INITIAL TRAINING NETWORK
RAINBOW

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X-RAY DIFFRACTION : an overview of the technique

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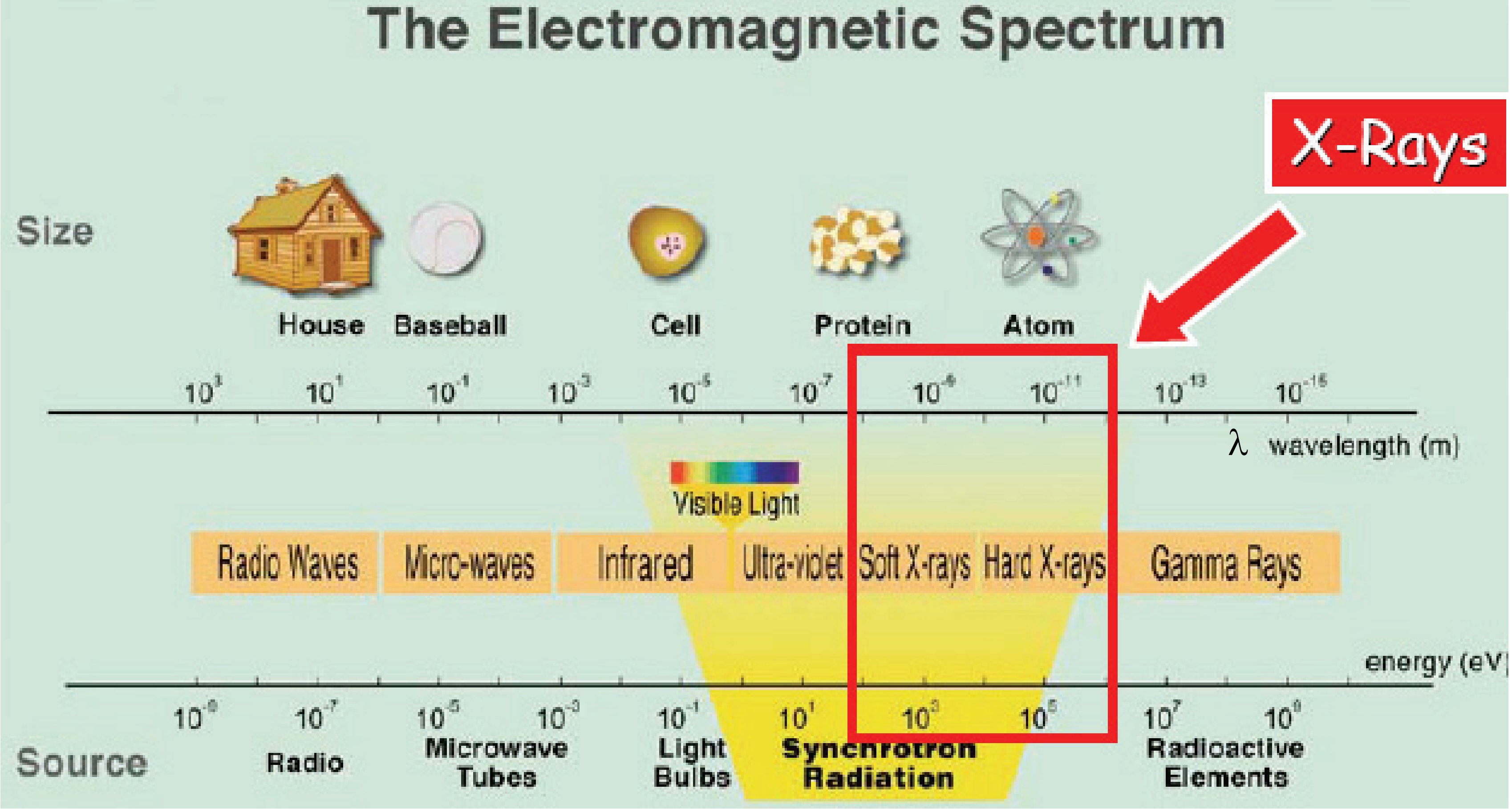
CiMap

OUTLINE :

- * Electromagnetic properties of X-rays
- * Production of X-rays
- * Interactions of X-rays with matter
- * X-ray diffraction : investigation of the fine structure of the matter
- * Some basics of crystallography
- * X-ray diffraction by crystals
- * Summary

X-rays = Electromagnetic (EM) radiations lying between Ultraviolet light and Gamma rays in the electromagnetic spectrum

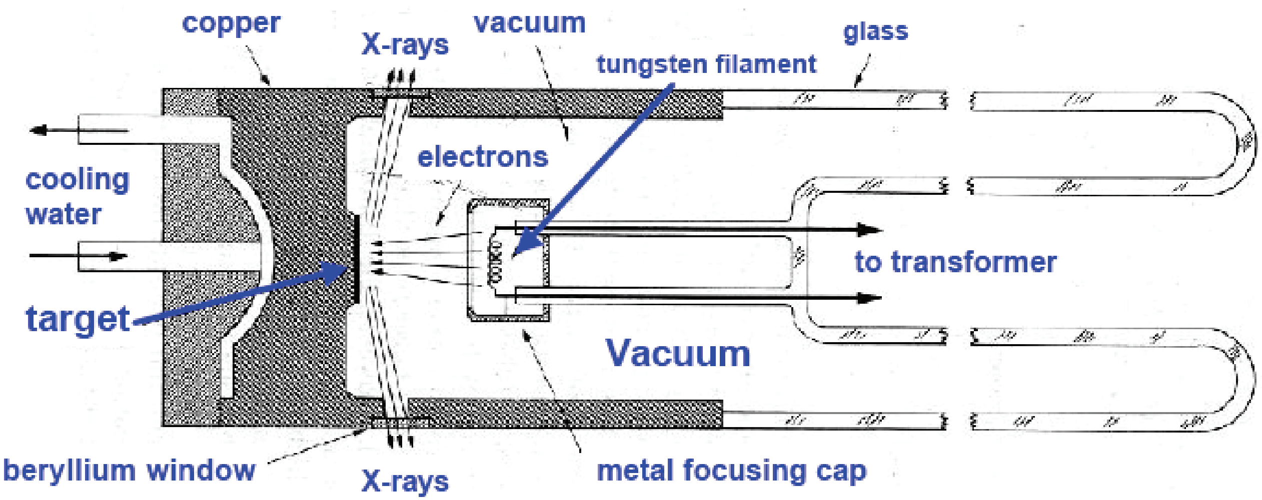


- **Associated particle = photon (no charge, no mass) ; photon energy** $E = \frac{hc}{\lambda}$

- **X-rays are characterized by relatively short wavelength : $\lambda = 0.01\text{-}100$ Å (10^{-10}-10^{-6} cm), with Hard X-Ray on one hand and Soft X-Ray on the other.**

X-ray production = X-ray tube

X-rays are conventionally produced by either conversion of kinetic energy of charged particles into radiation (continuous X-ray spectrum) or by atom excitation in a metal target (Cu, Al, Mo, Mg) upon which fast moving electrons impinge.



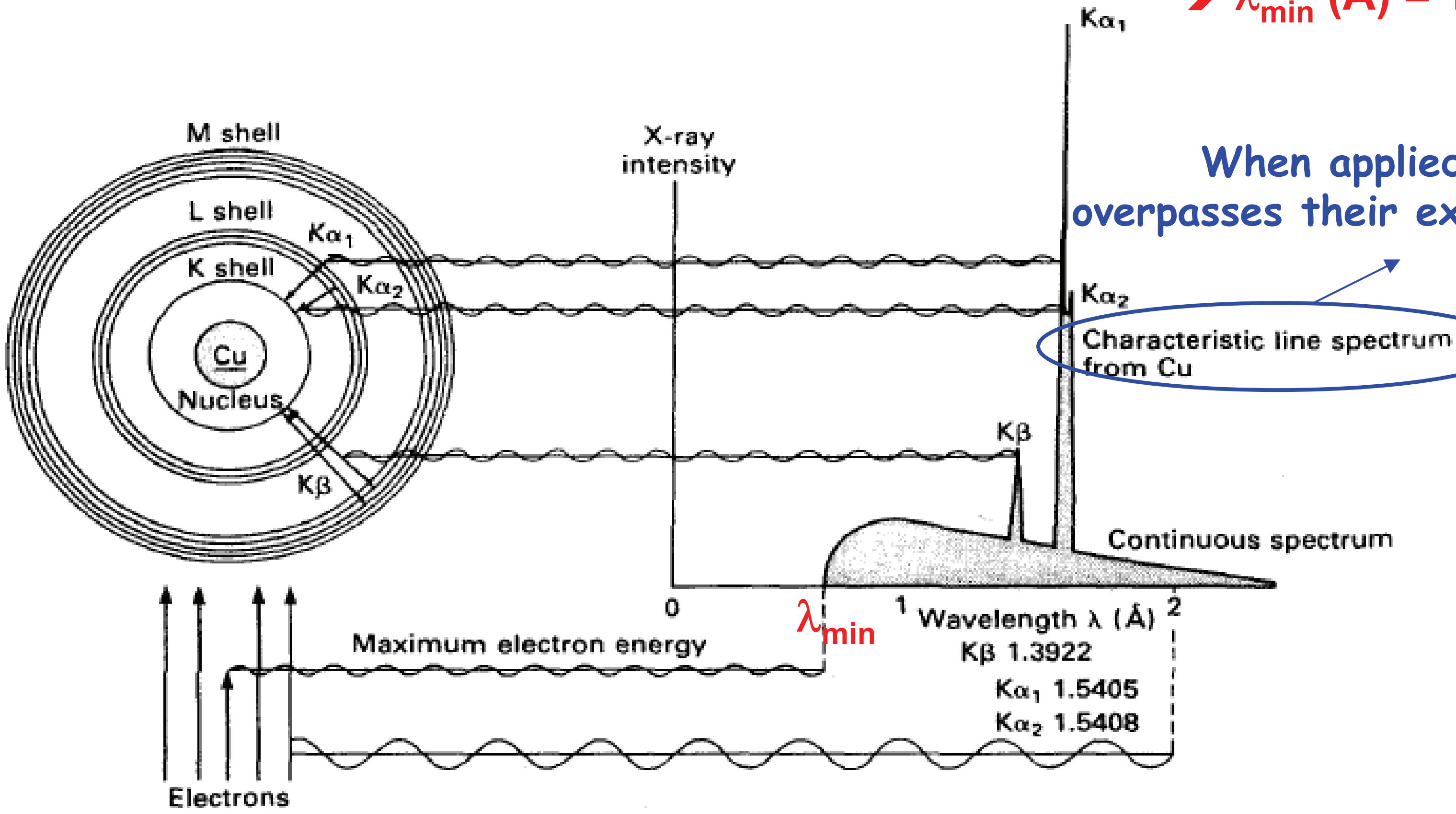
Sealed-off filament X-ray tube

- Anode is a water-cooled Cu block containing the desired target

- Source of electrons = hot W filament (Joule effect, I ~ 40 mA), accelerated by application of high accelerating voltage V (~ 45 kV) between cathode (W) and anode (target).

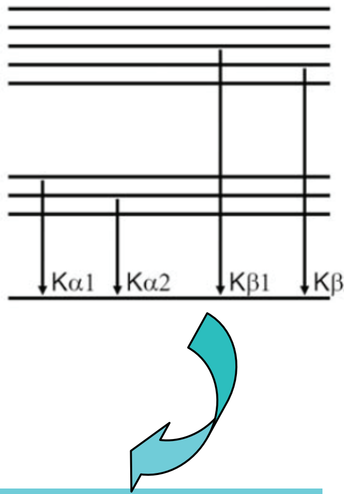
Continuous spectrum generation = electron deceleration in the target (complete or partial electrons collision)

➔ λ_{min} (Å) = 12.4 / V (kVolts)



λ_{min}

When applied voltage V overpasses their excitation threshold



Bombarding electrons can eject electrons from the metal target atom inner shells: vacancies will be quickly filled by electrons dropping down from higher levels, emitting x-rays with sharply defined frequencies associated with the difference between the atomic energy levels of the target atoms.

