Types of powder diffraction and high pressure experiments

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solid state matter = periodical arrangement of atoms
(crystallographic structure -> notion of crystal)

~100 different kinds of atoms
in the nature,
but
infinite ways to arrange these atoms

→ physical and chemical properties of solid

↓

precise knowledge of the crystallographic structure
of a material to understand these properties.

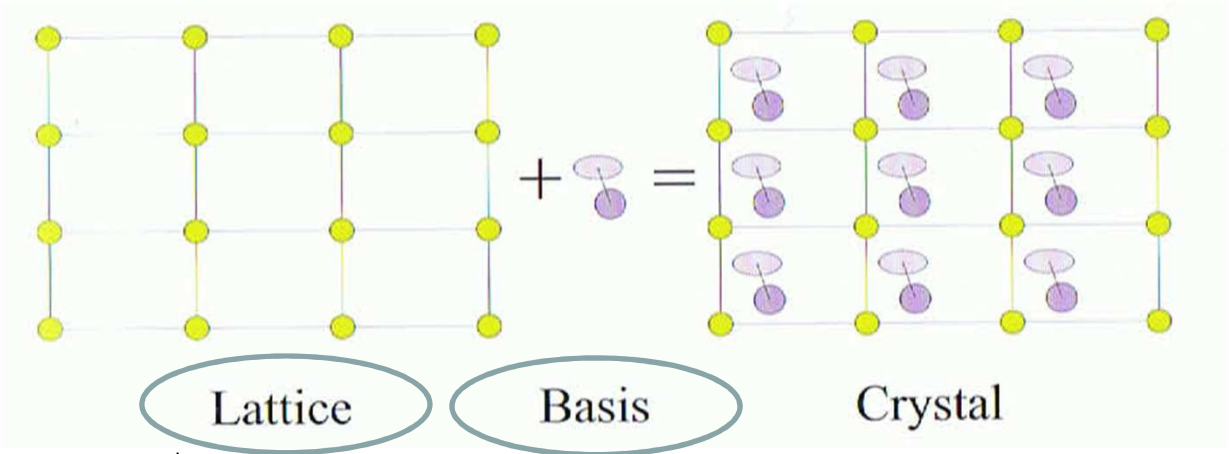
↓

The elastic scattering of a radiation beam (diffraction) represents the most suited technique to determine crystallographic structures (nowadays widely used in chemistry, physics, engineering, and biology).

What is a crystal ?

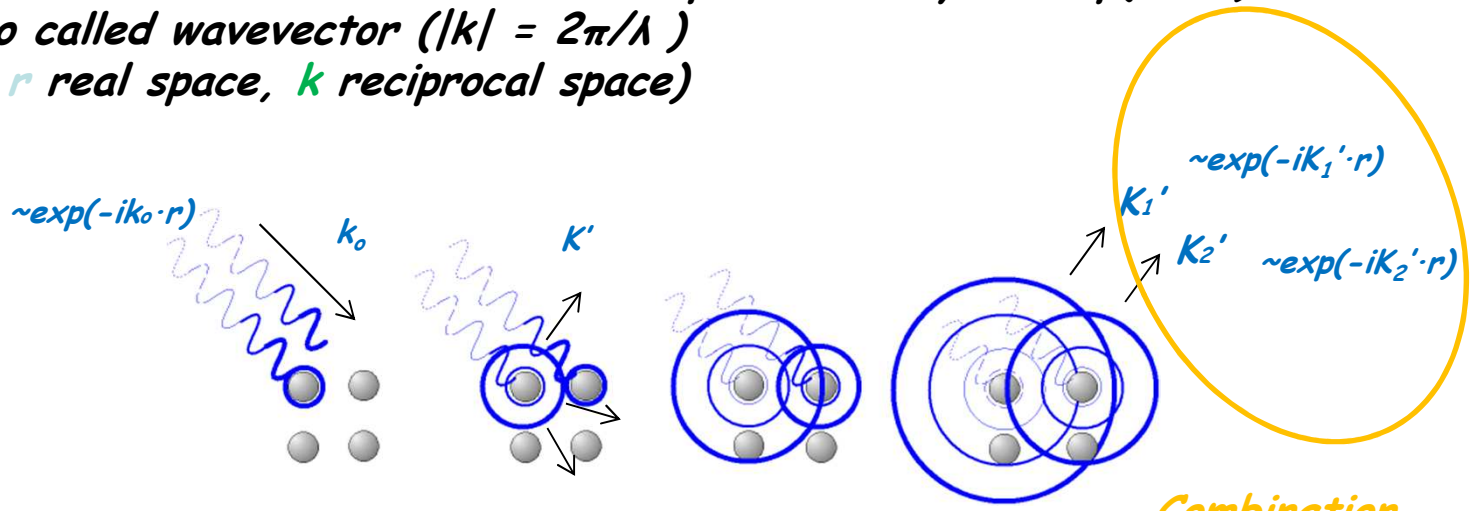
- Well it is neither a material in liquid, nor in gaseous state -> not amorph
- So it is a solid

A crystal is a 3-dim periodic (and ideally infinite) arrangement (lattice) of identical scatterers (basis). (basis = molecule)
Each point has an identical surrounding

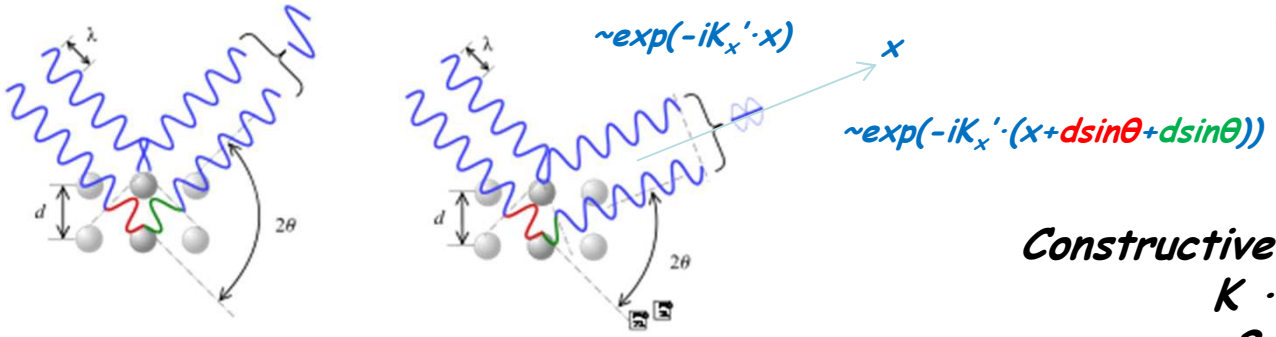


Position of peaks ***Intensity of peaks***

Reminder : electronic radiation is represented by A exp(-ik·r)
k so called wavevector (|k| = 2π/λ)
(ps r real space, k reciprocal space)



Combination of scattered waves



Constructive interference for
K ·2dsinθ = 2π·n
or 2dsinθ = nλ

Crystallography and Bragg law

Bragg formulation of X-ray diffraction

"crystals, at certain specific wavelengths and incident angles, produce intense peaks of reflected radiation (known as Bragg peaks)"

"W. L. Bragg explained this result by modeling the crystal as a set of discrete parallel planes separated by a constant parameter d"

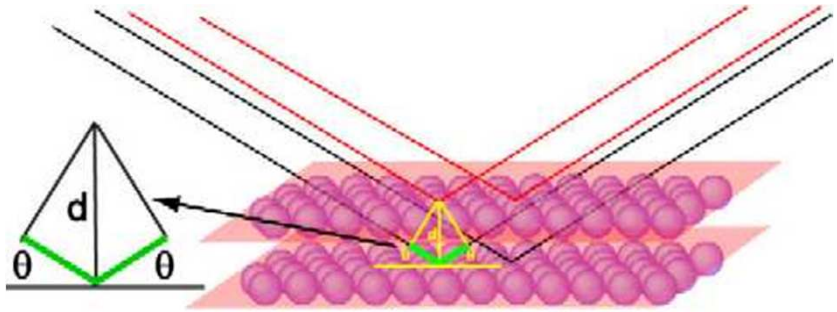
$\lambda = 2d\sin\theta$

d spacing between planes

λ wavelength of radiation

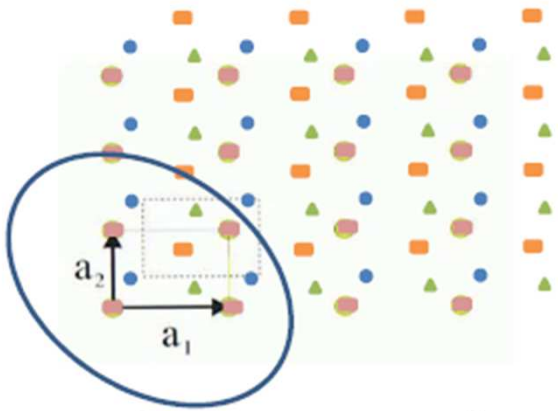
d,λ must have same order of magnitudes

planes are formed by layer of atoms -> ~1-100 Å



Typical wavelength typical wavelength of X-rays or neutrons -> ~1-100 Å

Crystallography and Bragg law



In unit cell system, the 4 atoms are at

- $x_0=0, y_0=0$
- x_1, y_1
- x_2, y_2
- x_3, y_3

$$F_{hkl} = f_o * \exp(iQ_{hkl} \cdot r_o) + f_1 * \exp(iQ_{hkl} \cdot r_1) + f_2 * \exp(iQ_{hkl} \cdot r_2) + f_3 * \exp(iQ_{hkl} \cdot r_3)$$

The intensity of a specific Bragg reflection hkl is proportional to the so called structure factor

$$F(\mathbf{Q_{hkl}}) = \sum_{r_j} f_j e^{i\mathbf{Q_{hkl}}\mathbf{r}} t_j(\mathbf{Q_{hkl}})$$

summation over all atoms in the unit cell

position of each atom in unit cell

temperature factor

scattering strength power of the atom (radiation and energy dependent)

X-ray : $f_j = f_o + f'(E) + i\, f''(E)$

f_o = number of electrons

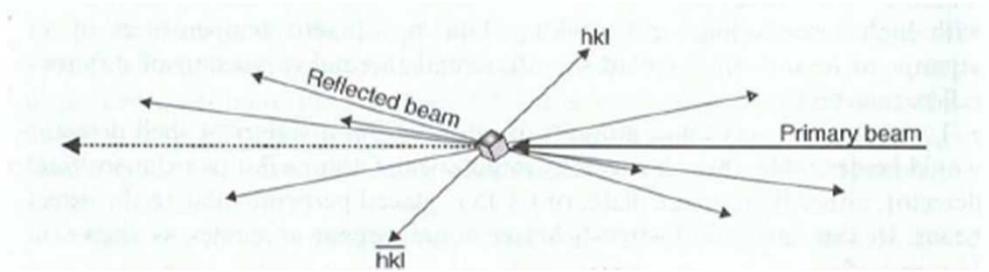
$f', f'' \neq 0$ around absorbtion edge

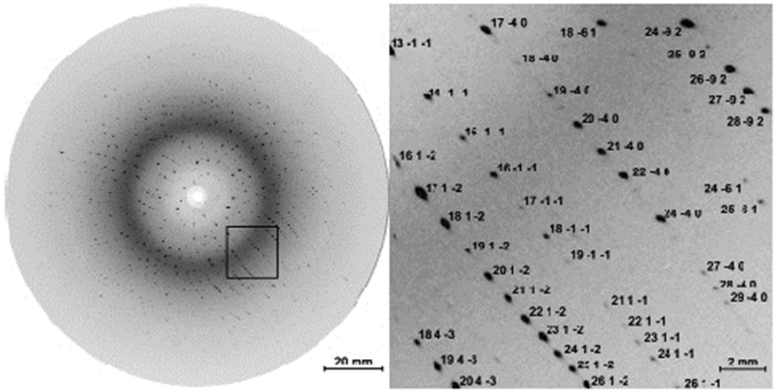
Neutrons : $f_j = b$

b no systematics vs. periodic table, it is related to atom nucleus

b can be related to the intrinsic magnetic moment of atoms

detector

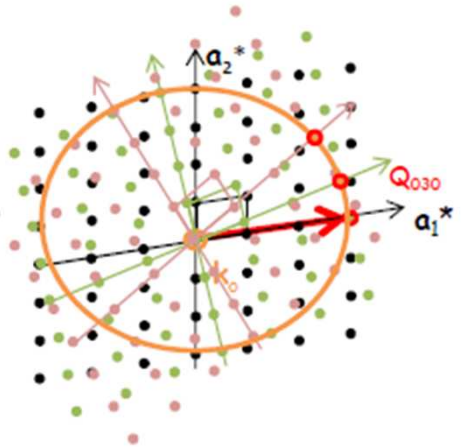


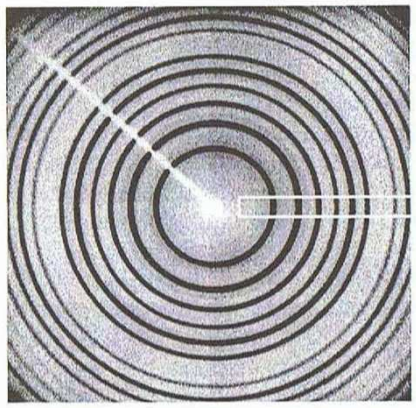


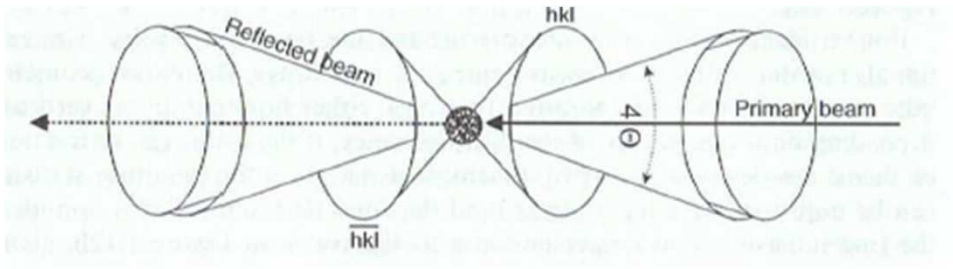
Rotate the crystal to get other Bragg reflections
Complete experiment :
100-300 frames at various crystal orientation

Powder diffraction

- *A powder is a multitude of single crystals, the diffraction by a powder is then the superposition of all contributing crystals. (a powder grain is not necessarily a single crystal)*
- *The hkl bragg peak on the detector is no longer a single spot in space but the sum of spots arising form each contributing crystal (problematic of statistical averaging)*
- *All the hkl spots are at 2θ angle from incoming beam, they hence all lie on the so called Debye-Scherrer cone.*
- *Intercept of detector plane with cone produces rings*





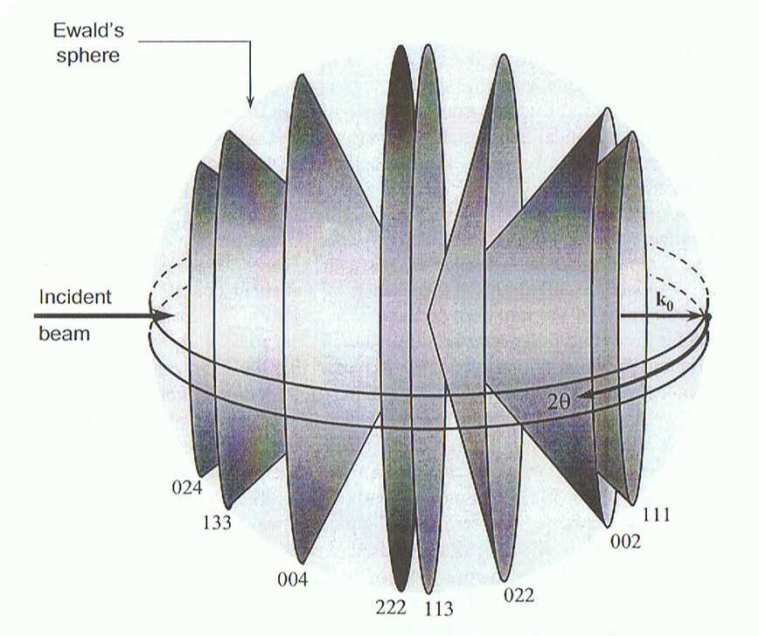


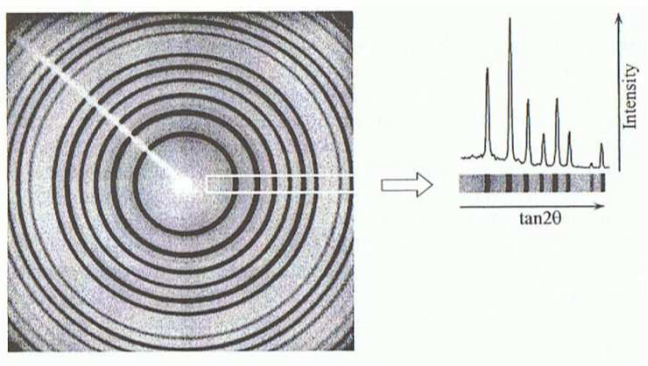
Debye-Scherrer cone

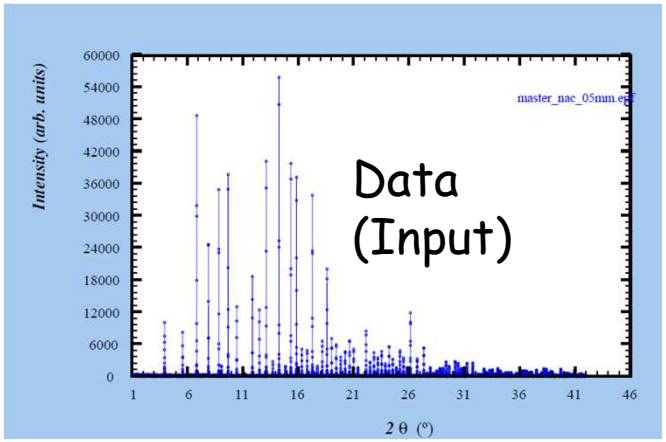
A diffraction experiment consists in

1) setting a crystal or a powder (= statistical ensemble of crystals with "random" orientation) in a well known radiation beam

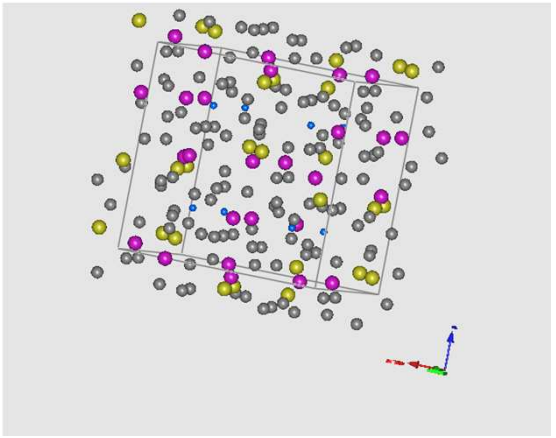
2) measure the (non-isotrop) level of radiation scattered by this material.

