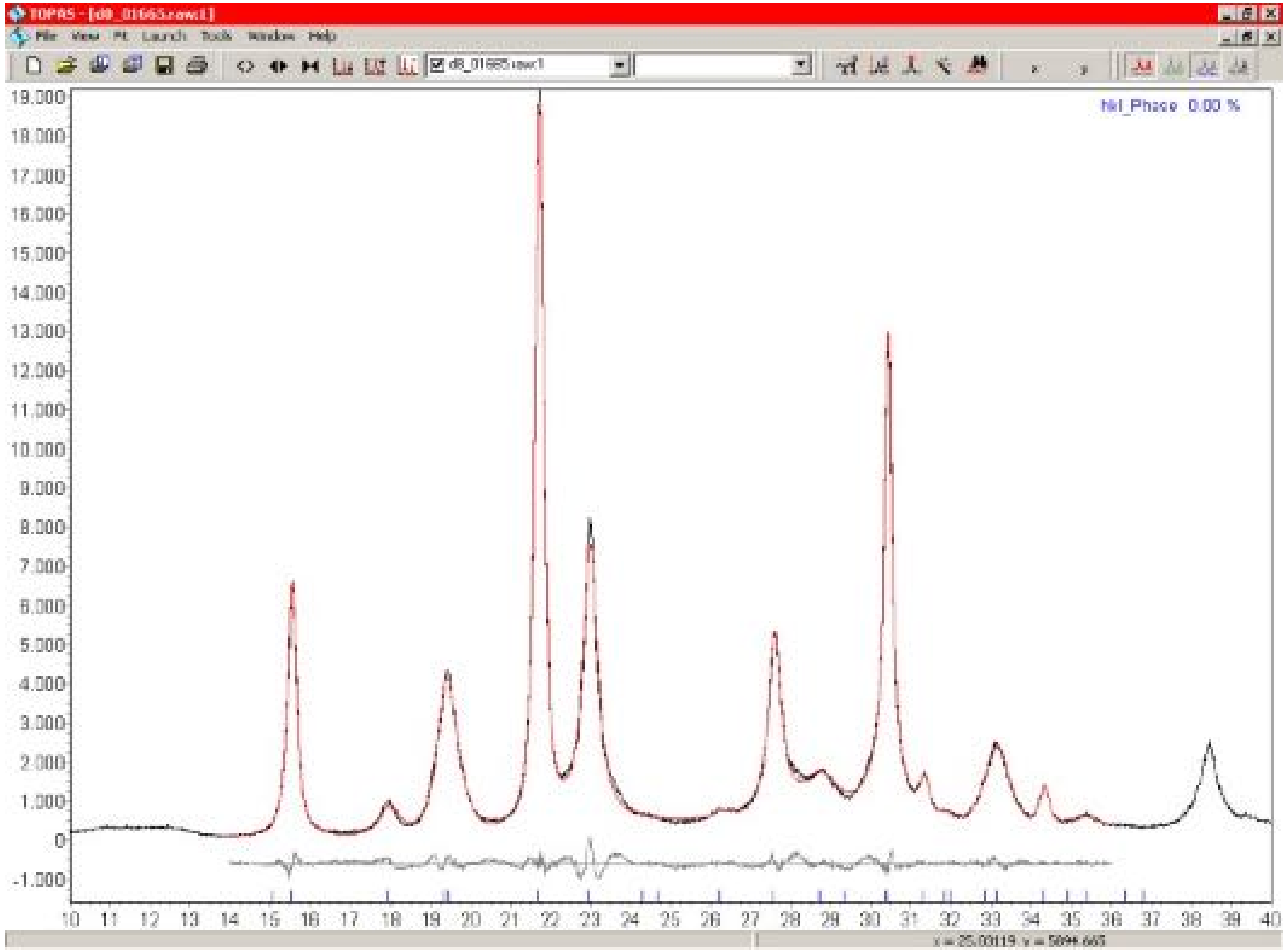


Amorphous 1.00%

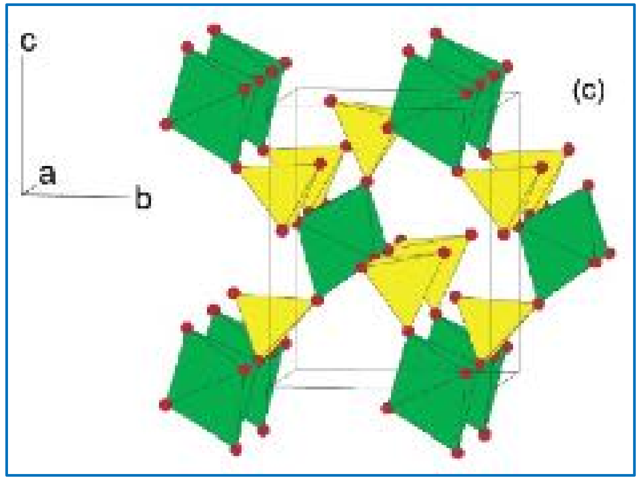
Data	Corundum [%]	Quartz [%]	Glass [%]
NS-A	50.01	0	49.99
NS-B	49.98	15.03	34.99
NS-C	49.99	30	20
NS-D	50	40	10
NS-E	50	45	5
NS-F	50.01	48	1.99
NS-G	50	49	1
NS-H	50.02	49.48	0.5
NS-I	49.87	50.13	0

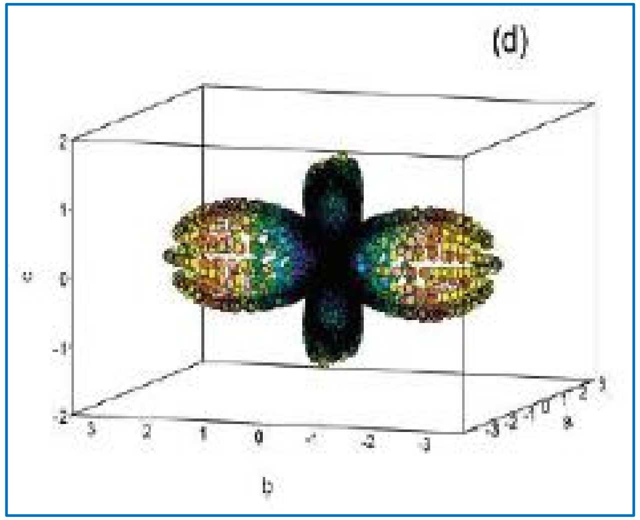
Z. Kristallogr. 226 (2011) 944–955

Anisotropic Microstrain



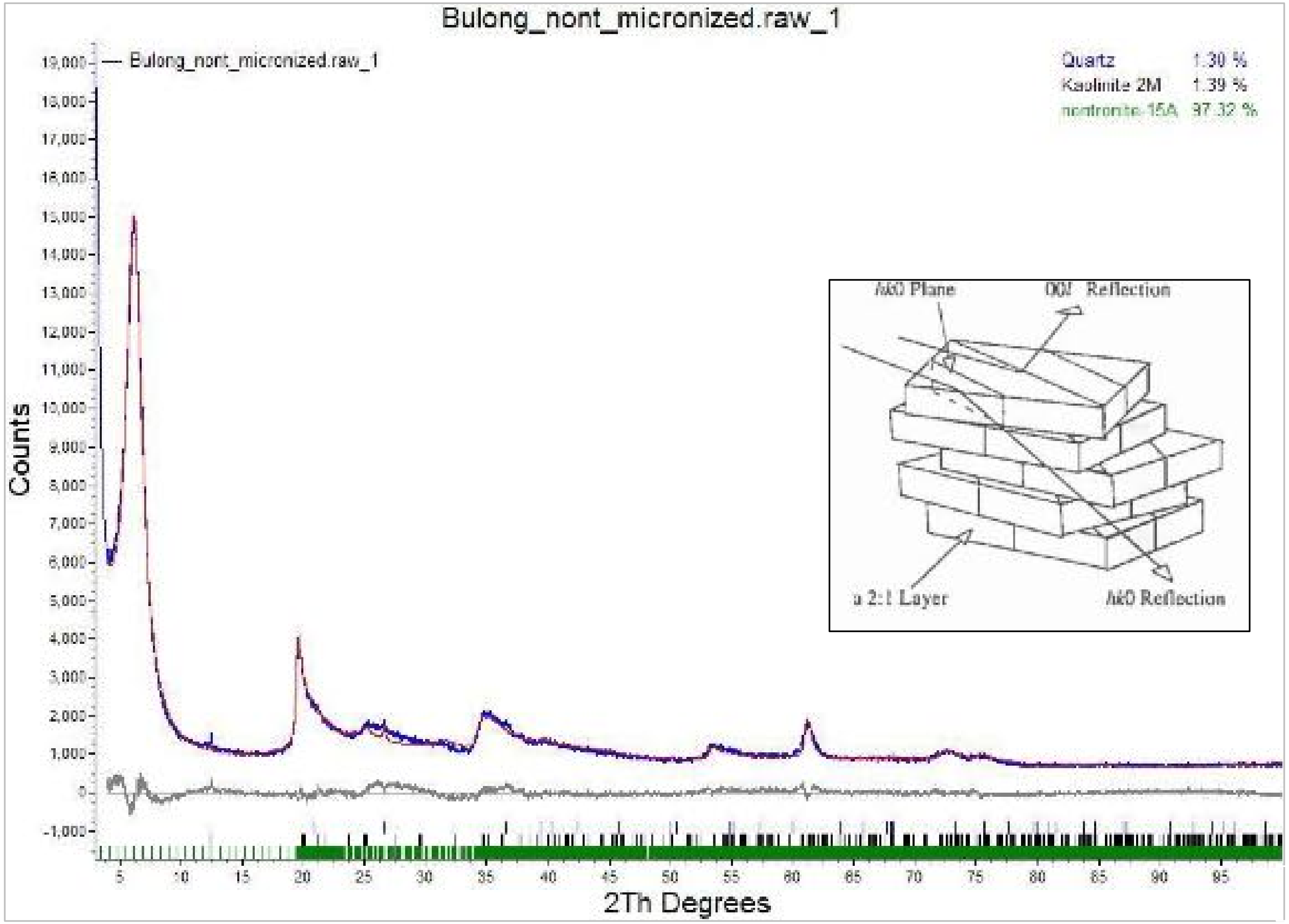
Allen et al, 2003

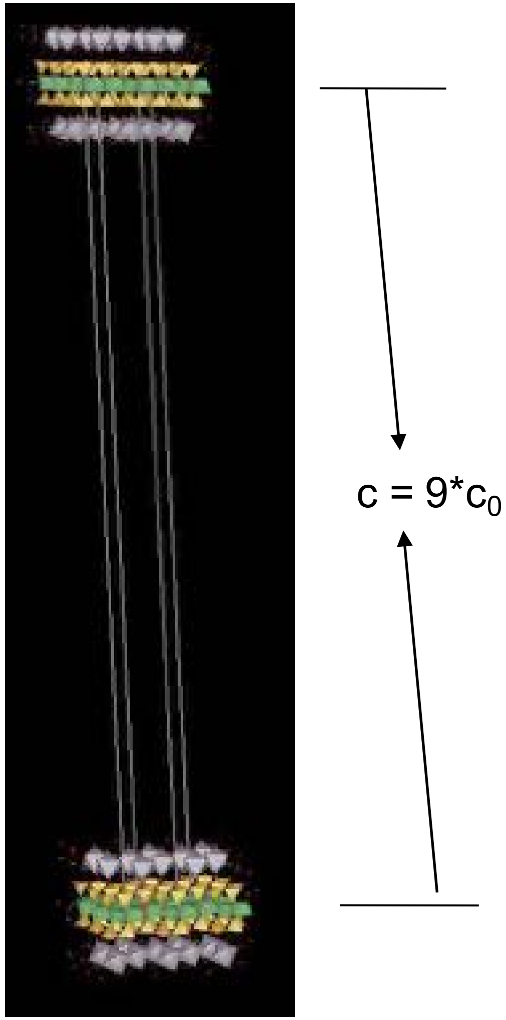




Spherical harmonics function used to model lattice strain in b-direction

Supercell Modelling of Clay Minerals



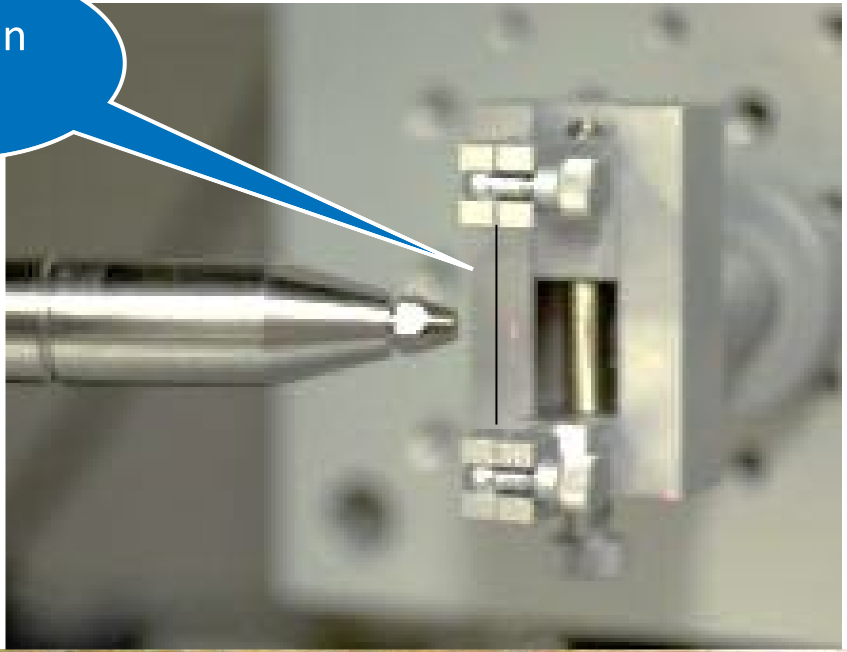


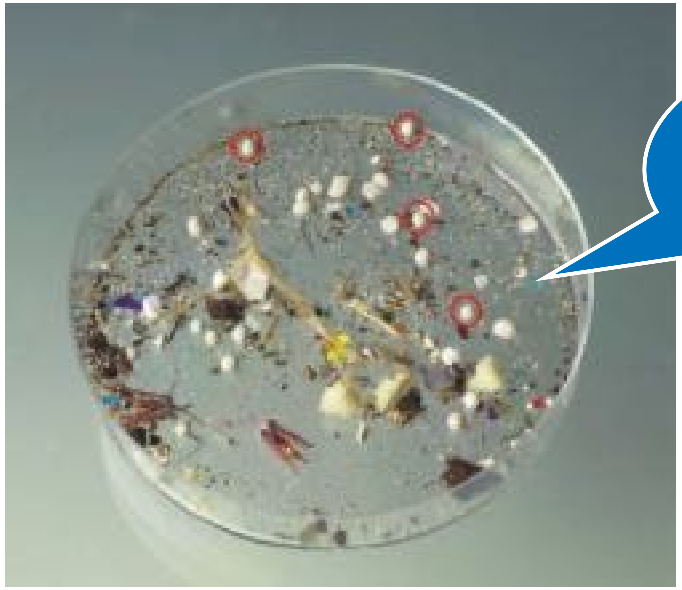
Supercell

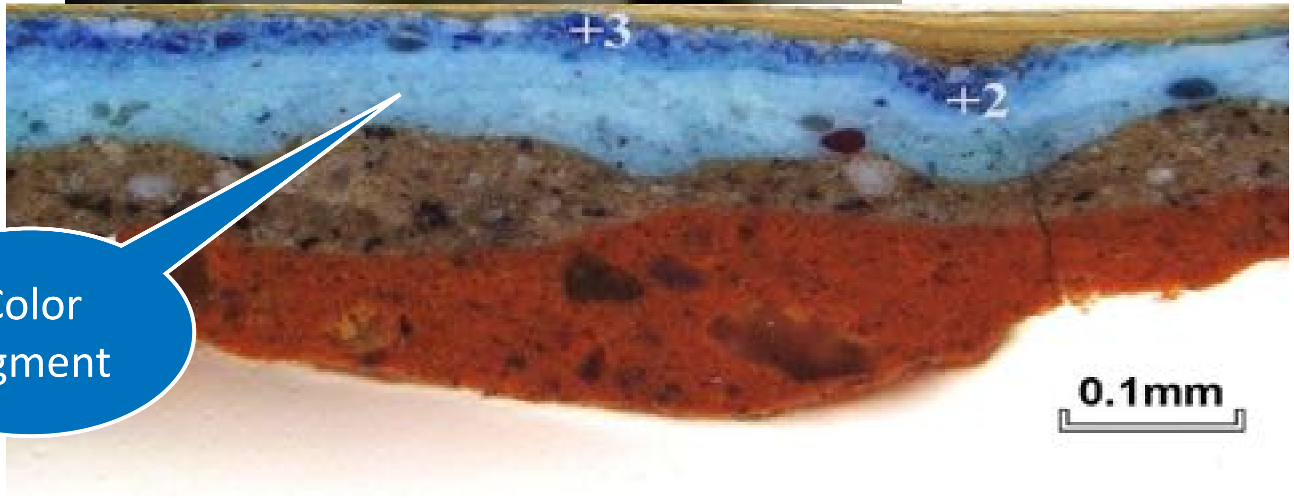
Ufer et al, 2004, Wang, 2012

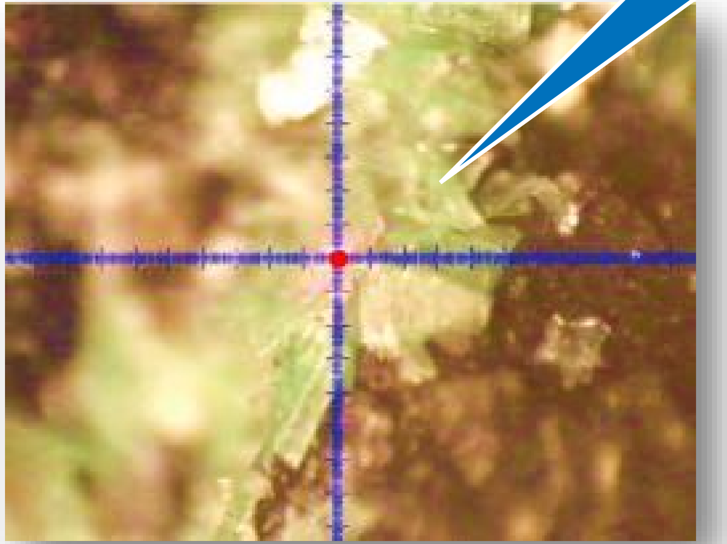
- What is Powder
- X-ray generation
- Peak Profiles
- Data Acquisition
- LOD & LOQ
- Qualitative Analysis
- Quantitative Analysis
- **ADVANCES**

Micro-Diffraction

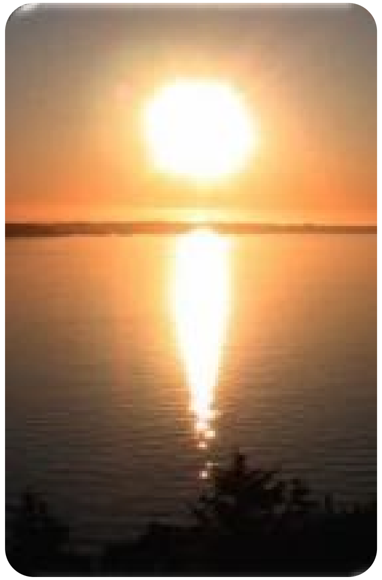
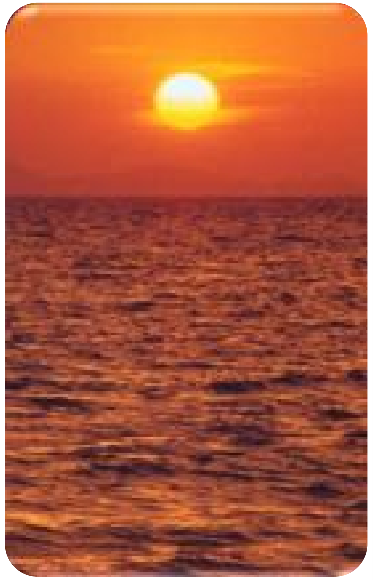
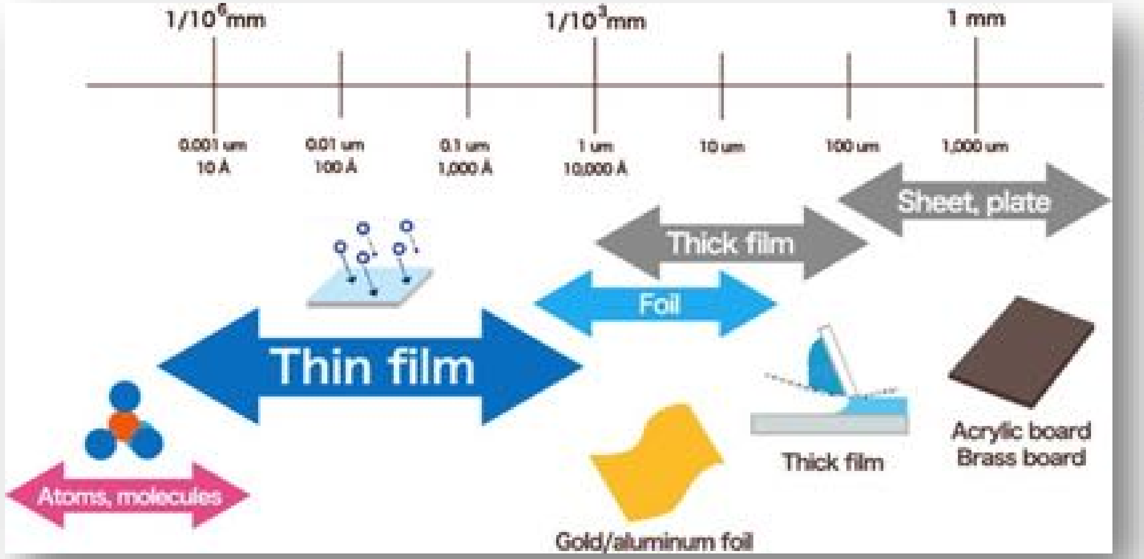


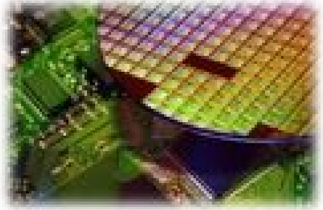






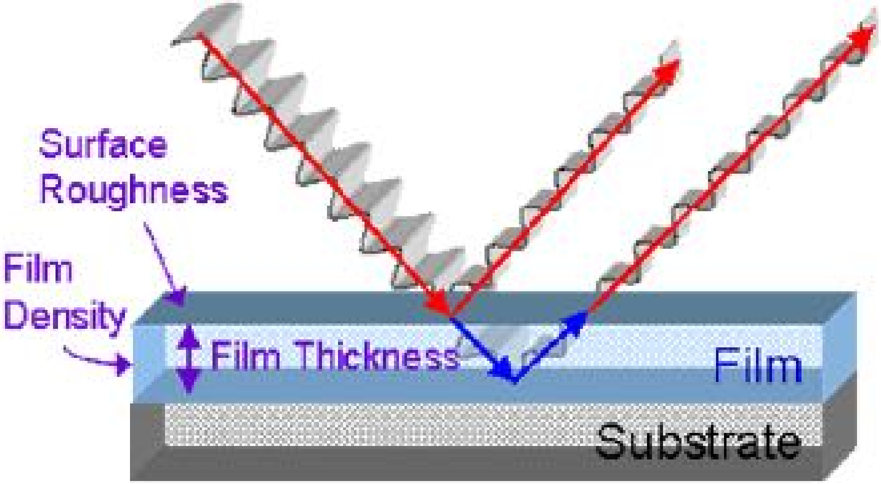
Thin Film - XRR

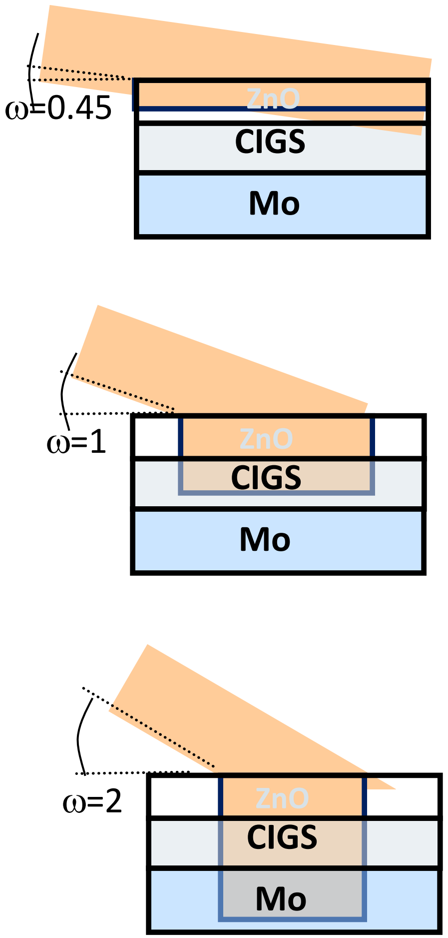

Reflectivity information:

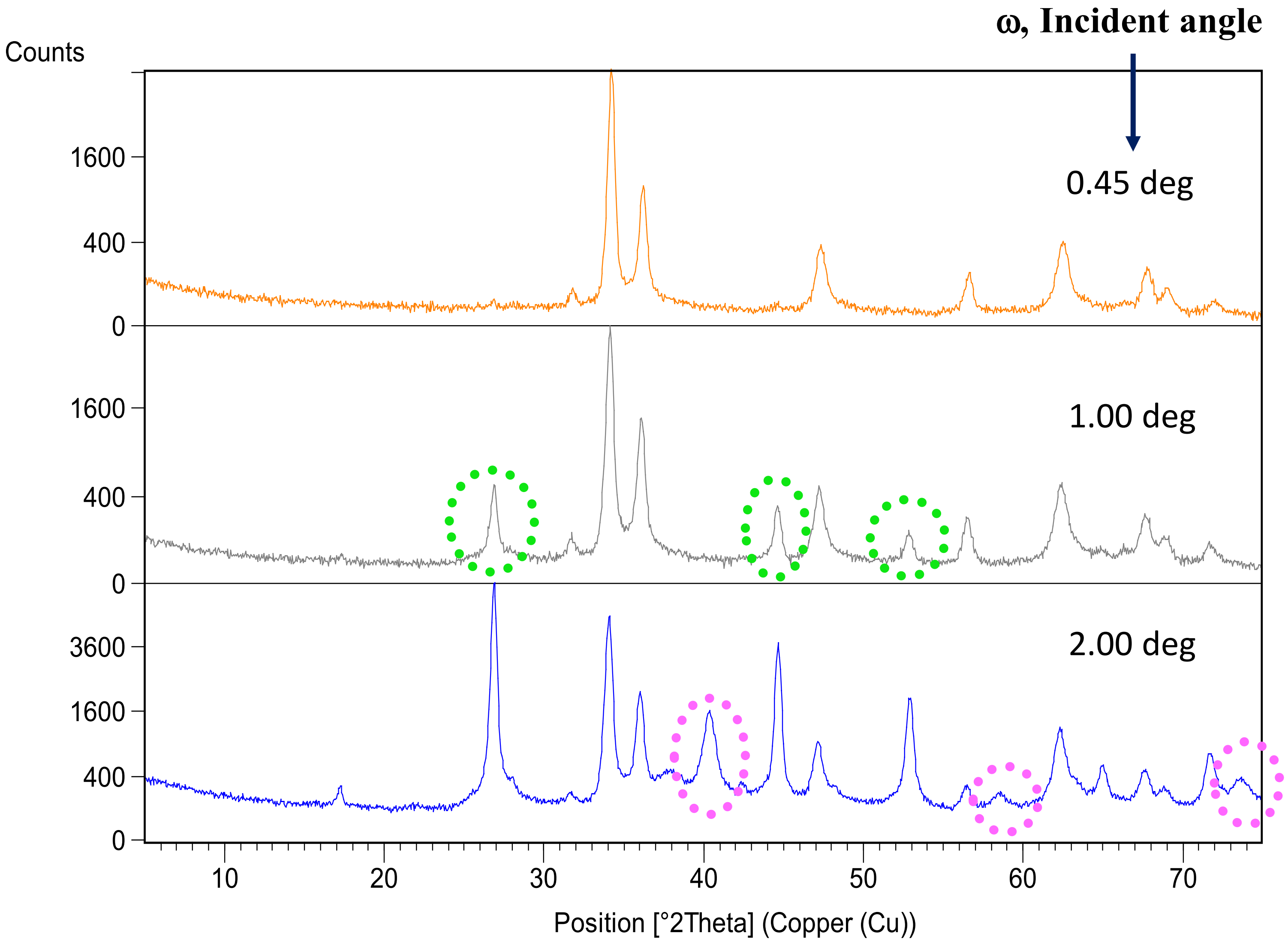
- ✓ Density
- ✓ Thicknesses
- ✓ Roughness
- ✓ Interface quality



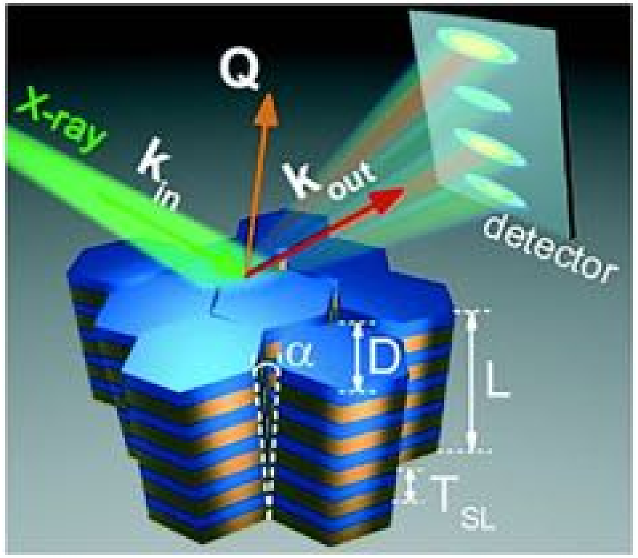
- ❖ X-rays interact with the whole film
- ❖ Thickness 0.1 - 1,000 nm
- ❖ Structural scale > nm measurement
- ❖ $\omega < 7°$ or ($2\theta < 14°$)

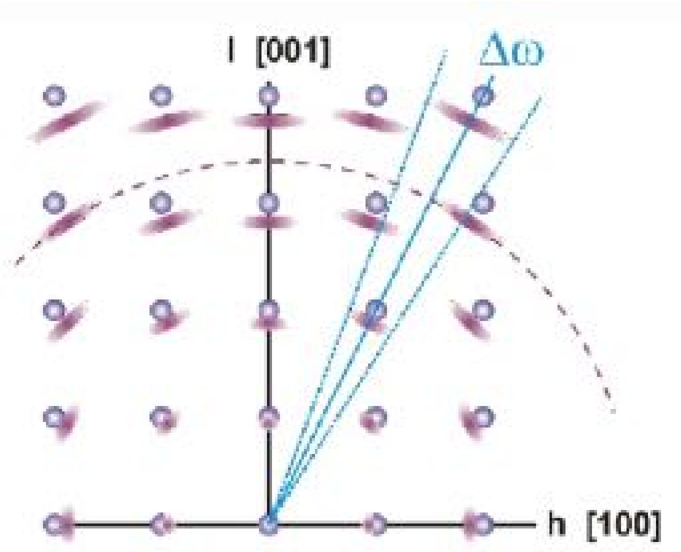
Thin Film - Depth Profile Analysis

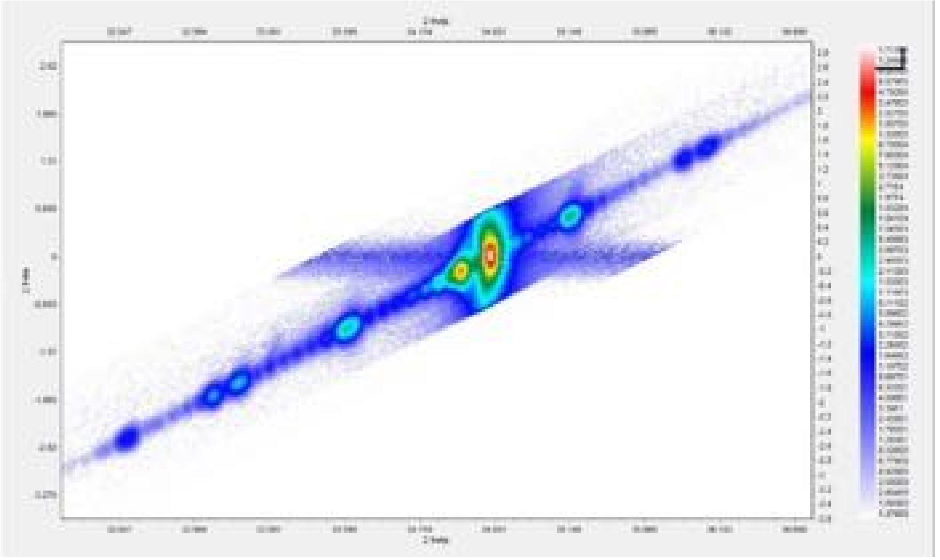


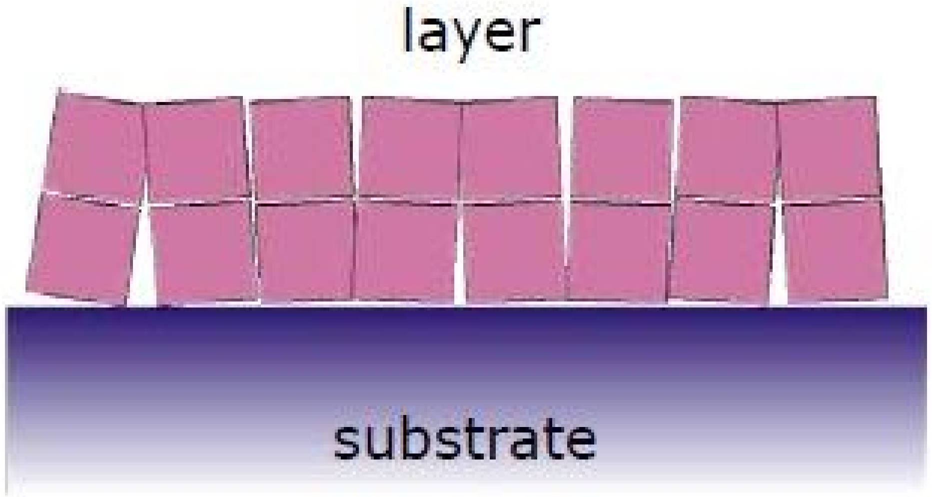


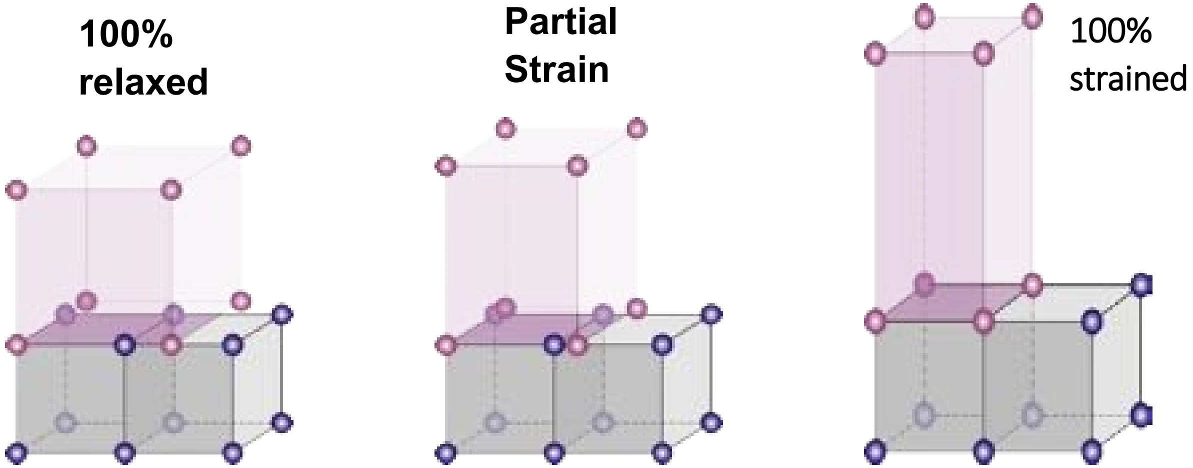
Thin Film - Reciprocal Space Map



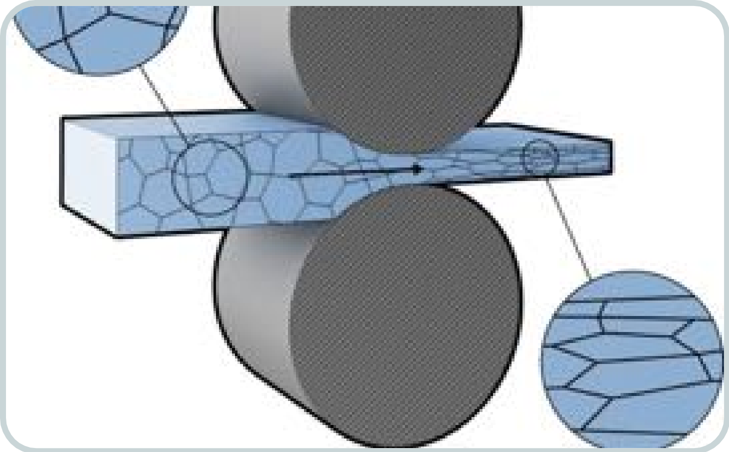








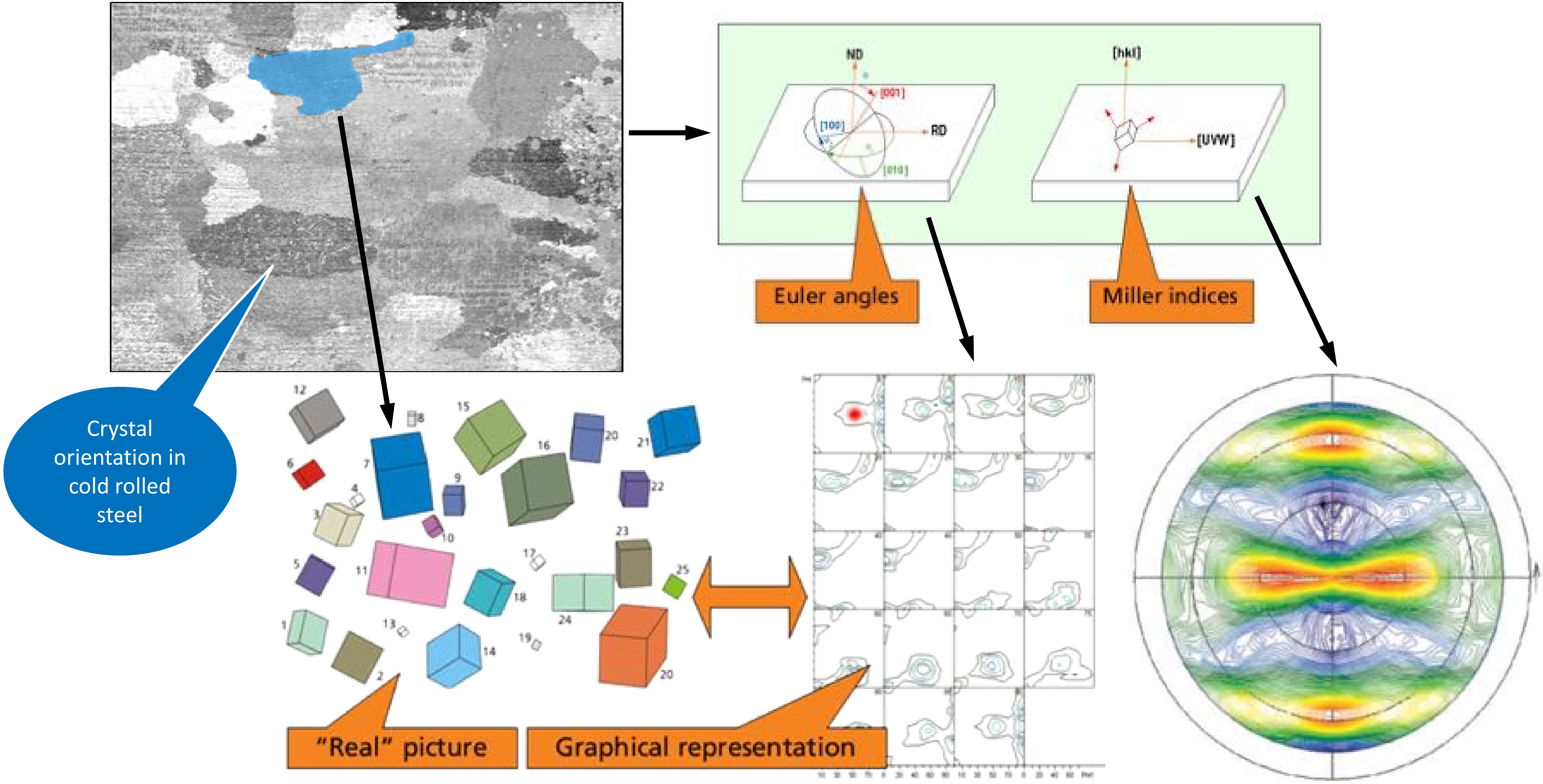
Texture and Residual Stress



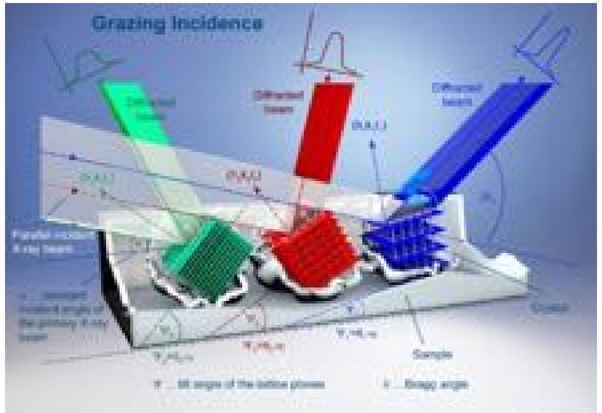
Crystal orientation dictates:

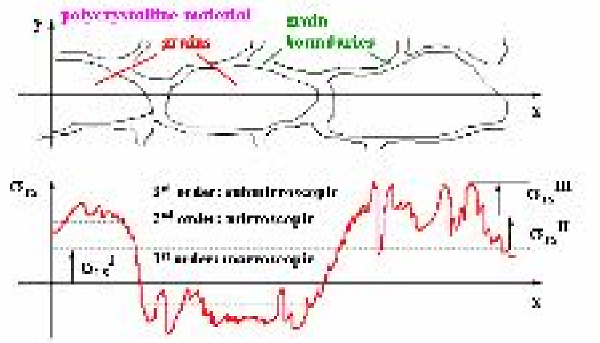
- Strength
- Elasticity
- Hardness
- Thermal expansion
- Conductivity
- Optical properties
- Magnetic properties
- etc