Refractive index of X-rays :

$$n = 1 - \delta - i\beta$$

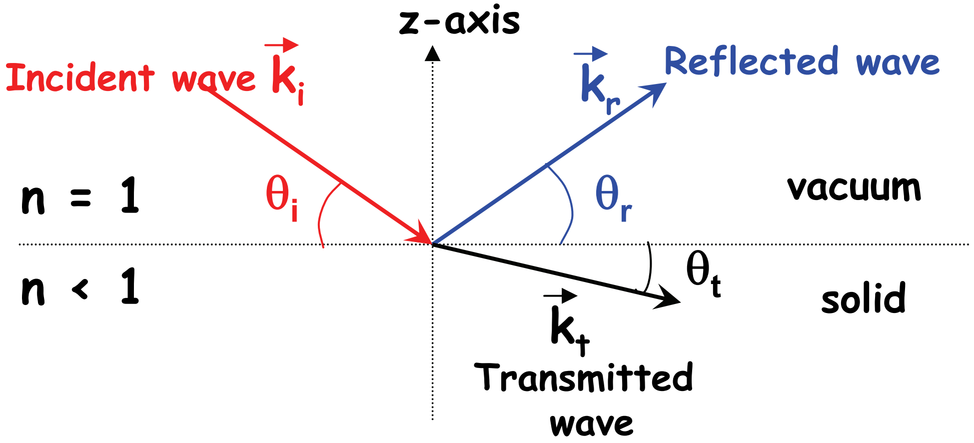
$$\delta = \frac{r_e}{2\pi}\lambda^2\rho_e$$

dispersion

$$\beta = \frac{\lambda\mu}{4\pi}$$

absorption

r_e: classical radius of the electron (0.28 10^{-5} nm)
ρ_e: electron density of the material
μ : linear absorption coefficient



Typically δ: 10^{-5} to 10^{-6}, and $\beta \sim \delta/10$;

$\delta > 0$, *i.e.* n < 1 but slightly
➔ total reflection possible if :

$$\cos\theta_c = n = 1 - \delta \approx 1 - \frac{\theta_c^2}{2}$$

➩ At low angle (~ 0.1° to 1°), X-ray beam totally reflected and propagation in material is evanescent (penetration depth : 2 to 3 nm) : surface study + reflectivity

➩ At higher angle : penetration depth of some tens of µm thickness !
As n < 1 but slightly : X-ray linear propagation in the matter.

Interaction of X-rays with matter ($\theta >> 1°$)

X-rays mostly interact with atomic electrons ➔ 3 options:

1) Penetrate
2) Scatter = change in direction of electromagnetic waves with or not an energy exchange between photons and matter
3) Be absorbed (partially or totally)

5 basic interactions of X-rays with matter:

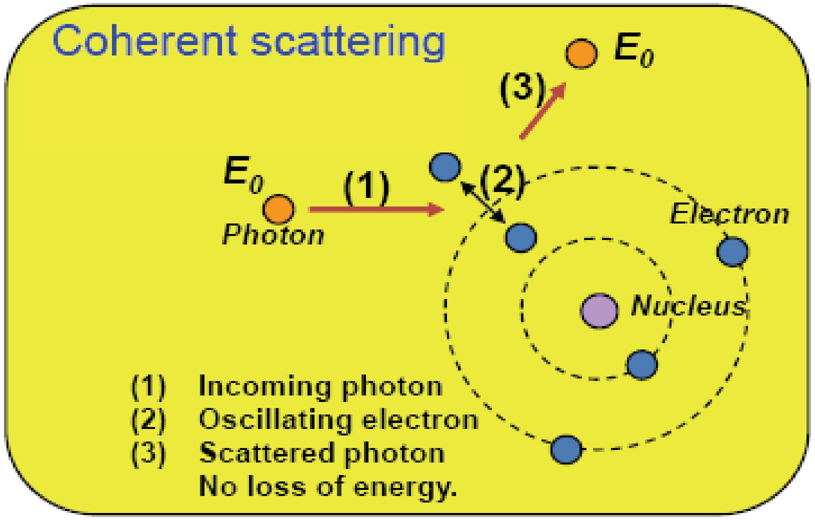
(different competiting processes that are involved simultaneously)

- Coherent scattering : Thomson diffusion (elastic) if $h\nu < E_{binding}$
- Incoherent scattering : Compton diffusion (inelastic) if if $h\nu >> E_{binding}$
- Photoelectric absorption effect if $h\nu \geq E_{binding}$
- ~~Pair production (electron – positron pair) : E > 1 MeV~~
- ~~Photodisintegration E > 10 MeV~~

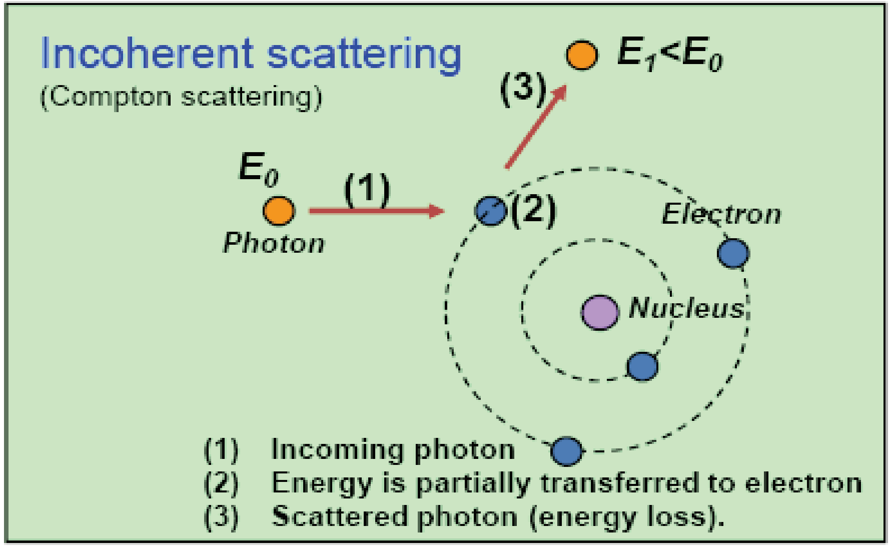
For 10 eV – 30 KeV :
pair production and photodisintegration are negligible

Interactions of X-rays with matter:

- **Coherent scattering** : Thomson diffusion (elastic) if $h\nu < E_{binding}$ (at low energy : Rayleigh diffusion)
- **Incoherent scattering** : Compton diffusion (inelastic) if if $h\nu >> E_{binding}$ (at low energy : Raman diffusion)

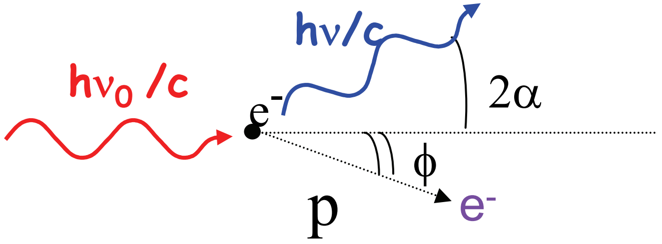

Coherent scattering

(1) Incoming photon
(2) Oscillating electron
(3) Scattered photon
No loss of energy.


Incoherent scattering
(Compton scattering)

(1) Incoming photon
(2) Energy is partially transferred to electron
(3) Scattered photon (energy loss).

Compton scattering energy loss :

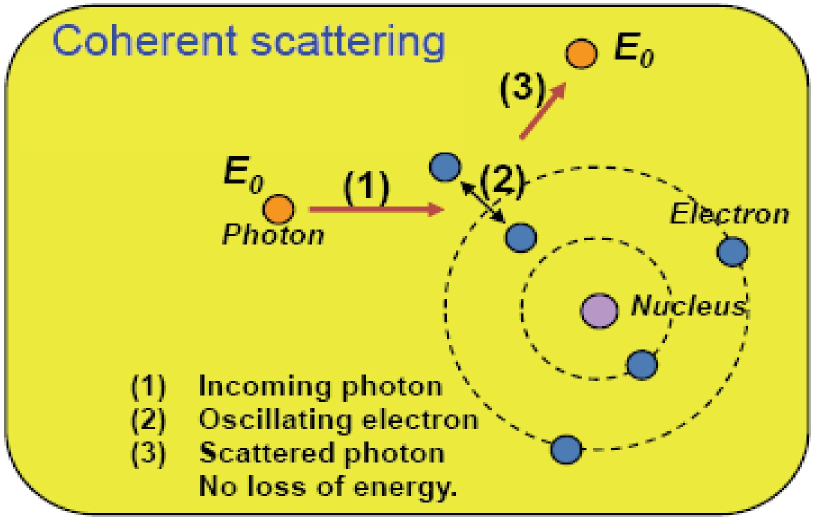


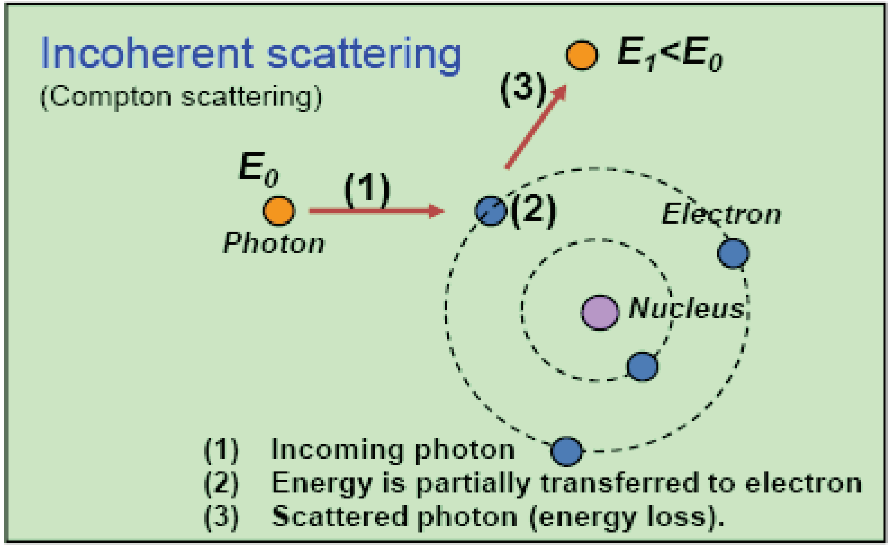
$$\lambda - \lambda_0 = \Delta\lambda = \frac{h}{m_0 c}(1 - \cos(2\alpha)) = 0.0242\ (1 - \cos(2\alpha))$$

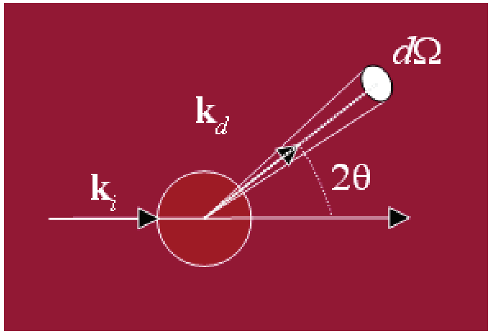
Interactions of X-rays with matter:

- **Coherent scattering** : Thomson diffusion (elastic) if $h\nu < E_{binding}$ (at low energy : Rayleigh diffusion)