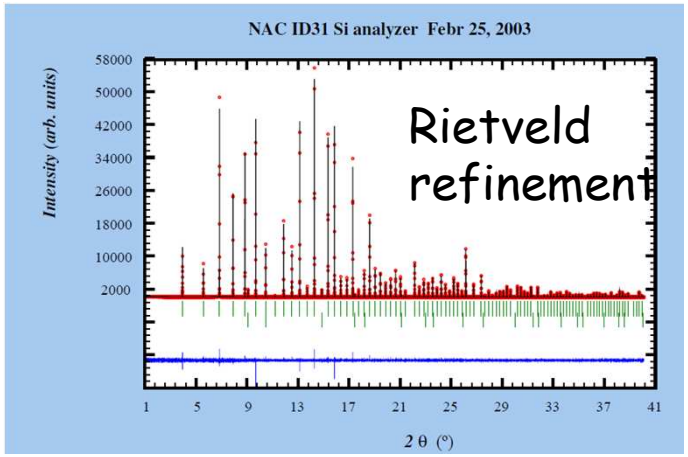


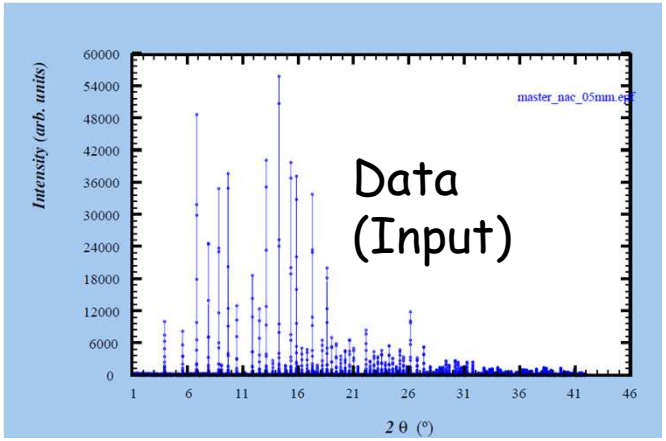




Model (Input/outpout)





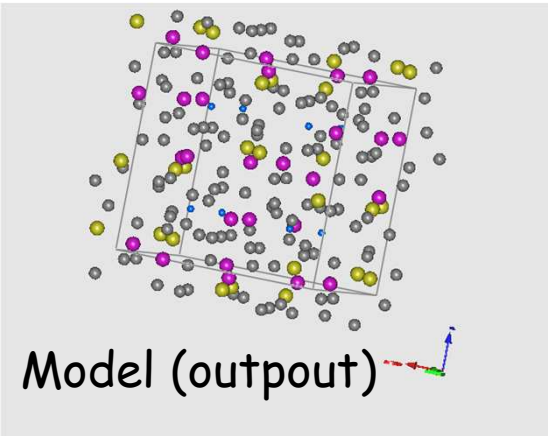


- Assign hkl for each of the peak
  -> (extract space group)

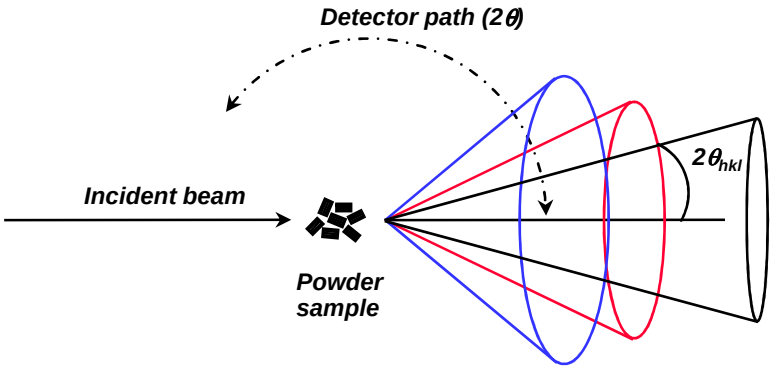
-extract integrated intensity
of each individual peak
  -> extract structure factors

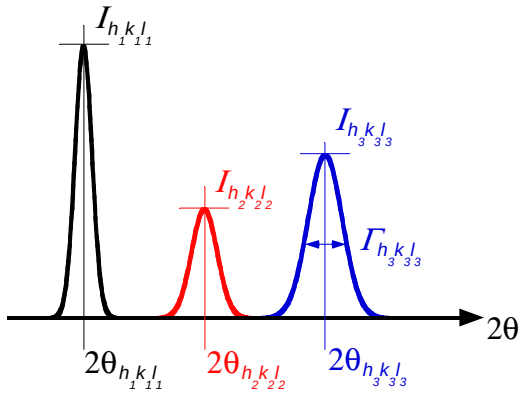
Alternative to Rietveld ?

So called ab-initio or direct methods



Powder diffraction





The center θ_g

Bragg law :
$\lambda=2*d_{hkl}*\sin\theta_{hkl}$

=> $d_{hkl}=d_{hkl}(a,b,c)$
=> information on overall peridical arrangement of atoms

The intensity $I_g=f(\theta_g)$

Structure factor:

$$F_{hkl} = \sum_j f_j \exp[-i2\pi(x_j h + y_j k + z_j l)]$$

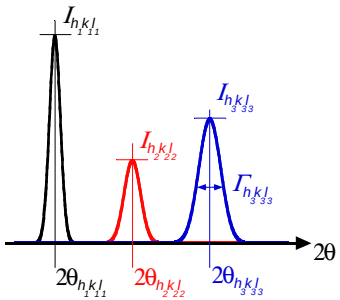
=> kind of atom (f_j) in material
=> position (x_j, y_j, z_j) of atoms in the structure (unit cell)

The fwhm Γ_g

($f(\theta_g+\Gamma_g)=1/2*f(\theta_g)$)

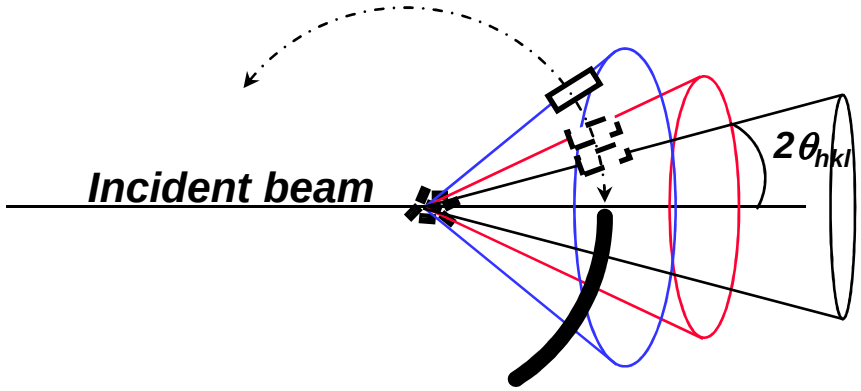
=> microstructural features (strain, size)

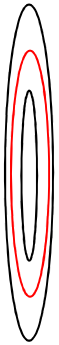
strongly instrument dependent



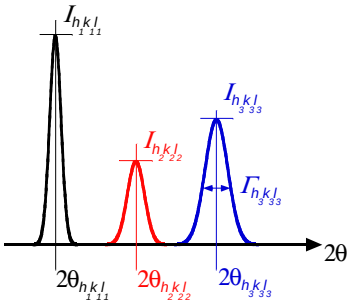
direct 1D acquisition
either scanning a point detector (0D)
or collecting on a 1D steady state detector

Key elements :
the source, the detectors
the mechanics holding the sample and detector

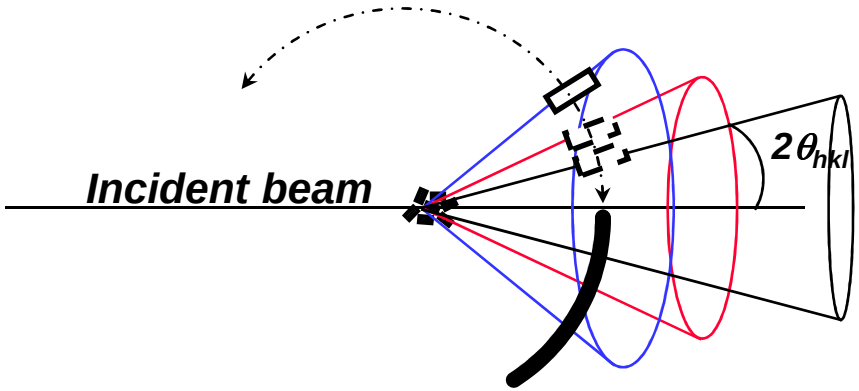


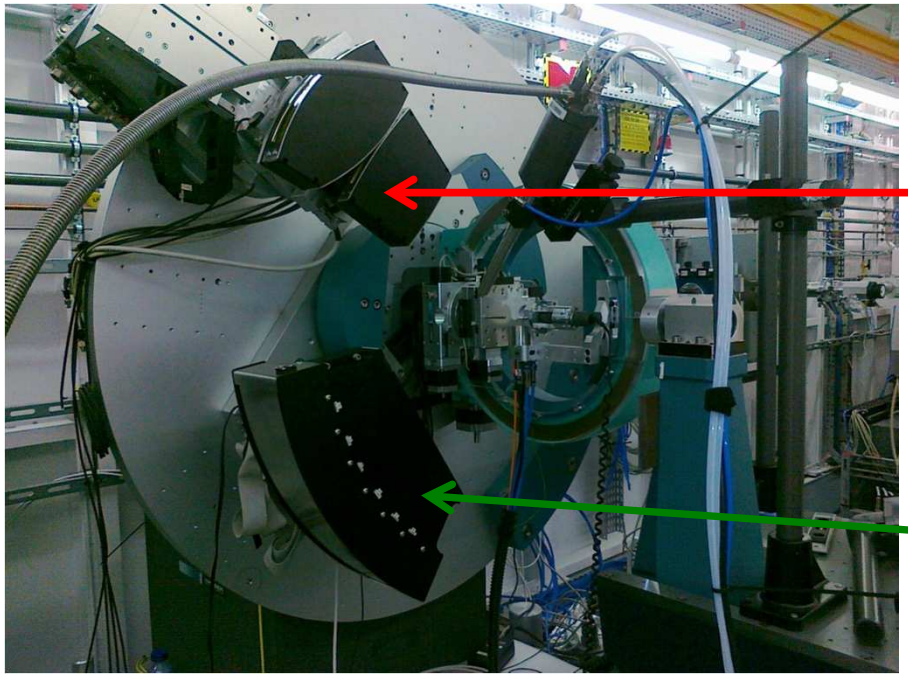


2D-> 1D conversion



Powder diffraction Instrumentation





Multi Analyzer Detector (10? – 50 keV)
(to be commissioned Nov 2012)

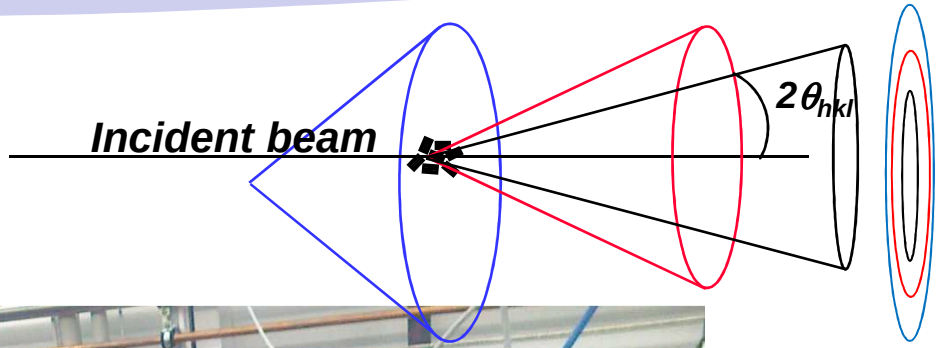
- 13 channels with 1.5 deg pitch
- Si_{111} or Si_{220} Bragg reflection
- YAP scintillator + PMT

Mythen detector (7-30? keV)

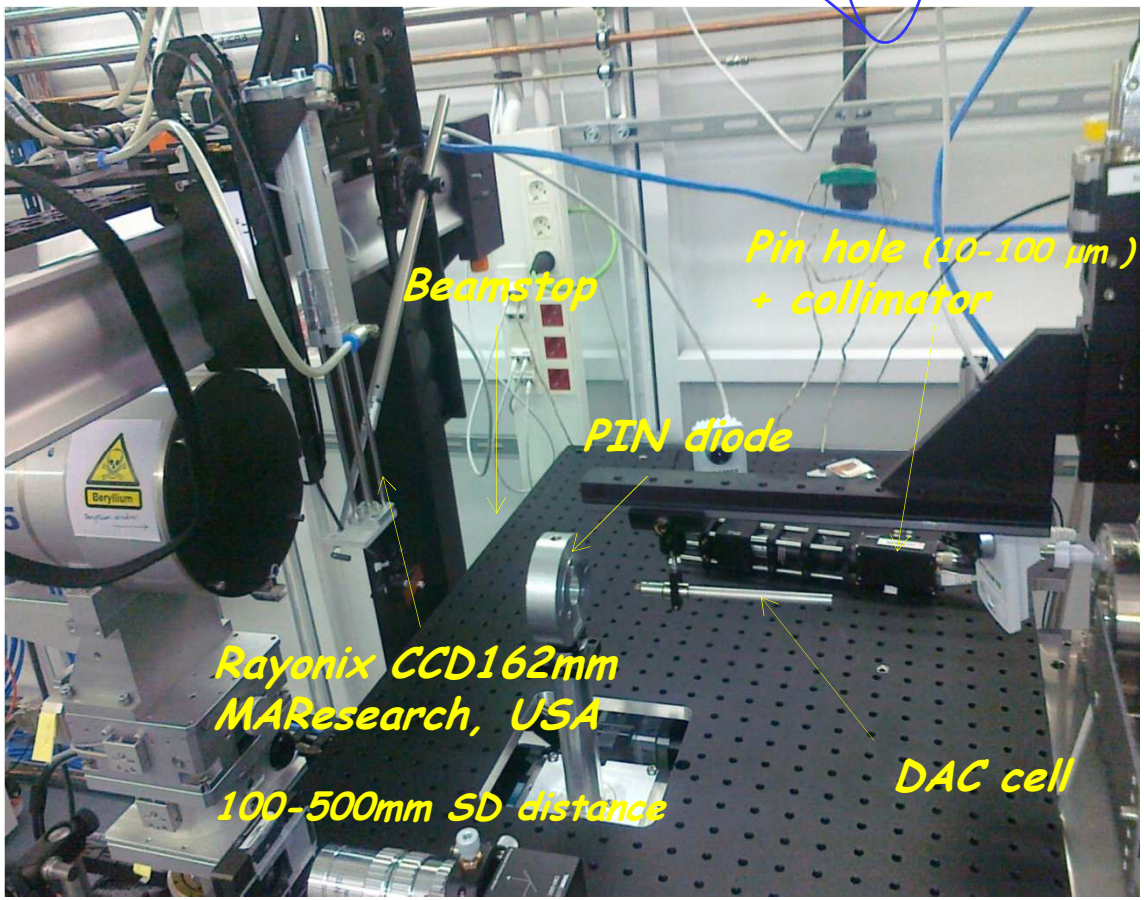
- 6 modules (1280 channels, 50 µm pitch)
- D_SD= 550mm
- ms time resolution

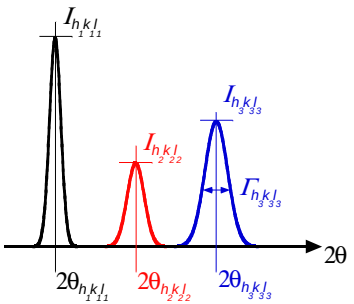
-> ~40deg in 0.005 deg pitch

Powder diffraction Instrumentation



2D-> 1D conversion

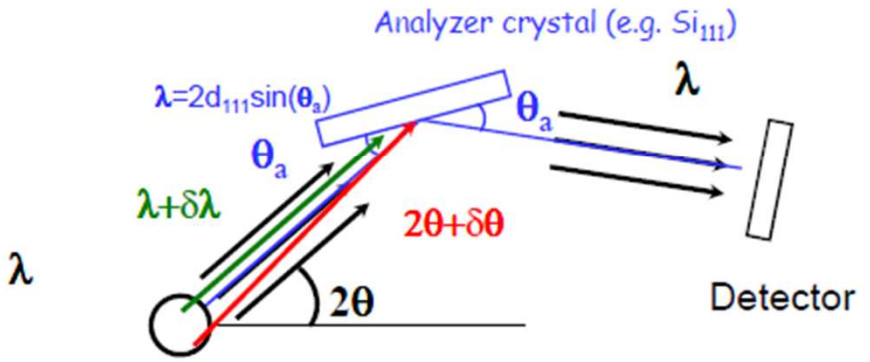




How to achieve higher resolution better signal to noise

1- minimize incoming beam divergence and energy resolution
(no focusing, collimating mirror, DCM monochromator, synchrotron source
(eg. Wiggler => high horizontal divergence, low vertical