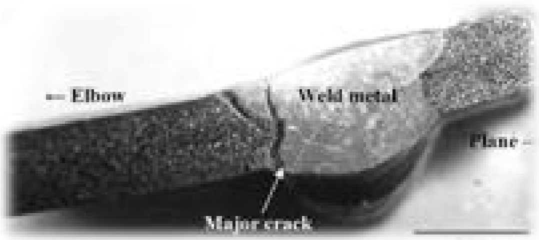
Bulk - Texture

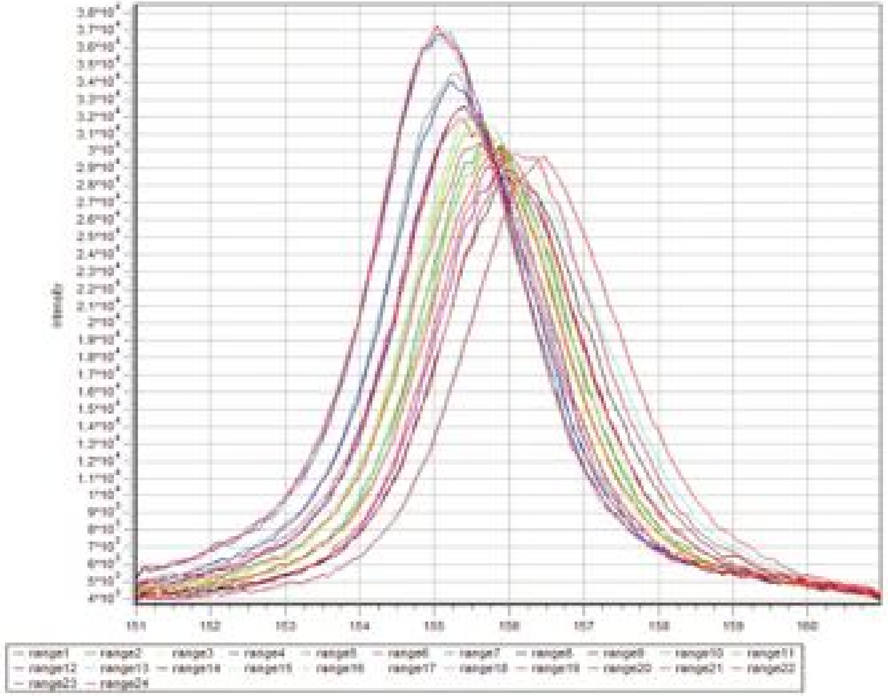


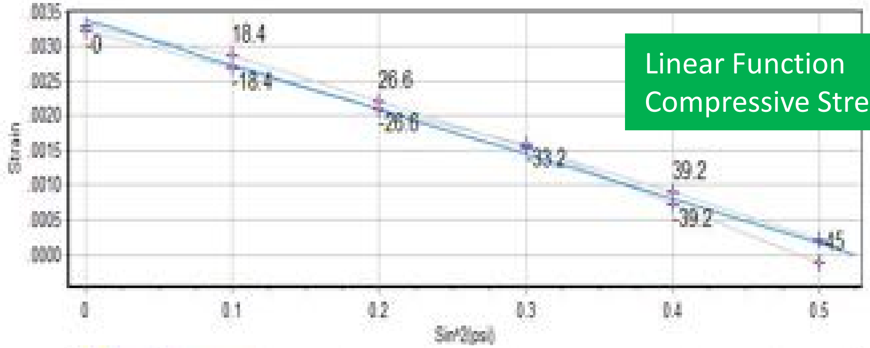
Residual Stress



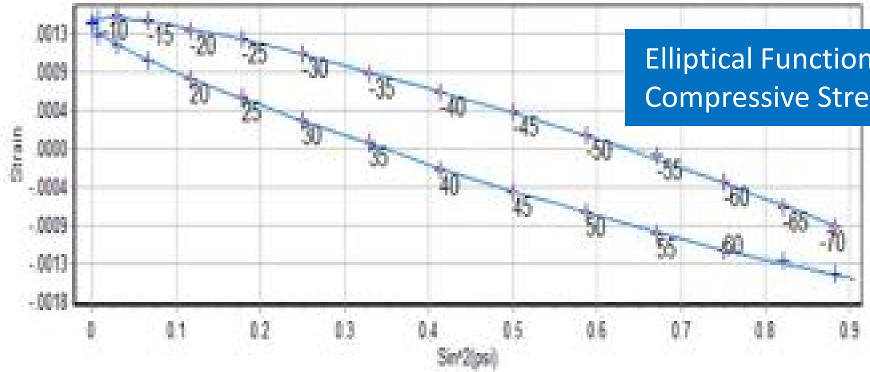




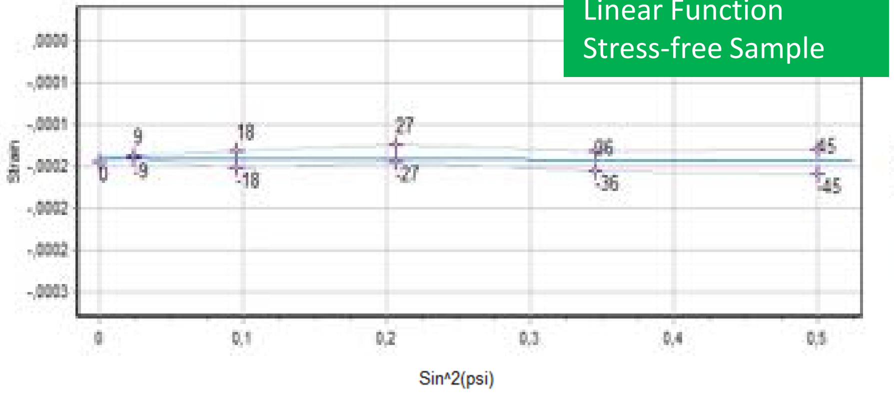




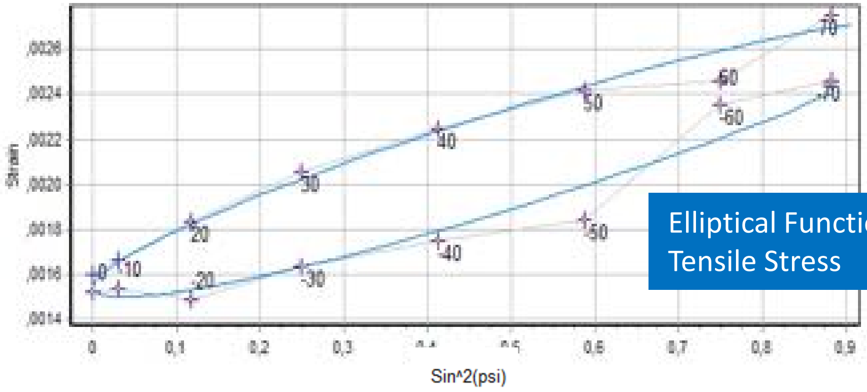
Linear Function
Compressive Stress



Elliptical Function
Compressive Stress



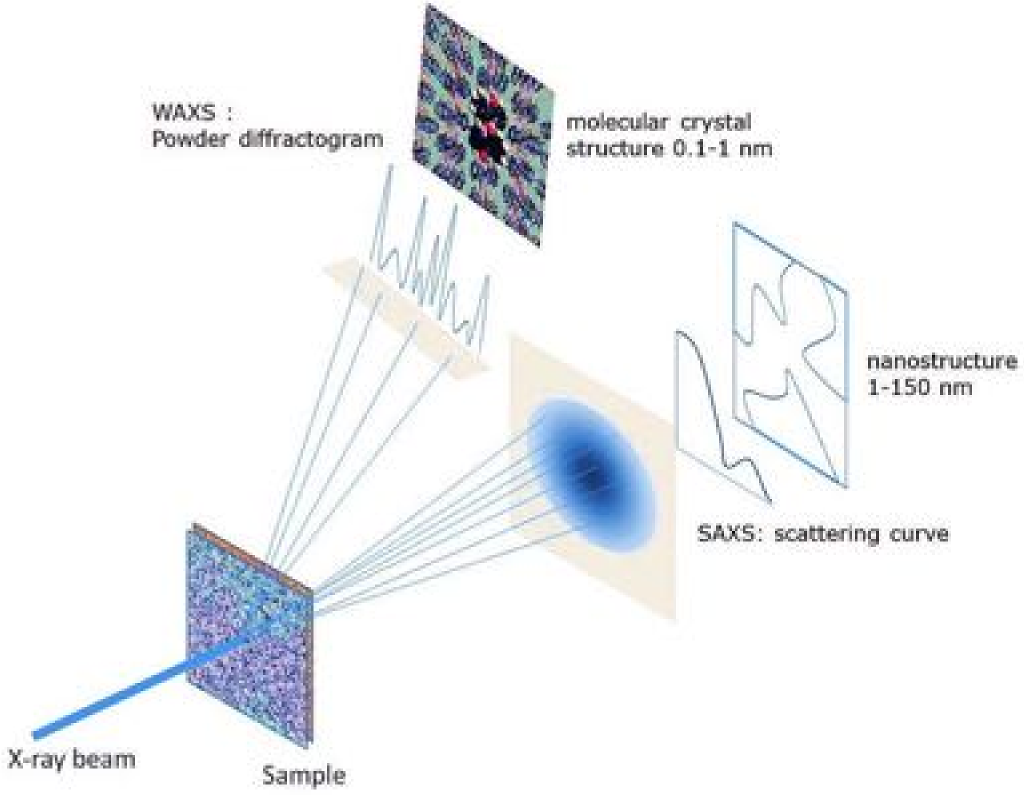
Linear Function
Stress-free Sample

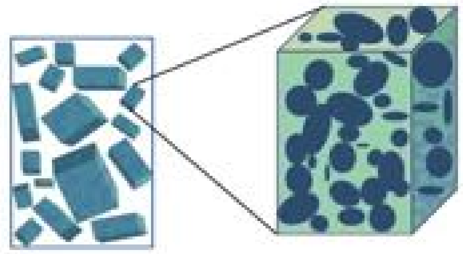


Elliptical Function
Tensile Stress

Nano Particles - SAXS

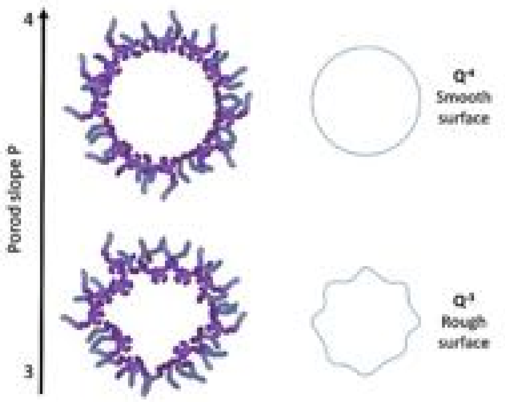
- Sensitive to density fluctuations at the nano-scale
- Covers range from 1nm - 150nm (Cu-source)
- Typically measured in transmission

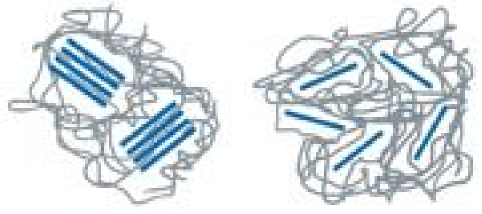




Outer surface
Particle envelope

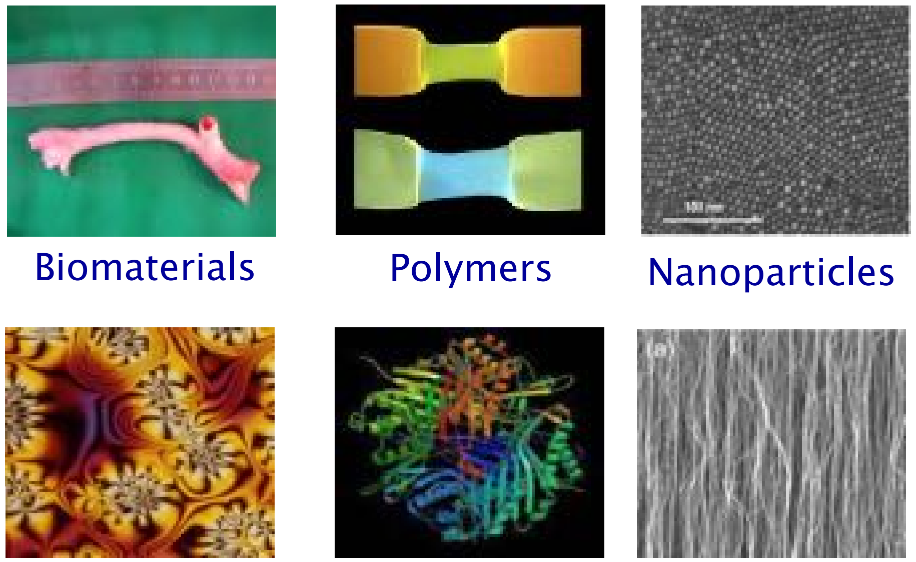
Inner surface
Pores-cavities





Intercalated

Exfoliated



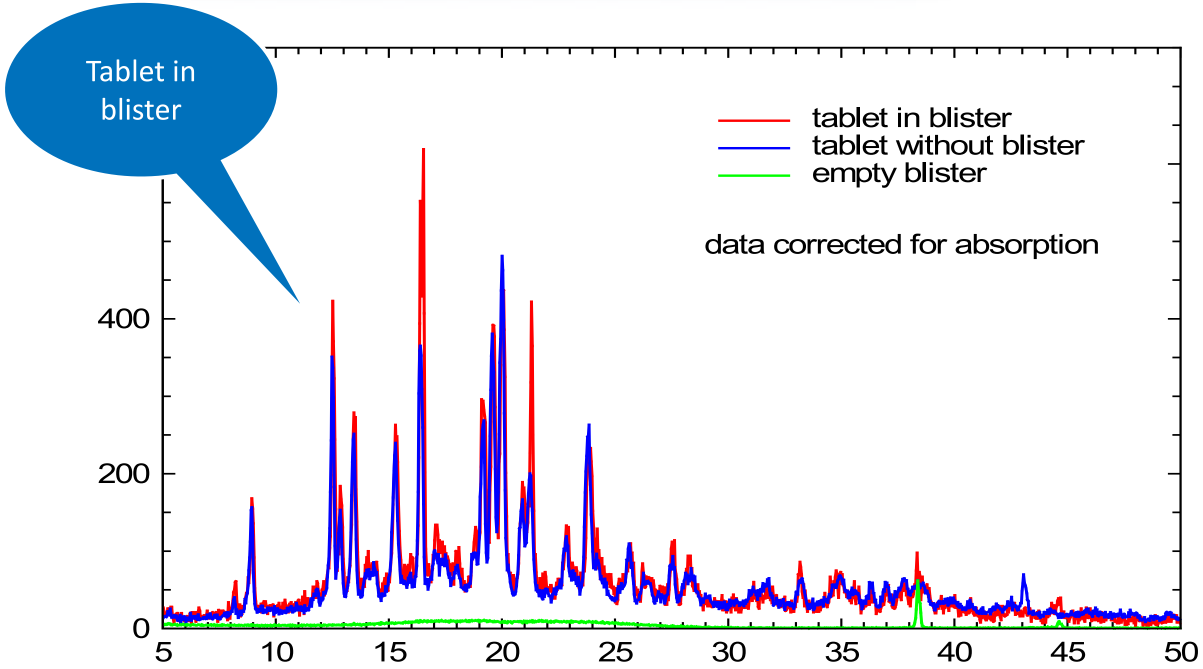
Biomaterials | Polymers | Nanoparticles

Liquid crystal | Proteins | Nanotubes

Which information can be extracted?

- Typical size, size distribution
- Distance between particles
- Arrangement of Nano-particles
- Texture of large structures
- Details about system (volume fraction degree of crystallinity)
- Porosity
- Shape and dimensions of Nano-particles
- Strain, etc.

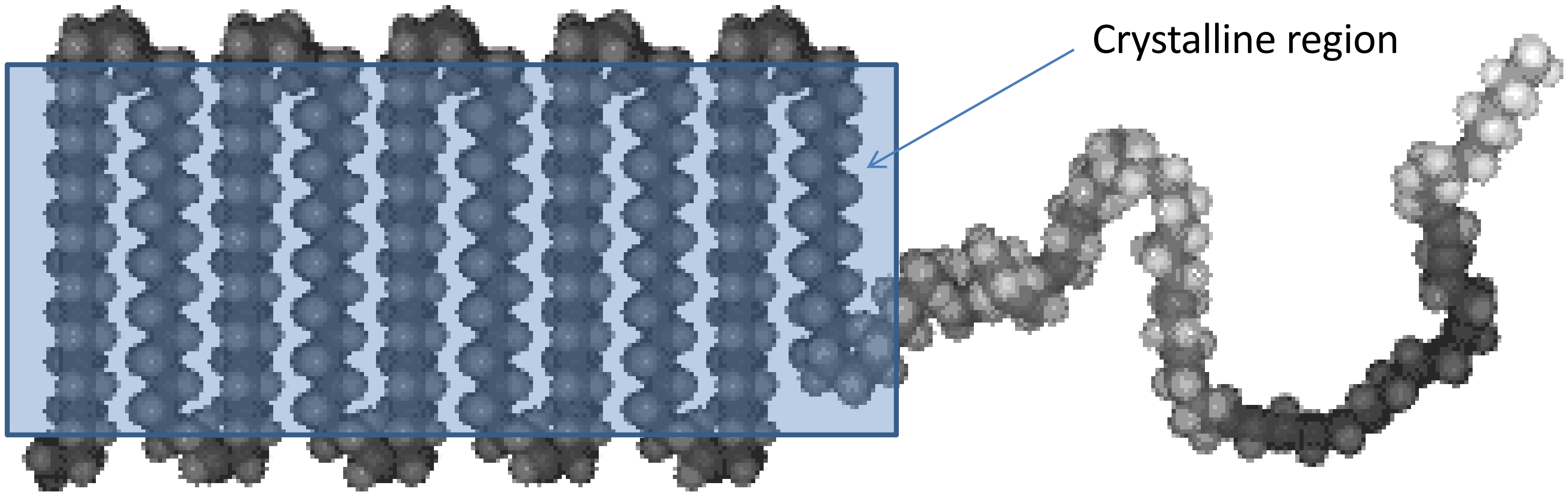
Pharmaceuticals



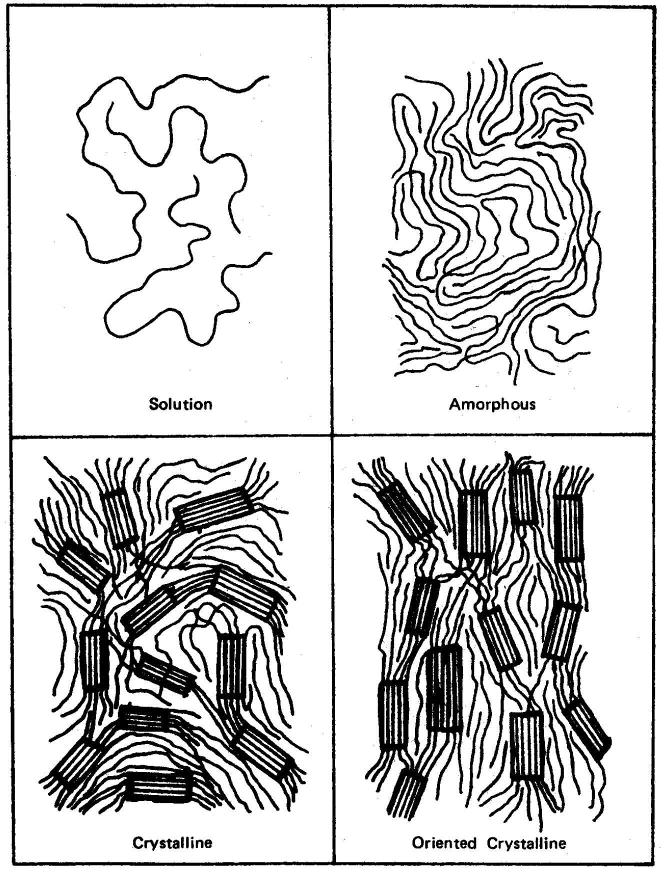
Polymers

Once the monomers link together they form long chains sort of like strands of spaghetti.

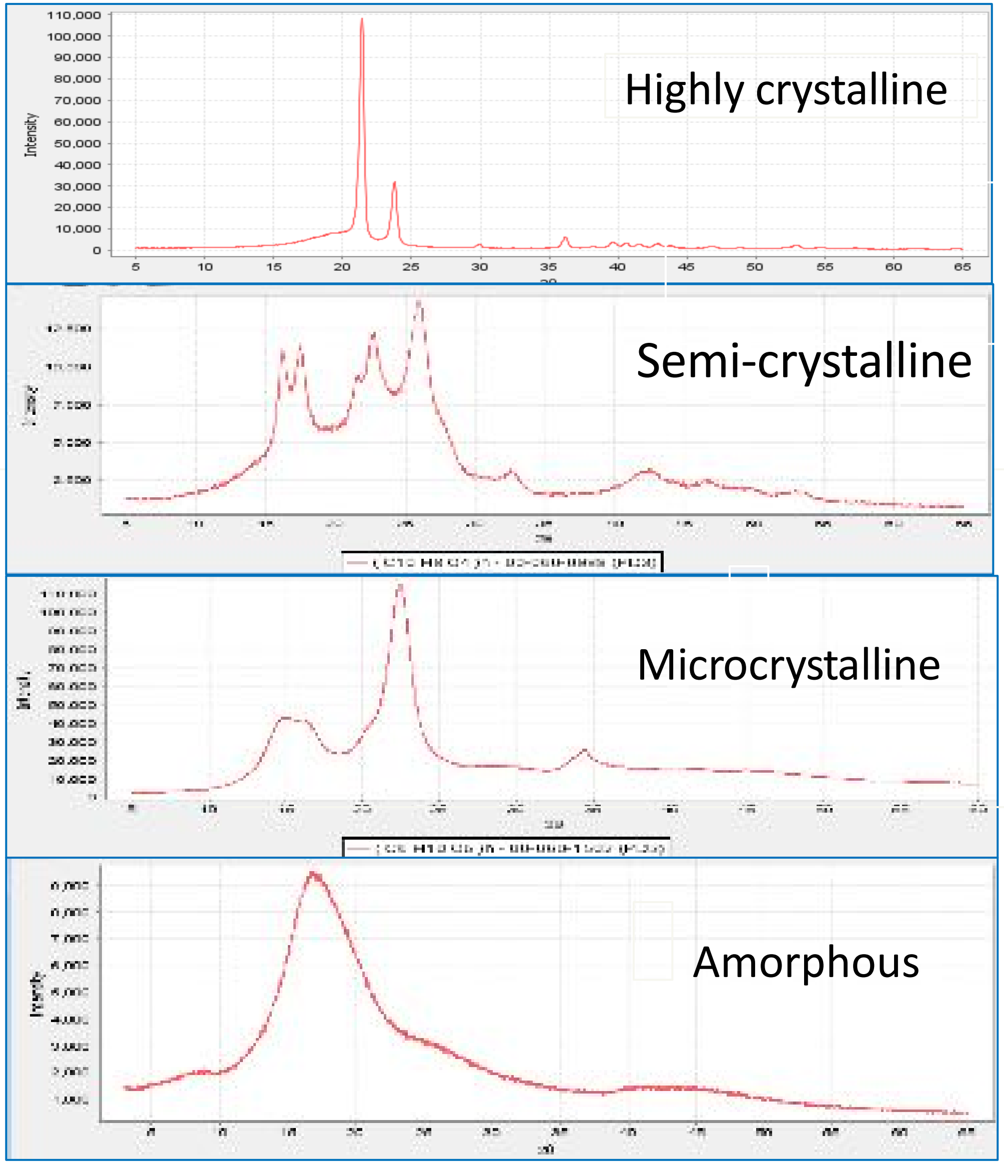
The chains can move and fold – chain folding is one means by which crystalline regions can form, hydrogen bonds (and other types of intermolecular forces) often help in linking chains together.



Polimer Crystalline States



From “Selected Papers Of Turner Alfrey”, Marcel Dekker Inc, 1986



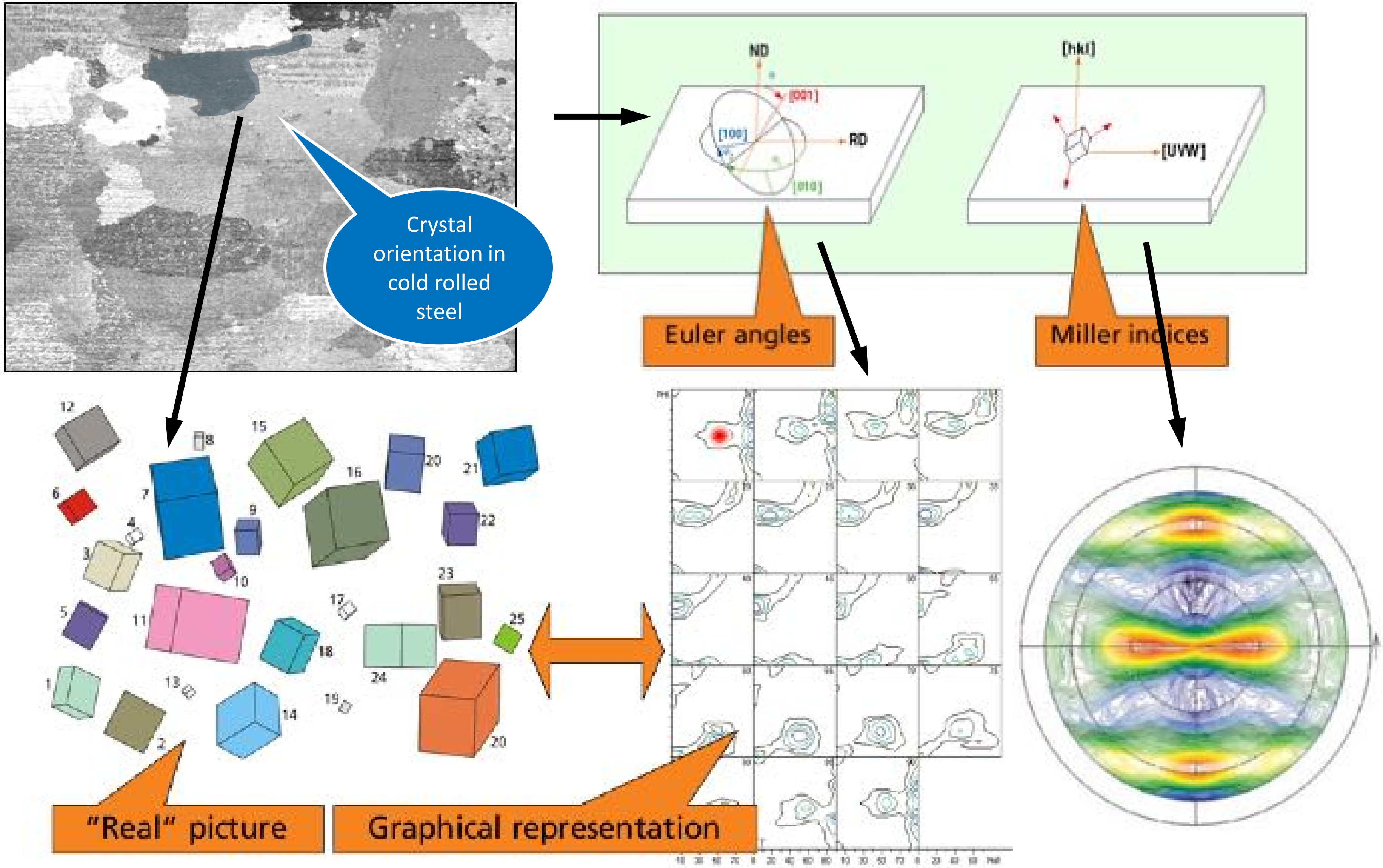
Non-Ambient

In-situ XRD can yield quantitative analysis to study reaction pathways, rate constants, activation energy, and phase equilibria

Application area:

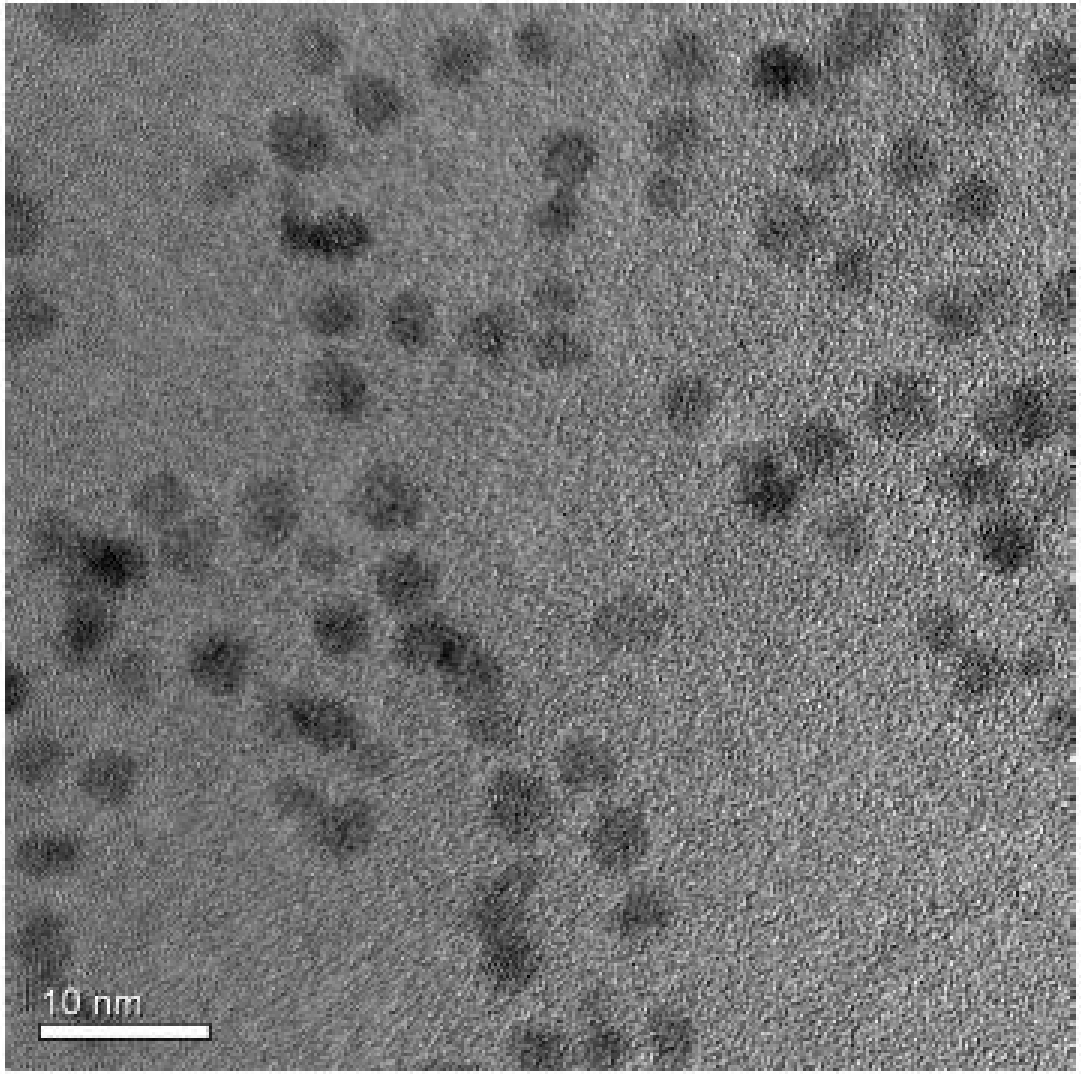
- Heat treatment and annealing
- Calcination and sintering
- (De)Hydration processes
- Material formation / structure changes
- Material changes during operation
- Li-ion Charge/Discrage

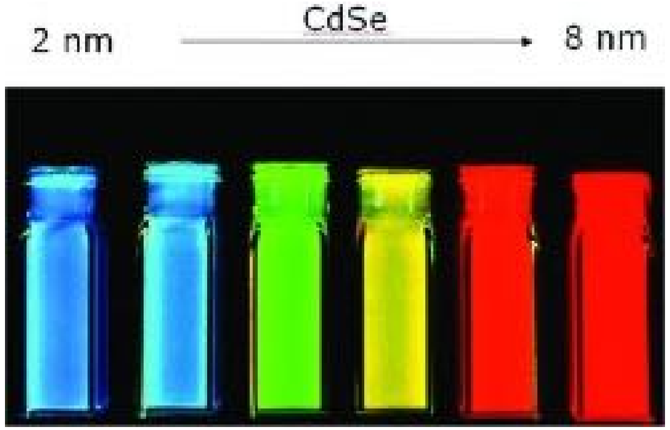
Textures

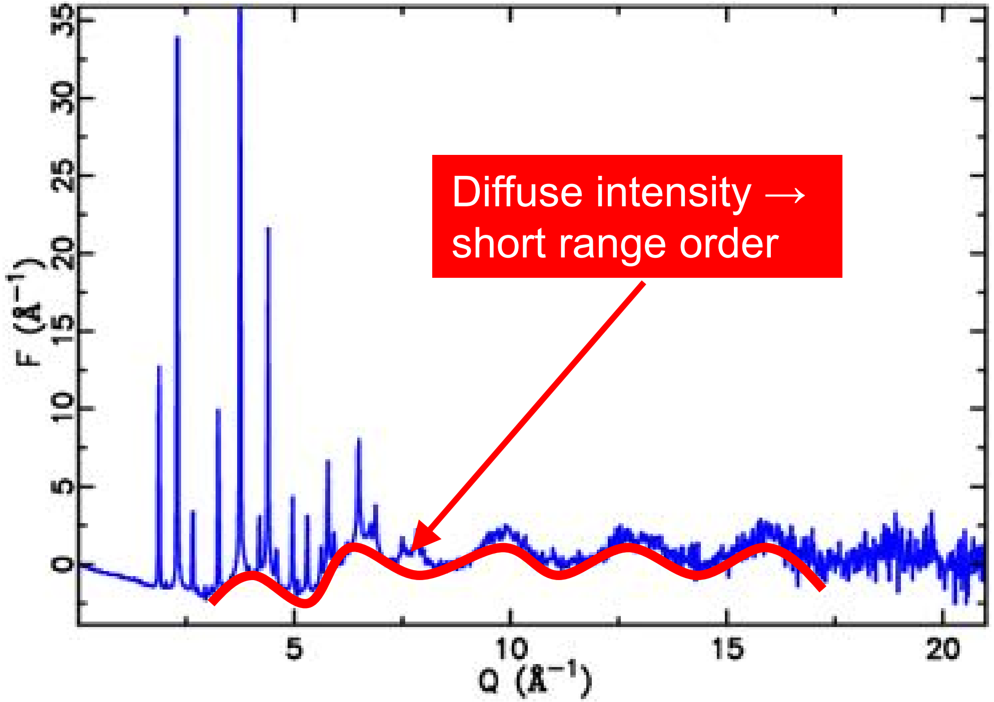


Crystal orientation in cold rolled steel
ND
[001]
[100]
RD
[010]
[hkl]
[UVW]
Euler angles
Miller indices
"Real" picture
Graphical representation

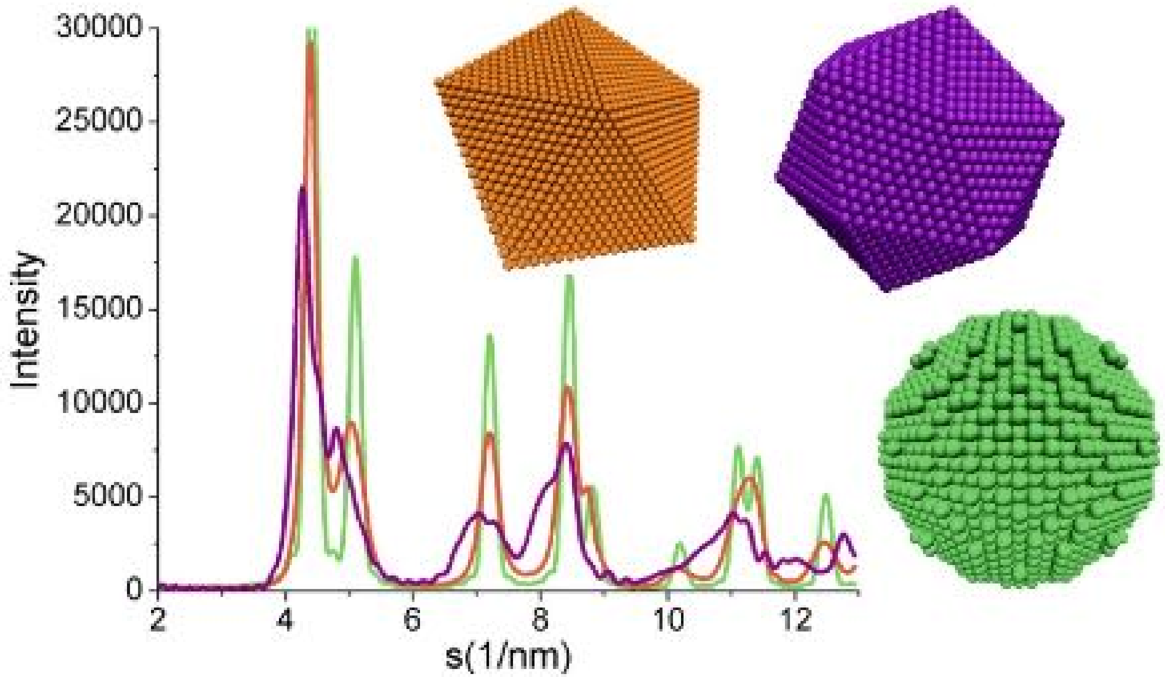
Short Range Order

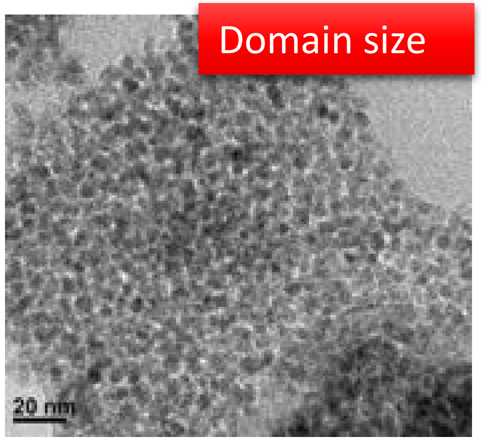


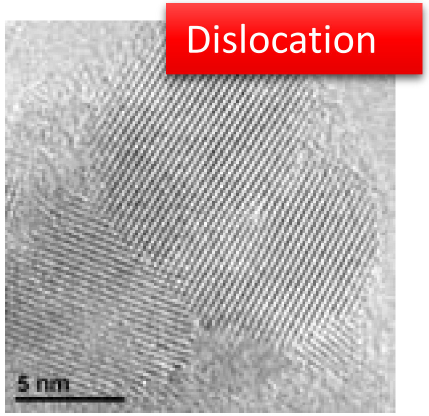


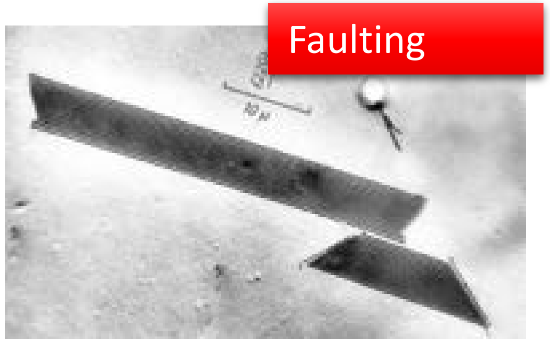


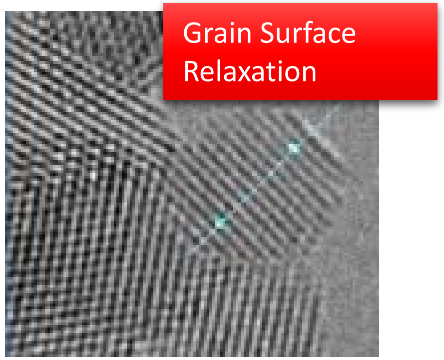
Whole Powder Pattern Modeling

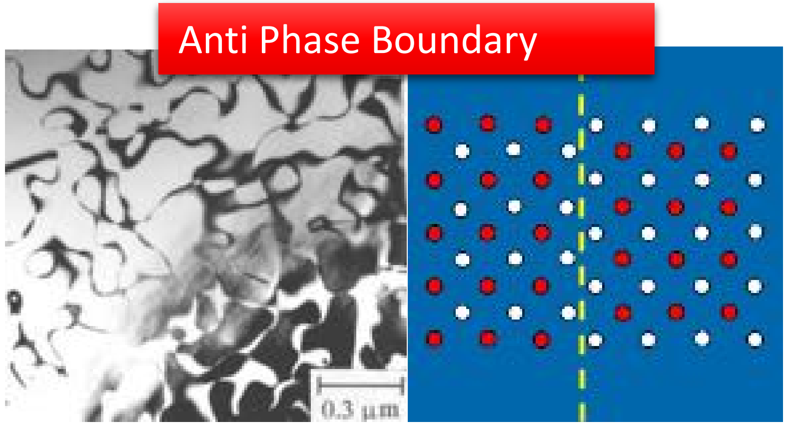


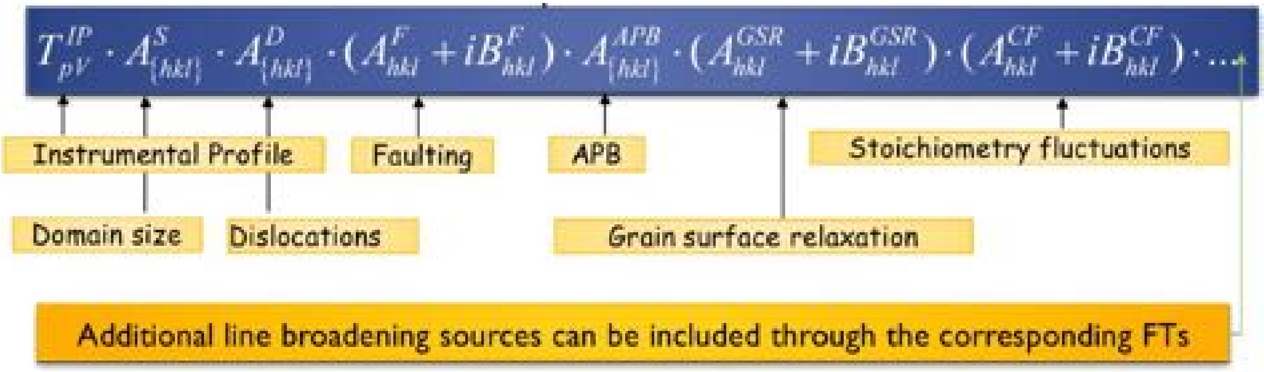












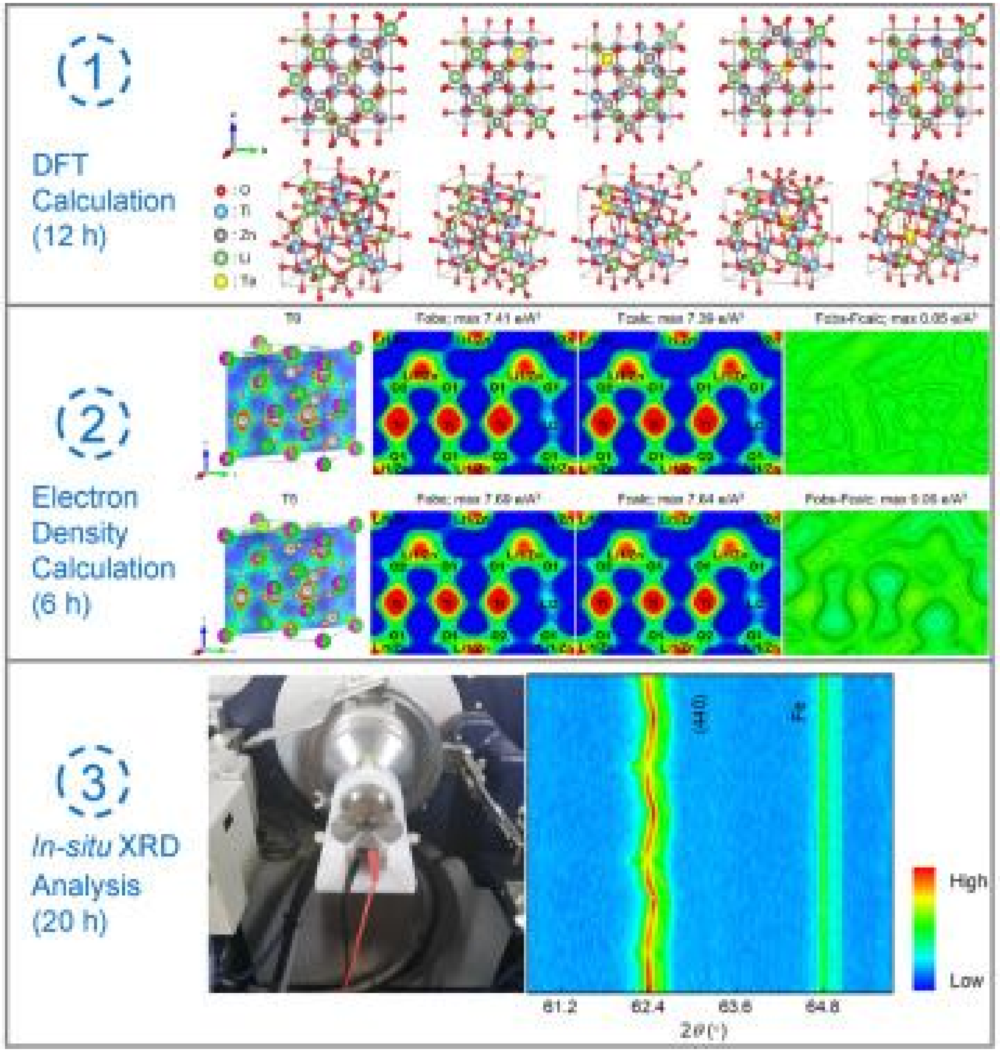
Refer to Prof. Matteo Leoni, Matteo.Leoni@unitn.it

- Published Articles
- under
- ================
- Crystallography
- X-Ray &
- Neutron diffraction
- ================
- Diffraction Lab. Result

Visualizing crystal structure evolution of electrode materials upon doping and during charge/discharge cycles in lithium-ion batteries

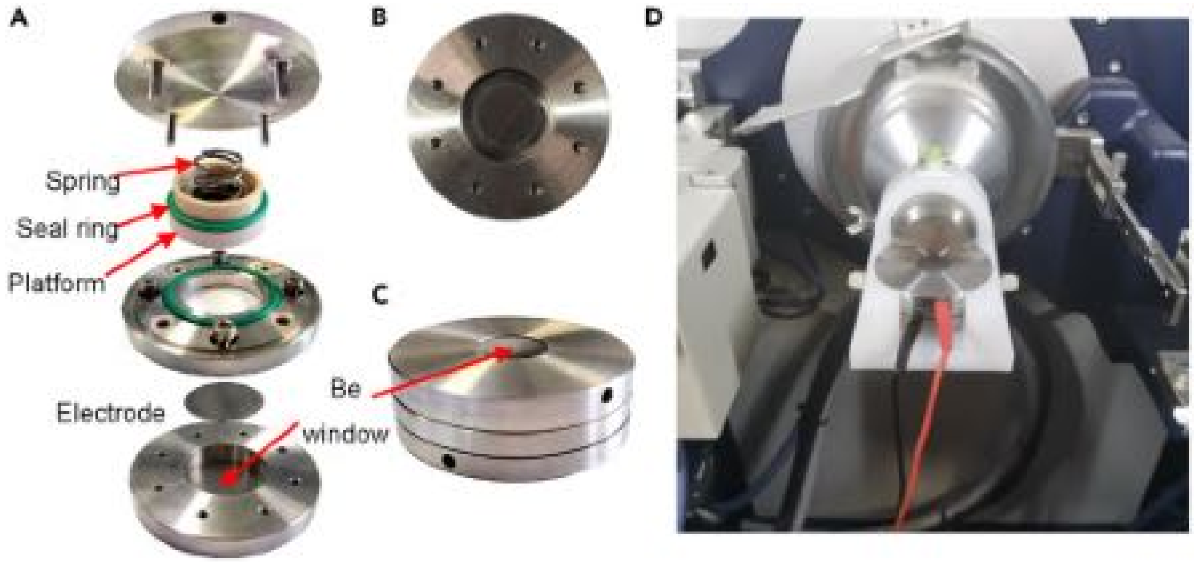
Dongwei Ma, Jing Yang, Masfid Manawan, Chenglu Yang, Jiahui Li, Yongli Liang, Ting Feng, Yong Wei Zhang, Jia Hong Pan

https://doi.org/10.1016/j.xpro.2021.101099



Highlights

- Monodisperse Ta^{5+}-doped $Li_2ZnTi_3O_8$ spheres from TiO_2 spheres as self-template
- DFT and electron density calculations for crystal structure parameters
- *In-situ* XRD analysis to visualize crystal structure evolution of electrodes

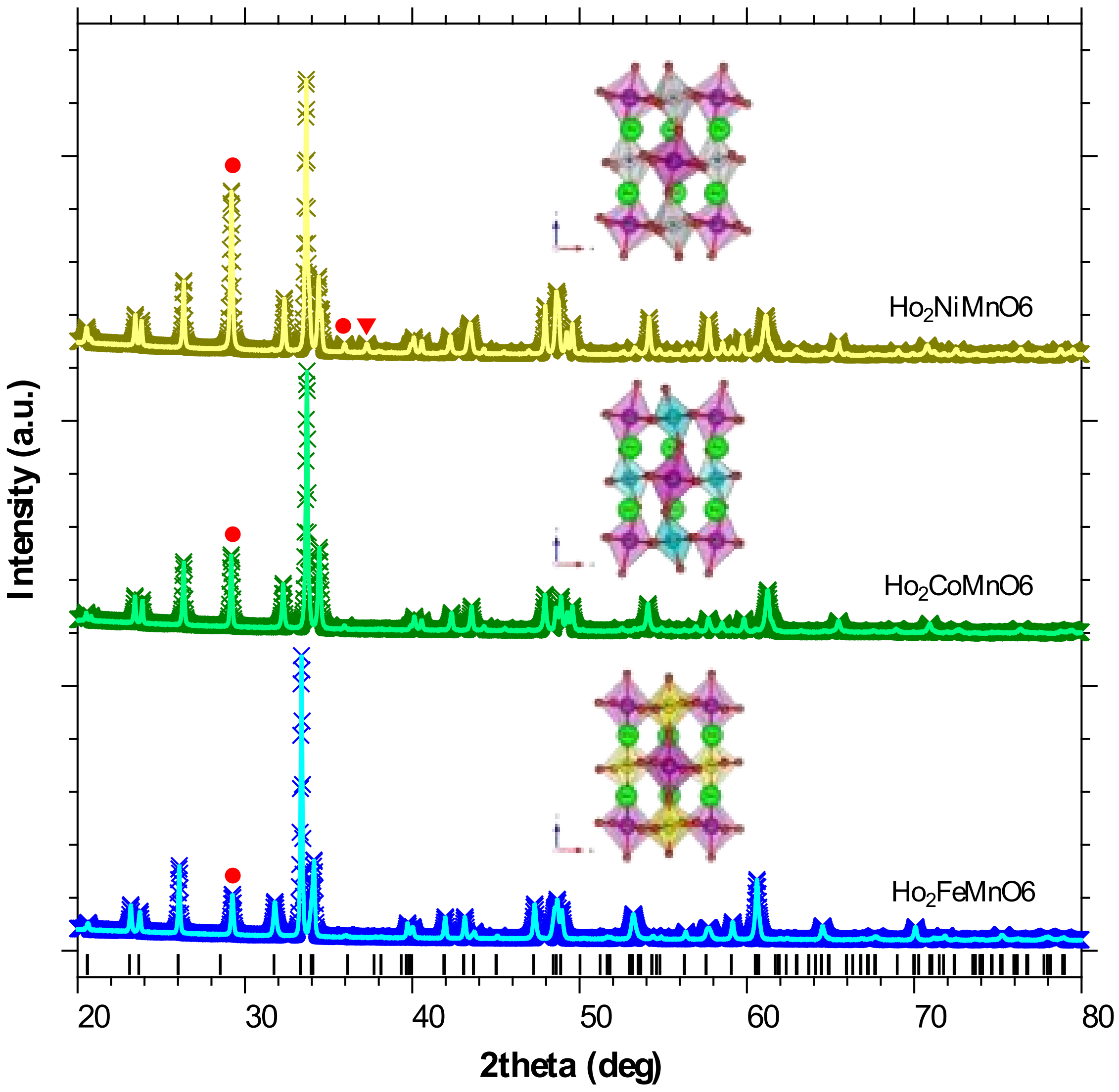


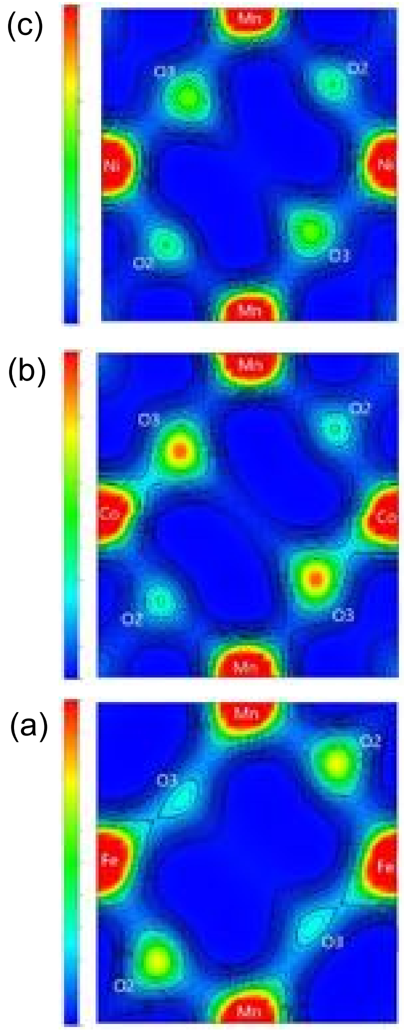
ELSEVIER

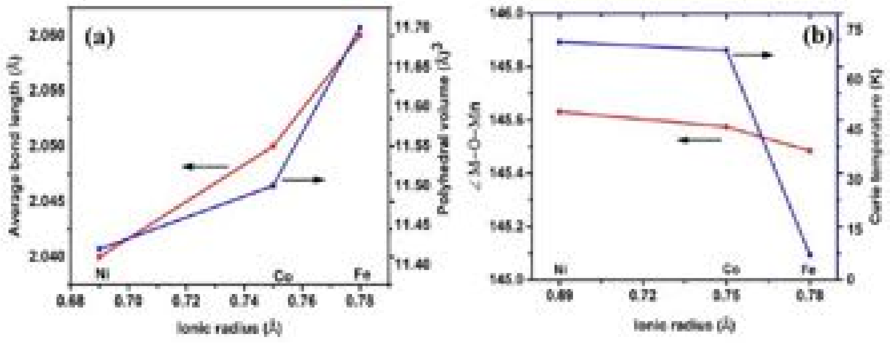
Structural, magnetic and magnetocaloric properties of double perovskite Ho_2MMnO_6 (M = Fe, Co, and Ni)