- **Incoherent scattering** : Compton diffusion (inelastic) if if $h\nu >> E_{binding}$ (at low energy : Raman diffusion)


Coherent scattering


Incoherent scattering
(Compton scattering)



Some definitions :

$\vec{k_i}$ and $\vec{k_d}$: incident and diffuse scattering vectors

2θ = diffusion angle

Elastic scattering : $k_i = k_d$

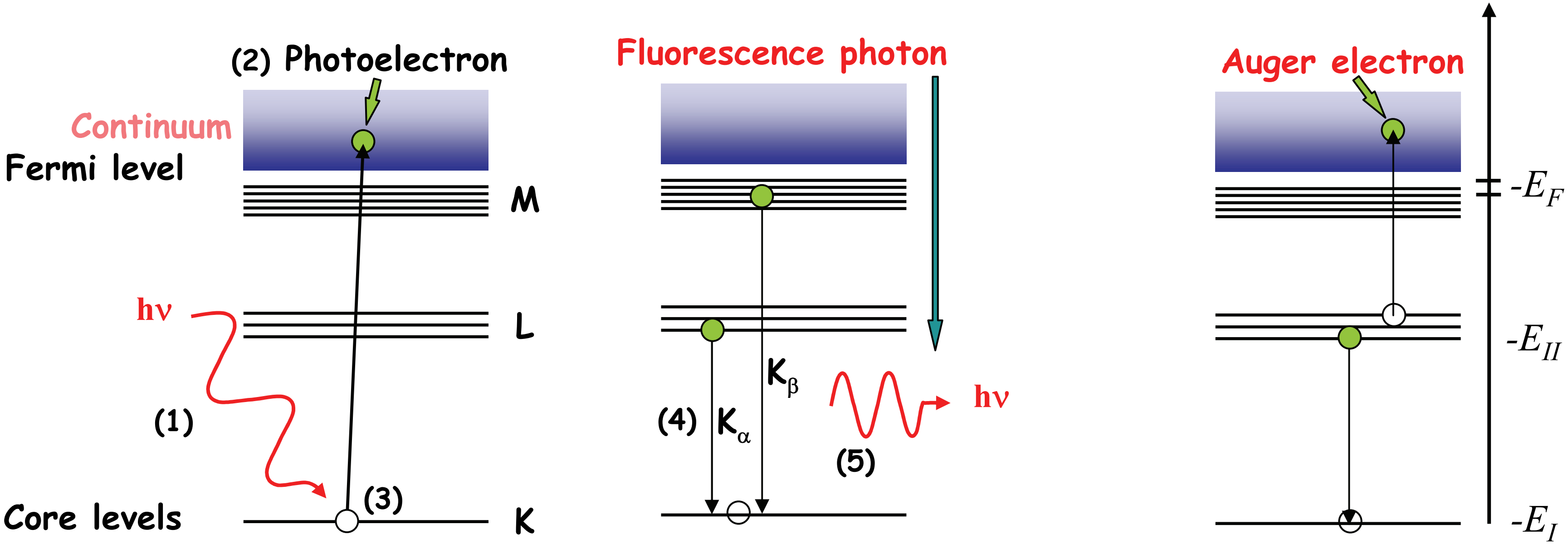
Inelastic scattering $k_i \neq k_d$

X – ray absorption

For E < 1000 keV photoelectric effect is dominant

- Photoelectric effect = photon absorption interaction
  - Absorbed photon if $h\nu > E_B$ (E_B electron binding energy)
  - Excitation : emitted photoelectron ($E = h\nu - E_B - \Phi$)

- Desexcitation mechanisms : fluorescence photon ($h\nu = E_I - E_{II}$)
  or Auger electron ($E = E_I - E_{II} - E_{III}$)



Excitation

Secondary X-ray = photon emission

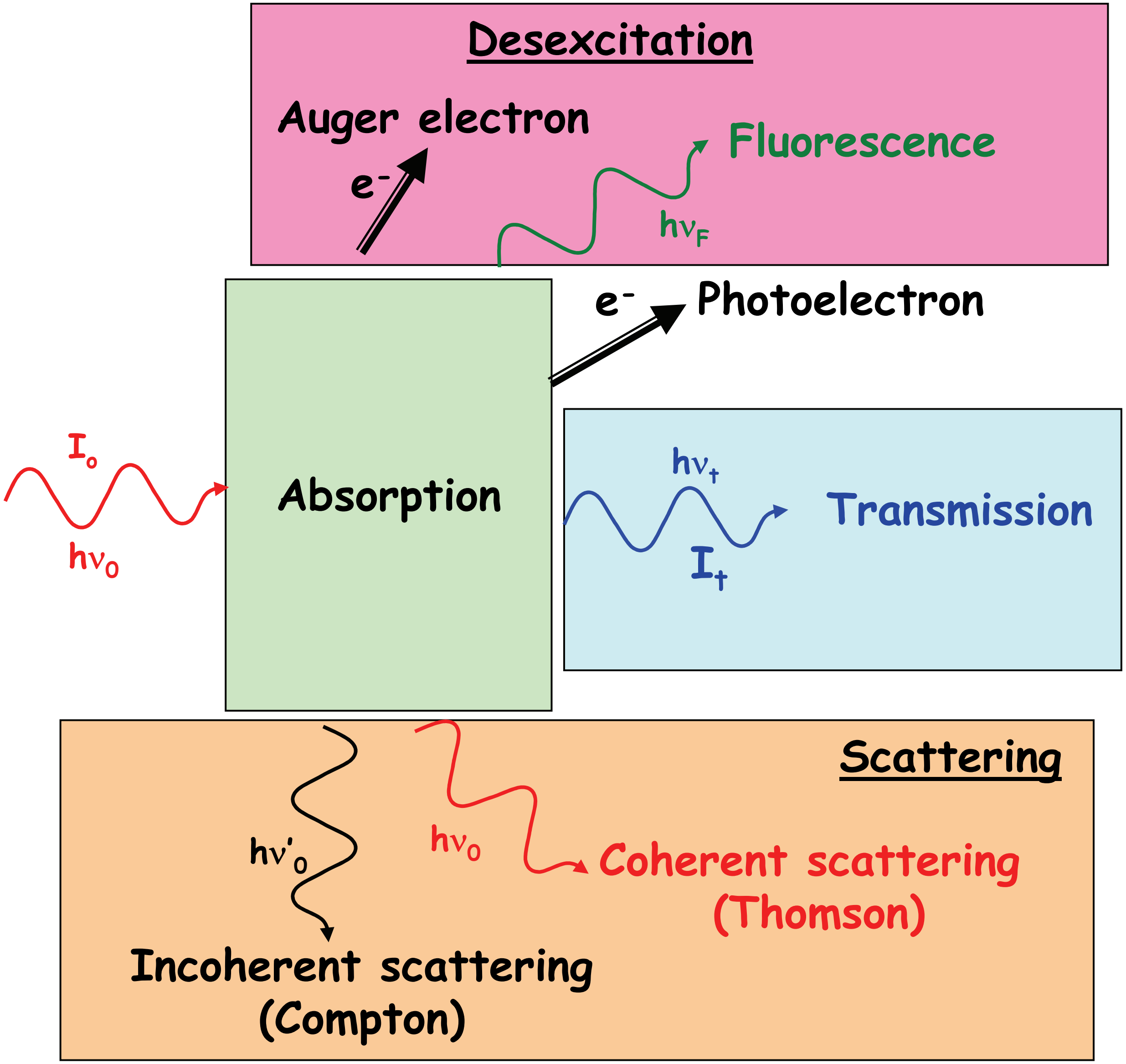
Electron emission from
The upper levels

(1) Incoming photon
(2) Expelled electron
(3) Hole is created in shell

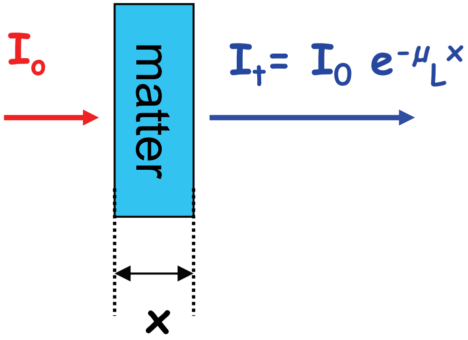
(4) Outer shell electron moves to inner shell hole
(5) Energy excess emitted as photon.

(4') Hole is occupied by outer e⁻
(5') Excitation energy transferred to e⁻
(6') Electron ejected from atom

Interaction of X-rays with matter



Absorption



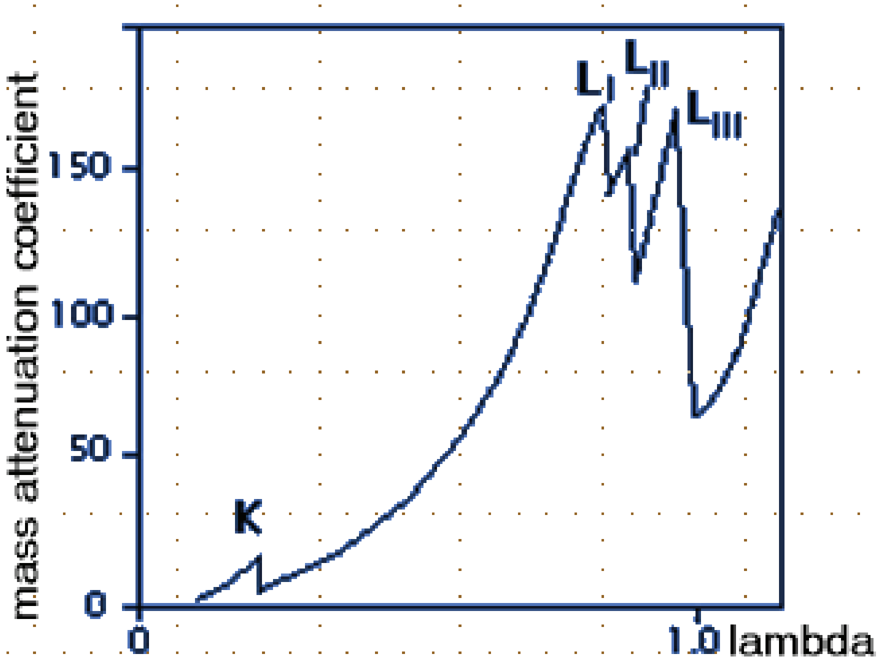
$$I_t = I_0\, e^{-\mu_L x}$$

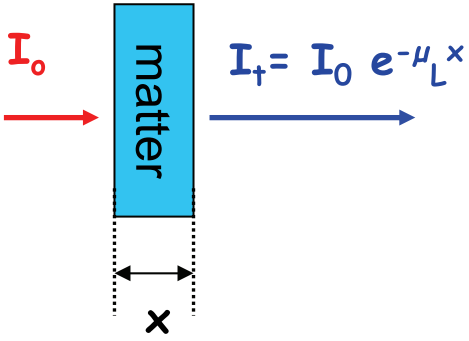
$$\mu_L = \text{linear absorption coefficient} = \left(\frac{\mu_L}{\rho}\right)\rho = \mu\rho$$

with ρ = volumic mass ($g.cm^{-3}$) and μ = mass absorption coefficient in $g^{-1}.cm^{-2}$ (tabulated in the International Tables of Crystallography)

$$I_t = I_0\, e^{-\mu\rho x}$$

Interaction of X-rays with matter





μ_L = linear absorption coefficient $= \left(\frac{\mu_L}{\rho}\right)\rho = \mu\rho$

with ρ = volumic mass ($g.cm^{-3}$) and μ = mass absorption coefficient in $g^{-1}.cm^{-2}$ (tabulated in the International Tables of Crystallography)

$$I_t = I_0\, e^{-\mu\rho x}$$

↳ Attenuation globally increases with λ and for a given λ, μ increase with Z.