2- use analyzers (back monochromator)

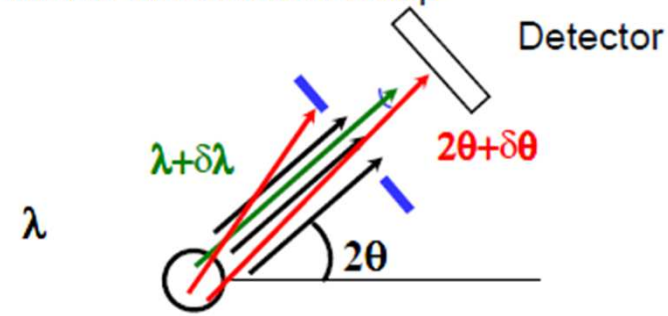


high signal to noise ratio
(elimination of inelastic processes)

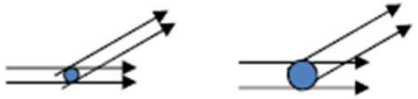
higher angular resolution
(analyzer crystal acts as a tiny slit:
$d\theta$ ~ 10" ~ intrinsic width of the analyzer crystal)

no sample size/tranparency effect
Not sensitive to displacement

Slit/No Slit-detector setup



high intensity rate
(since no loss (discrimination)
from analyzer-radiation interaction)
Angular resolution is proportional to
sample size in the beam



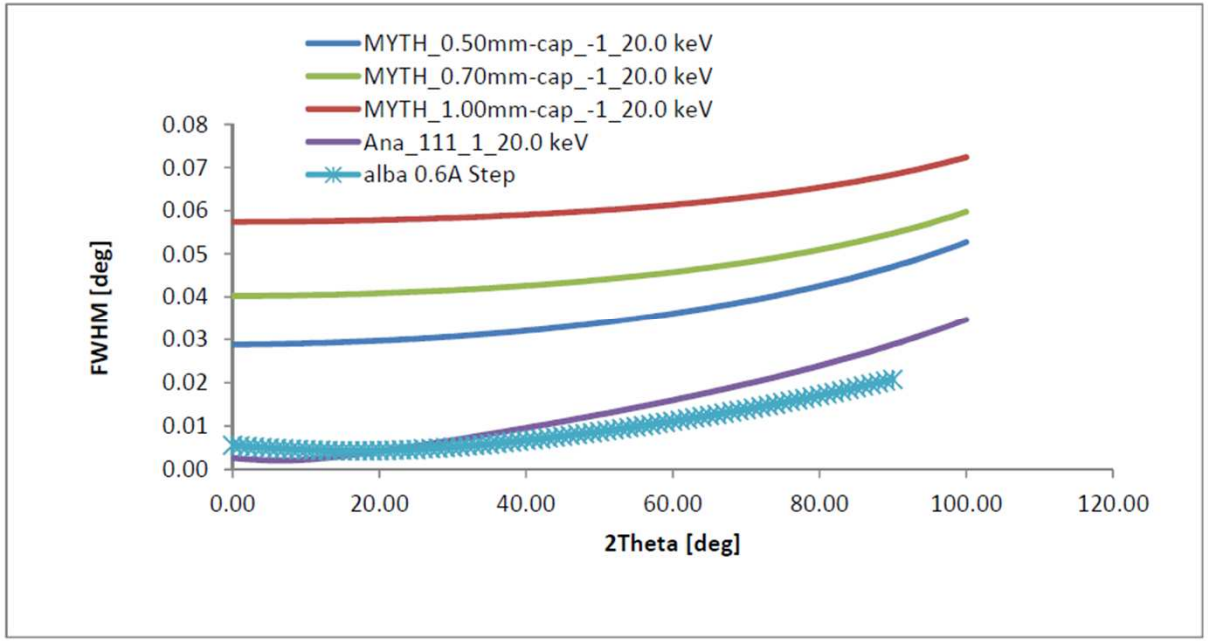
Powder diffraction, angular resolution

$$\Gamma_{ana} = \sqrt{\omega_a + \tfrac{1}{2}\omega_m \left\{\frac{\tan(\theta_a)}{\tan(\theta_m)} - 2\frac{\tan(\theta_s)}{\tan(\theta_m)}\right\}^2 + \varphi_{M1}^2 \left\{\frac{\tan(\theta_a)}{\tan(\theta_m)} - 2\frac{\tan(\theta_s)}{\tan(\theta_m)} \pm 1\right\}^2 + \varphi_{M2}^2}$$

$$\Gamma_{mythen} = \sqrt{\sqrt{d^2 + p^2} + \tfrac{1}{2}\omega_m \left\{-2\frac{\tan(\theta_s)}{\tan(\theta_m)}\right\}^2 + \varphi_{M1}^2 \left\{-2\frac{\tan(\theta_s)}{\tan(\theta_m)} \pm 1\right\}^2 + \varphi_{M2}^2}$$

$\omega_{a/m}$	Darwin width mono/analyzer Xtal
$\varphi_{M1/M2}$	Beam divergence on before/after
$\theta_{a/m/s}$	Angle sana/mono/sample (energy)
p, d	Pitch between stripe, capillar diamteer

+ additional horizontal divergence and sample size contribution

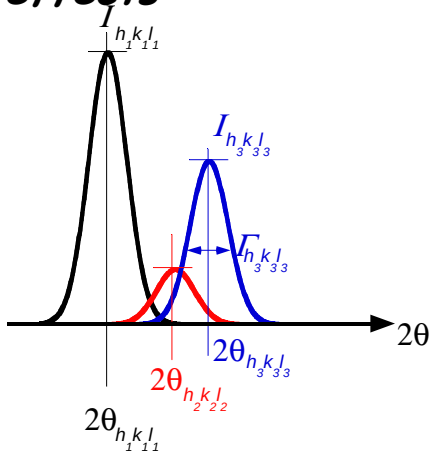


Powder diffraction, angular resolution, accuracy

Why high resolution and high accuracy ?

Not only to get accuracy of cell parameter up to forth or fifth digit
As seen before, in order to detect microstructural effects
But as well to minimize the overlap problem

In some circonstances (short λ, high 2θ, big unit cells) Bragg reflections strongly overlap



characteristic values
(θ_g, I_g, Γ_g) difficult to extract

inaccuracy in the determination of a crystal structure

Powder diffraction, peak shape

Reflections in a powder pattern can be described by various profile functions
The most important are

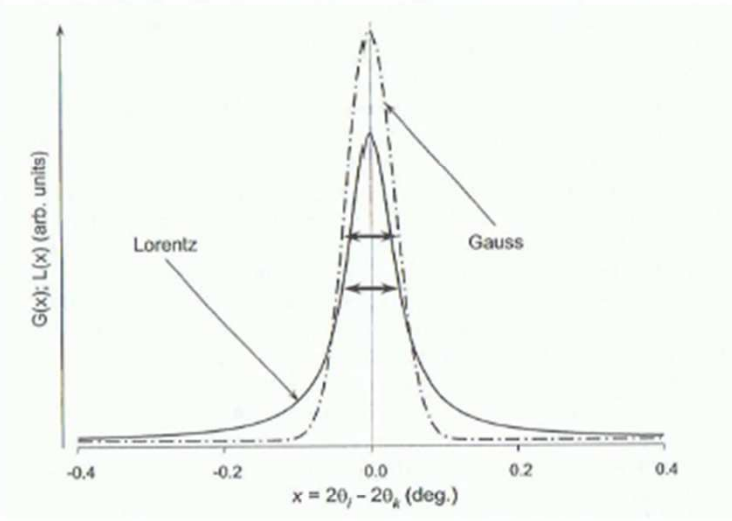
- **Gaussian** function G(x)
- **Lorentz** (or Cauchy) function L(x)
- **pseudo Voigt** function pV(x)

$$G(x) = \frac{\sqrt{C_G}}{\sqrt{\pi}H} e^{-C_G x^2} \qquad L(x) = \frac{\sqrt{C_L}}{\pi H}(1 + C_L x^2)^{-1}$$

$C_G = 4\ln 2$ $\qquad$ $C_G = 4$

$$pV(x) = \eta L(x) + (1-\eta)G(x)$$

H: full width at half maximum FWHM $pV(x \pm H/2) = \frac{1}{2}\, pV(x)$



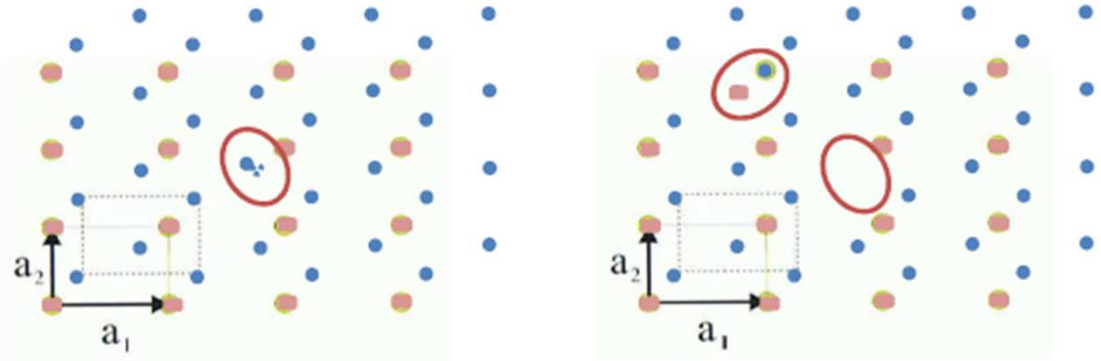
Why is the easy modelisation of shape function important ?

Data analysis of powder patterns by so called Rietveld refinement implies comparison of the measured spectrum with a trial calculated pattern. This process involves calculation of thousends of Bragg reflection.
Rapide calculation only possible as long as Bragg reflection are easily modelized.

Why is the knowledge of shape function important ?

Width Γ_g information on microstructural features,
that is any form of atomic disorder or imperfections exhibited by the periodical arrangement of atoms
(crystallite-size effects, dislocations, stacking fault, variations in interatomic distances due to internal stresses or non-stoechiometry)

-> all what contributes to reduce the infinite periodicity of the lattice



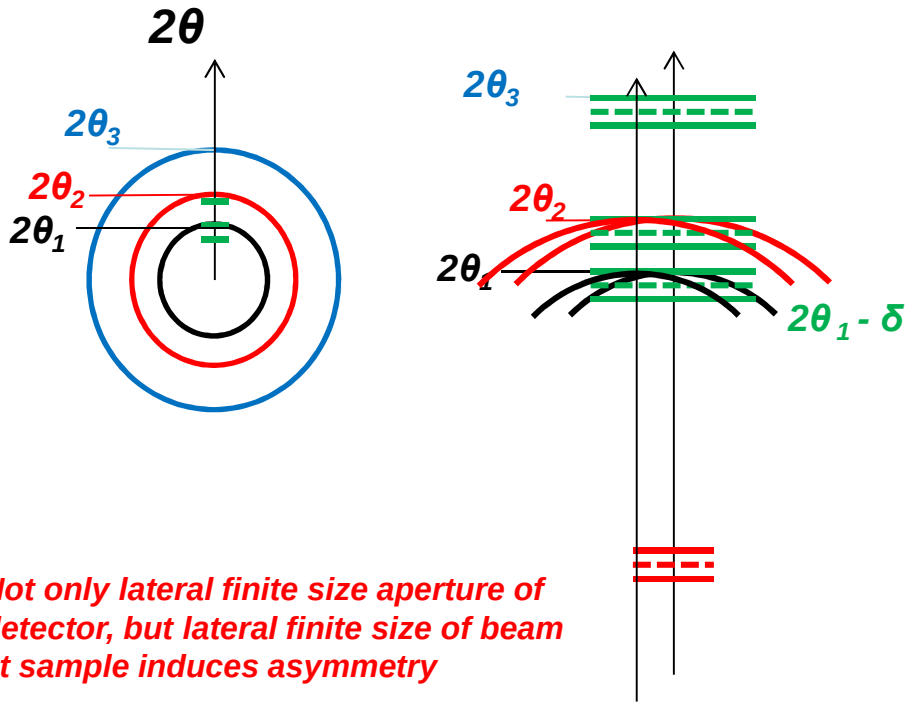
- *mixed site*

  2-3 types of atoms (generally of the same size), occupy the same lattice site

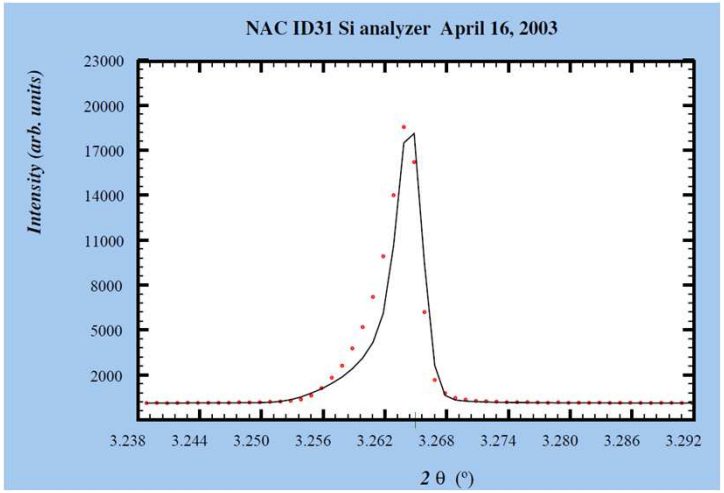
These features are very small and generally contribute marginally to the width.
The main contribution comes from the instrument itself -> high resolution needed

Powder diffraction, asymmetry

Sever peak shape asymmetry at low angles
-> due to limited size aperture of detector and beam



Not only lateral finite size aperture of detector, but lateral finite size of beam at sample induces asymmetry



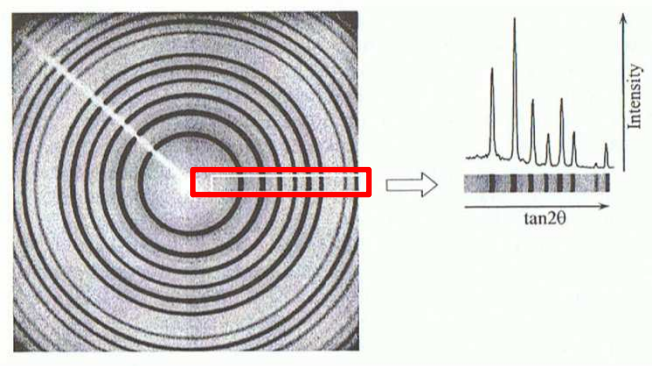
So called "umbrella" effect can Be 'hardly' corrected using so called Thomson-Cox-Hasting asymmetry correction function

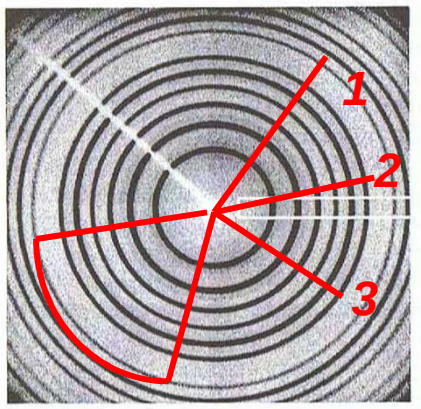
depends on S/D and L/D where
S lateral dimension of beam at sample
L lateral dimension of detector aperture
D sample to detector distance

How to get rid of asymmetry

- *Minimize S and/or L –> lost of intensity*
- *Increase Sample to deetctor distance D -> lost of intensity*
- *Use higher wavelength so to have Bragg peaks at higher 2theta*

No asymmetry if using 2D detectors and properly convert from 2D to 1D





1+2+3

Why using Synchrotron Powder diffraction

Why using Synchrotron ?
(compared to Lab X-ray sources)

- *Energy tunability (not limited Cu K, Mo K edges)*
- *Broader range of energies (8-50 keV at MSPD@ALBA)*
- *High energies = higher penetration depth:*
  - *-> transmission geometrie*
  - *-> sample environments*
  - *(cryostats, furnace, ...all what includes windows)*

- *High flux*
  - *-> high throughput*
  - *-> high resolution*
    - *- we can afford putting "beam cleaning elements (DCM monochromator, analyzer crystals)*
    - *- very low natural beam divergence*
  - *-> small spot size*

- *Big diffractometers*
  - *Space to put ancillary equipments*

Why using Synchrotron Powder diffraction

Why using Synchrotron ?