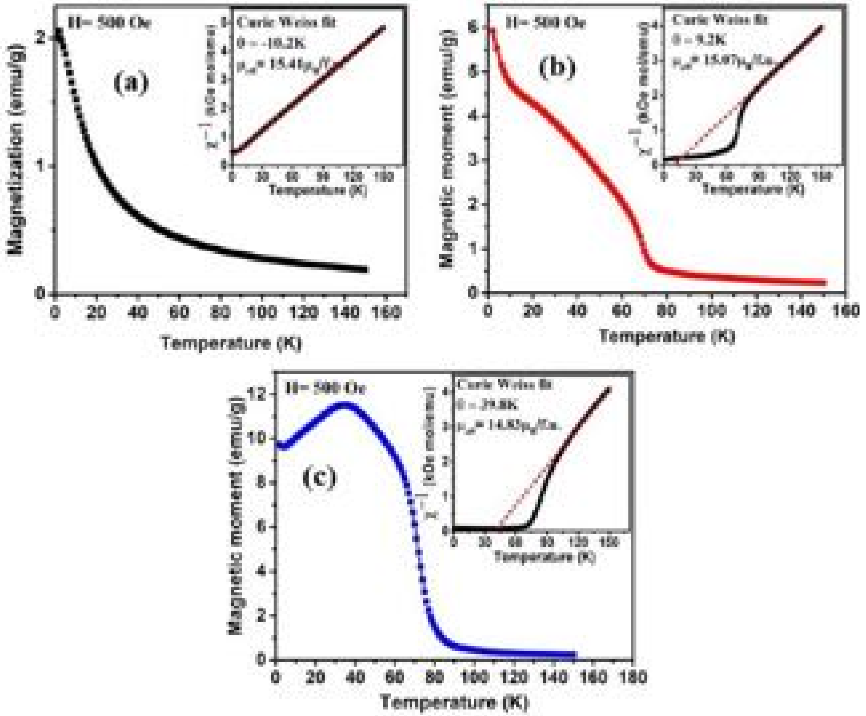
K.P. Shinde, M. Manawan, S.Y. Park, Y. Jo, V.M. Tien, Y. Pham, S.C. Yu, N. Kalanda, M. Yarmolich, A. Petrov, D.H. Kim

https://doi.org/10.1016/j.jmmm.2021.168666







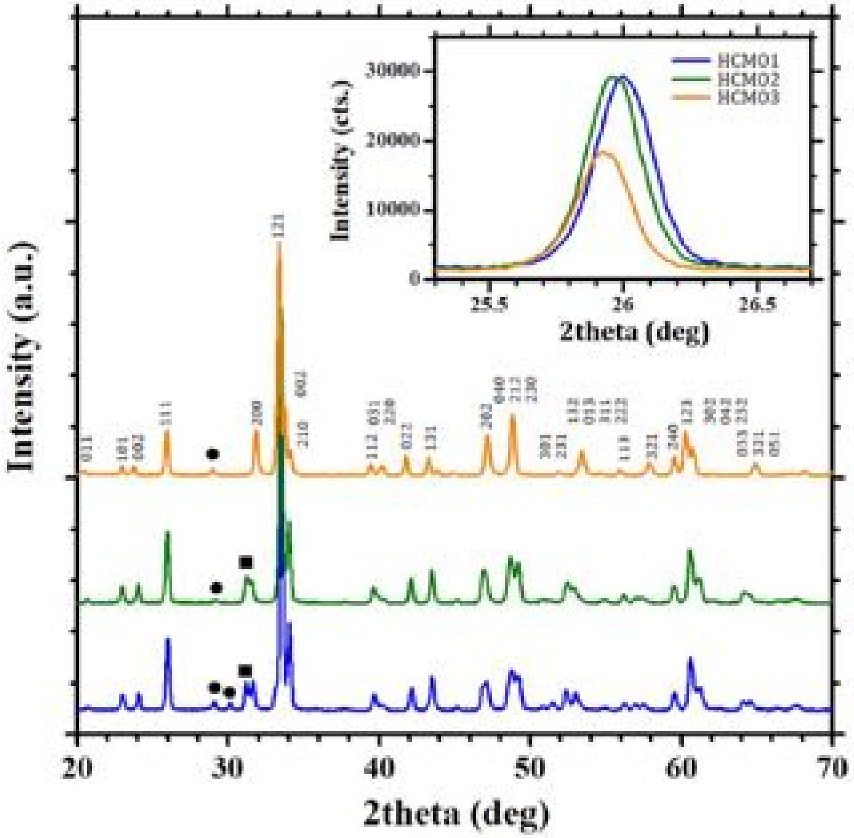


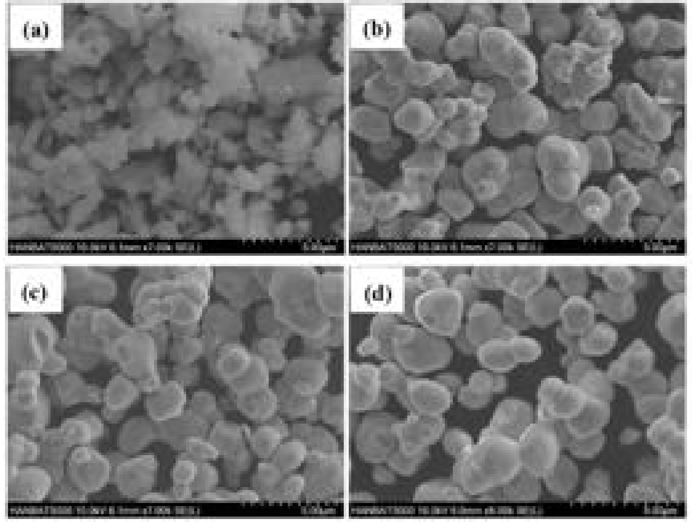
Springer

Study of structural, magnetic, and magnetocaloric properties of $Ho_{1-x}Ca_xMnO_3$

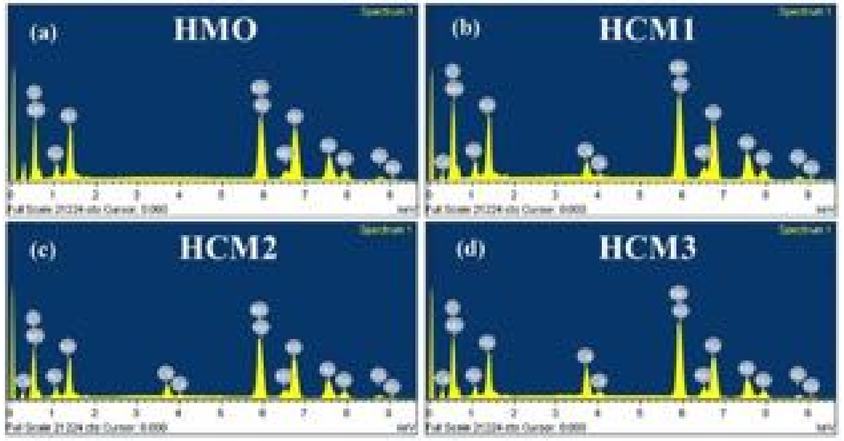
K. P. Shinde, E. J. Lee, M. Manawan, A. H. Lee, S.-Y. Park, Y. Jo, B. K. Koo & J. S. Park

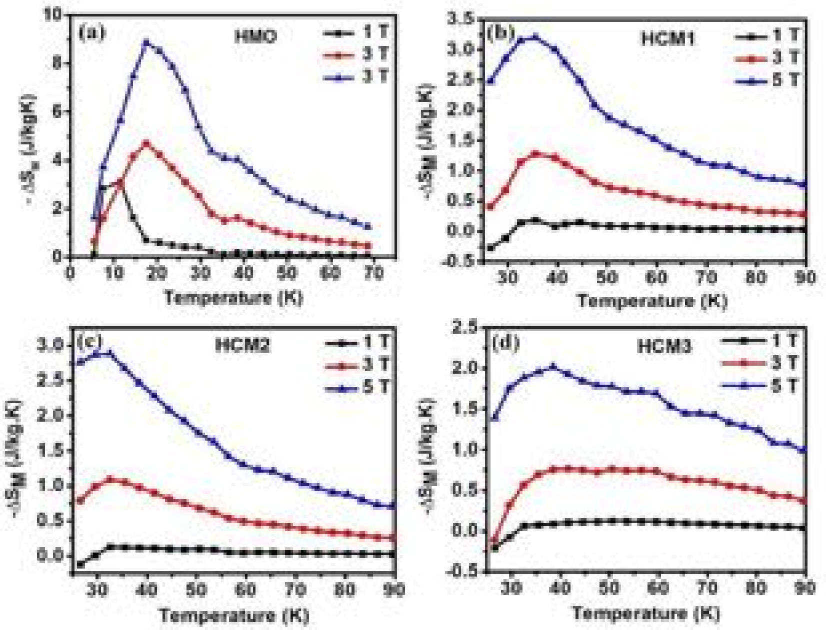
https://doi.org/10.1007/s00339-021-04991-y





		HMO	HCM1	HCM2	HCM3
Space Group		*P6₃cm*	*Pnma*	*Pnma*	*Pnma*
Cell Mass (g/mol)		1670.20(1)	987.99(2)	971.14(0)	921.64(1)
Cell Volume (Å^3)		373.30(2)	221.30(3)	223.01(2)	224.57(5)
Crystal Density (g/cm^3)		7.149(0)	7.399(0)	7.273(1)	6.880(0)
Crystal Size (nm)		105(2)	26.6(1)	38.5(7)	54.7(4)
Lattice Parameters:	a (Å)	6.1481(2)	5.6711(5)	5.7090(4)	5.7102(2)
	b (Å)	6.1481(2)	7.4107(5)	7.4153(4)	7.4419(2)
	c (Å)	11.4057(4)	5.2658(3)	5.2678(3)	5.2848(1)
Ho	X	–	0.42212	0.42161	0.43312
	Y	–	0.25000	0.25	0.25
	Z	–	0.01370	0.01744	0.01565
	Occupancy	–	0.8307	0.7991	0.6819
Ca	X	–	0.42498	0.42221	0.43140
	Y	–	0.25000	0.25	0.25
	Z	–	0.01439	0.1613	0.01551
	Occupancy	–	0.1693	0.2009	0.3181
Rexp		2.05	1.60	1.64	1.83
Rwp		7.23	4.45	4.57	5.75
GoF		3.53	2.78	2.79	3.14

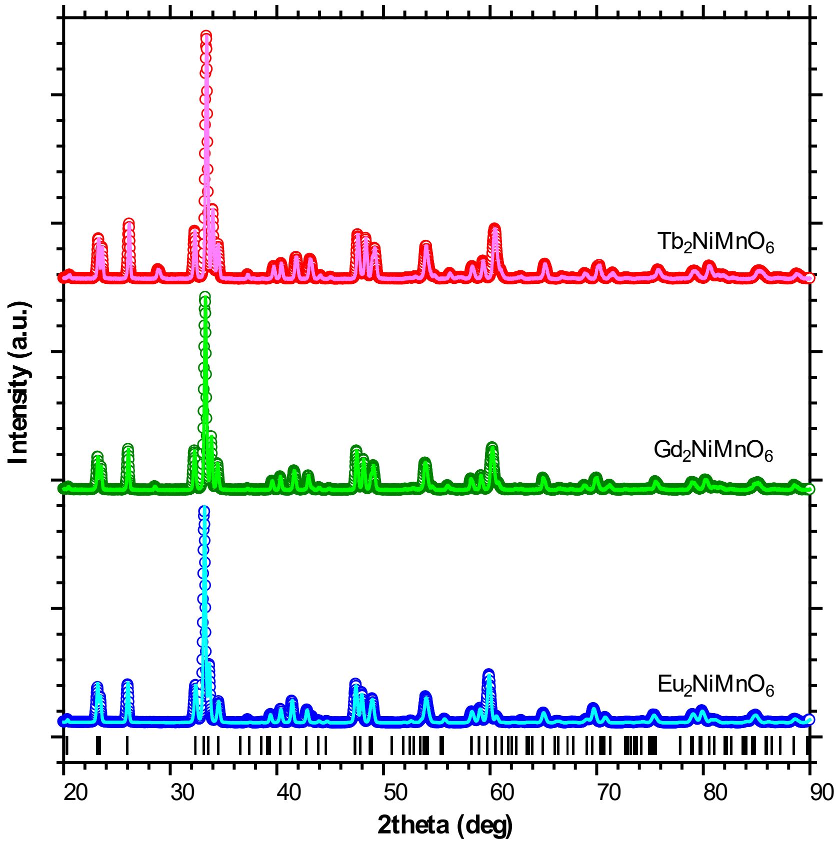


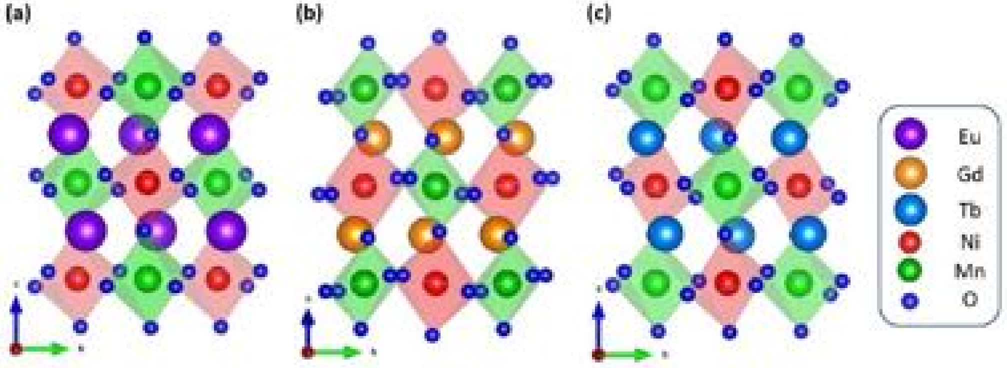


Structural, magnetic, and magnetocaloric properties of R_2NiMnO_6 (R = Eu, Gd, Tb)

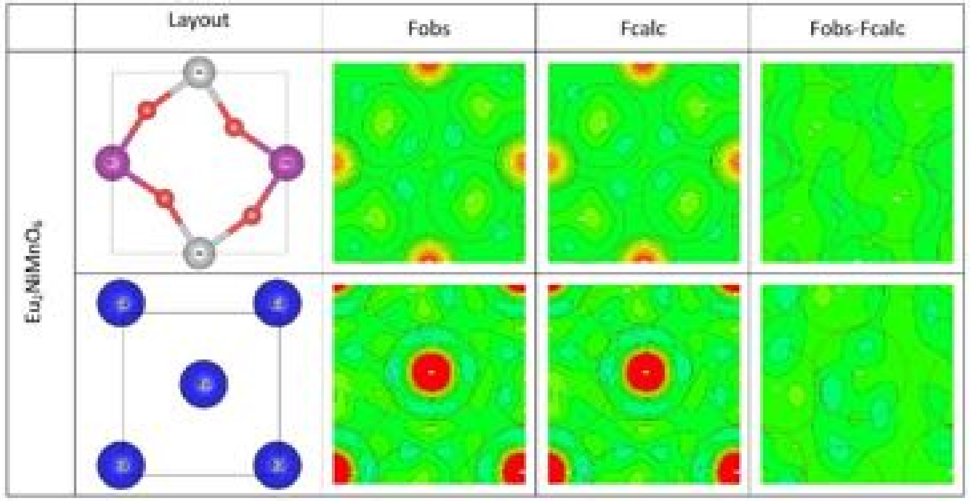
K. P. Shinde, E. J. Lee, M. Manawan, A. Lee, S.-Y. Park, Y. Jo, K. Ku, J. M. Kim & J. S. Park

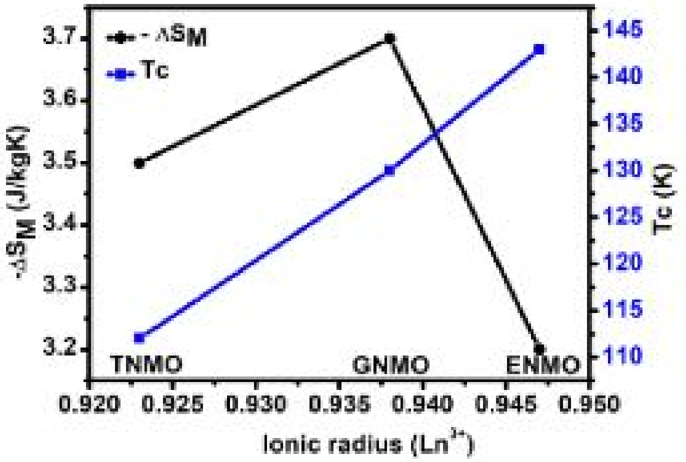
https://doi.org/10.1038/s41598-021-99755-2





	Bond length (Å)		
	Eu_2NiMnO_6	Gd_2NiMnO_6	Tb_2NiMnO_6
Ni - O1	1.9961(1)	2.0781(2)	1.8256(1)
Ni - O2	2.0051(2)	1.6247(1)	1.7962(0)
Ni - O3	1.9391(0)	2.0505(2)	1.9416(4)
Mn - O1	1.9481(2)	1.9120(1)	2.0444(2)
Mn - O2	1.9611(2)	2.2756(3)	2.2468(0)
Mn - O3	1.9731(0)	1.8169(1)	1.9057(2)
	Bond Angle (°)		
	Eu_2NiMnO_6	Gd_2NiMnO_6	Tb_2NiMnO_6
Ni - O1 - Mn	153.07(2)	147.30(4)	161.84(2)
Ni - O2 - Mn	150.99(1)	157.80(1)	141.65(3)
Ni - O3 - Mn	151.47(1)	155.12(3)	155.95(1)

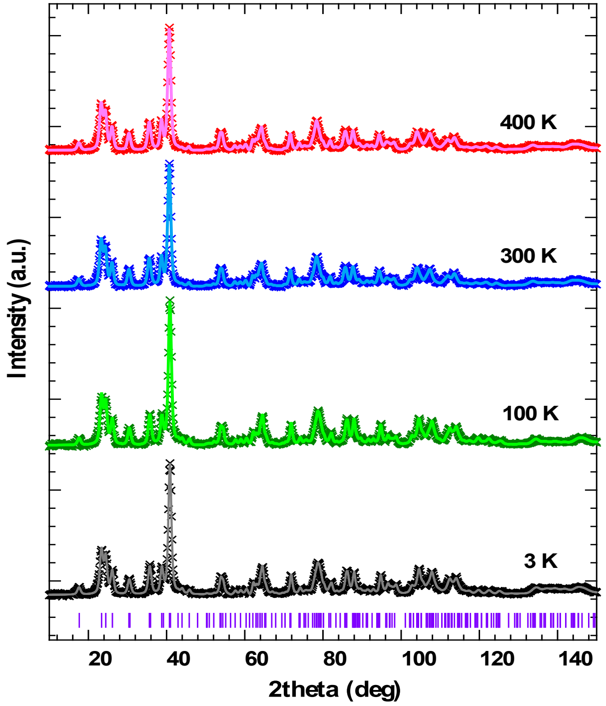


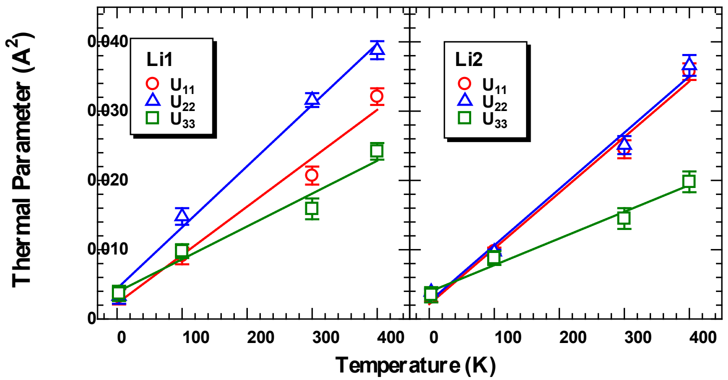


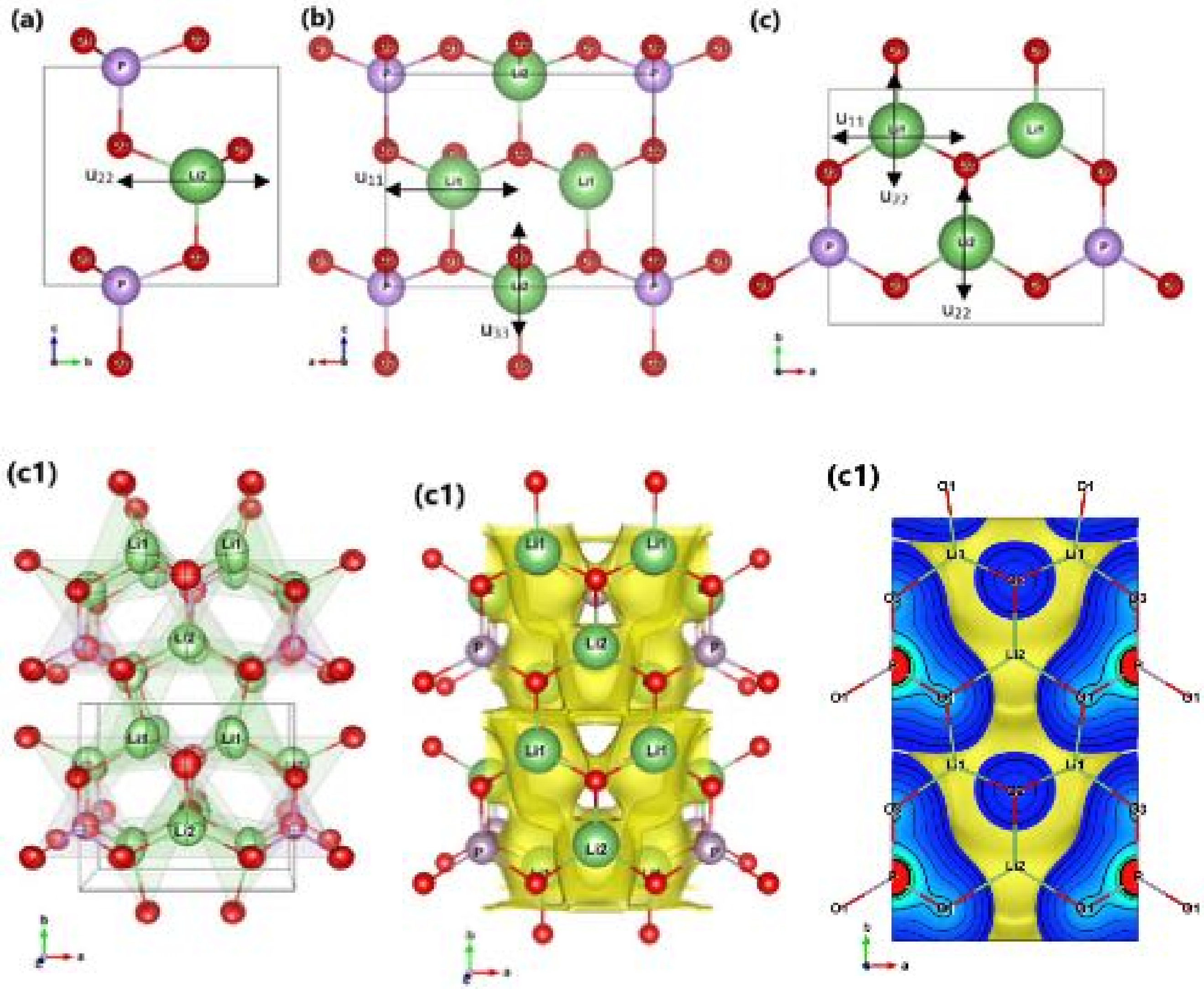
Visualizing lithium ions in the crystal structure of Li_3PO_4 by *in situ* neutron diffraction