↳ Absorption discontinuities (K, L_I, L_{II}, L_{III}) can be evidenced : they appear when energy of incident photon, $W = h.c/\lambda \geq W_K$

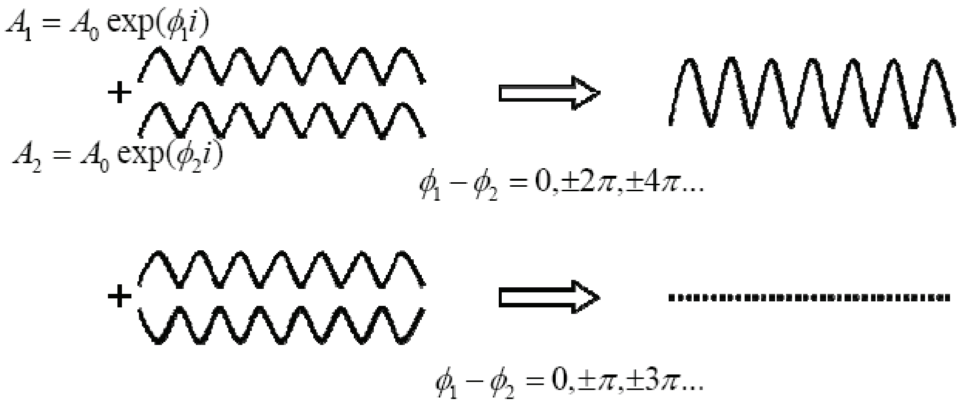
X-ray diffraction

- Diffraction is the effect following the interaction of an EM wave with an object having size dimension comparable with the period of the wave.

- X-rays have wavelengths on the order of a few angstroms, the same as typical interatomic distances in crystalline solids = tool for investigation of fine structure of matter

↓

X-ray diffraction is based on elastic scattering and results from the coherent sum of all EM waves that are diffused from each atom of a periodic structure constituting the matter (the same that occurs with visible light interaction with a grating).



$A_1 = A_0 \exp(\phi_1 i)$

$+$

$A_2 = A_0 \exp(\phi_2 i)$

$\phi_1 - \phi_2 = 0, \pm 2\pi, \pm 4\pi \ldots$

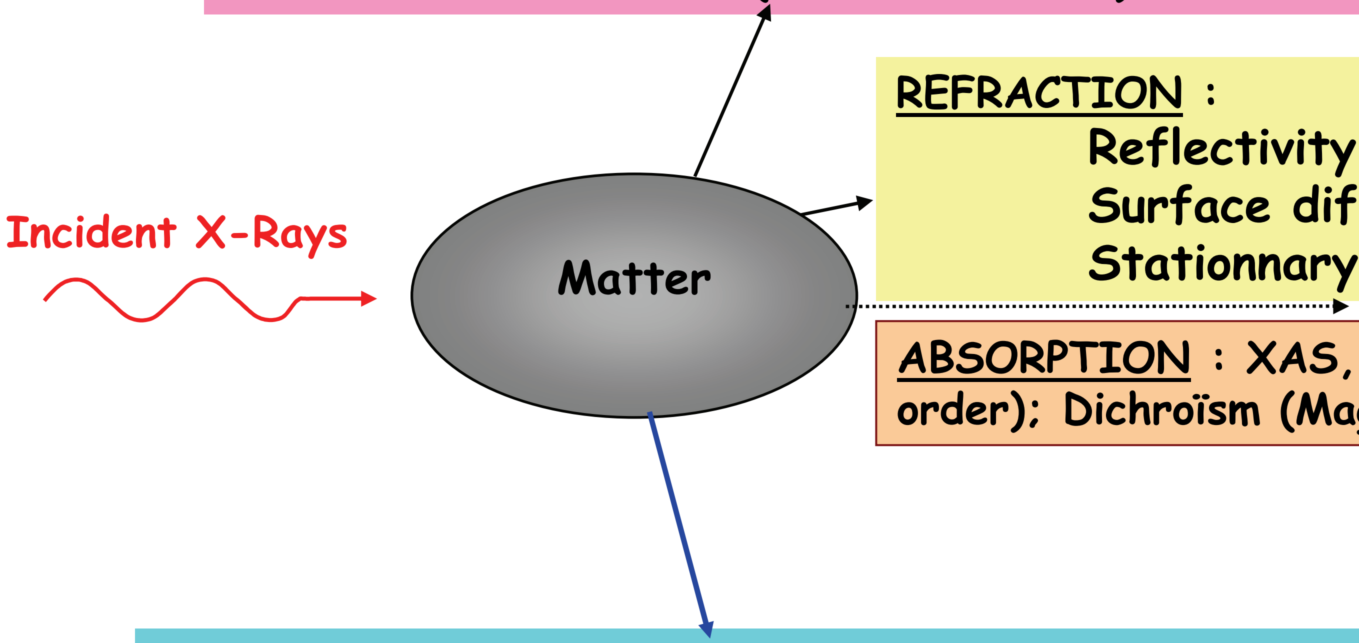
Coherent sum : Constructive interference

$+$

$\phi_1 - \phi_2 = 0, \pm\pi, \pm 3\pi \ldots$

Non coherent sum: Destructive interference

Interaction of X-rays with matter : associated techniques



EMISSION :
- * X-rays : Fluorescence (chemical analysis)
- * Electrons:
  - Photoelectrons, Auger electrons (analysis)
  - Photoelectrons diffraction (local structure)
  - Photoemission (band structure)

REFRACTION :
- Reflectivity (surface, interface)
- Surface diffraction (surfaces)
- Stationnary waves (surfaces)

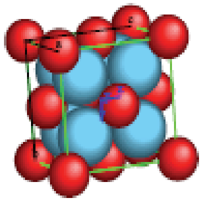
ABSORPTION : XAS, EXAFS, XANES (local order); Dichroïsm (Magnetism, surfaces)

DIFFUSION:
- Diffraction (Structures); diffuse diffusion (Disorder, liquids)
- Compton diffusion (Electronic structure)
- Small angles diffusion (Polymers, liquid crystals, agregates)
- Magnetic, inelastic , coherent diffusion… (synchrotrons)

Now we need some basics of crystallography !

↳ <u>Crystallised solid</u> : 3D periodic array of atoms

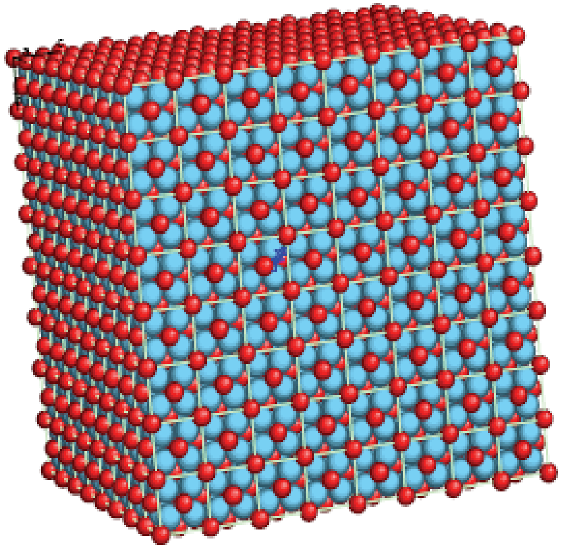
CaF_2



🟥 Ca^{2+}

🟦 F^-

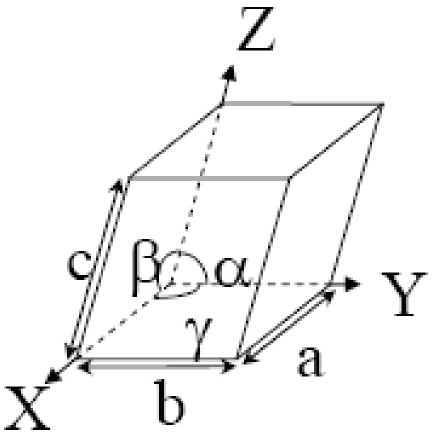
<u>Unit cell</u> :
The smallest
set of atoms
arranged in a
particular way.



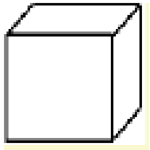
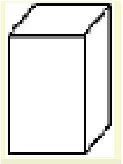
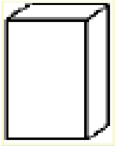
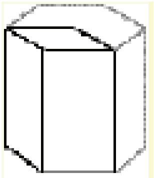
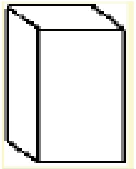
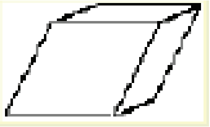
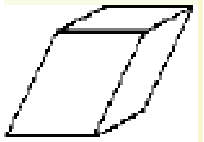
periodically repeated
in three dimensions

Macroscopic crystal =
crystal lattice

In 3D, the unit cell =
parallelepiped built on the crystallographic
basis vectors a, b, c of the direct lattice,
is determined by 6 parameters: a, b and c
cell lengths and the α, β and γ angles
which can be determined by XRD

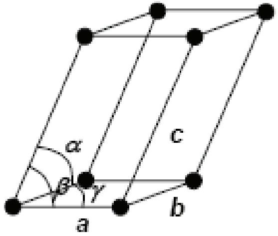


Cristalline structures classification (function of their symmetry) ➔ 3D Bravais lattice systems (7)

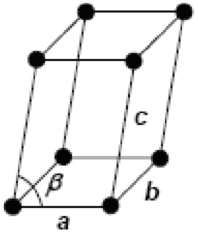
cubic		$a = b = c$ $\alpha = \beta = \gamma = 90°$
tetragonal		$a = b \neq c$ $\alpha = \beta = \gamma = 90°$
orthorhombic		$a \neq b \neq c$ $\alpha = \beta = \gamma = 90°$
hexagonal		$a = b \neq c$ $\alpha = \beta = 90°$ $\gamma = 120°$
monoclinic		$a \neq b \neq c$ $\alpha = \gamma = 90°$ $\beta \neq 90°$
rhombohedric		$a = b = c$ $\alpha = \beta = \gamma \neq 90°$
triclinic		$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma \neq 90°$

Seven crystal systems + four lattice centering types = 14 Bravais lattices

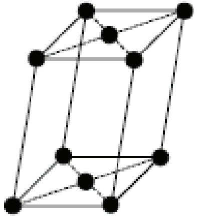
14 Bravais lattices Λ(r)