Practical examples

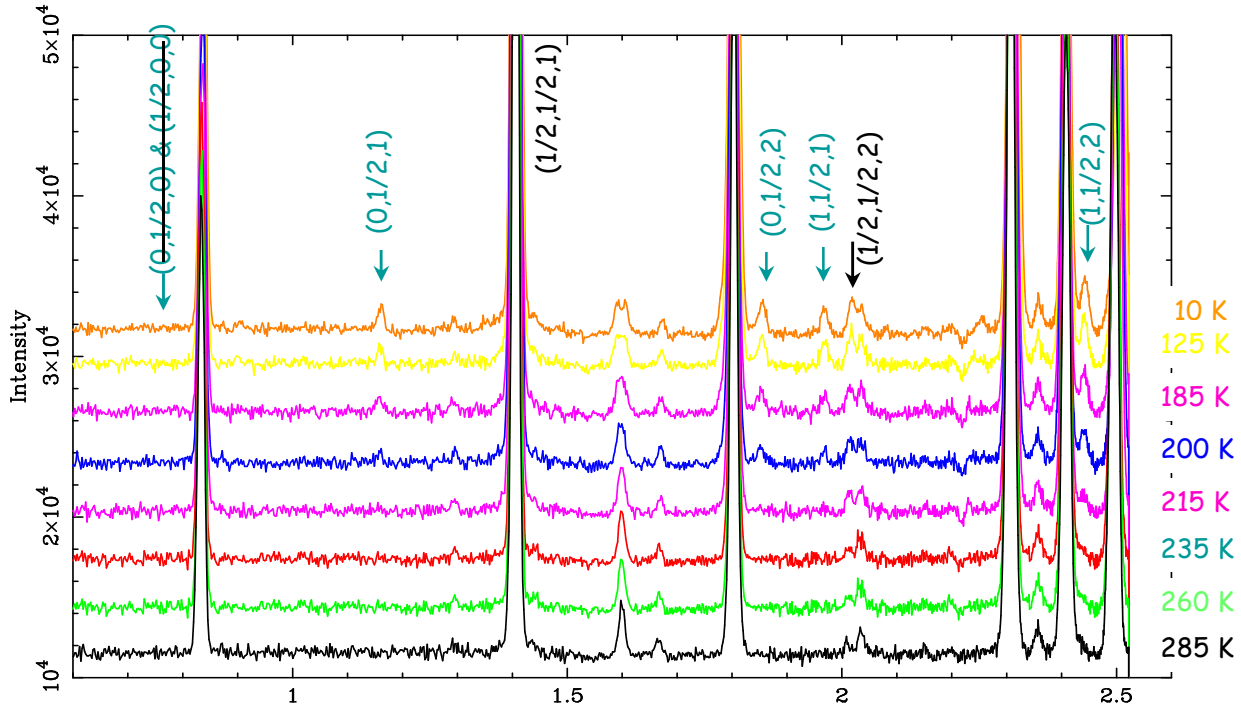
- *When good signal to noise is needed*
- *When high angular resolution is needed -> resolve peaks*
- *When energy tunability is needed -> resonant diffraction*
- *When high throughput is useful*
- *When temperature helps*
- *Beyond the reasonable*
- *When small spot size is needed -> high pressure*
- *When high energy and controlled spot size is needed*
- *When high angular resolution is needed -> microstructural effects*

When good signal to noise is needed

$LnBaCo_2O_{5.0}$ (Ln=Ho[1,2],Tb[2], Dy[2], Y[3]) Charge Ordering

1) Suard et al., Phys. Rev. B61 (2000) R11871
2) Fauth et al., Eur. Phys. J B21 (2001) 163
3) Vogt et al., Phys. Rev. Lett. 84 (2000) 2969

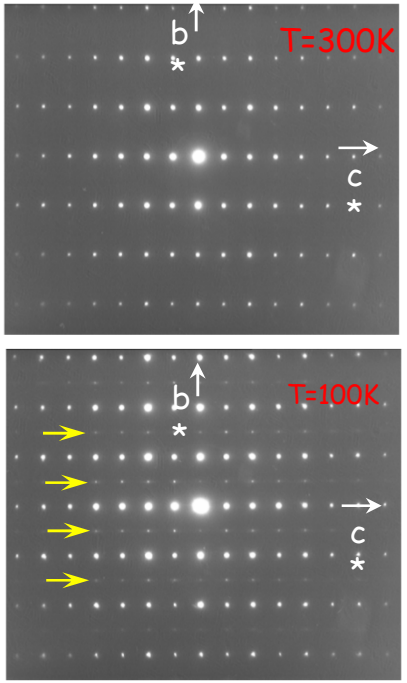
Neutron Powder Diffraction : G61@LLB (l=4.73 Å)
$HoBaCo_2O_5$: (indexation in $a_p \times a_p \times 2a_p$ setting)



no (0,1/2,0) ➔ S.G. Pmmb

no (1/2,0,0) => magnetic moments along the a-axis

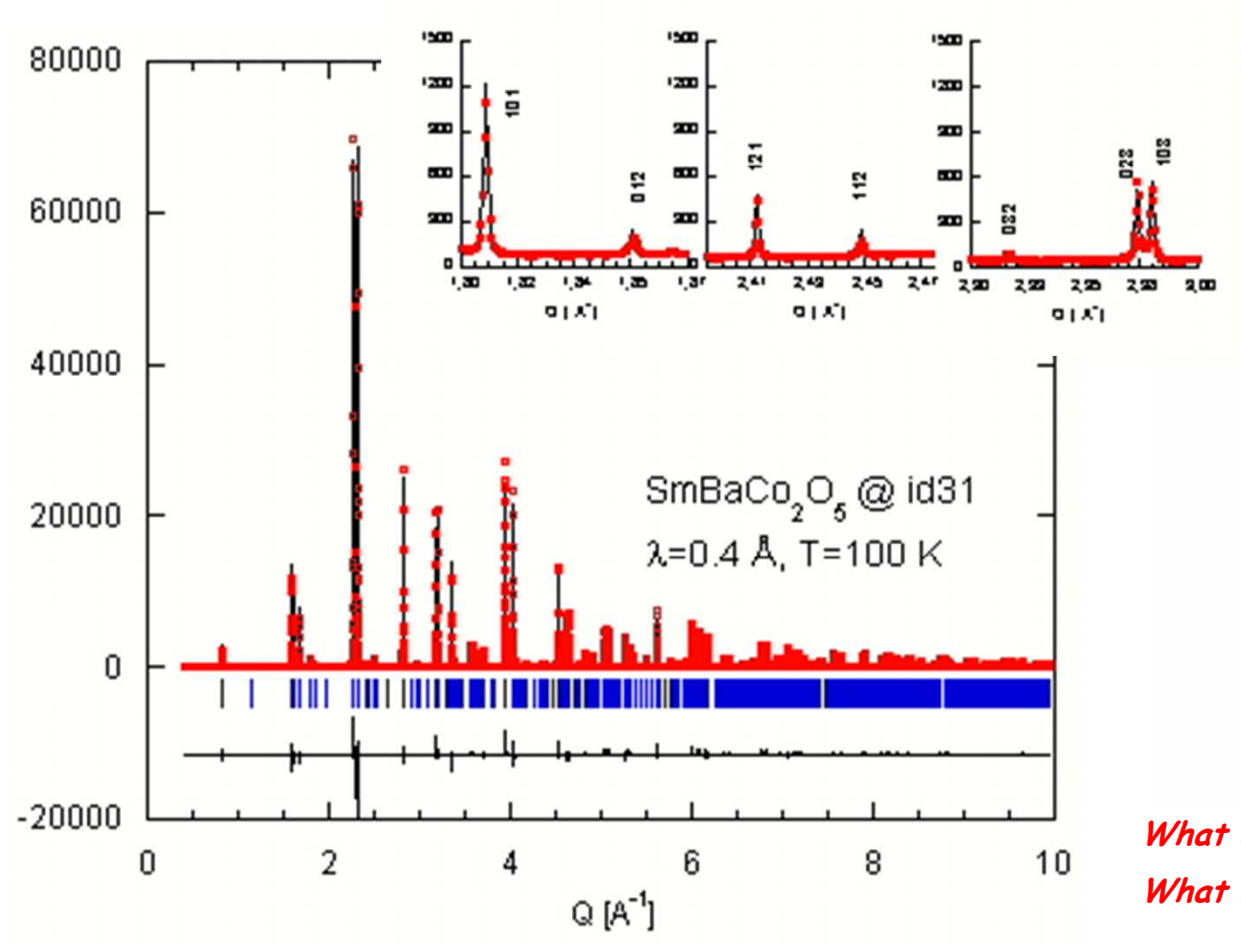
Standard Electron diffraction
$TbBaCo_2O_5$ (+ microdiffraction)

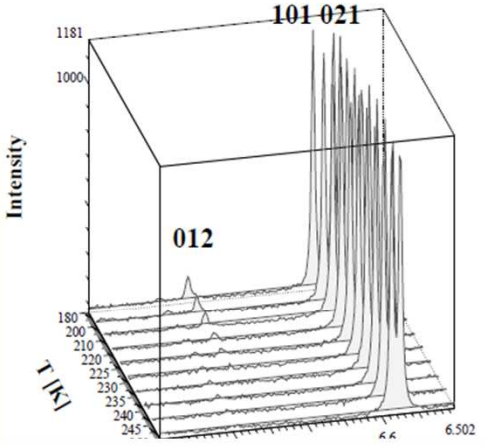


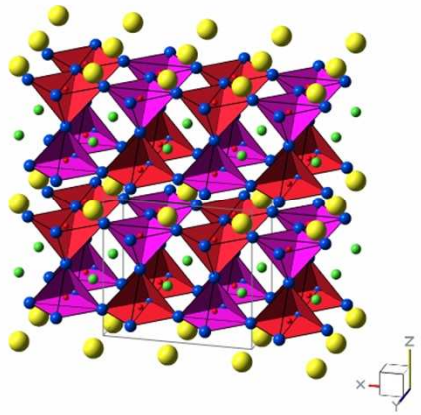
Pmmb extinction
0k0, hk0, k=2n+1

Charge Ordering in Ln=Y,Ho,Tb, Dy and $SmBaCo_2O_{5.0}$

Synchrotron Powder Diffraction :
Pmmb Co structure







What about CO in $LnBaCo_2O_5$ Ln=Nd,Pr ?

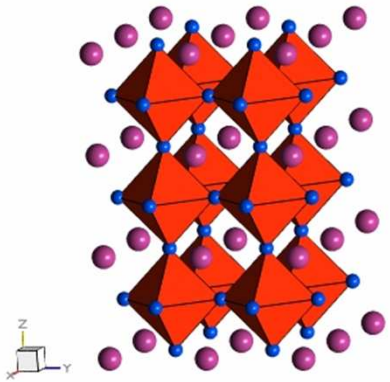
What about CO in $LnBaCo_2O_{5+e}$? (e_{max} ?)

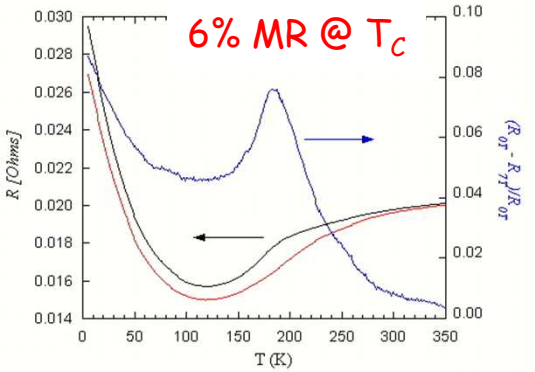
$LaBaCo_2O_{6.0}$ [1] : Orbital Ordering

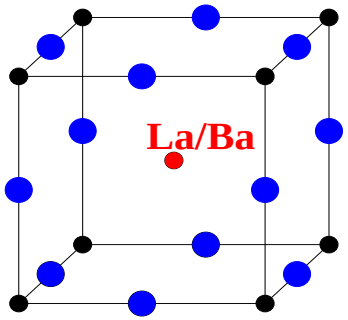
1) Fauth et al., Phys. Rev. B65 (2002) 060401(R)

Reminder

- Equal amount of Co^{4+} and Co^{3+}
- Co ions in octahedral coordination
- $La_{1/2}Ba_{1/2}CoO_3$ (~ $La_{1-x}Sr_xCoO_3$)

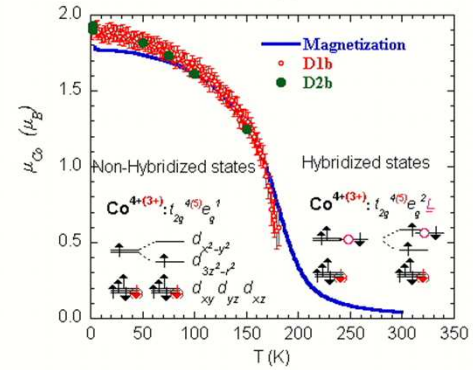






Perosvkite structure Pm3m at RT (a_p x a_p x a_p)

➔La and Ba statistically distributed on the body center site



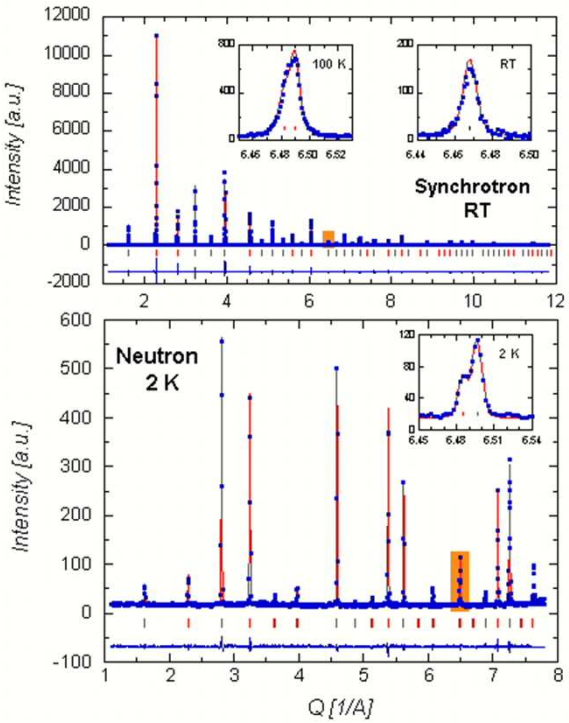
Ferromagnetism
T_C ~ 180 K, $m_o(Co)$= 1.9m_B
(weak value !)

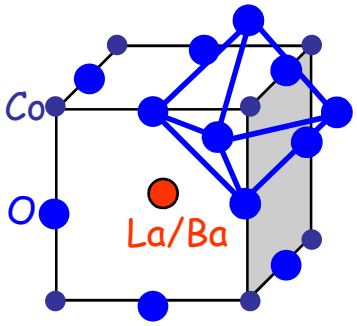
When high angular resolution is needed

Pm3m -> P4/mmm

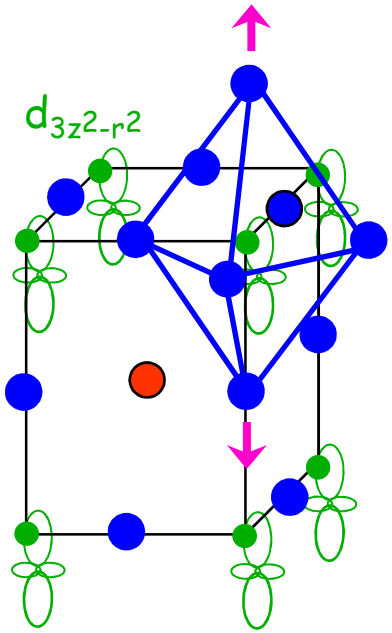
axaxa axaxa′ (a<a′)

(a′-a)/a ~ 0.16% @ 2K

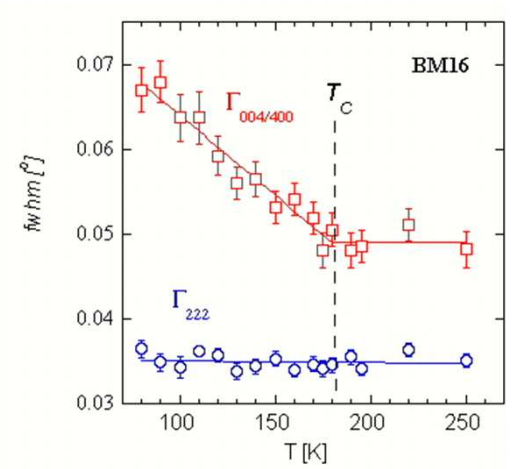




"Elongated" octahedra



↳Associated magnetic and structural transitions



↳No phase segregation

Ferro-orbital ordering of the $d_{3z^2-r^2}$

Jahn-Teller induced ferrodistortive transition

-> favored if IS-Co^{3+} : $|t_{2g}^5 e_g^1>$

and/or IS-Co^{4+} : $|t_{2g}^4 e_g^1>$

Resonant X-ray scattering (often mentioned, raraly applied in powder)

EUROPHYSICS LETTERS 1 January 2001
Europhys. Lett., **53** (1), pp. 72–78 (2001)

Direct observation of 1-dimensional charge order below the first-order "metal-insulator" transition in Yb_4As_3

U. Staub[1], B. D. Patterson[1], C. Schulze-Briese[1], F. Fauth[1], M. Shi[1], L. Soderholm[2], G. B. M. Vaughan[3] and A. Ochiai[4]

[1] *Swiss Light Source, Paul Scherrer Institute - CH-5232 Villigen PSI, Switzerland*
[2] *Chemistry Division, Argonne National Laboratory - Argonne, IL 60439, USA*
[3] *European Synchrotron Radiation Facility - F-38043 Grenoble Cedex, France*
[4] *Department of Material Science and Technology, Niigata University Niigata 950-21, Japan*

PHYSICAL REVIEW B **71**, 075115 (2005)

Temperature dependence of the crystal structure and charge ordering in Yb_4As_3

U. Staub,[1] M. Shi,[1] C. Schulze-Briese,[1] B. D. Patterson,[1] F. Fauth,[2] E. Dooryhee,[3] L. Soderholm,[4] J. O. Cross,[5] D. Mannix,[6] and A. Ochiai[7]