M. Manawan, E. Kartini and M. Avdeev

https://doi.org/10.1107/S1600576721008700



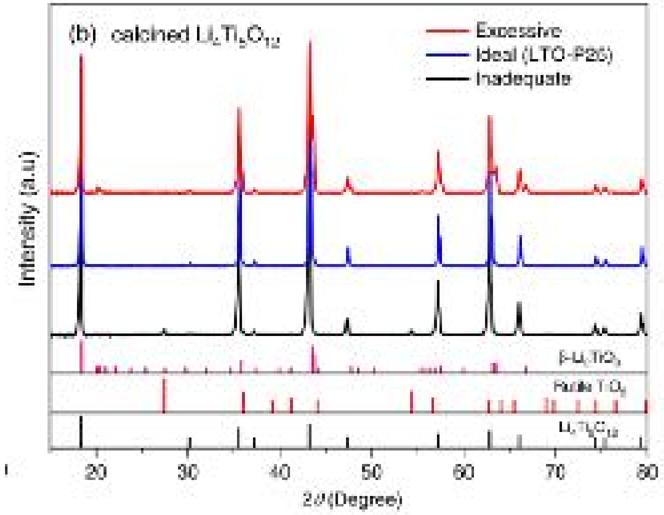


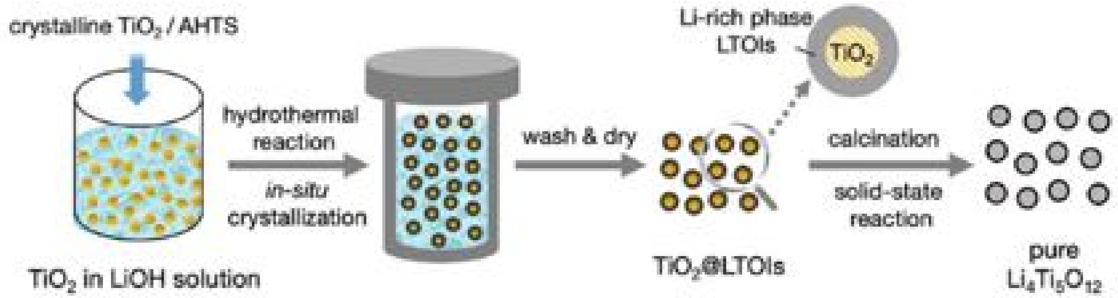


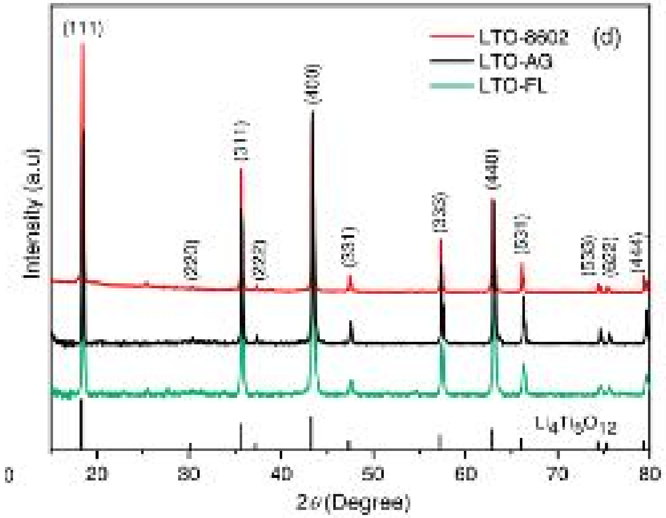
Unveiling the Formation Mechanism and Phase Purity Control of Nanostructured $Li_4Ti_5O_{12}$ via a Hydrothermal Process

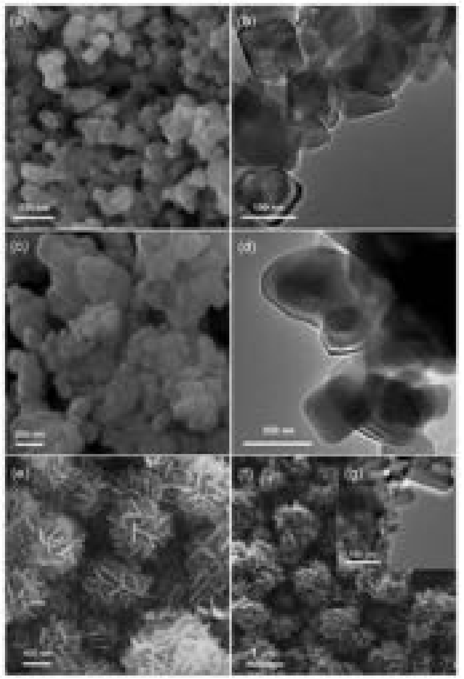
Kaiming Li, Xundong Dai, Maykel Manawan, Qing Wang*, and Jia Hong Pan*

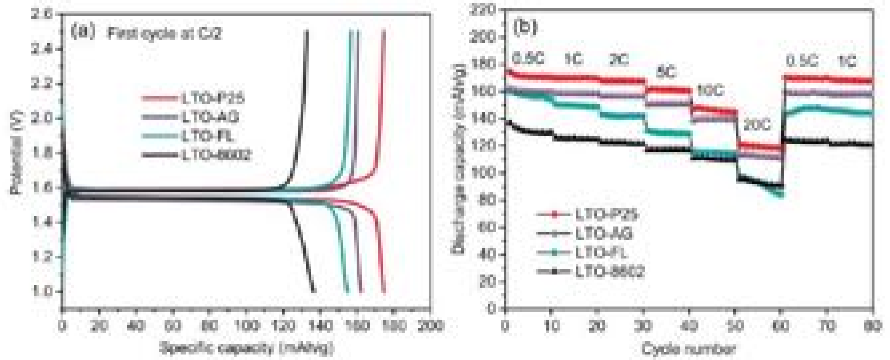
https://doi/10.1021/acs.cgd.1c00727

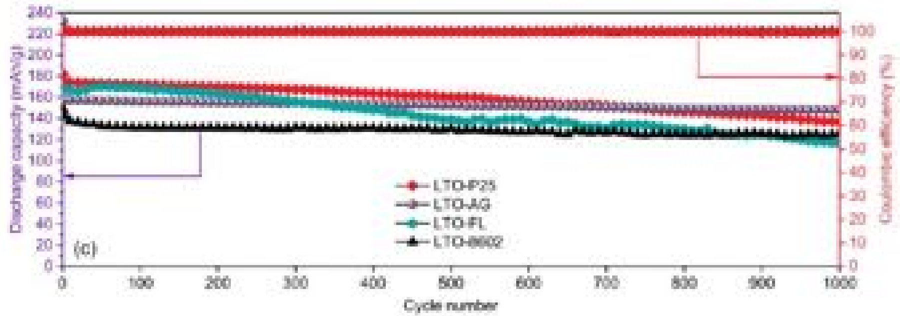












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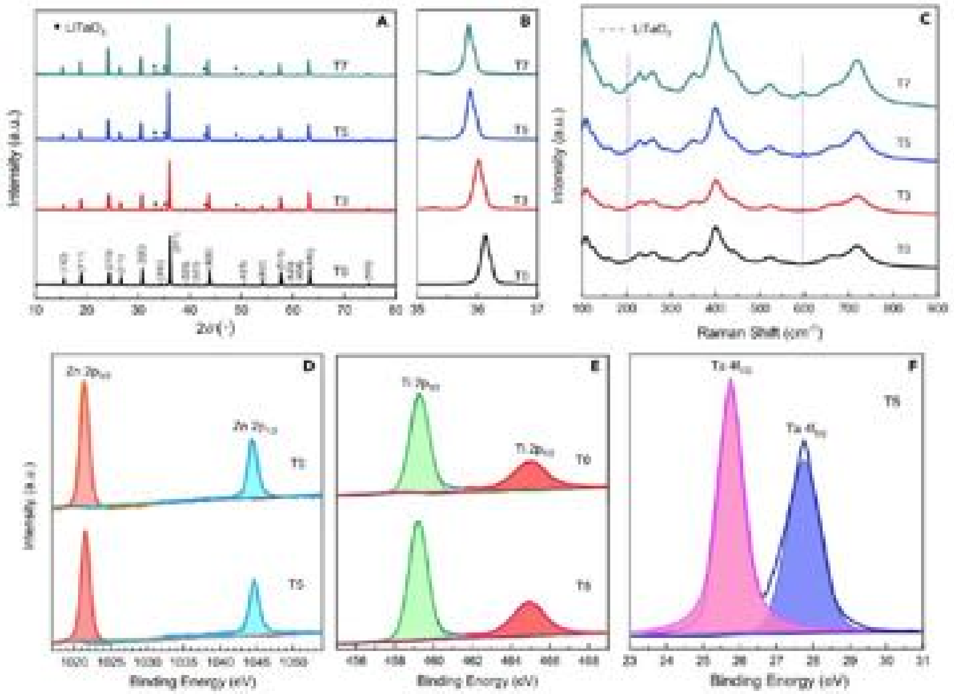
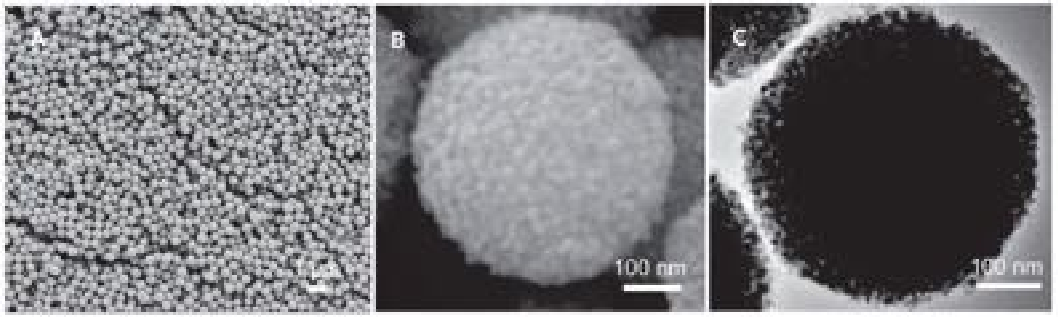
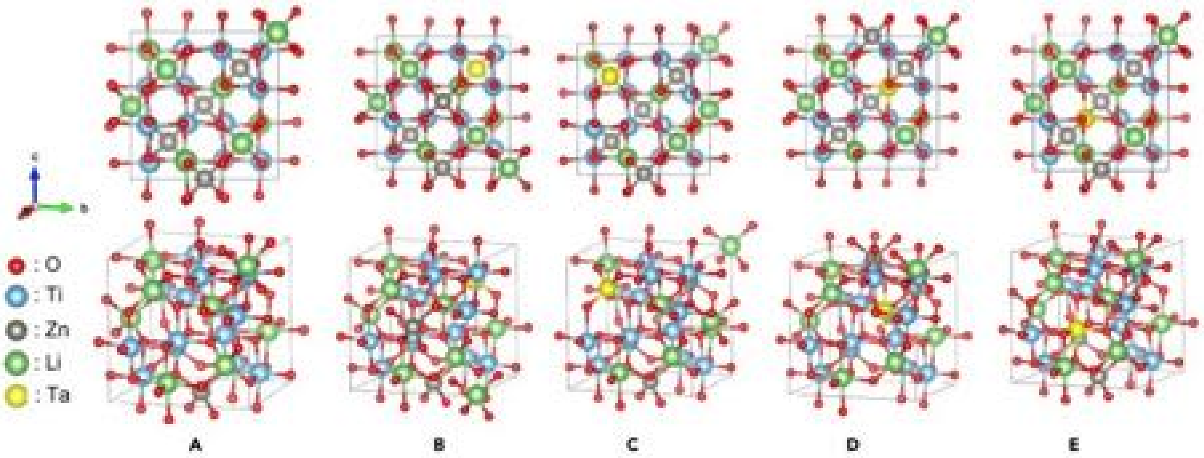
Solid-state self-template synthesis of Ta-doped $Li_2ZnTi_3O_8$ spheres for efficient and durable lithium storage

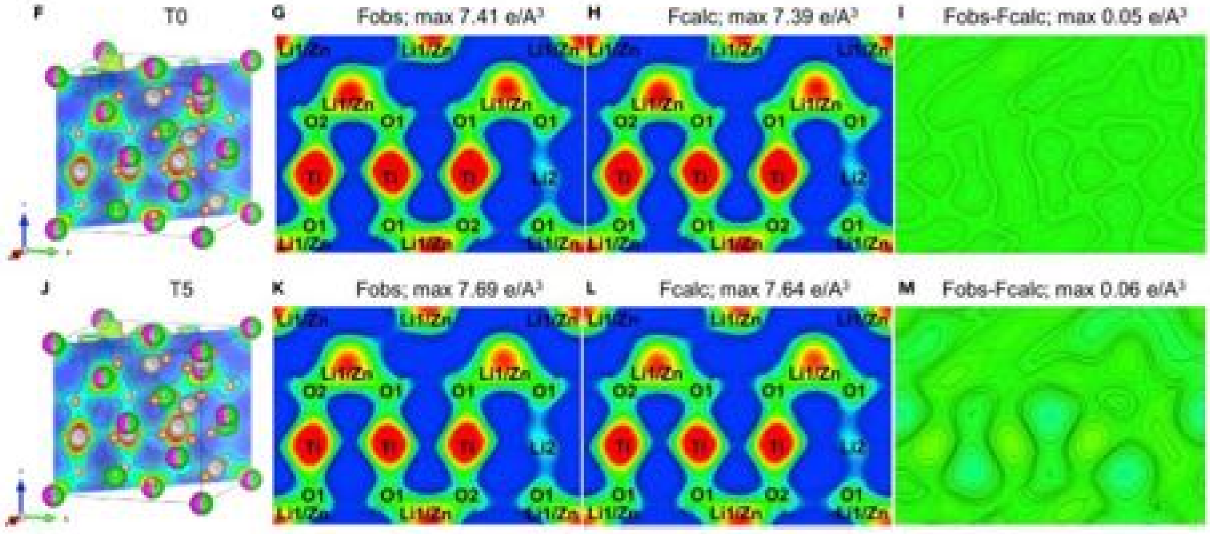
Dongwei Ma, Jiahui Li, Jing Yang, Chengfu Yang, Maykel Manawan, Yongri Liang, Ting Feng, Yong-Wei Zhang, and Jia Hong Pan*

https://doi.org/10.1016/j.isci.2021.102991





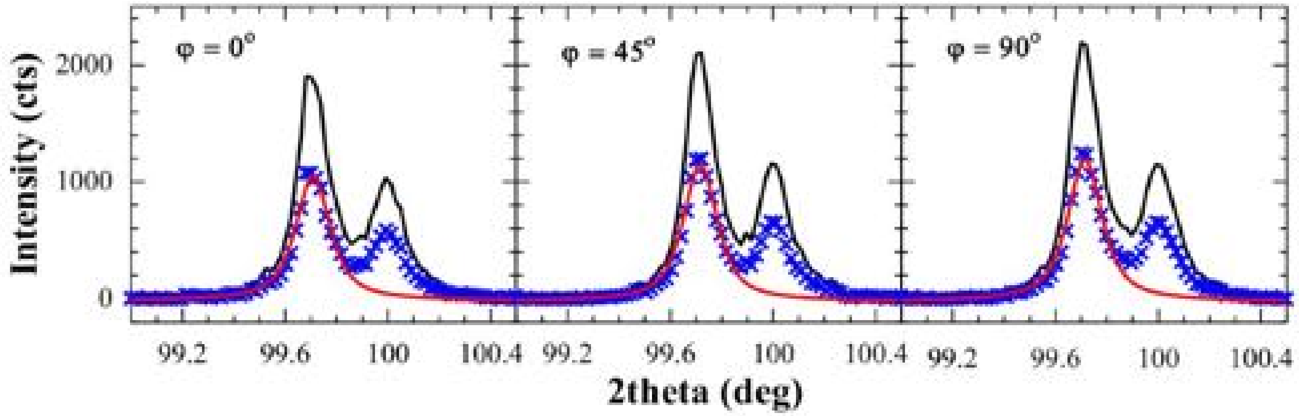


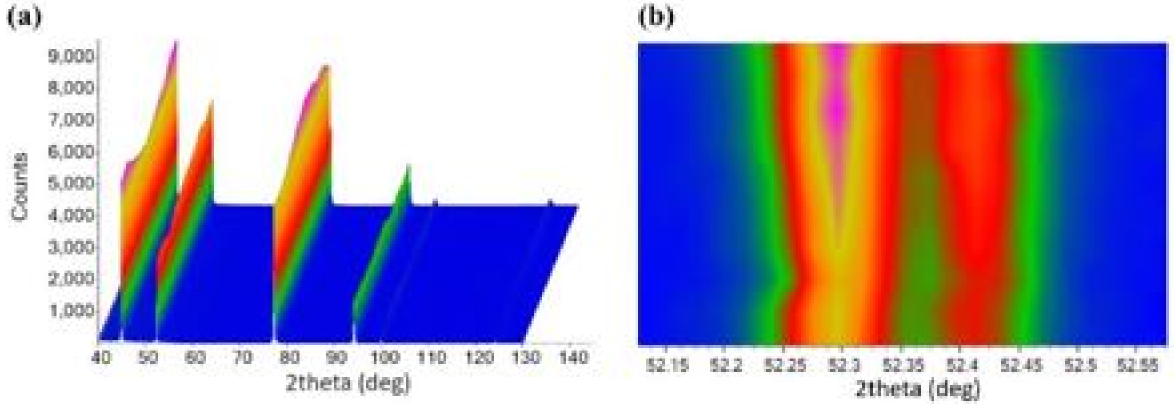


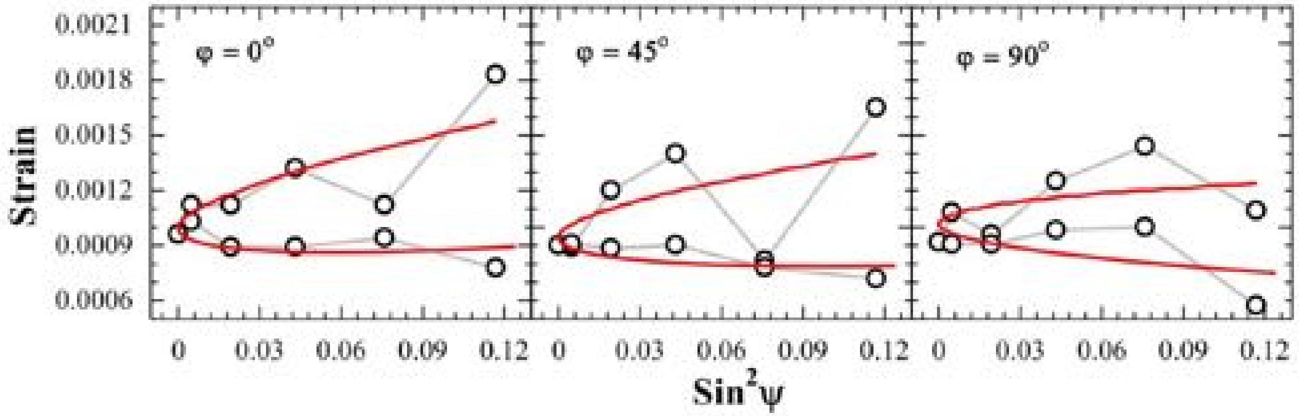
XRD Residual Stress and Texture Analysis on 6082T Aluminum Alloy