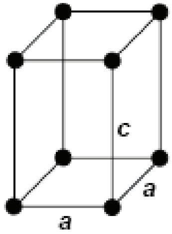
Triclinic P



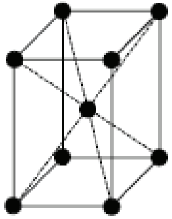
Monoclinic P



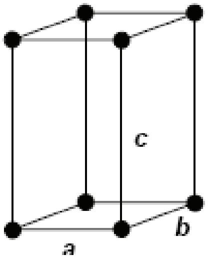
Monoclinic C



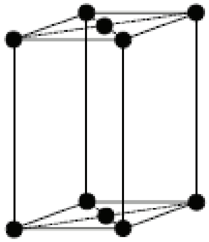
Tetragonal P



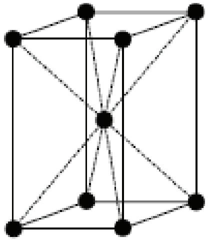
Tetragonal I



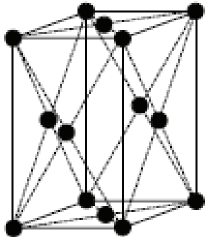
Orthorhombic P



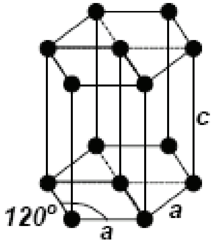
Orthorhombic C



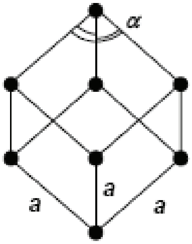
Orthorhombic I



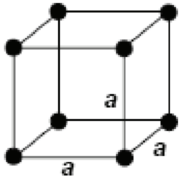
Orthorhombic F



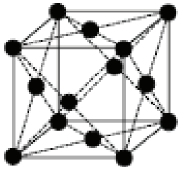
Hexagonal P



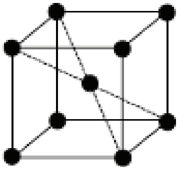
Rhombohedric P



Cubic P



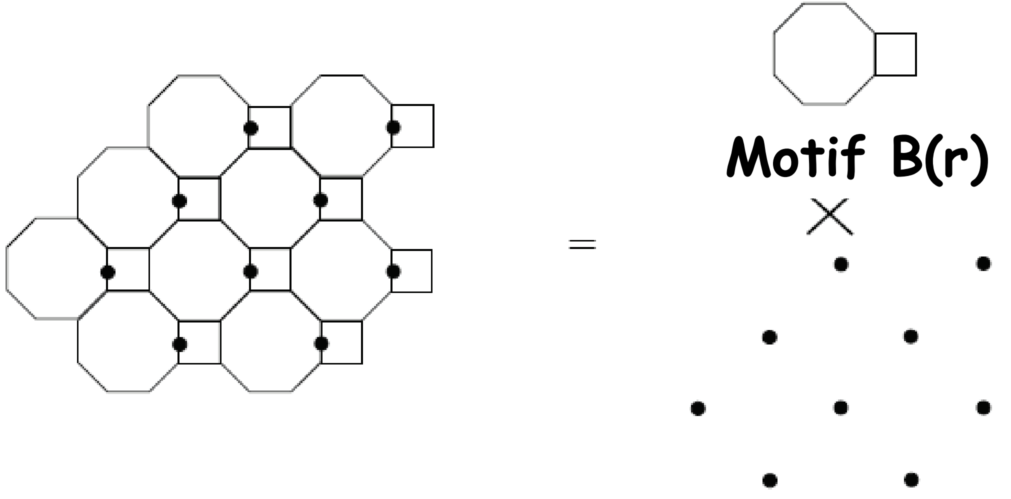
Cubic F



Cubic I

Motif and crystal lattice:

- The crystal structure C(r) can be considered to arise from the convolution of a basis domain B(r) also called the motif with the Bravais lattice Λ(r) : C(r) = B(r) × Λ(r).



=

Motif B(r)

×

Lattice Λ(r)

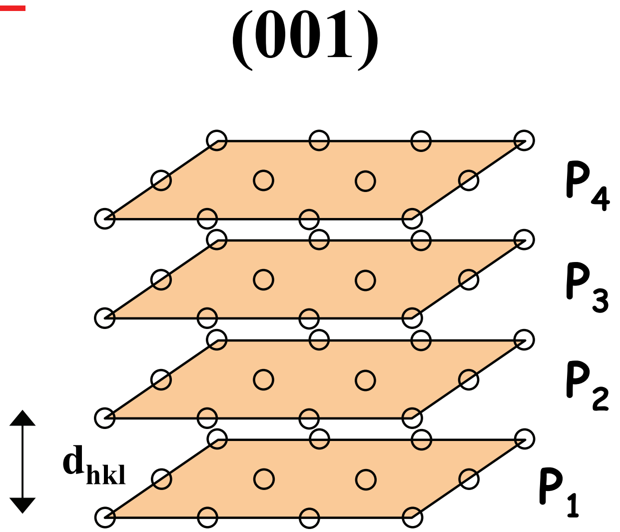
The motif B(r) = smallest possible unit of atoms which by application of all translational symmetry generates the whole crystal

- The lattice Λ(r) can be viewed as a set of periodically spaced Dirac distributions, i.e. a Dirac $\delta(\vec r - \vec r_0)$ distribution located on each node of the lattice:

$$\Lambda(\vec r) = \sum_{u=-\infty}^{+\infty}\sum_{v=-\infty}^{+\infty}\sum_{w=-\infty}^{+\infty}\partial(\vec r - u\vec a - v\vec b - w\vec c)$$

with Direct lattice point coordinates : $u\vec a + v\vec b + w\vec c$ with u, v and w integers

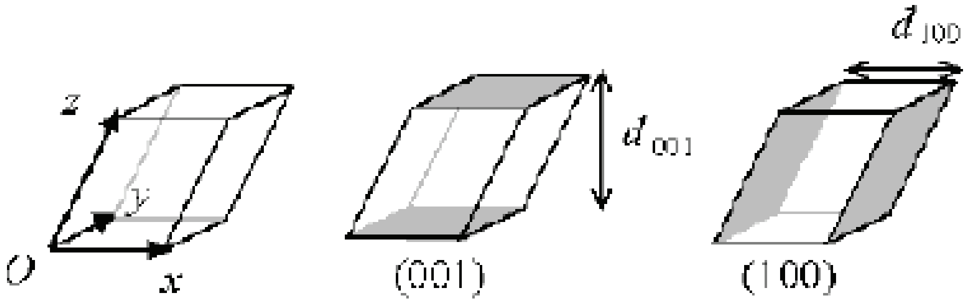
Families of lattice planes:



- Lattice plane (h k l) = plane which passes through lattice nodes that do not all lie on the same straight line and labelled by three prime integer numbers h, k, l named Miller indices.

- Family of lattice planes = set of parallel lattice planes P_i.

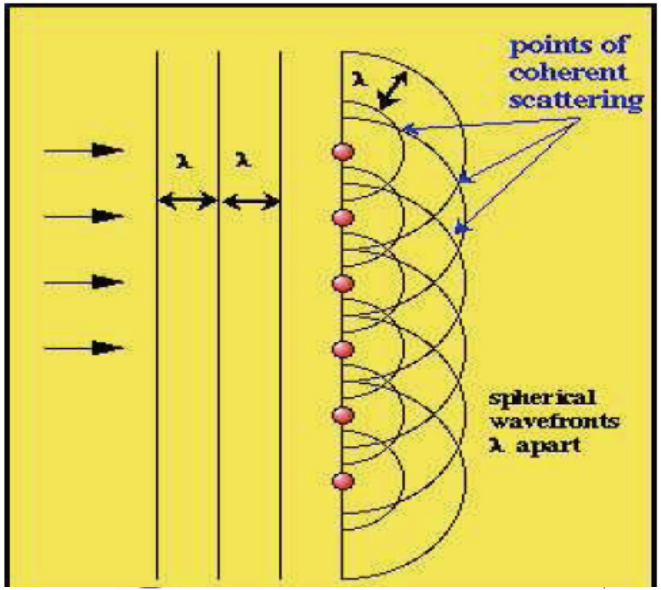
Distance between two neighbouring lattice planes = spacing d_{hkl} accessible by XRD.



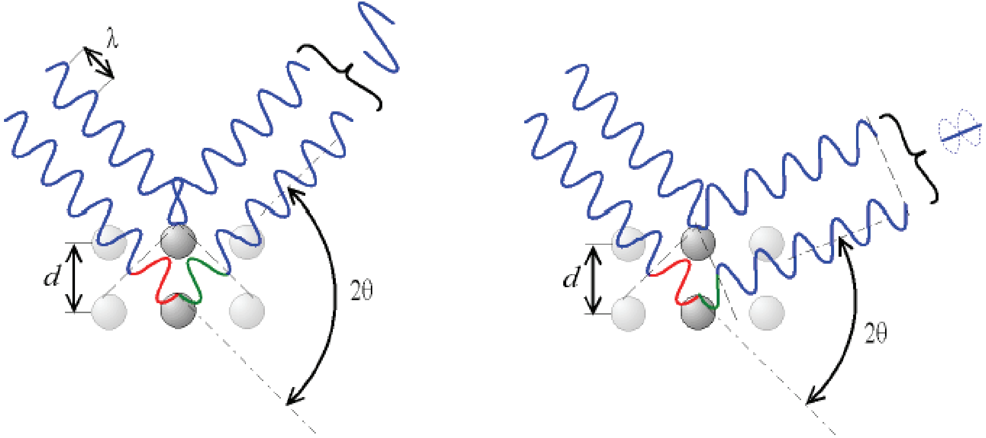
X-ray diffraction and Bragg law

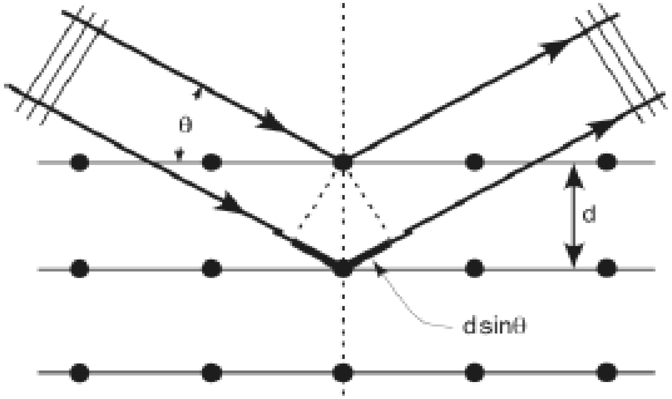
- When X-rays interact with a single particle, it scatters the incident beam uniformly in all directions (spherical wave).

- When X-rays interact with a solid material the scattered beams can add together in a few directions and reinforce each other to yield diffraction.



Diffraction =
Elastic diffusion +
constructive interferences





Constructive interference : Path length difference 2 d sinθ = integral number of λ

Bragg law :
2 d sinθ = m λ with m integer

Reciprocal space: Geometric definition

Introduced by Bravais and used again by Ewald (1917) : an essential concept for the study of crystal lattices and their diffraction properties.