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Charge order and crystal structure below the first-order "metal–insulator" transition in Yb_4As_3

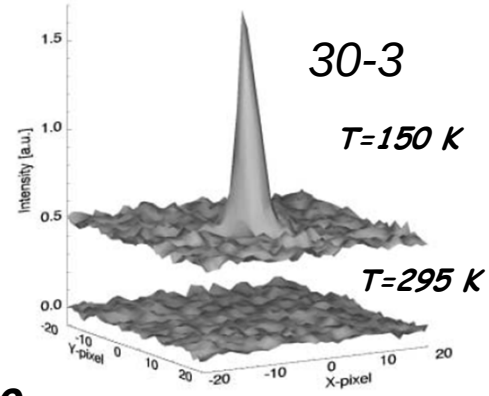
U. Staub[a,*], B.D. Patterson[a], C. Schulze-Briese[a], F. Fauth[a,c], M. Shi[a], L. Soderholm[b], G.B.M. Vaughan[c], E. Dooryhee[c], J.O. Cross[d], A. Ochiai[e]

[a] *Swiss Light Source, Paul Scherrer Institute, CH-5232 Villigen PSI, Switzerland*
[b] *Chemistry Division, Argonne National Laboratory, Argonne, IL-60439, USA*
[c] *European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble Cedex, France*
[d] *PNC-CAT, Advanced Photon Source, Argonne National Laboratory, IL 60439, USA*
[e] *Center for Low Temperature Science,Tohoku University, Aramaki aza Aoba, Aoba-ku, Sendai, Miyagi 980-8578, Japan*

When energy tunability can be used

Yb_4As_3 : charge localization at metal-insulator transition T_{MI}=290 K
I-43d -> R3c transition

Above T_{MI} : Yb in 2.25 valence state (assuming As^{3-})
Below T_{MI} : charge ordering, two Yb species (Yb^{3+} and Yb^{4+})



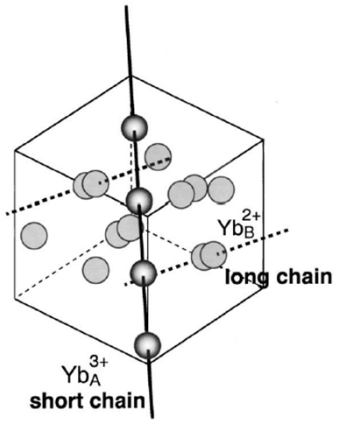
$$F(\mathbf{Q_{hkl}}) = \sum_{r_j} f_j e^{i\mathbf{Q}_{hkl}\mathbf{r}_j},\ f_j = f_o + f'(E) + i \cdot f''(E)$$

$$\sum_A \exp[iQR_A] = -\sum_B \exp[iQR_B]$$

F ≠ 0 if f', f'' ≠ 0

Single crystal CCD detector

[111]



Intensity have to be corrected for absorption

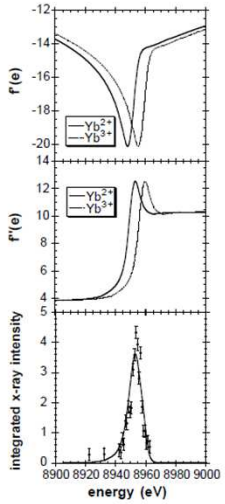


Fig. 3

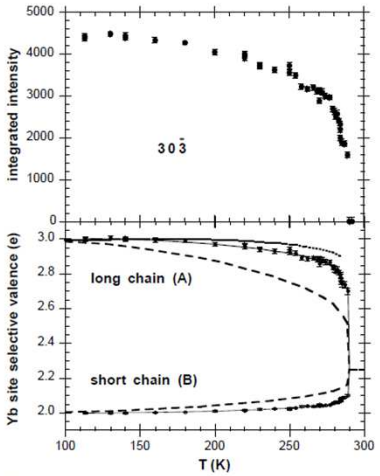


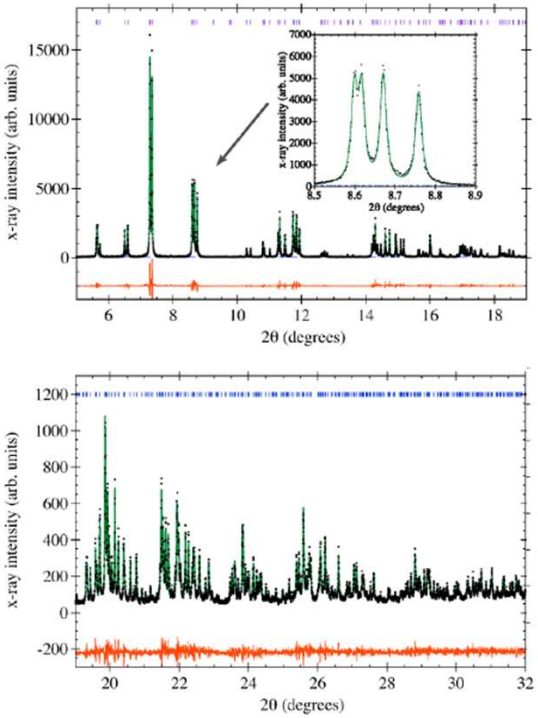
Fig. 4

Fig. 3 – Energy dependence of the real (f') (top), and the imaginary (f'') (middle) parts of the scattering factor of Yb^{2+} and Yb^{3+}, and of the 303 reflection together with a fit to $\alpha|f_{Yb^{2+}} - f_{Yb^{3+}}|^2$, in the vicinity of the Yb L_3-absorption edge in Yb_4As_3.

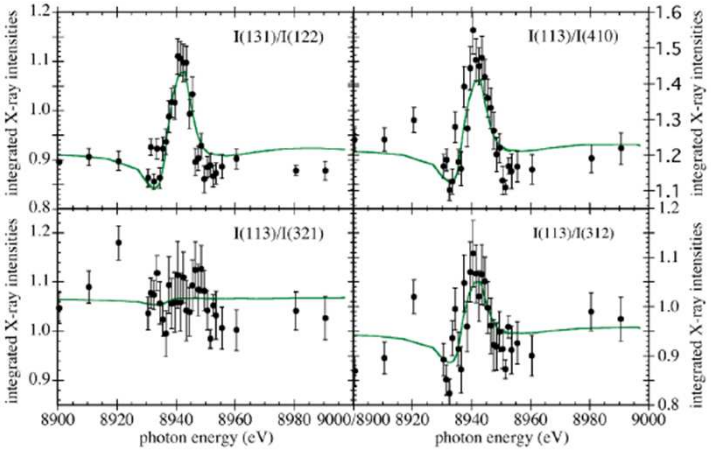
When energy tunability can be used

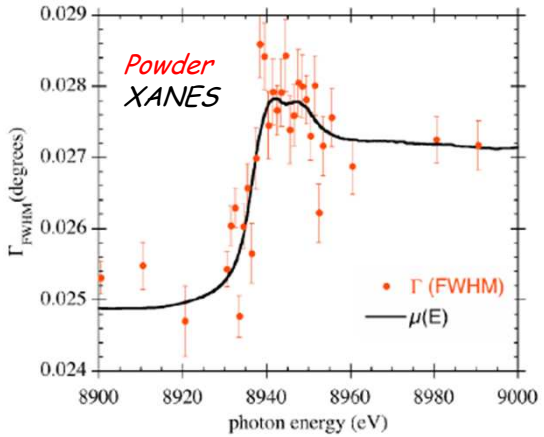
What about using powder

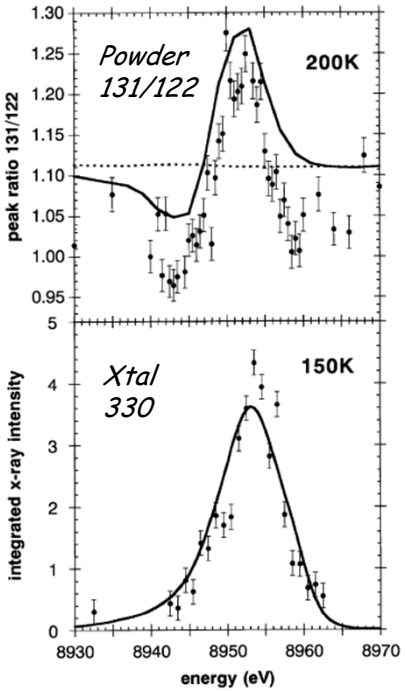
No need to correct for absorption since intensity ratio

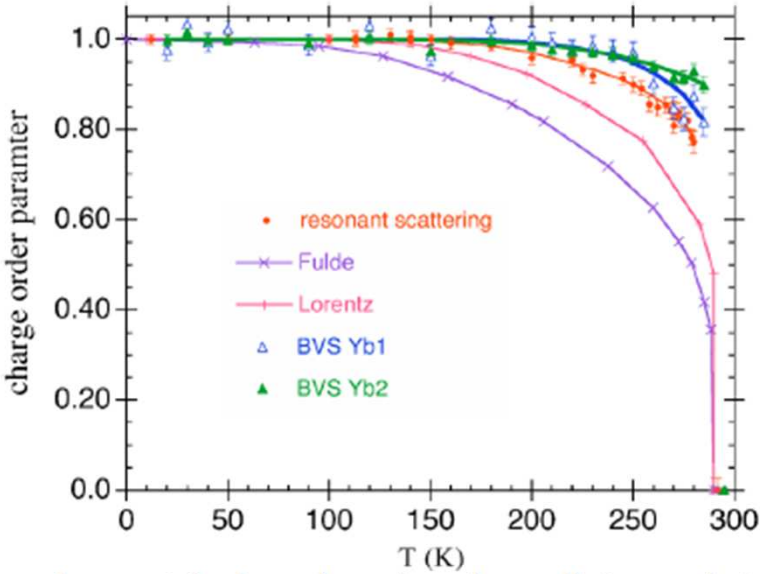


T=180 K, R3c
a=12.503 Å, c=7.496 Å







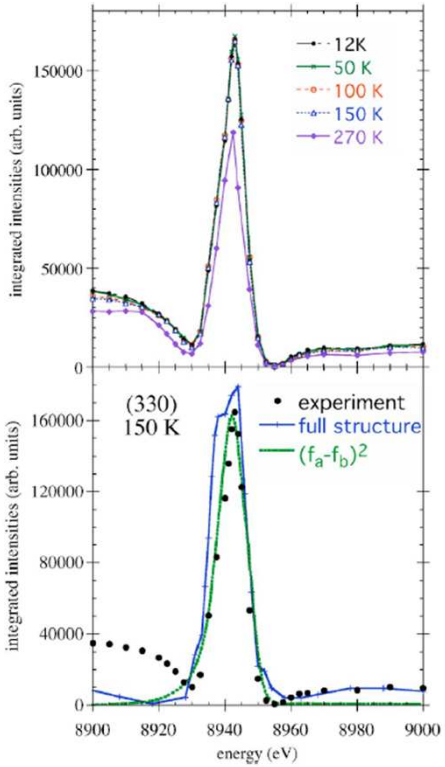


the expected valence change is weak as well. A convenient way to correlate the bond-length changes with the valence changes is to calculate the "bond valence sum" (BVS), defined as

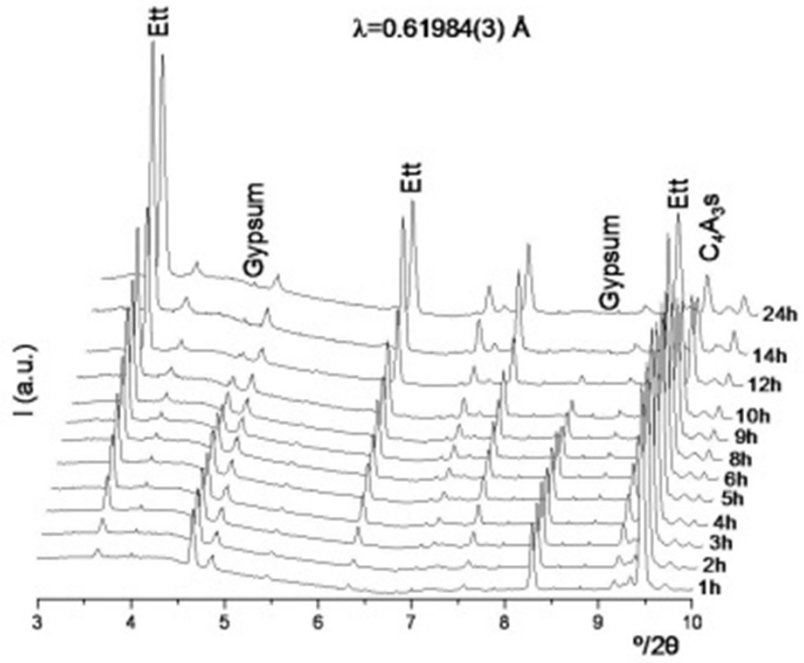
$$V_i = \sum_j \exp[(R_{ij} - d_{ij})/b],$$

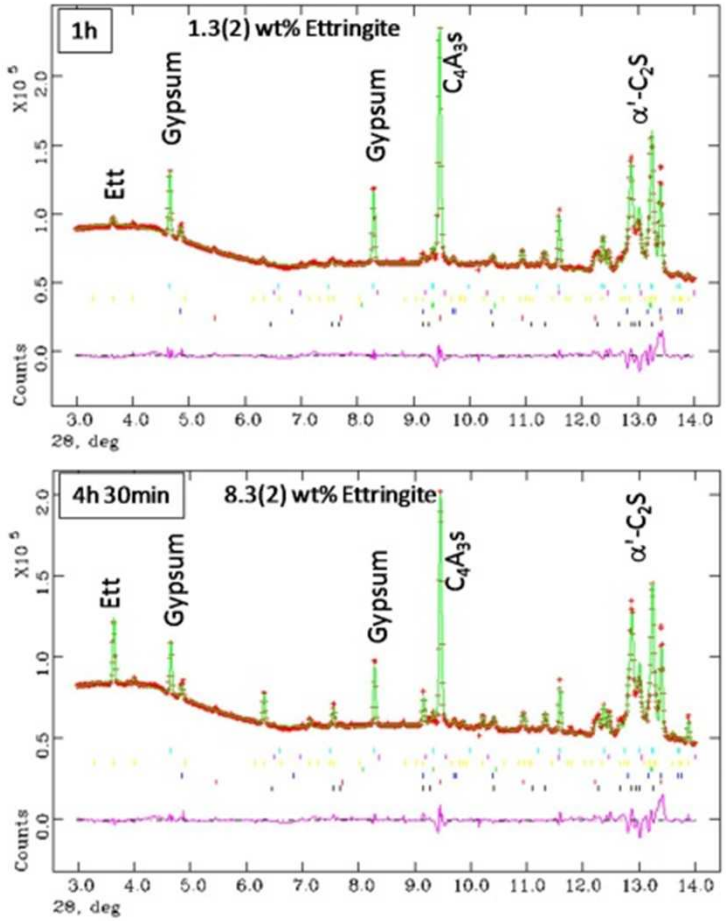
Bon valence sum forumulation

with b=0.37 as the general parameter, R_{ij} as the bond length between the ith and jth ion and d_{ij}=2.59 Å[52,53] the parameter for the Yb—As bond, which gives the correct Yb^{3+} valences for Yb in YbAs. To correct for thermal expansion, we rescale the bond lengths with the cube root of the temperature-dependent cell volume. The obtained low temperatures value for Yb(1) and Yb(2) are 2.27 and 1.81, respectively, which deviate significantly from the expected 3+ and 2+ values. Finally, the temperature-dependent valence is obtained by scaling the corresponding BVS by the expected valence changes of 3−2.25=0.75 e and 2.25−2=0.25 e, for the



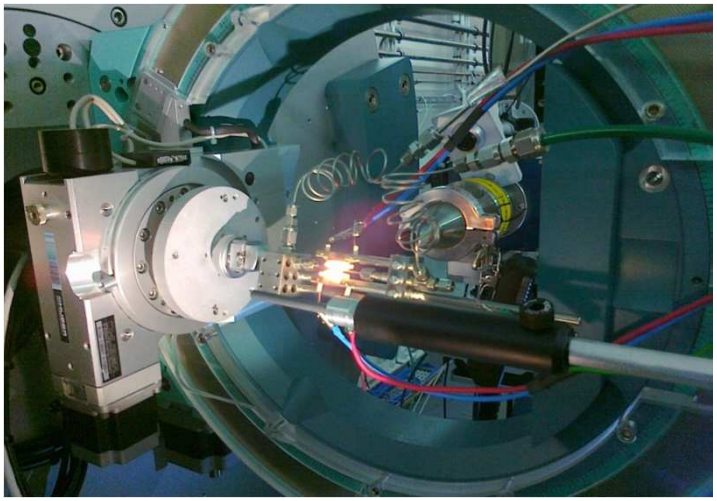
First PD User experiment (October 2, 2012), Malaga University Group
Hydration of eco cement (Miguel A.G. Aranda[⊥,±], Ángeles G. De la Torre*[⊥])