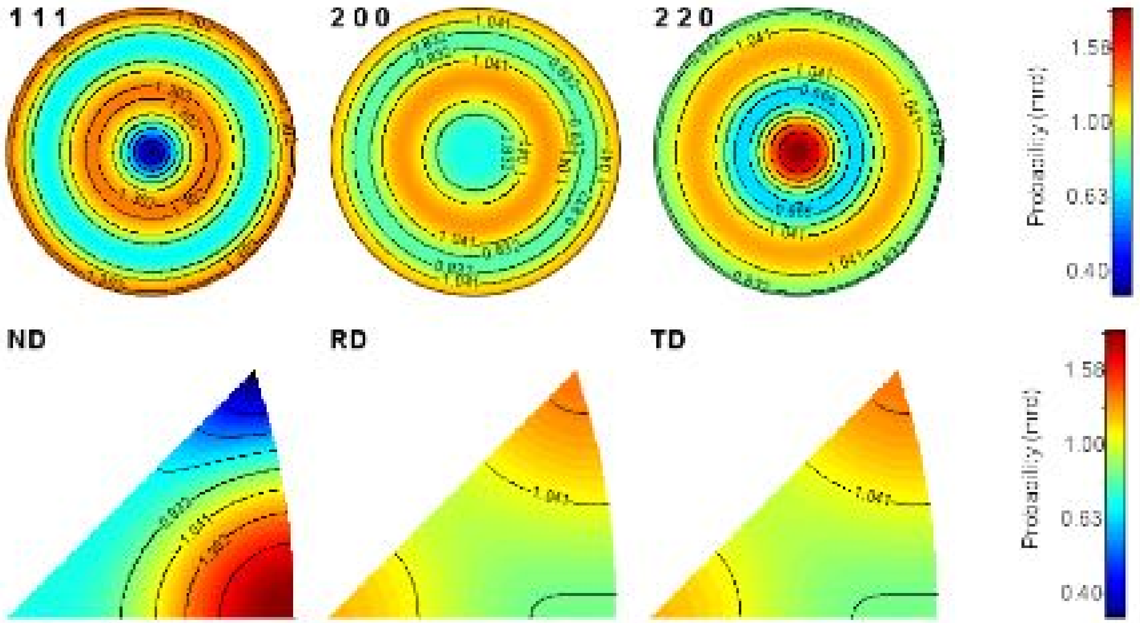
Maykel Manawan[1,a*], Sovian Aritonang[1,b], Masayu Elita[1,c],
Antonius Suban Hali[2,d], Nono Darsono[3,e], Toto Sudiro[4,f],
Permono Adi Putro[5,g], Risdiana[6,h]

https://doi.org/10.4028/www.scientific.net/MSF.1028.409







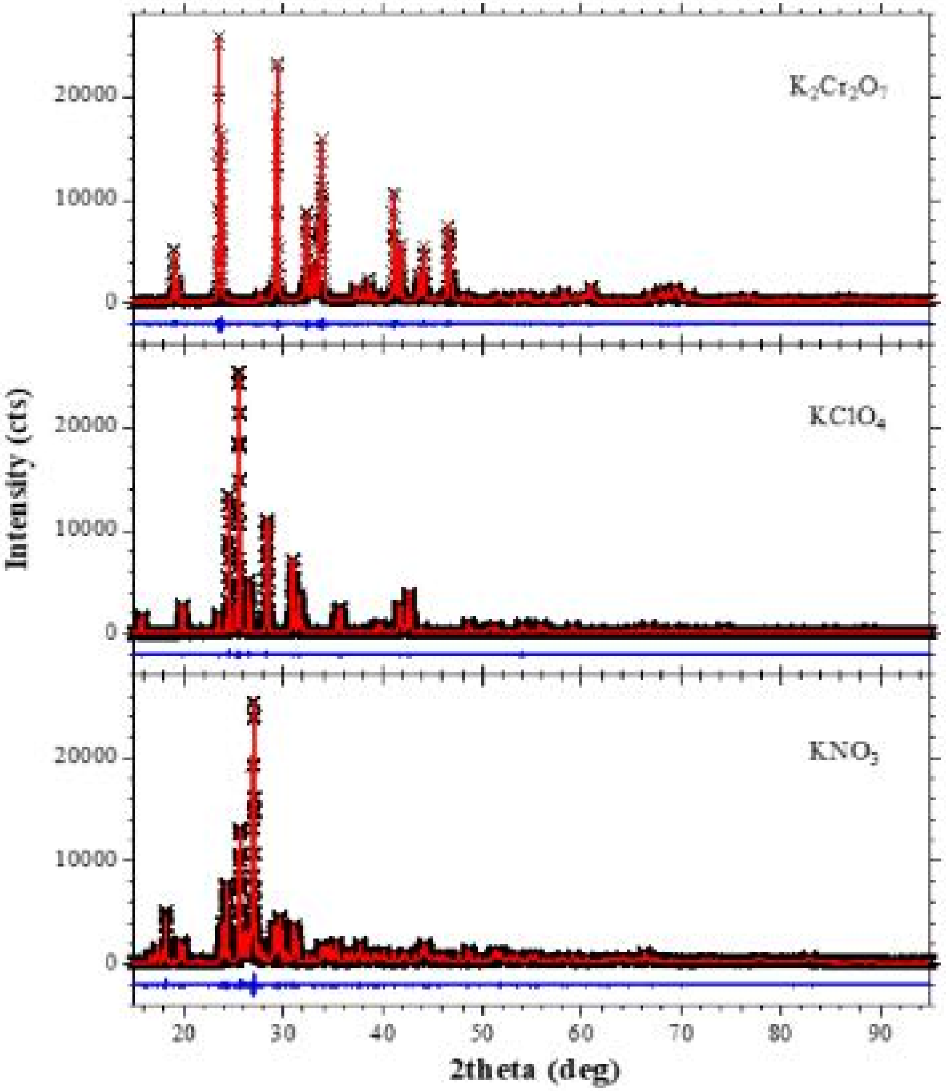


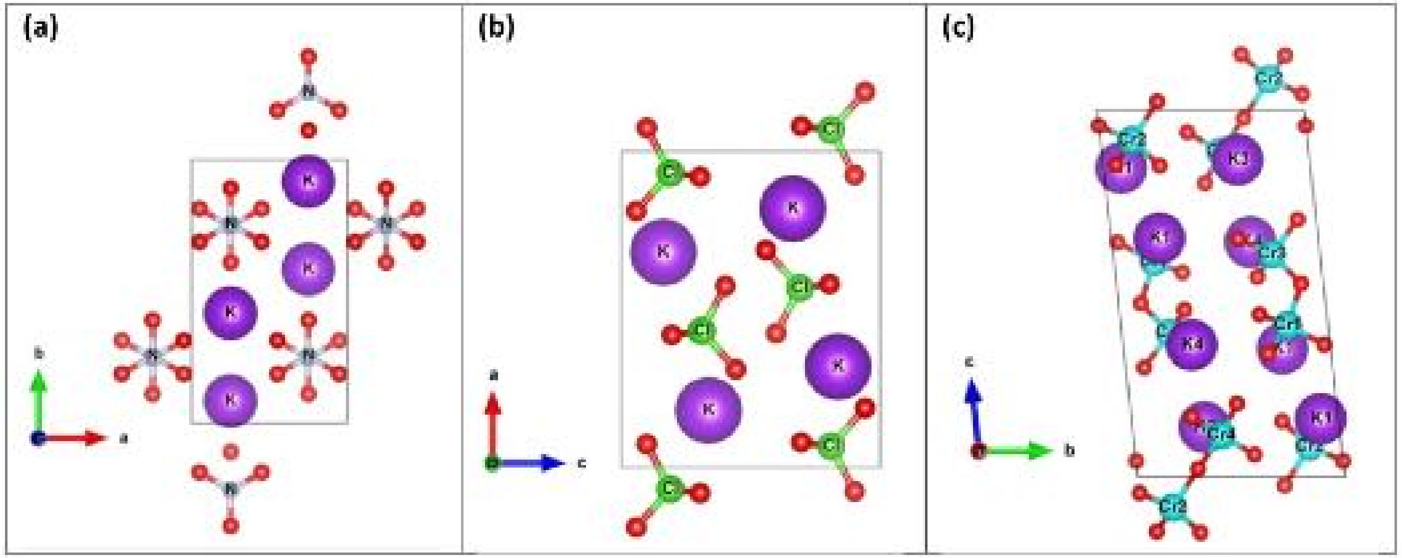
φ (°)	Normal Stress (MPa)	Shear Stress (MPa)
0	109.3 ± 21.2	-28.7 ± 6.1
45	68.9 ± 19.4	-25.6 ± 8.5
90	-9.9 ± 9.4	-20 ± 8.5

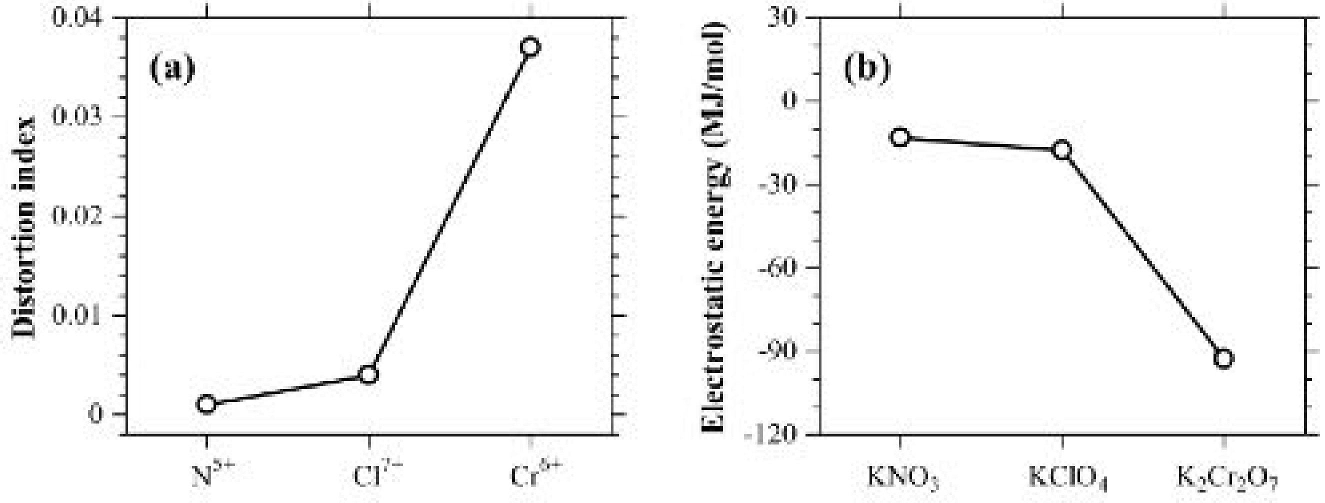
Crystal Characterization of Anionic Salt Compounds as Composite of Solid Propellant Oxidizing Agent

Sovian Aritonang[1,a], Maykel Manawan[1,b*], Mas Ayu E. H[1,c], Timbul Siahaan[1], Shofi Muktiana S.[1], Hanung Bayu Setiawan[1], Sih Wuri Andayani[2], Gaos Abdul Karim[2], Alfiz Muhammad Qizwini[2], Otong Nurhilal[3], Togar Saragi[3], Risdiana[3]

https://doi.org/10.4028/www.scientific.net/MSF.1028.269







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Original research

Solid electrolyte composite $Li_4P_2O_7$–Li_3PO_4 for lithium ion battery

Evvy Kartini[a,*], Valentina Yapriadi[b], Heri Jodi[a], Maykel Manawan[c], Cipta Panghegar[d], Wahyudianingsih[a]

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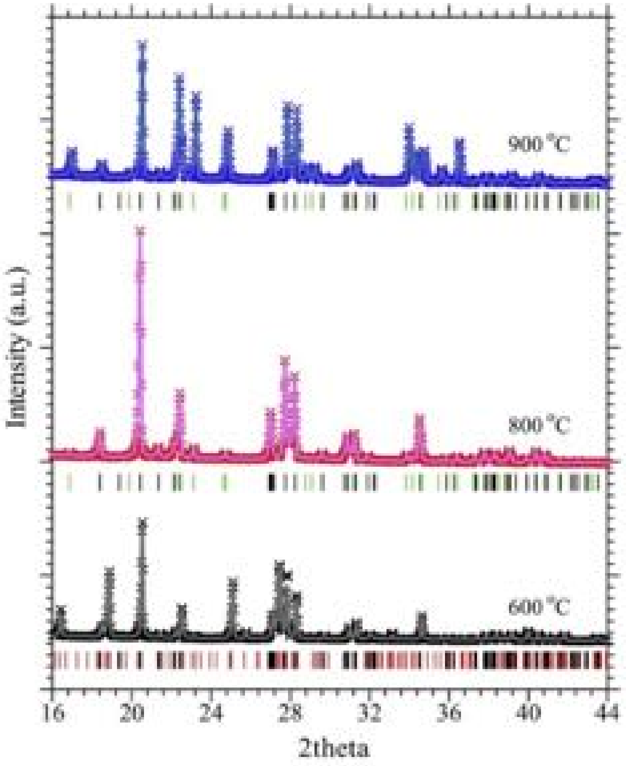
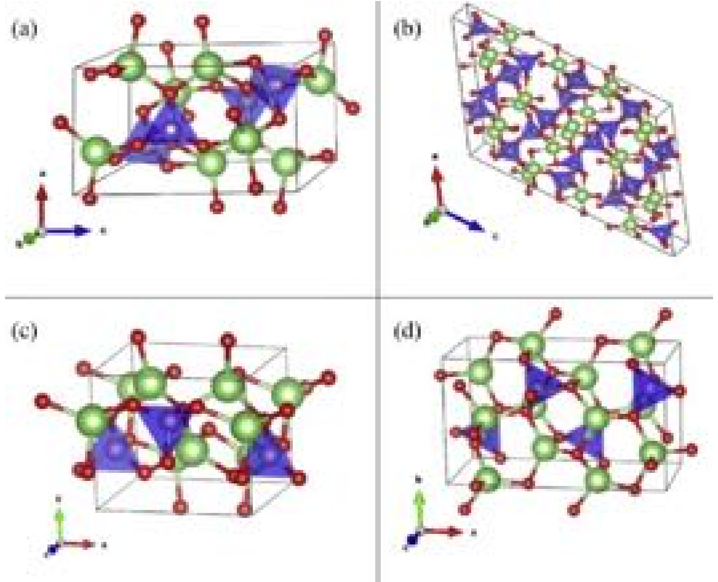
Table 2
Bond distance calculation of quenched samples at 600 °C, 800 °C and 900 °C.

600 °C						800 °C						900 °C					
Li4P2O7			LiPO3			Li4P2O7			Li3PO4			Li4P2O7			Li3PO4		
Bond		Distance	Bond		Distance	Bond		Distance	Bond		Distance	Bond		Distance	Bond		Distance
Li1	- O6	1.87	Li1	- O3	1.912	Li1	- O6	1.869	Li1	- O1	1.947	Li1	- O6	1.869	Li1	- O1	1.946
	- O1	1.95		- O14	1.967		- O1	1.95		- O3	1.994		- O1	1.949		- O3	1.994
	- O4	1.976		- O1	1.978		- O4	1.974		- O3	1.994		- O4	1.975		- O3	1.994
	- O4	1.976		- O7	1.989		- O4	1.974		- O2	2.047		- O4	1.975		- O2	2.047
Li2	- O6	1.913	Li2	- O3	1.965	Li2	- O6	1.913	Li2	- O1	1.93	Li2	- O6	1.913	Li2	- O1	1.93
	- O2	1.94		- O8	1.967		- O2	1.94		- O2	1.944		- O2	1.94		- O2	1.944
	- O5	2.009		- O11	1.996		- O5	2.008		- O3	1.95		- O5	2.009		- O3	1.949
	- O1	2.01		- O13	1.987		- O1	2.008		- O3	1.992		- O1	2.01		- O3	1.992
Li3	- O3	1.865	Li3	- O14	1.877	Li3	- O3	1.864				Li3	- O3	1.865			
	- O2	1.874		- O10	1.898		- O2	1.872					- O2	1.873			
	- O7	2.085		- O10	1.997		- O7	2.084					- O7	2.085			
	- O5	2.105		- O1	1.997		- O5	2.105					- O5	2.104			
Li4	- O5	1.926	Li4	- O6	1.907	Li4	- O5	1.926				Li4	- O3	1.926			
	- O3	1.926		- O3	1.934		- O3	1.926					- O5	1.926			
	- O4	2.008		- O6	1.935		- O4	2.008					- O4	2.008			
	- O1	2.011		- O8	1.996		- O1	2.01					- O1	2.011			
			Li5	- O7	1.914												
				- O7	1.914												
				- O2	1.973												
				- O2	1.973												





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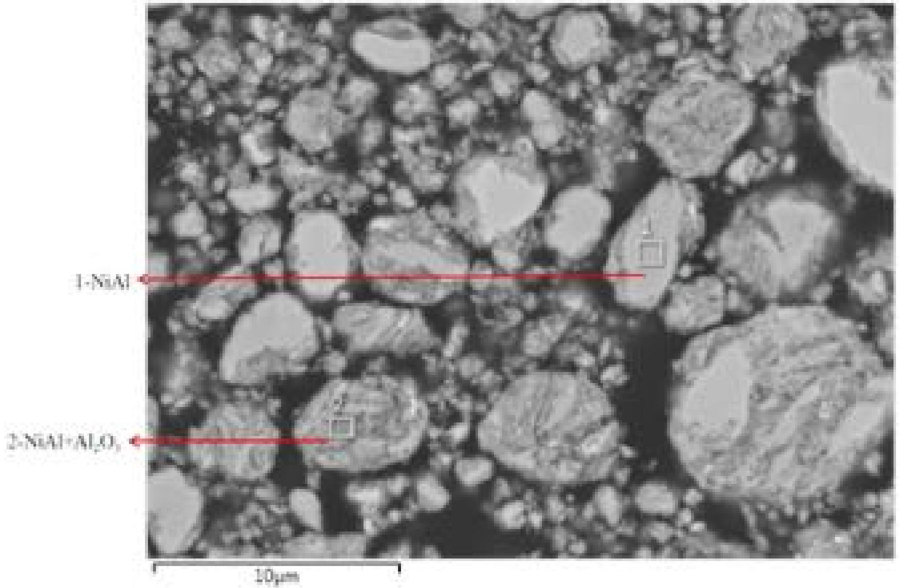

In-situ synthesis and characterization of nano-structured NiAl-Al_2O_3 composite during high energy ball milling

Maryam Beyhaghi [a,*], Jalil Vahdati Khaki [b], Maykel Manawan [c,d], Alireza Kiani-Rashid [b], Mehrdad Kashefi [b], Stefan Jonsson [e]

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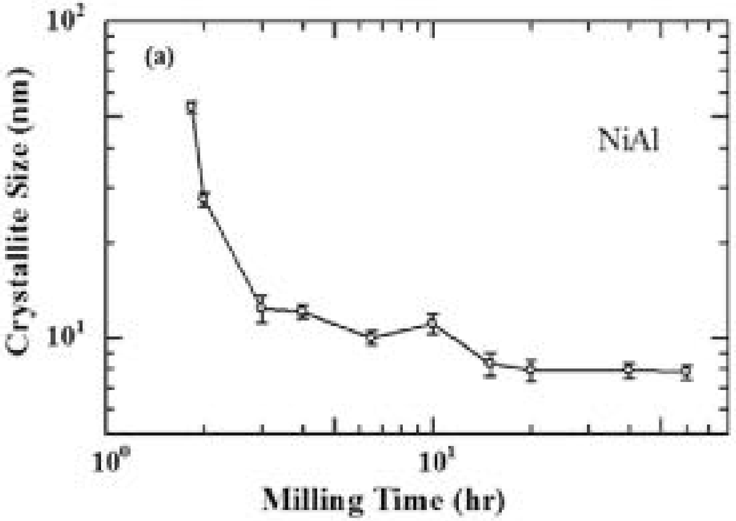

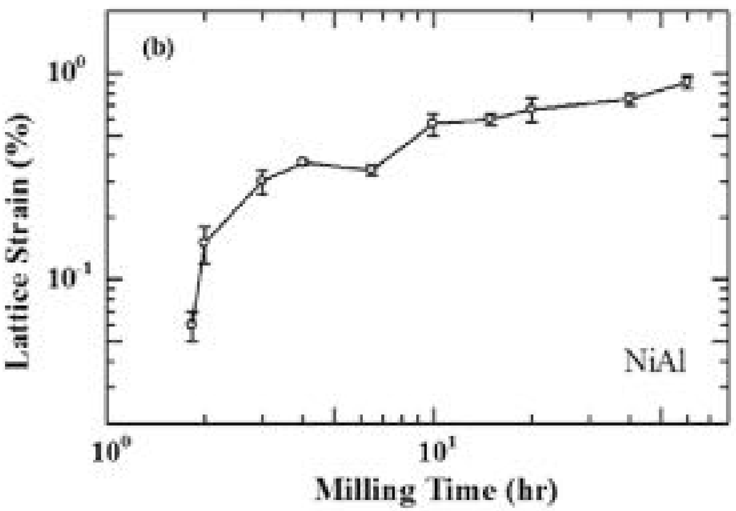

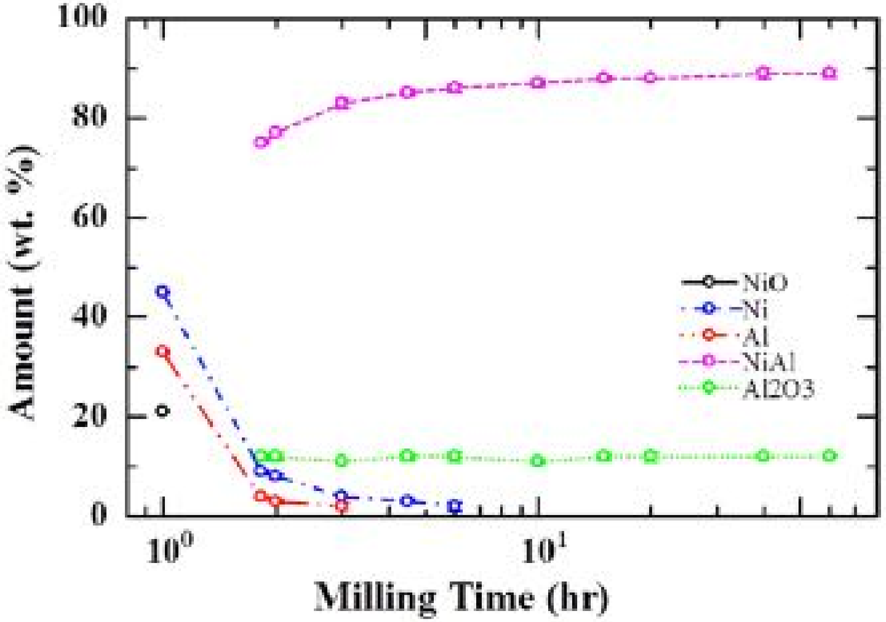

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Anatase and rutile in evonik aeroxide P25: Heterojunctioned or individual nanoparticles?