- Basic vector definitions:

$$\vec{a}^* = \frac{\vec{b} \wedge \vec{c}}{V}, \quad \vec{b}^* = \frac{\vec{c} \wedge \vec{a}}{V}, \quad \vec{c}^* = \frac{\vec{a} \wedge \vec{b}}{V}$$

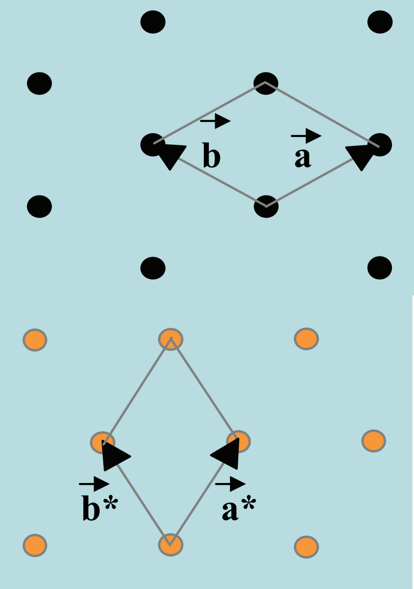
with $V=(a,b,c)$ direct cell volume
and $V^*=(a^*,b^*,c^*)=1/V$ reciprocal cell volume

- Equivalent definitions (2D, 3D...)

$$\begin{array}{lll} \vec{a}^*.\vec{a}=1 & \vec{b}^*.\vec{a}=0 & \vec{c}^*.\vec{a}=0 \\ \vec{a}^*.\vec{b}=0 & \vec{b}^*.\vec{b}=1 & \vec{c}^*.\vec{b}=0 \\ \vec{a}^*.\vec{c}=0 & \vec{b}^*.\vec{c}=0 & \vec{c}^*.\vec{c}=1 \end{array}$$

Direct space

Reciprocal space

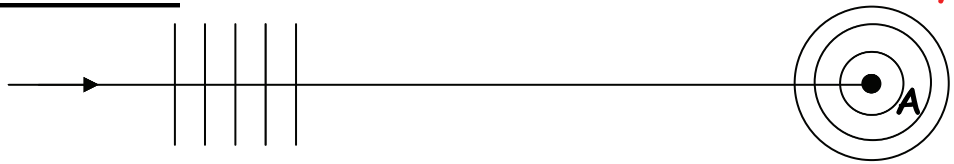


- Reciprocal lattice point coordinates: $\overrightarrow{Q_{hkl}} = n(h\vec{a}^* + k\vec{b}^* + l\vec{c}^*)$ with h,k,l and n integers

X-ray diffraction by crystals

➩ Elastic diffusion by 1 electron:

the electron (in A) acts
as a secondary X-ray source



Incident plane wave

Emitted spherical wave
by the electron

Amplitude diffused by electron A_e : proportional to $1/m^2$, q^4, ω^4, $\sin\theta$ (Thomson formula)

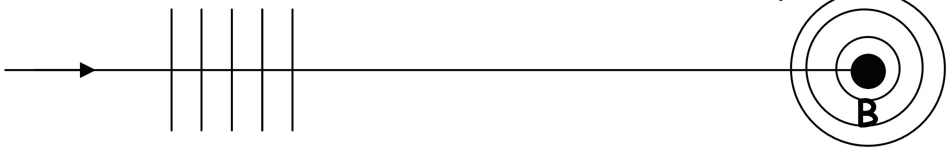
➩ Elastic diffusion by 1 atom = N electrons

All the N spherical diffused electromagnetic
waves with same phase must interfere

$\Downarrow$

amplitude diffused by
1 atom : $A_a = f(\theta)\ A_e$
with $f(\theta)$ form factor or
atomic diffusion coefficient

All the electrons of B atom
act as a secondary
X-ray source

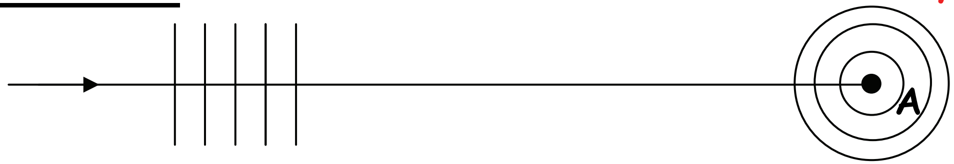


Incident plane wave

Emitted spherical
Wave by B atom

X-ray diffraction by crystals

↳ Elastic diffusion by 1 electron:



the electron (in A) acts
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Incident plane wave

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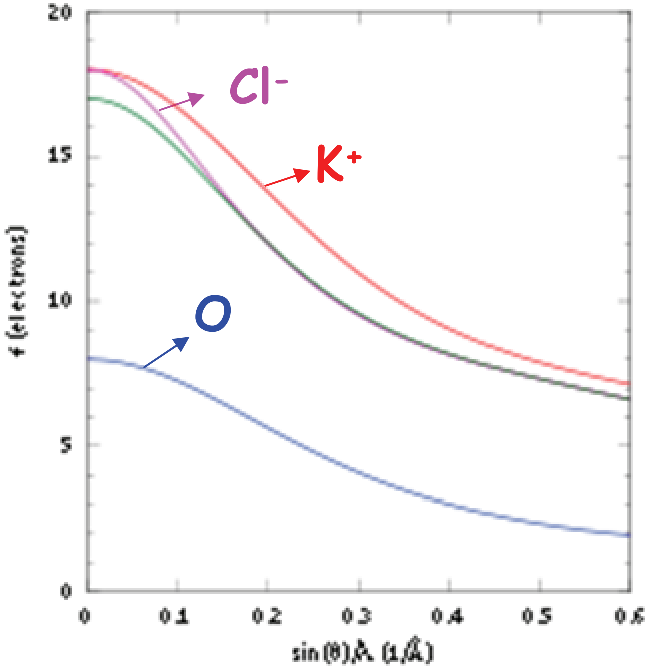
↳ Elastic diffusion by 1 atom = N electrons

All the N spherical diffused electromagnetic waves with same phase must interfere

↓

amplitude diffused by
1 atom : $A_a = f(\theta)\ A_e$
with $f(\theta)$ form factor or
atomic diffusion coefficient

Form factor $f(\sin\theta/\lambda)$ tabulated in the International Crystallography Tables



$f(\theta) = Z$ for $\theta = 0°$ + $f(\theta)$
fall down rapidly when θ ↗

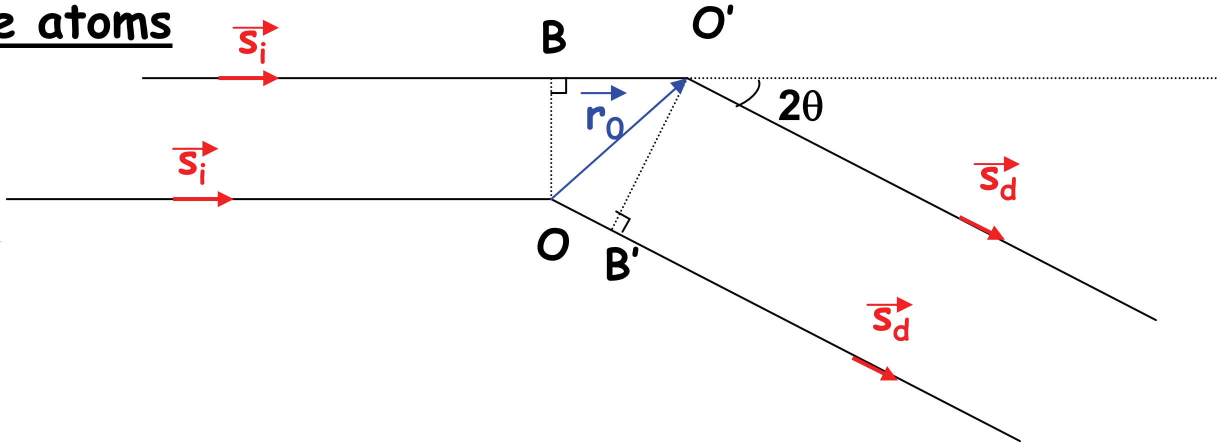
X-ray diffraction by crystals

↳ Elastic diffusion by 2 same atoms

$\vec{s}_i$ and $\vec{s}_d$: unitary incident and diffused vectors

$OB' = \vec{r}_0 . \vec{s}_d$ et $O'B = \vec{r}_0 . \vec{s}_i$

$$\partial = \vec{r}_0 .(\vec{s}_d - \vec{s}_i)$$



The path difference δ between the two scattered waves: δ = OB' – O'B gives the phase difference Δφ between these 2 waves: Δφ = 2π δ/λ

$$\Delta\varphi = \frac{2\pi}{\lambda}\vec{r}_0 .(\vec{s}_d - \vec{s}_i) = 2\pi\,\vec{r}_0 .\vec{H}$$

avec $\vec{H} = (\vec{s}_d - \vec{s}_i)/\lambda$: diffusion vector

Resulting wave amplitude : $A = A_a(1 + e^{2j\pi(\vec{r}_0 .\vec{H})}) = f\,A_e\,(1 + e^{2j\pi(\vec{r}_0 .\vec{H})})$

Constructive interference if Δφ = 2πn with n integer ➔ $\vec{r}_0 .\vec{H} = n$

et $\vec{r}_0 = u\vec{a} + v\vec{b} + w\vec{c}$ ➔ 3 relations that must be verified simultaneously

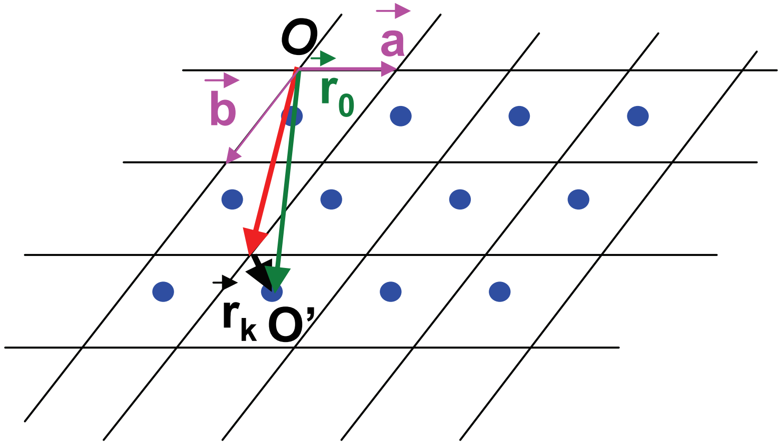
Laue conditions:

$$\vec{a}.\vec{H} = h$$
$$\vec{b}.\vec{H} = k$$
$$\vec{c}.\vec{H} = \ell$$

Constructive interference i.e. diffraction if diffusion vector $\vec{H}$ is a reciprocal space vector

Elastic diffusion by a monoatomic crystal

Diffused amplitude by O' atom with the phase origin in O: $f\, A_e\, e^{2j\pi(\vec{r_0}.\vec{H})}$



Motif = ● = 1 atom with (x, y, z) coordinates :
$\vec{r_k} = x_k\,\vec{a} + y_k\,\vec{b} + z_k\,\vec{c}$

$\vec{r_0} = \vec{r_k} + u\,\vec{a} + v\,\vec{b} + w\,\vec{c}$

motif

lattice

Crystal = n atoms : n diffused waves with different phase;

Diffused amplitude by n atoms :

$$A = \sum_{k=1}^{n} f\, A_e\, e^{2j\pi(\vec{r_0}.\vec{H})} = \sum_{k,u,v,w} f\, A_e\, e^{2j\pi(\vec{r_k}.\vec{H})} e^{2j\pi\left[u(\vec{H}.\vec{a})+v(\vec{H}.\vec{b})+w(\vec{H}.\vec{c})\right]}$$

Diffraction if $A \neq 0$ so $\vec{H}.\vec{a}, \vec{H}.\vec{b}$ and $\vec{H}.\vec{c}$ = integers.

$$A = A_e \sum_k f_k\, e^{2j\pi(\vec{r_k}.\vec{H})} \times u \times v \times w = NA_e \sum_k f_k\, e^{2j\pi(\vec{r_k}.\vec{H})}$$

Structure factor F = A/(NA$_e$)

$$F_{hkl} = \sum_k f_k\, e^{2j\pi(\vec{r_k}.\vec{H})} = \sum_k f_k\, e^{2j\pi(hx_k+ky_k+lz_k)}$$

Formula that can be generalized also for polyatomic crystals

Temperature factor = Debye-Waller factor

When t° ↗, atoms vibration amplitude ↗: $F_{hkl} = \sum_k f_k \, e^{2j\pi(hx_k + ky_k + lz_k)} \times e^{\left(\frac{-B\sin^2\theta}{\lambda}\right)}$

with (x_k, y_k, z_k) = atomic position in the unit cell and B = $8\pi^2\langle u^2\rangle$

$\langle u^2\rangle$ = standard deviation of the amplitude of atom vibration

↓

The influence of B is more important when θ ↗, i.e. when diffracted beam is far from the incident beam direction.