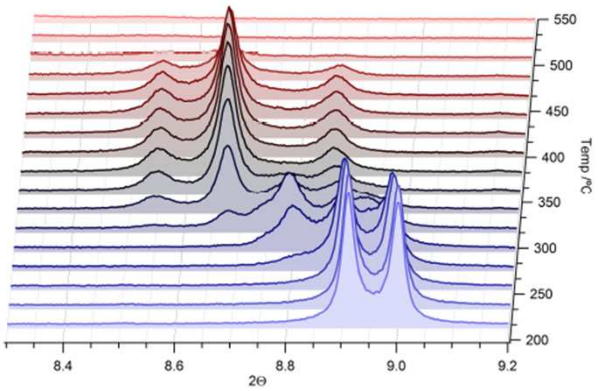




Gema Álvarez-Pinazo[⊥], et al submitted

When high throughput is useful





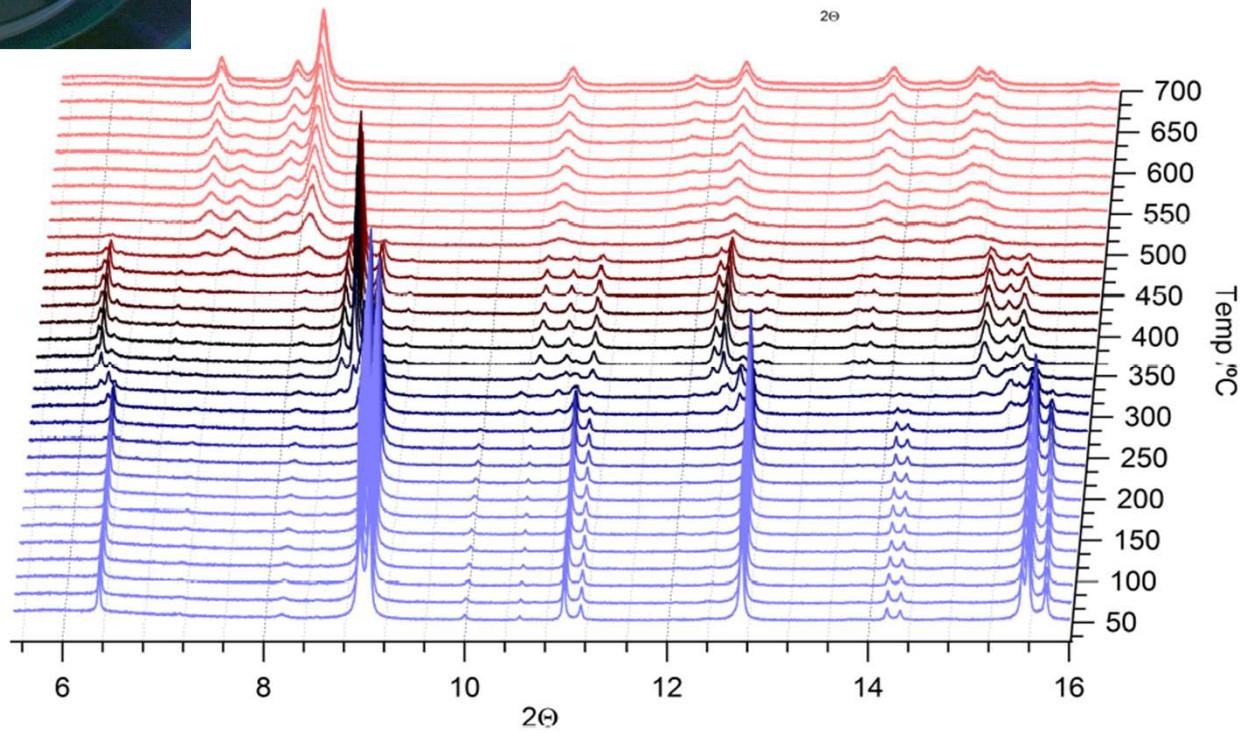
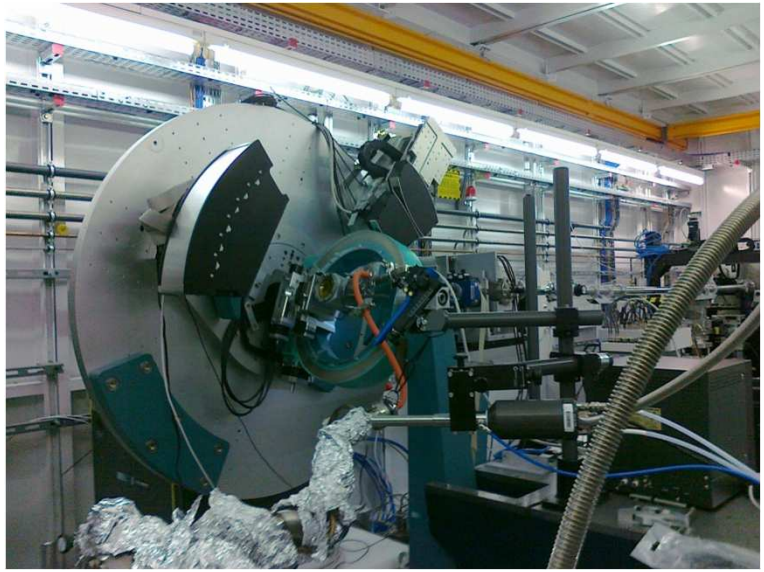
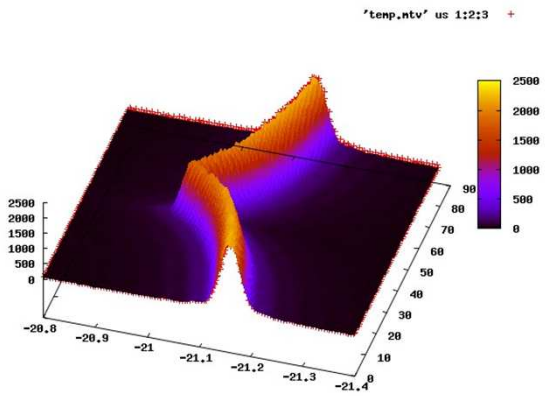


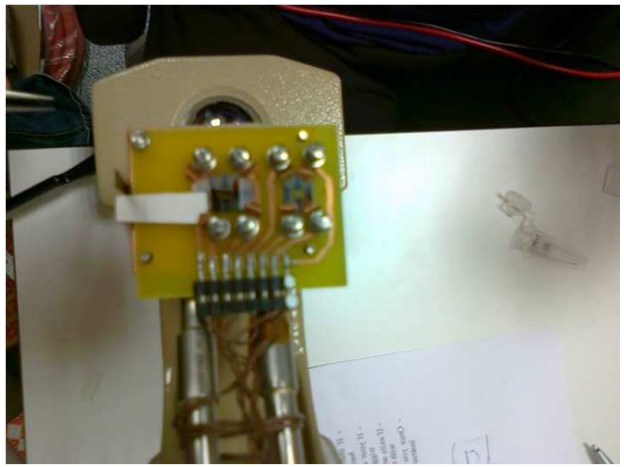
Fig 2 Reduction of LaCoO3 to La2O3 + Co under 5%H/He gas flow. The transition is passing through a brownmillerite intermediate. Each temperature scan was collected in 8 minutes.

When high throughput is useful





Pt peak on thin film heated up to 800 C
And down to RT at 15deg/sec 0.5 sec/frame



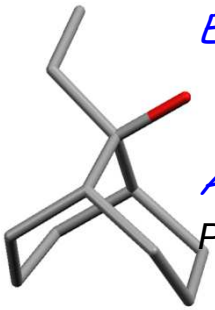
When high angular resolution is needed,…but temperature helps

Brunelli, Fitch, Mora, Vaughan and Wright,

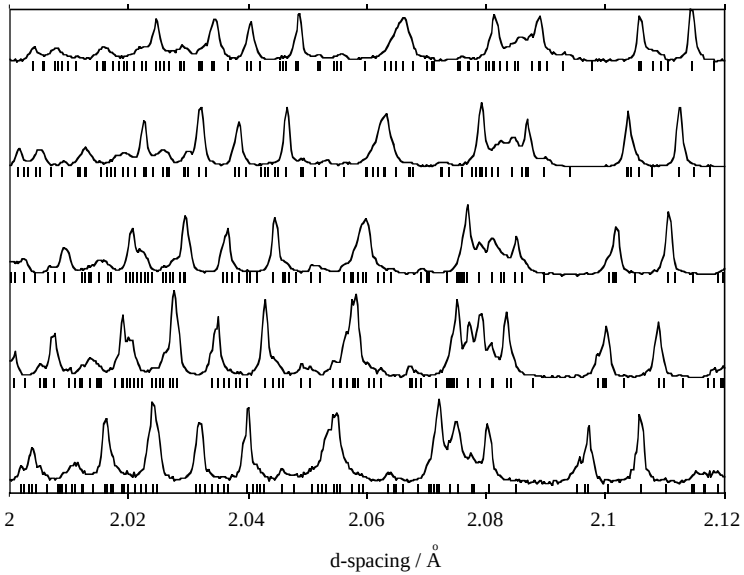
Structure determination by direct methods often limited by severe peaks overlap at high angle (d~1 1.2 Å) (e.g. big unit cell, low symmetry)

Idea: Exploit anisotropic expansion of cell parameters with temperature
Severe peaks overlap at T_1 resolved at T_2

-> 4 molecules and 48 (C+O) non-hydrogen atoms in he asymmetric unit
(attempt on BM16-PD failed, record 32 (C+O) non-hydrogen atoms in the asymmetric unit)



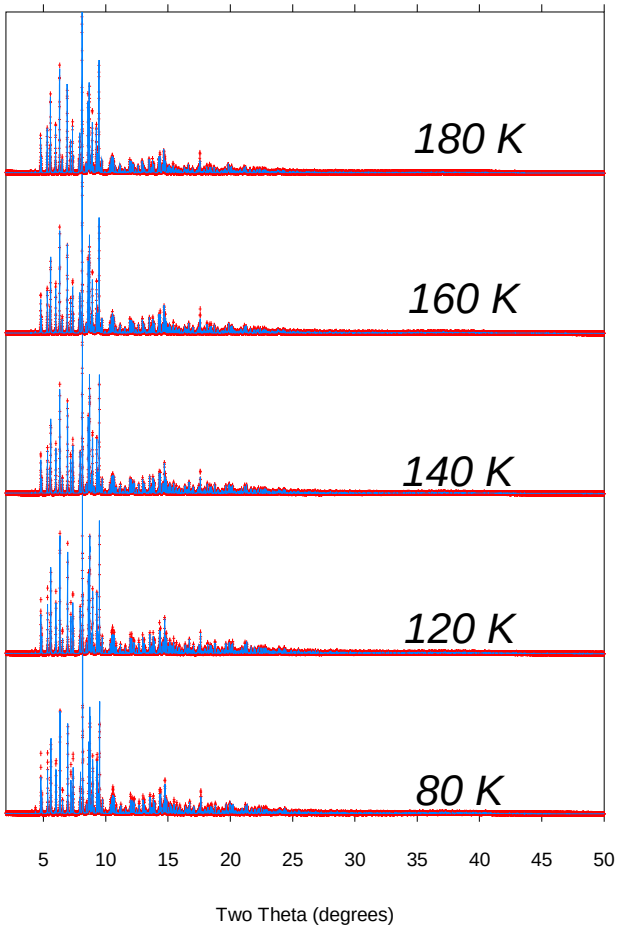
Example: Small molecule crystallography
9-ethyl-bicyclo[3.3.1]nona-9-ol
$C_{11}H_{20}O$
Amorph at high T, well cristallized below RT
Previously indexed in Pbca
a=21.02 Å, b=23.04 Å, c=16.38 Å

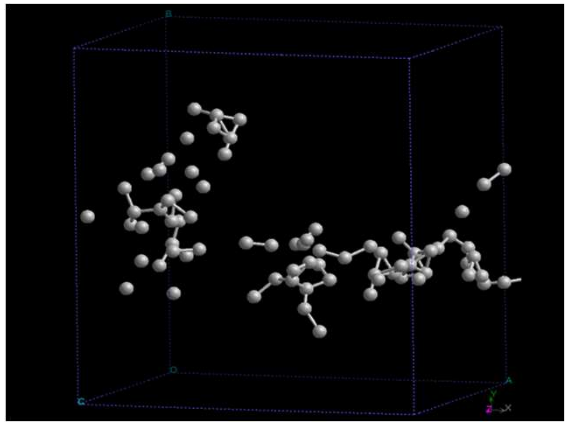


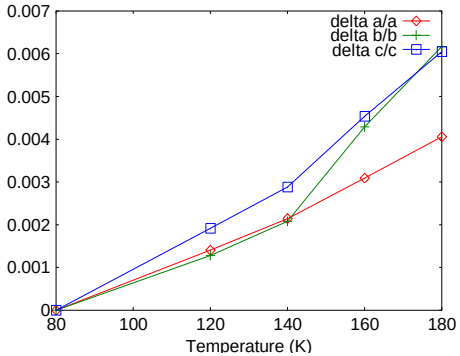
When high angular resolution is needed,…but temperature helps

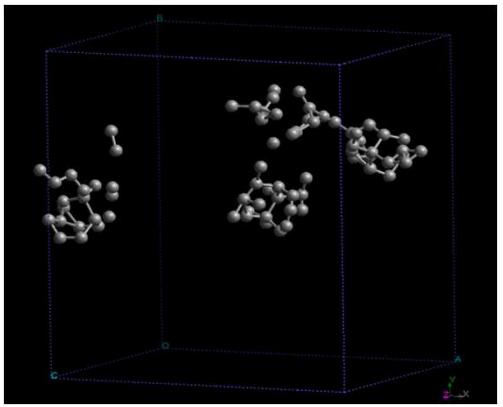
3 molecules identified from initial shelxs solution (direct methods) when 5 patterns were used.

First attempt with 2 datasets (80K, 120K) and feeding intensities to shelxs direct methods failed







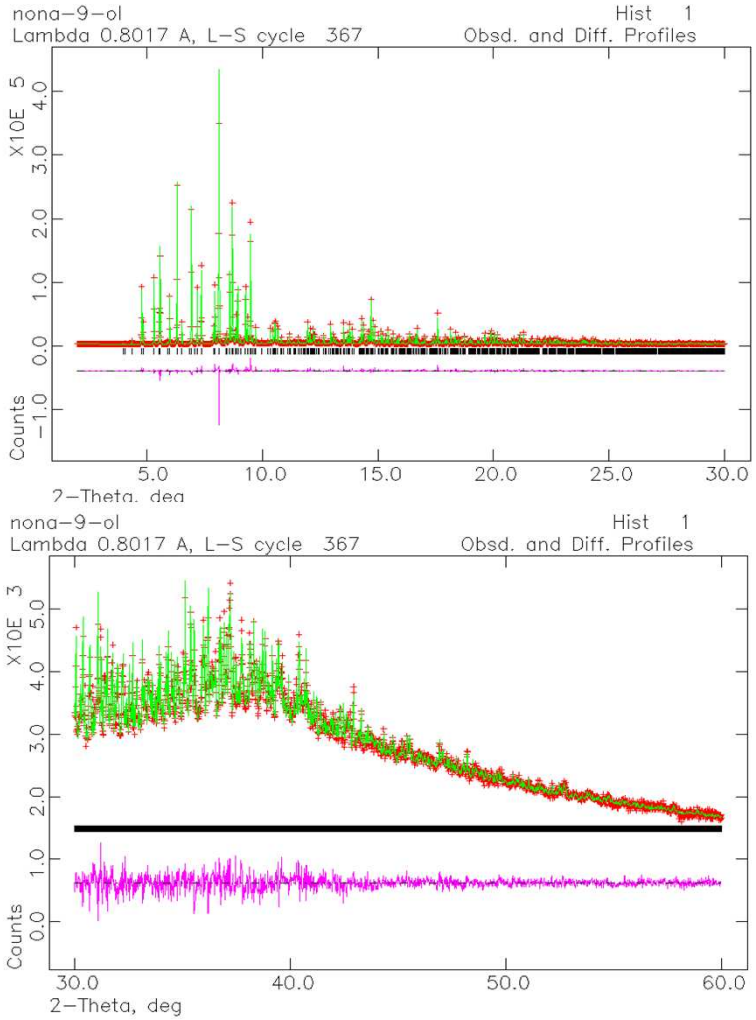


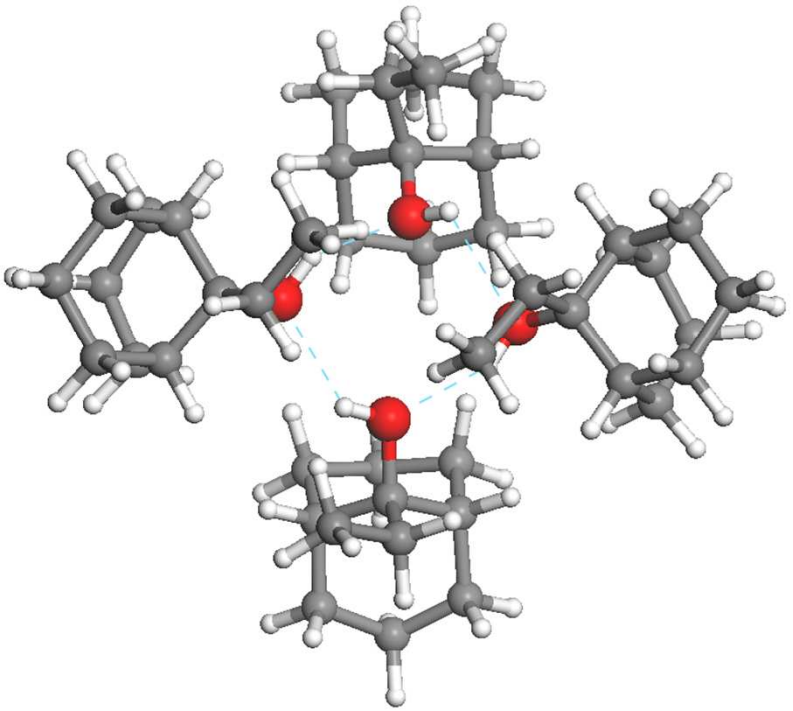
Structure completed by Fourier recycling against the extracted intensities from the 5 patterns

When high angular resolution is needed,…but temperature helps

Refined model against 140 K data
with stereochemical restraints (GSAS)

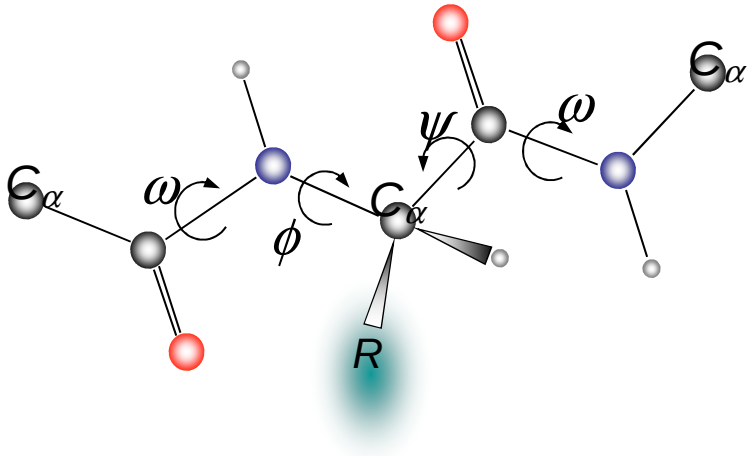
R_{wp}=5.6% R_p=4.7%





Synchrotron : Powder Methods + Proteins?