Xiongzhen Jiang[a,1], Maykel Manawan[b,1], Ting Feng[c], Ruifeng Qian[a], Ting Zhao[a], Guanda Zhou[a], Fantai Kong[d,*], Qing Wang[e,*], Songyuan Dai[a], Jia Hong Pan[a,*]

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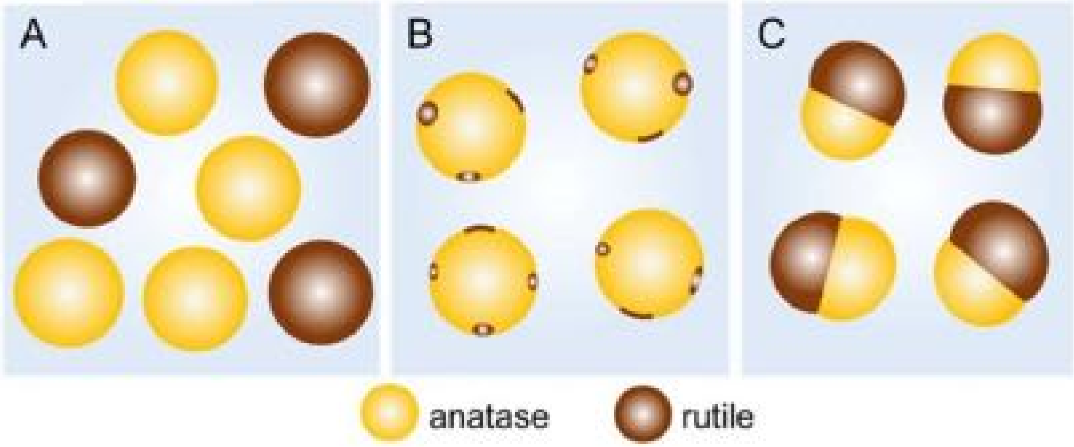
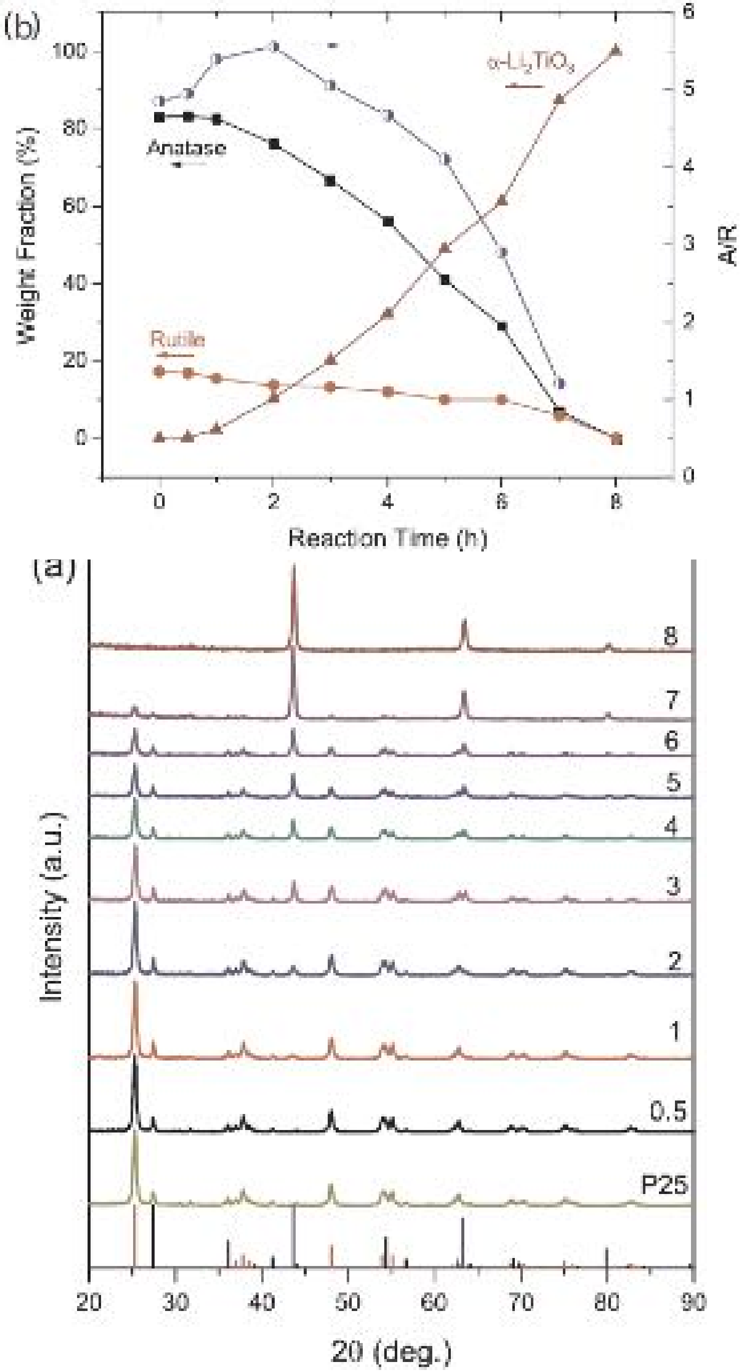
[e] Department of Materials Science and Engineering, Faculty of Engineering, National University of Singapore, Singapore 117576, Singapore





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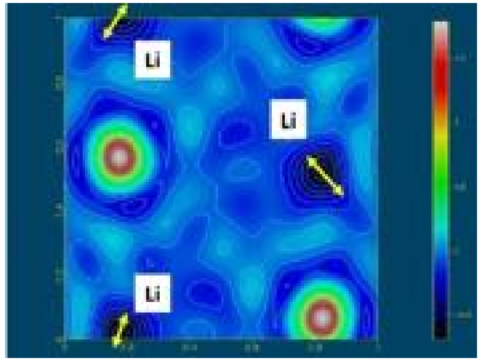

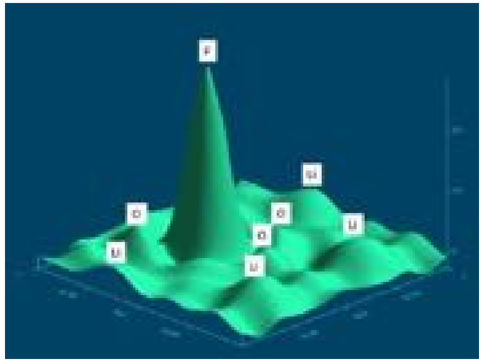

Neutron diffraction study on Li_3PO_4 solid electrolyte for lithium ion battery

Evvy Kartini [a,*], Maykel Manawan [b,c], Malcolm F. Collins [d], Maxim Avdeev [e]

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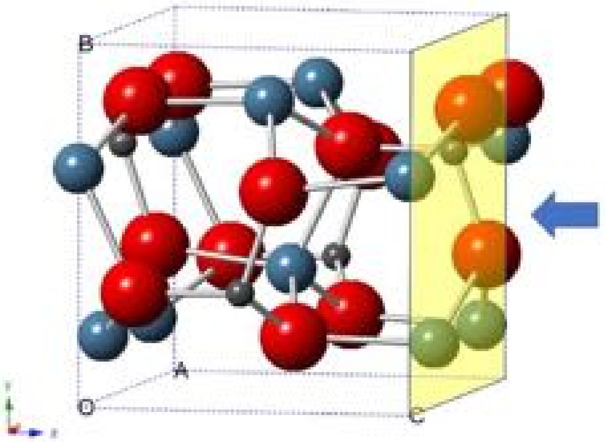
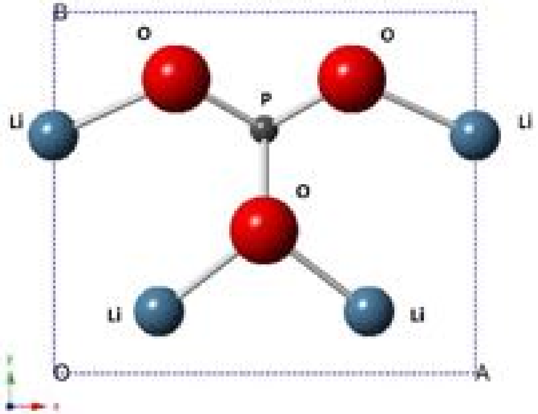
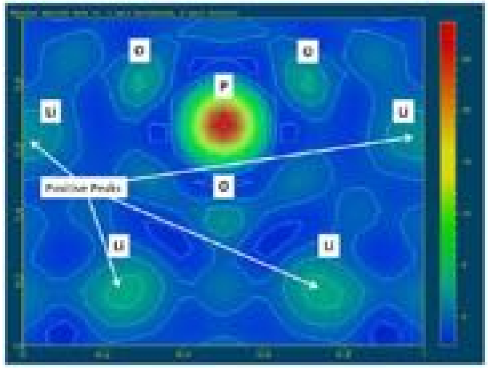
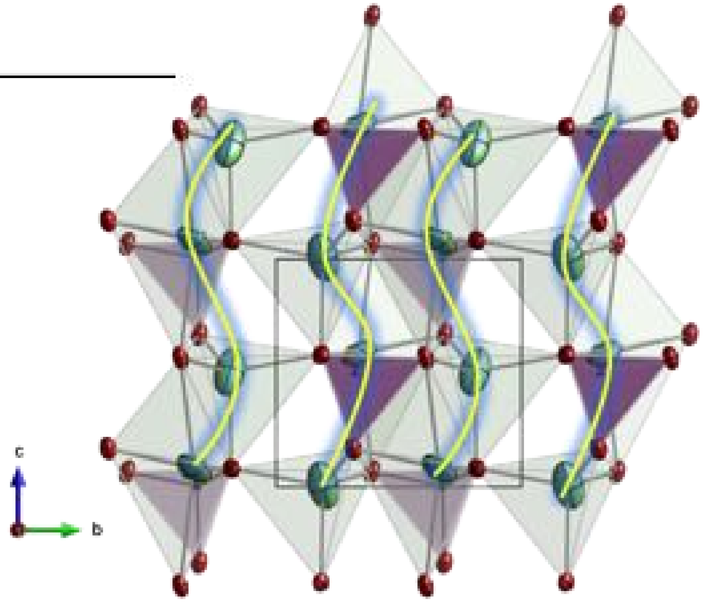
[d] Department of Physics and Astronomy, McMaster University, 1280 MainStreet West, Hamilton, ON L8S4L8, Canada
[e] Australian Centre for Neutron Scattering (ACNS), Australian Nuclear Science and Technology (ANSTO), Locked Bag 200, Kirrawee DC, NSW, 223, Australia









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Crystallite size determination of barium hexaferrite nanoparticles using WH-plot and WPPM

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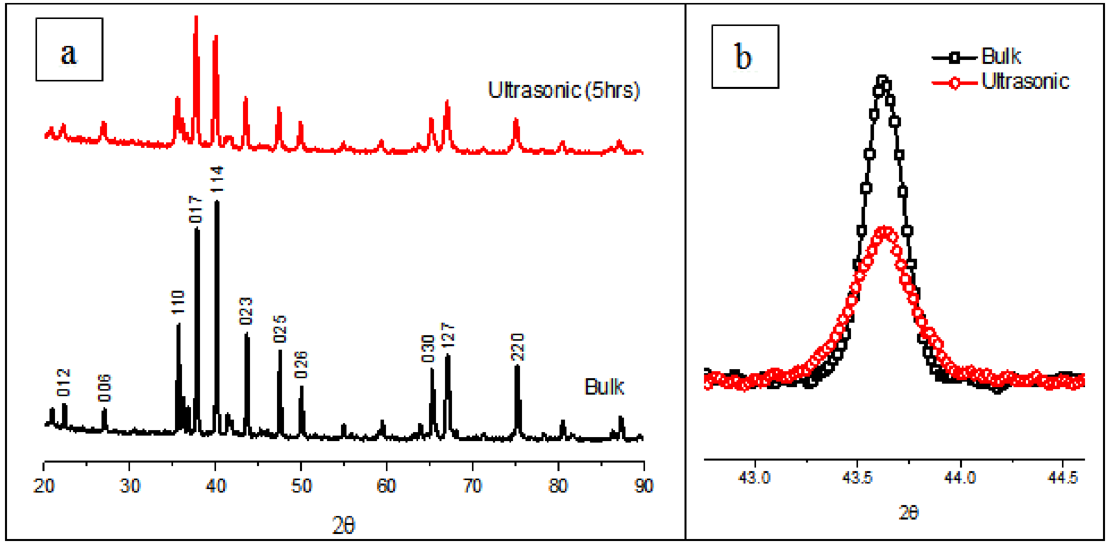
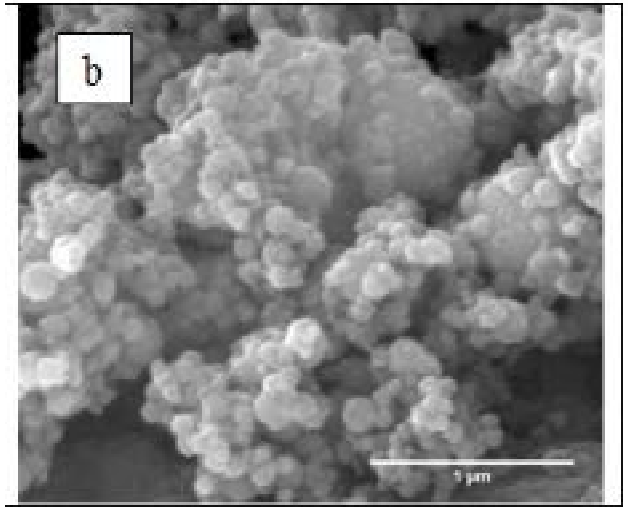
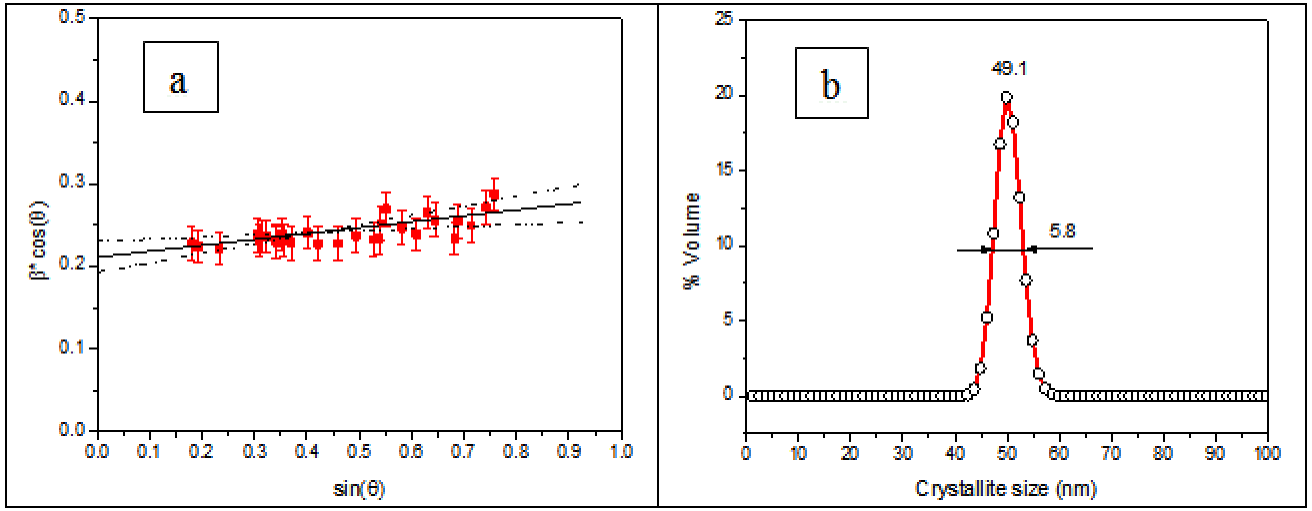






Future Project - MEM, BVS, BVEL, PDF

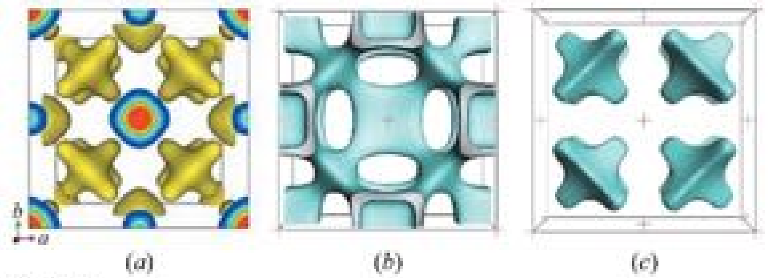


Figure 6
CuI at 773 K. (*a*) Experimental Cu-ion distribution determined by maximum entropy analysis of neutron diffraction data [adapted with permission from Adipranoto *et al.* (2009)]. (*b*) and (*c*) BVS map (isovalue ±0.1 v.u.) and BVEL (isovalue −1.4 eV, E_{min} −1.5 eV) data calculated for ICSD 163859 (Adipranoto *et al.*, 2009).

Figure 2
$(Pr_{0.9}La_{0.1})_2(Ni_{0.74}Cu_{0.21}Ga_{0.05})O_{4+\delta}$ (PLNCG) at 1288.75 K. (*a*) Experimental oxygen-ion distribution determined by maximum entropy analysis of neutron diffraction data [adapted with permission from Yashima *et al.* (2010)]; *A*, O2/O3 and the plus symbol denote (Pr,La) atoms, O2/O3 oxygen sites and the diffusion bottleneck, respectively. (*b*) and (*c*) BVS map [isovalue +0.2 valence units (v.u.)] and BVEL (isovalue −3.0 eV, E_{min} −6.6 eV) data calculated for ICSD 248032

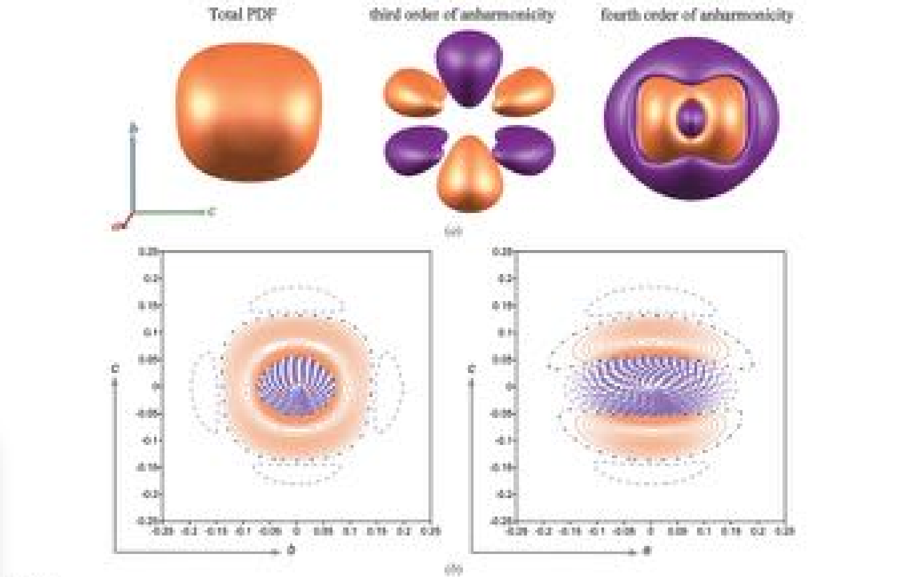


Figure 2
The orthorhombic $KNbO_3$ crystal: probability density distribution function for displacements of Nb from the equilibrium position. (a) 3D image of PDF and its components; isosurfaces are ±300 Å^{-3}; (b) 2D image of the anharmonic part of PDF: the step of isolines is 1000 Å^{-3}. All PDFs are depicted within a volume of 0.3 × 0.3 × 0.3 Å. Positive PDF values are marked in orange and negative ones are marked in violet. The directions of the unit cell axes are also indicated.

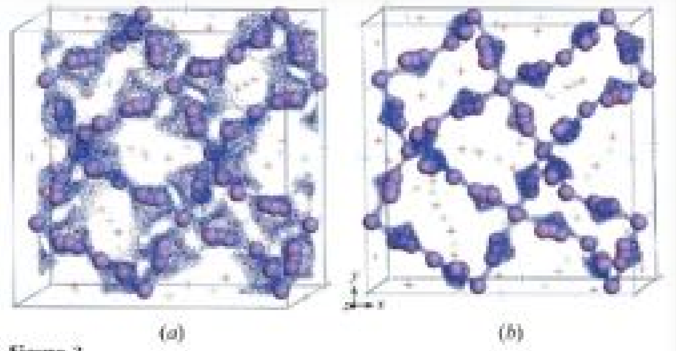


Figure 3
$La_3Li_5O_{12}Ta_2$. Lithium-ion distribution modelled in the diffraction data analysis by a large number of underoccupied atomic sites (spheres) overlaid with (*a*) a BVS map (isovalue ±0.1 v.u.) and (*b*) BVEL (isovalue −3.2 eV, E_{min} −4.0 eV) data calculated for ICSD 154400 (Cussen, 2006). The shaded areas represent regions of low BV mismatch and BVEL values.

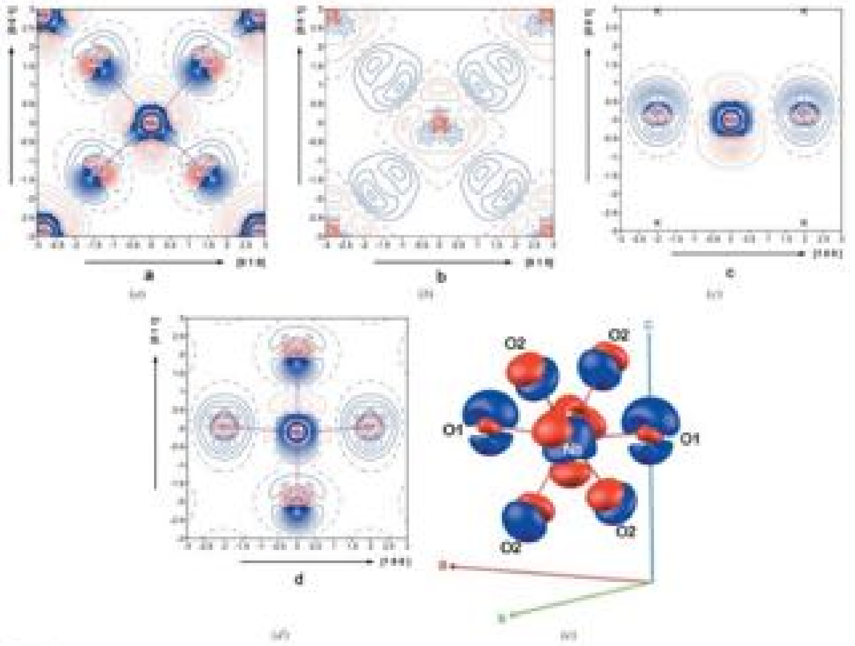


Figure 1
Deformation electron density in the NbO_6 octahedron along the Nb – O1 and Nb – O2 lines: (a)(c)(d) are experimental maps and (b) is a theoretical map. Areas of 2D electron-density accumulation relative to a procrystal are shown in blue. The step of the isolines is 0.1 e Å^{-3}. (e) The 3D image of the experimental deformation density consists of two surfaces: red (−0.3) and blue (0.3 e Å^{-3}).

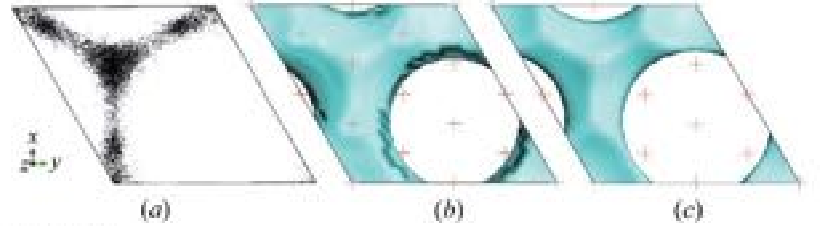
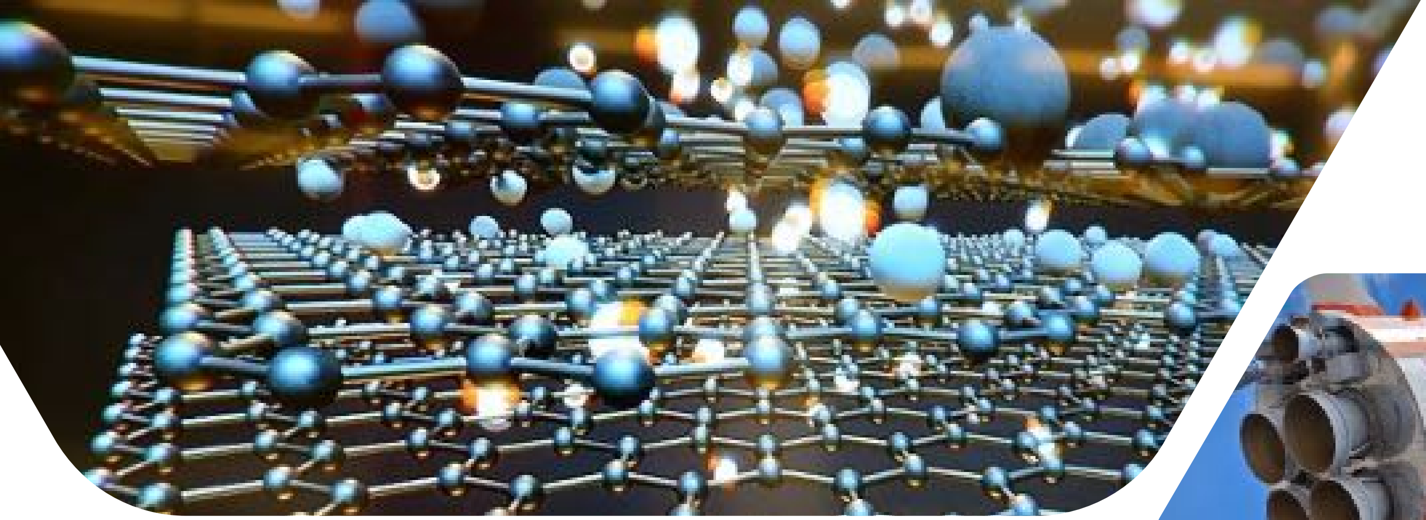


Figure 5
Na^+ β-alumina. (*a*) $Na_{1+x}Al_{11}O_{17+x/2}$ for $x \simeq 0.22$. *Molecular Dynamics* simulation of Na^+ ion distribution at 700 K [adapted with permission from Thomas (1992)]. (*b*) and (*c*) BVS map (isovalue ±0.15 v.u.) and BVEL (isovalue −1.0 eV, E_{min} −2.3 eV) data calculated for ICSD 60635 (Yamaguchi & Suzuki, 1968).



END
Session 4

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Maykel Manawan

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Introduction

Maykel Manawan currently works as a lecturer and researcher at Indonesia Defense University. An active member of the International Center for Diffraction Data, ICDD (Education sub-committee). Principal Investigator of the National Li-ion Battery Program (Battery Research Institute-Consortium). Member of Material Research Society Indonesia, MRS-INA. Member of Indonesian Magnetic Society. Member of Indonesia Neutron Scattering Society.

Skills and Expertise

Crystallography · Powder Diffraction · Neutron Diffraction · Protein X-ray Crystallography · Lithium Ion Batteries · Superconductivity and Superconduct... · Magnetic Materials and Magnetism · Density Functional Theory · Material Characterization · Ancient History

TERIMA KASIH