Intensity : the diffracted intensity in the direction $\vec{s}_d = \vec{s}_i + \lambda\vec{H}$

is proportional to FF*: $I_{hkl} \propto N^2 A_e F_{hkl} F^*{}_{hkl}$

with $F^*{}_{hkl} = \sum_k f_k \, e^{-2j\pi(hx_k + ky_k + lz_k)} \times e^{\left(\frac{-B\sin^2\theta}{\lambda}\right)}$

Friedel law: $F^*{}_{hkl} = F(\bar{h}\,\bar{k}\,\bar{l})$ and $F(\bar{h}\,\bar{k}\,\bar{l}) = F^*{}_{hkl}$ ➔ $I(\bar{h}\,\bar{k}\,\bar{l}) = I(hkl)$

if f_k is real : we cannot know if a structure is centrosymetric or not !

Systematic extinctions (due to the lattice) : I(hkl) = 0

* For a body centered lattice (I) : additional translation $\vec{t}=\left(\frac{1}{2}\vec{a}+\frac{1}{2}\vec{b}+\frac{1}{2}\vec{c}\right)$

To each atom in x_k, y_k, z_k corresponds systematically an atom in $x_k+1/2$, $y_k+1/2$, $z_k+1/2$

$$F_{hkl}=\left(\sum_{\text{half atoms}} f_k\ e^{2j\pi(hx_k+ky_k+lz_k)}+\sum_{\substack{\text{half atoms}\\ \text{deduced by }\vec{t}}} f_k\ e^{2j\pi\left(h\left(x_k+\frac{1}{2}\right)+k\left(y_k+\frac{1}{2}\right)+l\left(z_k+\frac{1}{2}\right)\right)}\right)\times e^{\left(\frac{-B\sin^2\theta}{\lambda}\right)}$$

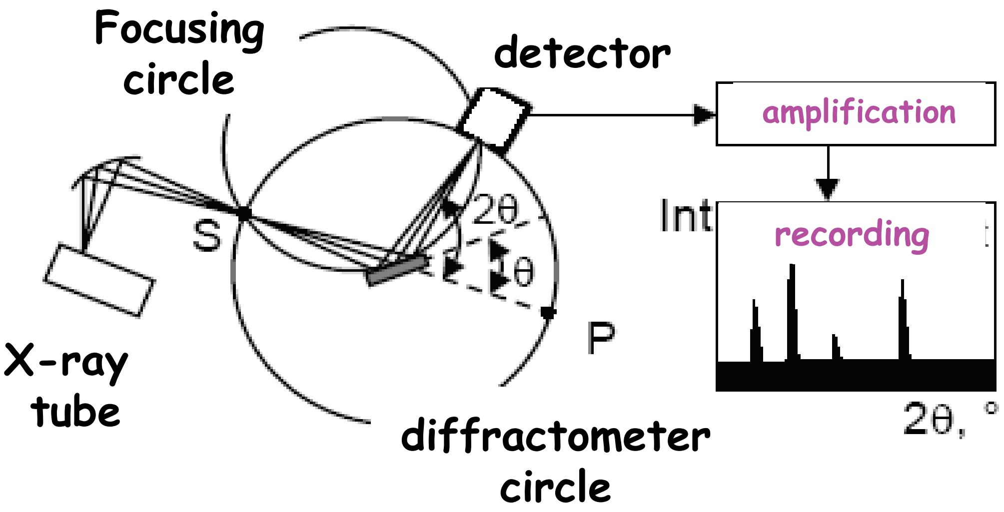
$$F_{hkl}=\underbrace{\sum_{\text{half atoms}} f_k\ e^{2j\pi(hx_k+ky_k+lz_k)}}_{\text{motif}}\underbrace{\left[1+e^{\frac{h+k+l}{2}}\right]}_{\text{lattice}}\times e^{\left(\frac{-B\sin^2\theta}{\lambda}\right)}$$

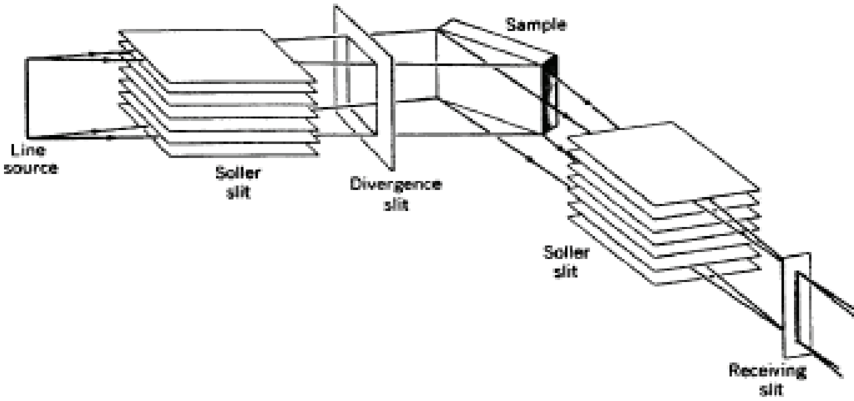
If h+k+l odd, F(hkl) = 0 :
systematic extinctions due to the I lattice if h+k+l=2n+1.

* For a face centered lattice (F) : 3 additional translations $\vec{t}=\left(\frac{1}{2}\vec{a}+\frac{1}{2}\vec{b}\right)$ $\vec{t}=\left(\frac{1}{2}\vec{a}+\frac{1}{2}\vec{c}\right)$ and $\vec{t}=\left(\frac{1}{2}\vec{b}+\frac{1}{2}\vec{c}\right)$; extinctions if h, k and l none of same parity.

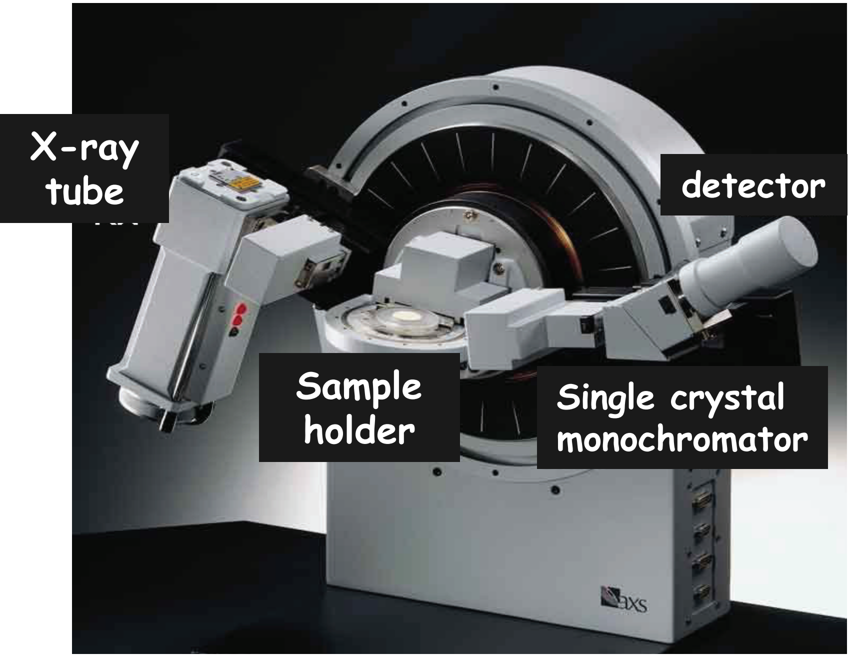
* For primitive lattice (P) : no extinction.

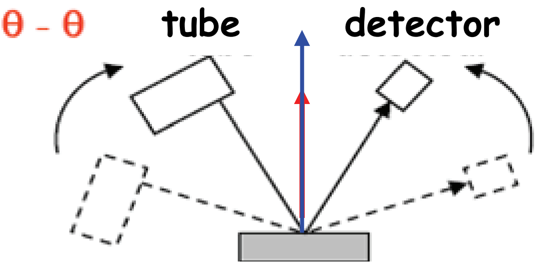
Powder diffractometer (2-circles)



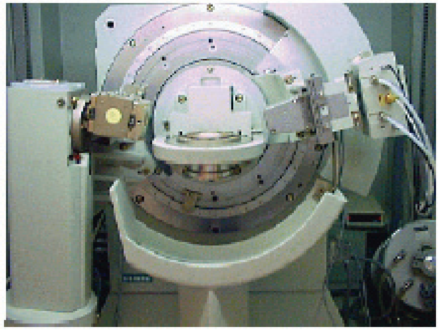


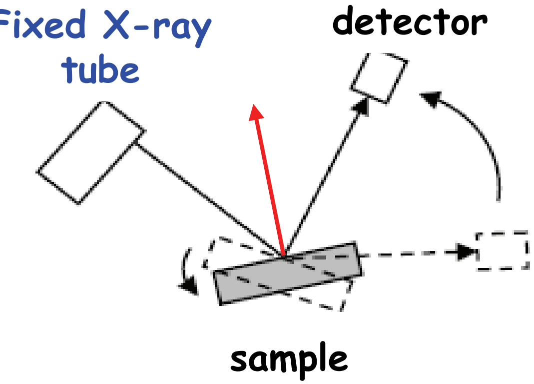
Bragg Brentano geometry

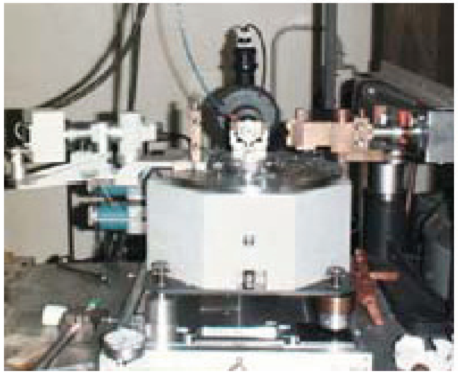




Fixed sample





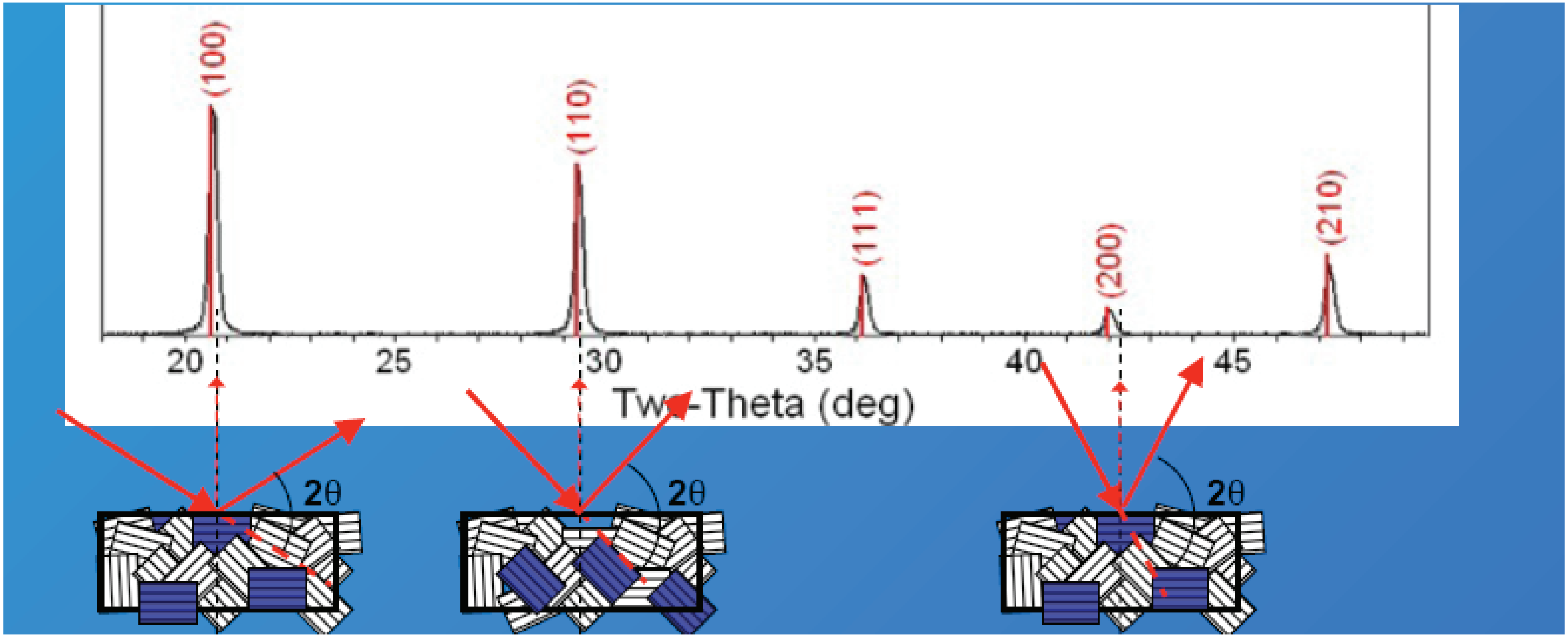


Polycrystalline sample = n single crystals

If n single crystals with random orientation = Powder

↓

All possible diffraction peaks (Bragg law) are observed



For every set of planes parallel to the sample surface, there will be some crystallites that are properly oriented to diffract (i.e. plane ⊥ bisects the incident and diffracted beams).