Myoglobin from horse skeletal muscle (Aldrich) recrystallised in 80% sat. $(NH_4)_2SO_4$



R. B. Von Dreele, "Combined Rietveld and stereochemical restraint refinement of a protein crystal structure." J. Appl. Cryst. ***32****, 1084-1089 (1999).*

*R. B. Von Dreele, "Binding of N-acetylglucosamine to chicken egg lysozyme: a powder diffraction study." Acta Cryst. D****57****, 1836-1842 (2001).*

J. Wright, I Margiolaki, ESRF

Samples easier to produce… but,
Large unit cells
Severe peak overlap
Many degrees of freedom in the structure

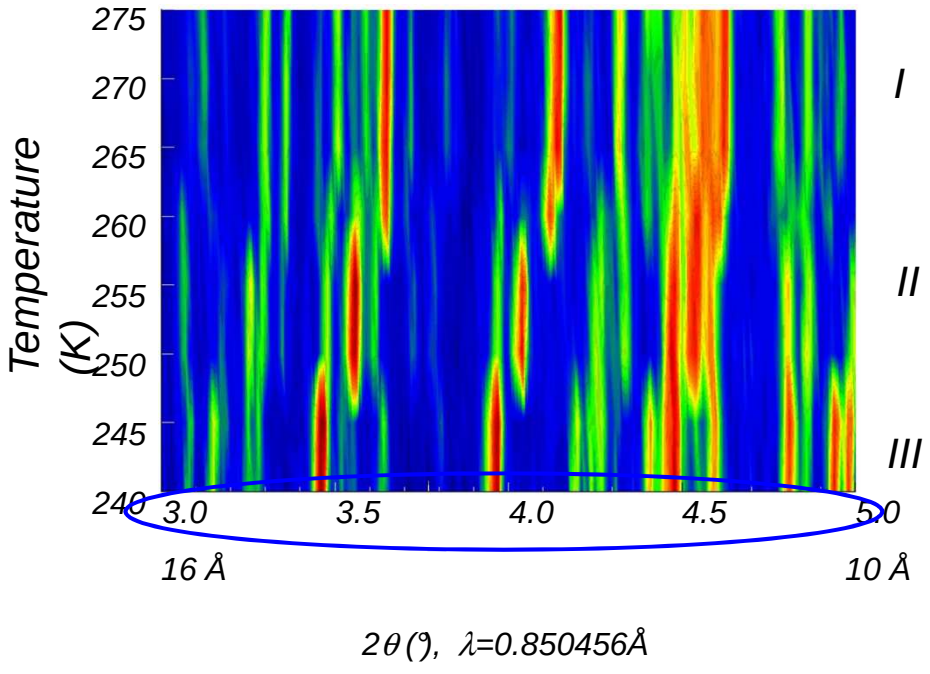
Fundamental differences to small molecule crystallography:

- *Well known geometric constraints (polypeptide chains)*
- *only ϕ, ψ and sidechain conformations which are unknown*

Improving the data quality…

Lower temperatures for better data quality (B factor & radiation damage)

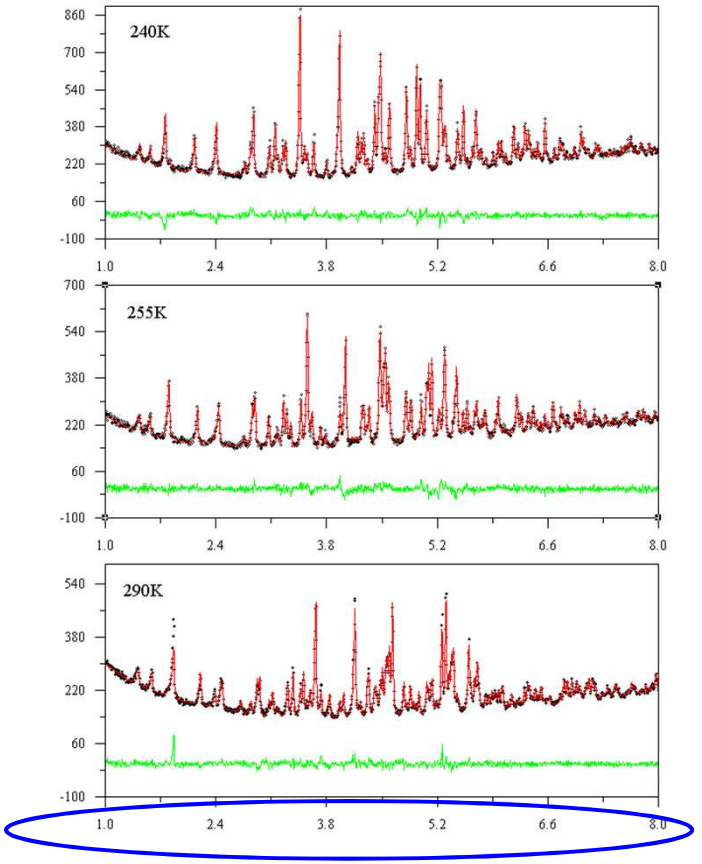
Exploit anisotropic thermal expansion to alleviate peak overlap



Combined fit for the 3 phases

Multipattern fit with the same intensities for each

Cell parameters and peak shape different for each pattern (indexed with DICVOL)



!! Small angular range !!

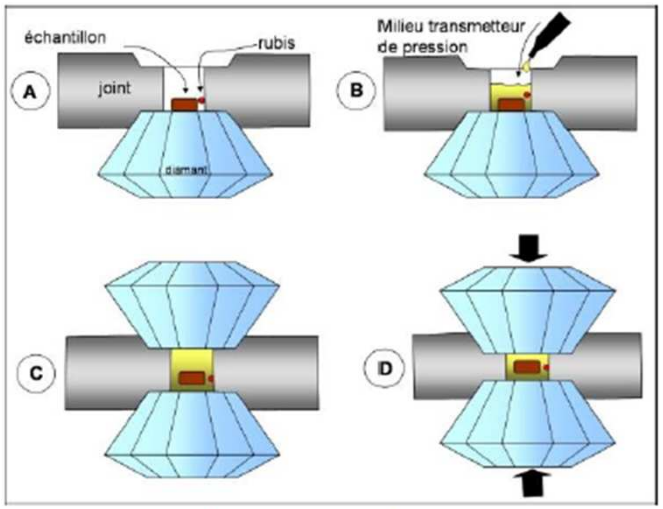
When small spot size is useful

Why using small spot size ?

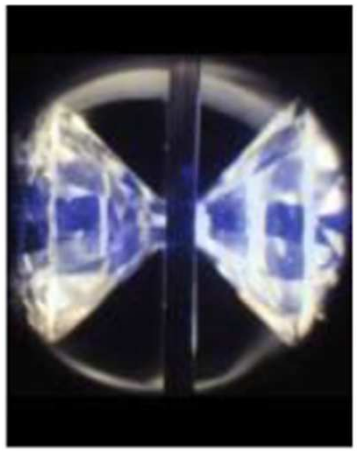
To illuminate a small part of the sample (spatial resoultion)

-> Useful for mesh mapping, e.g. cultural heritage samples
(e.g. determine from crystal structure different pigments used in paintings)
-> strain scanning experiments (engineering)

-> unavoidable in High Pressure experiments
(small samples -> high pressure, indeed Pressure = Force / Surface)



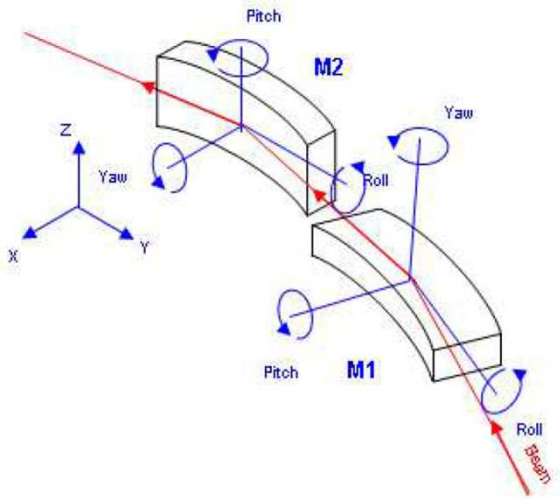
http://www.ens-lyon.fr/LST/HP



Incoming beam,

High energy to pass Through the dimonds

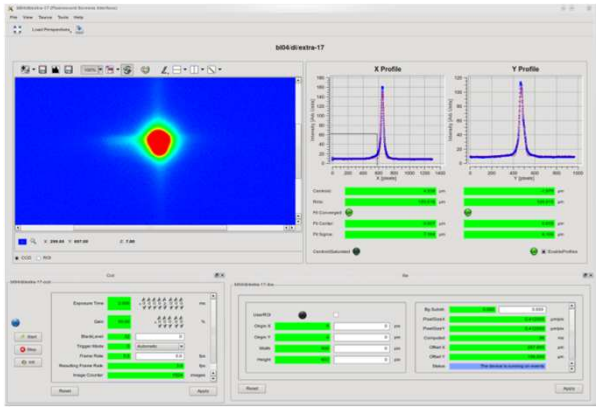
When small spot size is useful

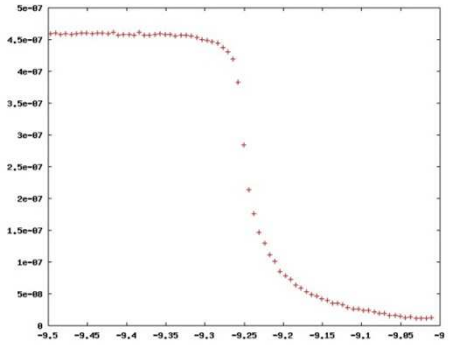


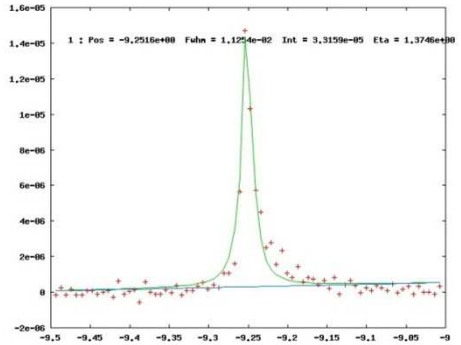
Kirkpatrick-Baetz focusing optics – Irrelec (ESRF licence), France

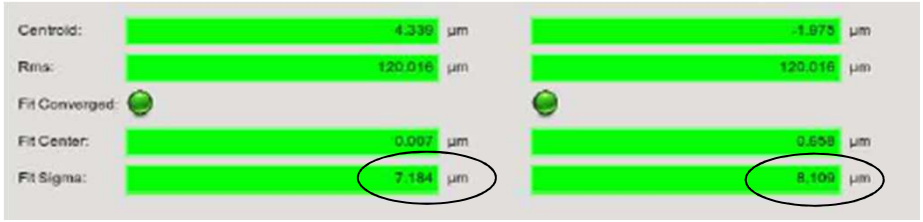
-Elliptical bendable,
-Grading [W/Si] multilayer :
Vert : p/q= 29.9/1.1, $[W/Si]_{100}$, d~3nm, slope error < 1 µrad
Hor : p/q= 30.3.9/0.7, $[W/Si]_{150}$, d~2.6nm, slope error < 1.5 µrad
-Focus spot of 12 µm (H) x 7 µm (V) at sample position over 20-50 keV

KB optimized to focus at the HP station, but designed to focus at the PD station as well









-> 10 X 10 µm beam

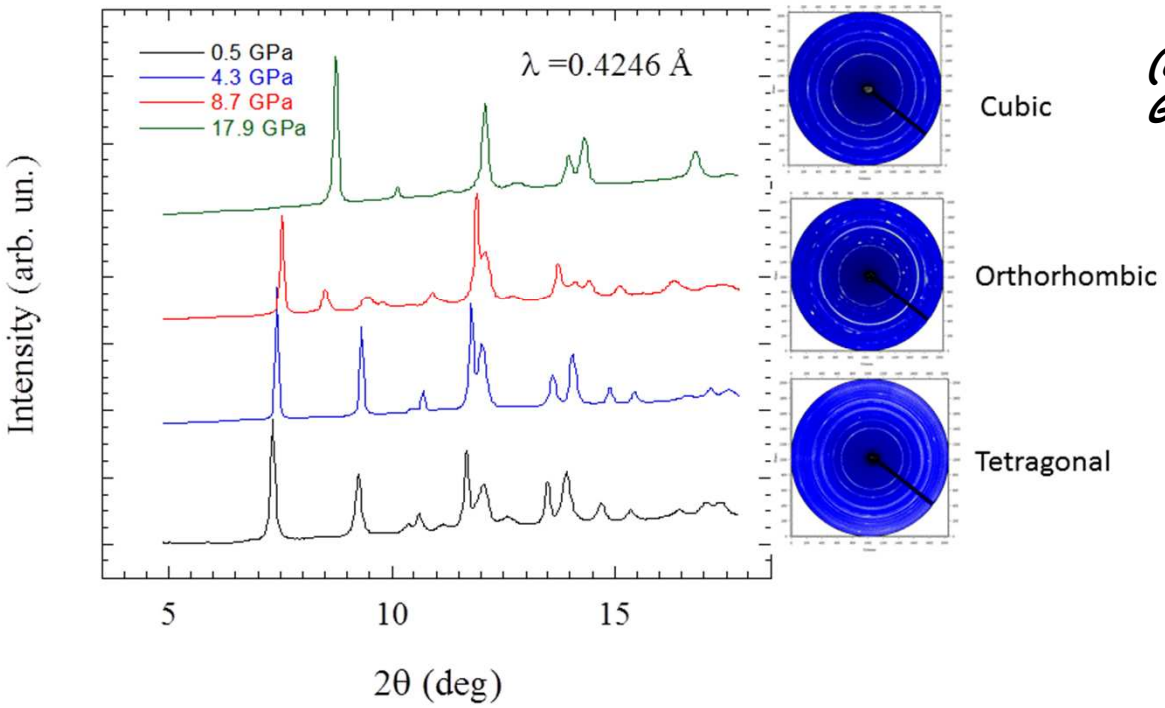
When small spot size is useful

First HP User experiment (June 27, 2012), Cantabria University Group

Pressure-induced structural-phase transition in $[(CnH_{2n+1})_4N]FeX_4$ (n=1,4; X: Cl,Br) associated with changes of Fe coordination.

Powder diffraction patterns versus pressure

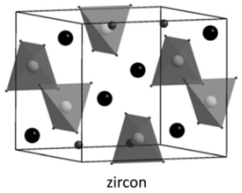
Pressure-induced phase transition sequence:
Tetragonal → Orthorhombic → Cubic

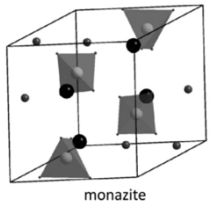


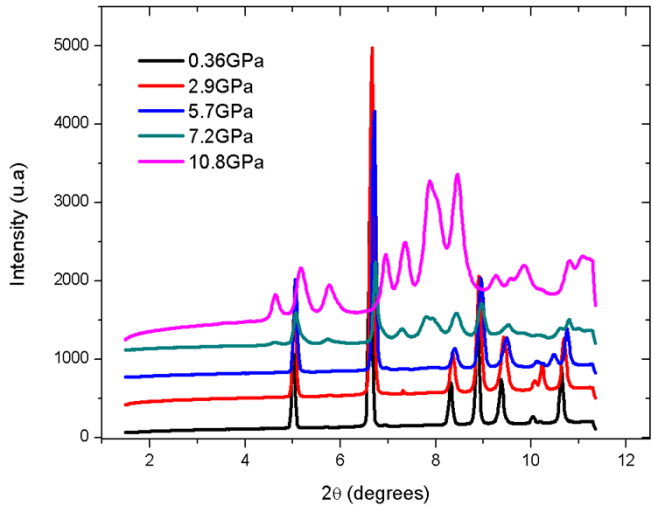
2D raw data to 1D patterns using A Hammersley's FIT2D
http://www.esrf.eu/computing/scientific/FIT2D

(evaluate possibility to use GSAS2 package)

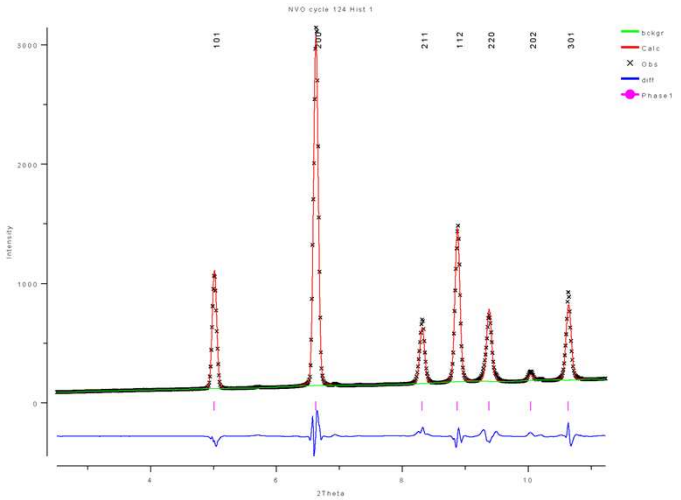
Zircon to monazite phase transition in NdVO4
(I41/amd, Z=4) > Monazite (P21/n, Z=4)

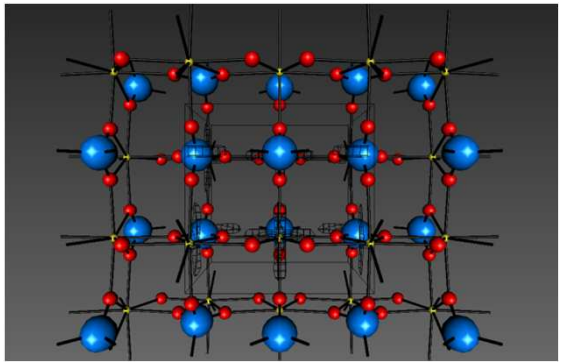






Evolution of the XRD patterns of NdVO4 as a function of pressure



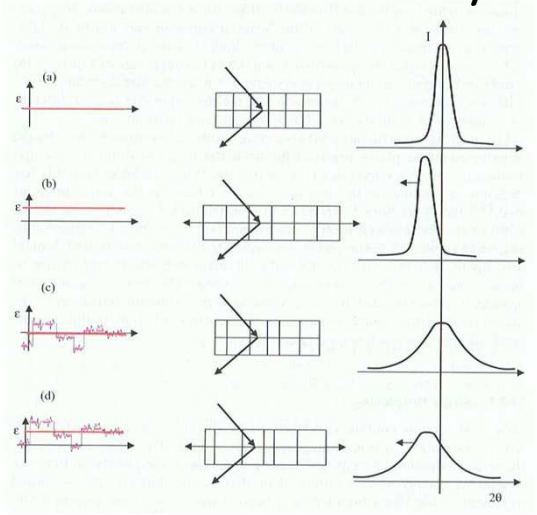


3D difference Fourier plot (GSAS)

Why high resolution and high accuracy ?

Sometimes the change in position are very tiny (e.g. change in temperature)

Strain ε lattice peak shape/shift

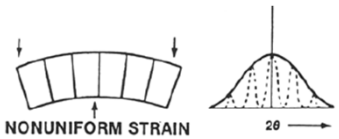


No strain

Macrostrain (e.g. tensile strain)

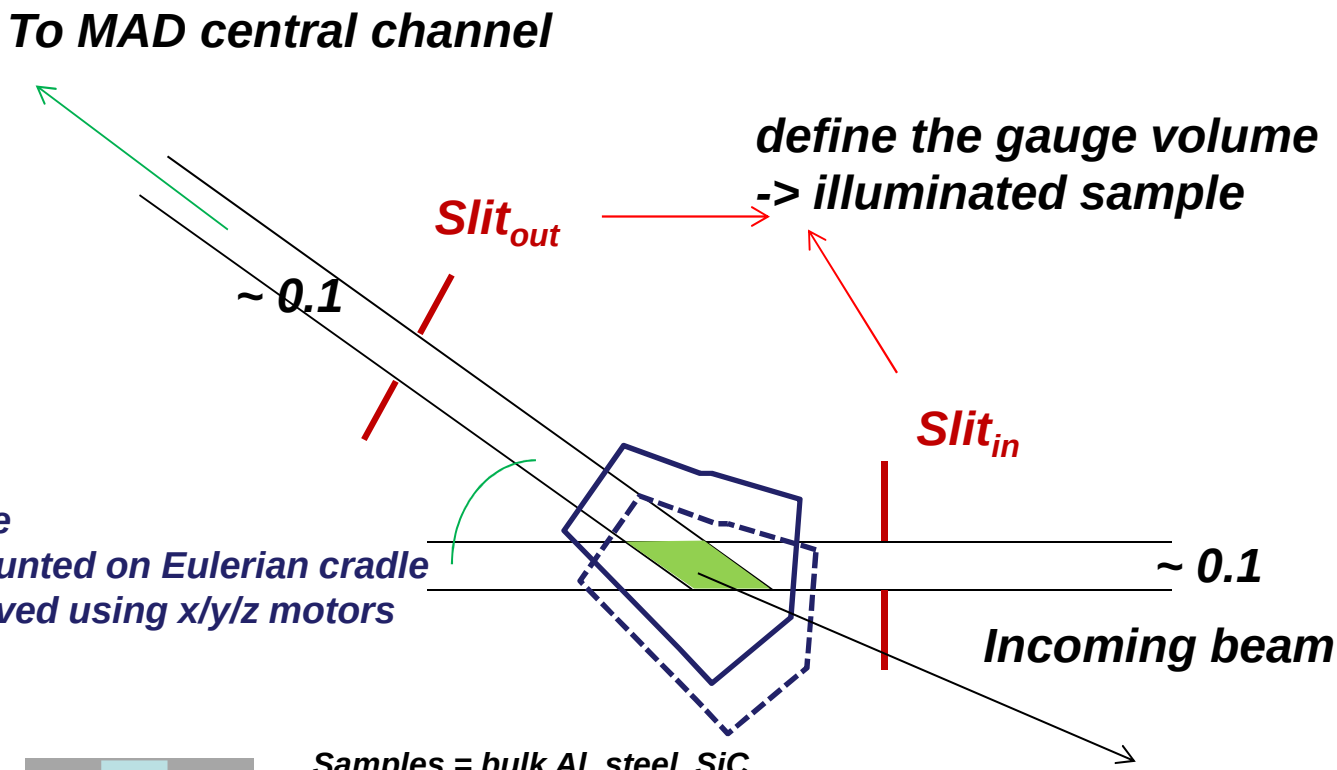
Microstrain

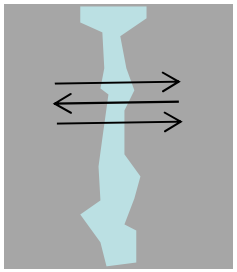
Macrostrain + Microstrain



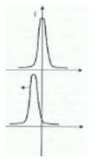
In a 'normal' powder without external forces no macrostrain is seen.
However, microstrain is important e.g in Metallurgy

When small spot size is useful





Samples = bulk Al, steel, SiC
-> high energy



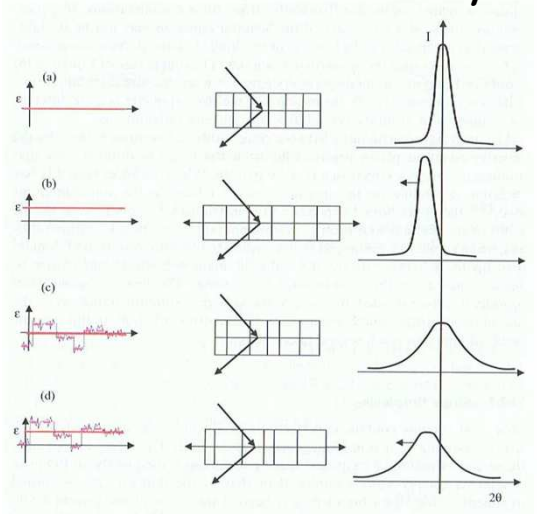
The smaller the gauge volume,
the better resolution
but no focusing otherwise
angular resolution is spoiled

2 pieces welded by verious techniques
-> goal is to have compressive strains at welding points

Why high resolution and high accuracy ?

Sometimes the change in position are very tiny (e.g. change in temperature)

Strain ε lattice peak shape/shift

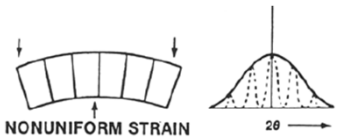


No strain

Macrostrain (e.g. tensile strain)

Microstrain

Macrostrain + Microstrain



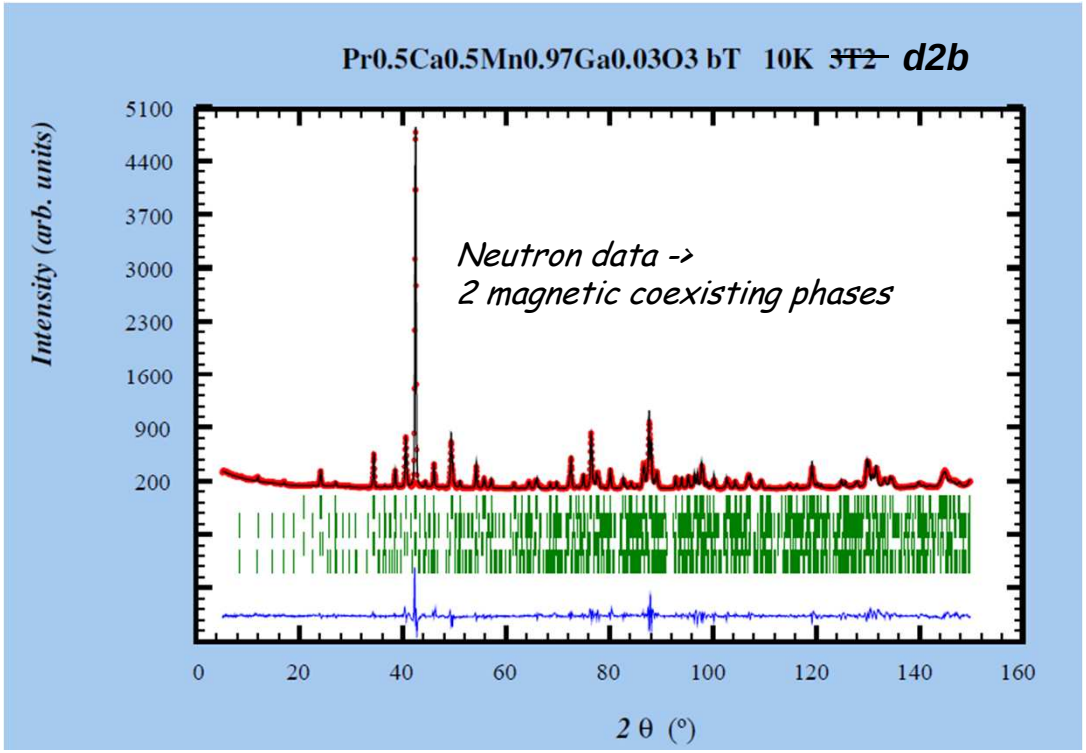
In a 'normal' powder without external forces no macrostrain is seen.
However, microstrain is important e.g in Metallurgy

When high angular resolution is needed…for microstructural effects

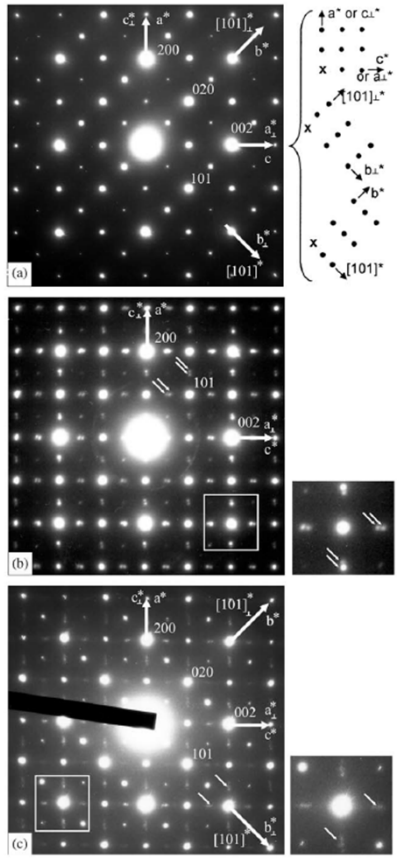
$Pr_{0.5}Ca_{0.5}Mn_{0.97}Ga_{0.03}O_3$, a strongly strained system due to the coexistence of two orbital ordered phases at low temperature

C. Yaicle[a,*], F. Fauth[b], C. Martin[a], R. Retoux[a], Z. Jirak[c], M. Hervieu[a], B. Raveau[a], A. Maignan[a]

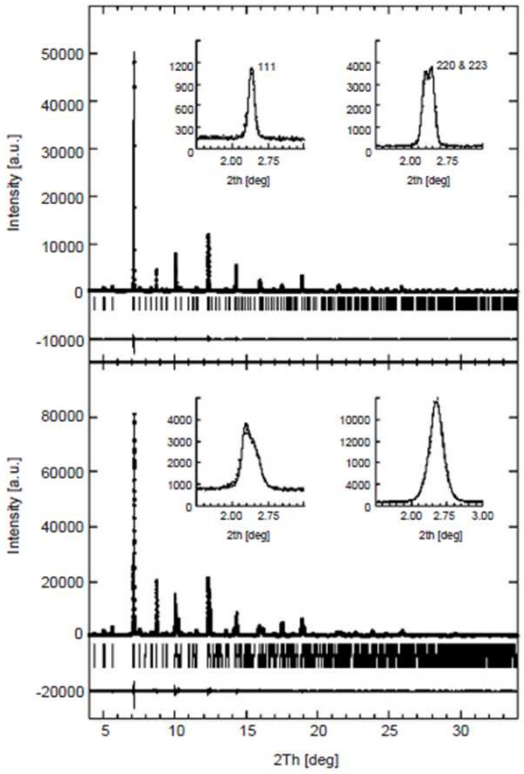
[a]*Laboratoire CRISMAT, UMR 6508CNRS ENSICAEN, 6 bd Maréchal Juin, 14050 CAEN Cedex, France*
[b]*ESRF, BP 220, 6 Rue Jules Horowitz, F-38043 Grenoble, France*
[c]*Institute of Physics ACSR, Cukrovarnicka 10, 162 53 Prague 6, Czech Republic*

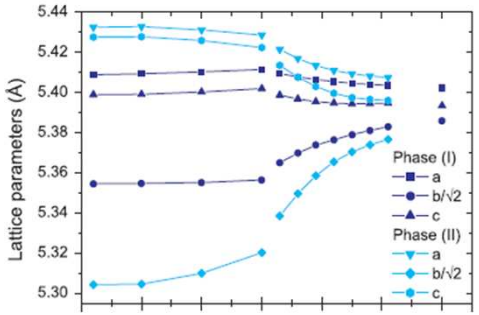


Elctron diffraction data ->
2 crystal coexisting phases



When high angular resolution is needed…for microstructural effects

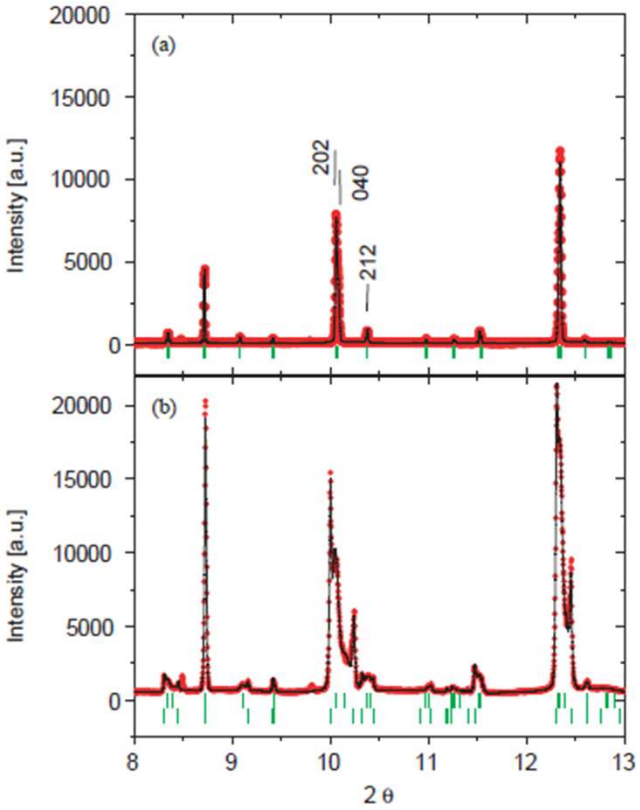




Anisotropic line broadening, using Stephens formalism

$$\Gamma^S(hkl) = \sqrt{\sigma^2(1/d^2_{hkl})}\,\tan(\theta)/(1/d^2_{hkl}),$$

$$\sigma^2(1/d^2_{hkl}) = \sum_{\substack{ij \\ \alpha_i=a,b,c,\alpha,\beta,\gamma}} \langle(\alpha_i - \langle\alpha_i\rangle)(\alpha_j - \langle\alpha_j\rangle)\rangle \times \frac{\partial(1/d^2_{hkl})}{\partial\alpha_i}\frac{\partial(1/d^2_{hkl})}{\partial\alpha_j} = \sum_{\substack{HKL \\ H+K+L=4}} S_{HKL}h^H k^K l^L.$$



The anisotropic line broadening reflects the local structural distortions observed by electron diffraction/ microscopy

Conclusion

They are as many types of synchrotron powder diffraction experiments as users' ideas

Make use of wide range of energies, high resolution, high throughput, small beam size, heavy duty diffractometers,
big space around sample position, low/high temperatures,
high pressures, gas flow,…

Make use of available complementary techniques :
Neutron powder diffraction, electron diffraction/microscopy,
transport properties (resistivity, DSC), chemical analysis

Sorry for not having mentioned Pair Distribution Function type data acquisition