Basic assumptions of powder diffraction = for every set of planes there is an equal number of crystallites that will diffract.

Random powder : $I_{RX\ calc.}(2\theta) = \sum_{hkl,phases} I_{hkl,phases}(2\theta)\, S_{hkl}(2\theta) + bkg\,(2\theta)$

$$I_{hkl}(2\theta) = A\, S\, |F_{hkl}|^2\, m_{hkl}\, \frac{L_p}{V_c^2}$$

absorption

scale factor
(quantitative phase amount)

structure factor
(includes Debye-Waller term)

multiplicity

Lorentz- polarisation factor

unit-cell volume

which atoms are ("motif");
Number of scattering electron

d_{hkl} : where the atoms are: due to "lattice";
position in reciprocal space

Random powder : $I_{RX\ calc.}(2\theta) = \sum_{hkl,phases} I_{hkl,phases}(2\theta)\, S_{hkl}(2\theta) + bkg\,(2\theta)$

$$\Rightarrow\ S_{hkl}(2\theta) = S^{I}_{hkl}(2\theta) * S^{S}_{hkl}(2\theta)$$

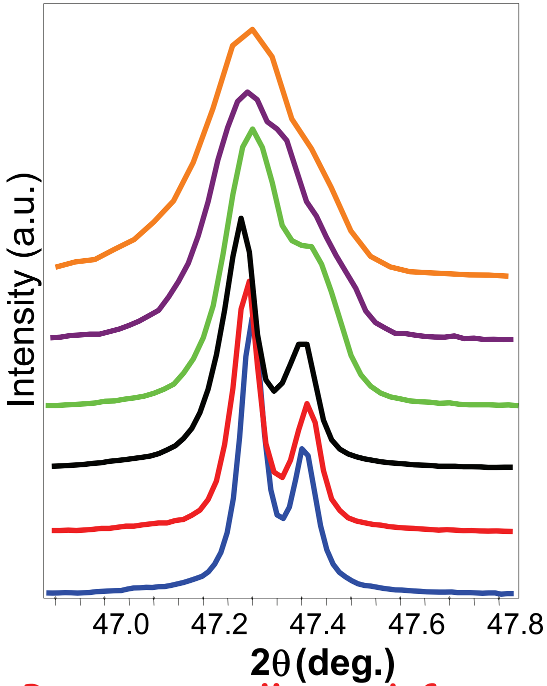
Peak profile function

instrumental broadening → powder standard calibration

Sample aberrations = crystallite sizes (isotropic or anisotropic)

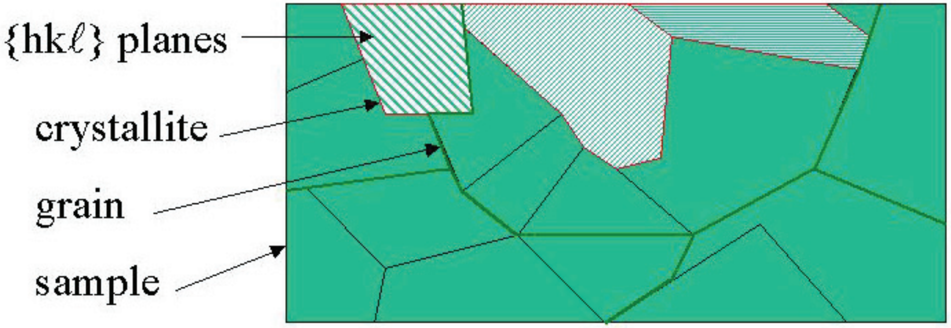
+

rms microstrains $\varepsilon^* = <\varepsilon>^2$ (Non-uniform Lattice Distortions, Faulting, Dislocations, Antiphase Domain Boundaries …)

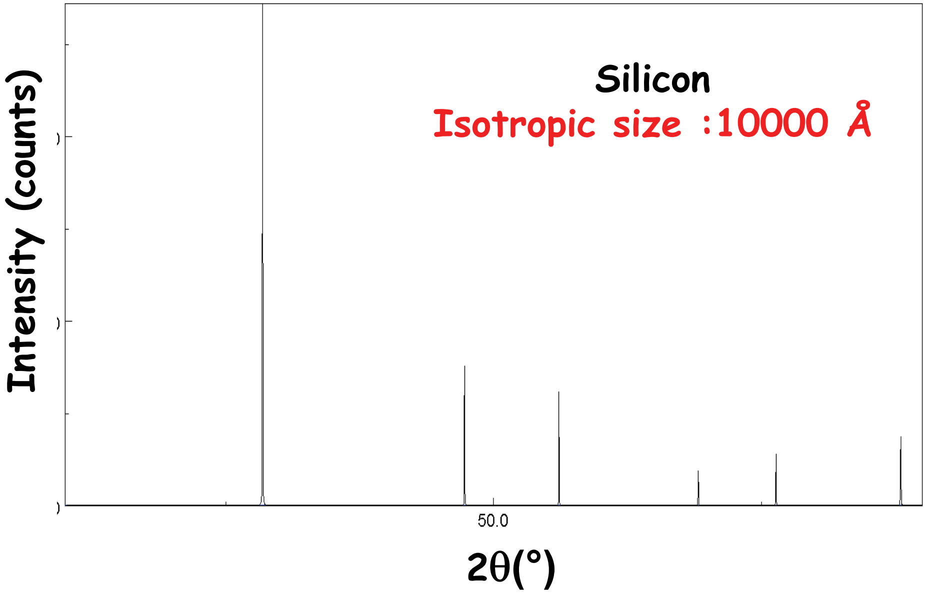


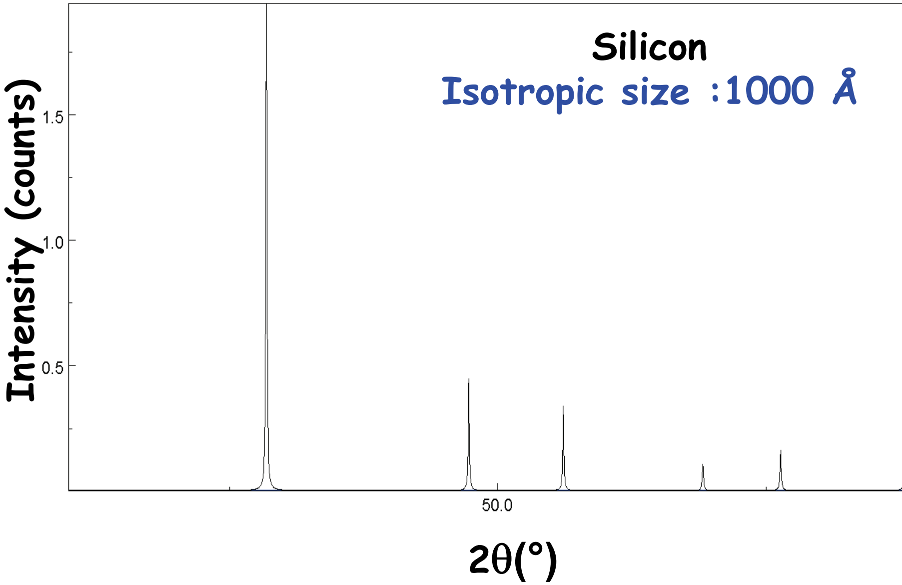
Patterns collected from the same sample with different instruments and configurations

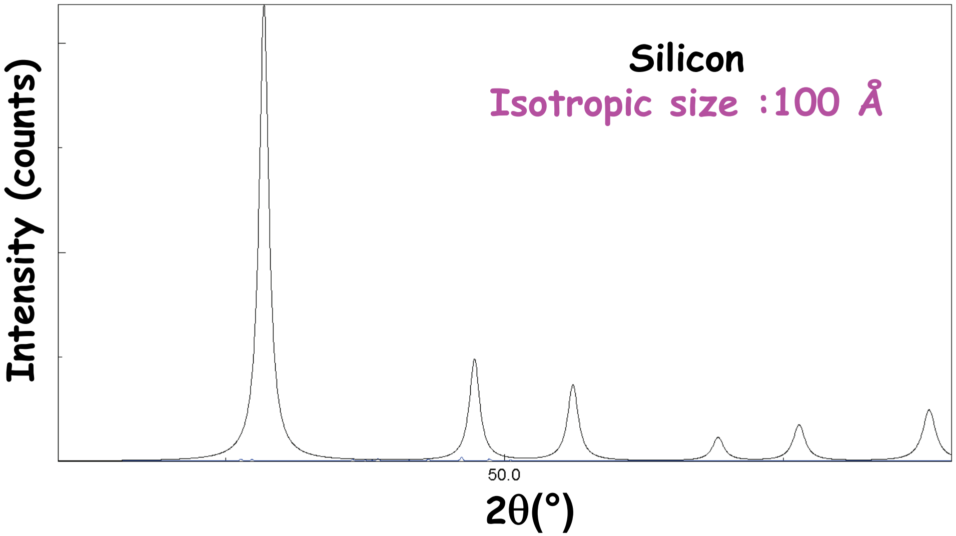
Take care:
crystallite size is different than grain size !!!

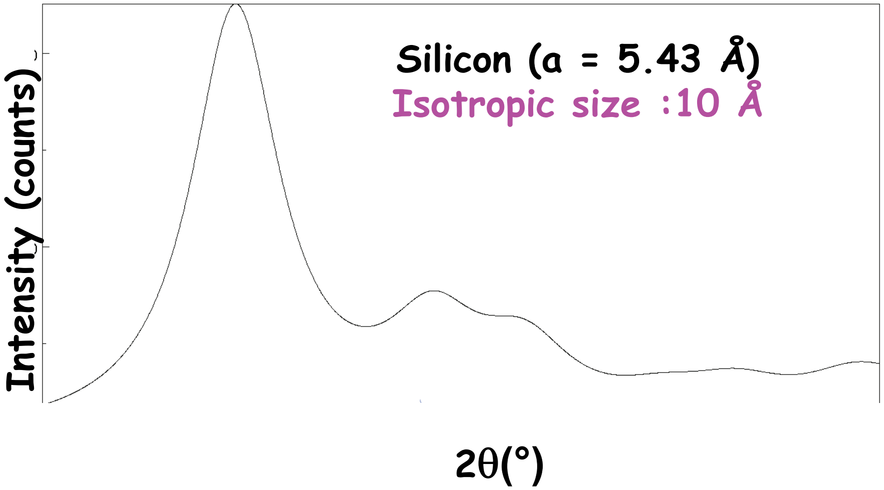


A grain may be made up of several different crystallites





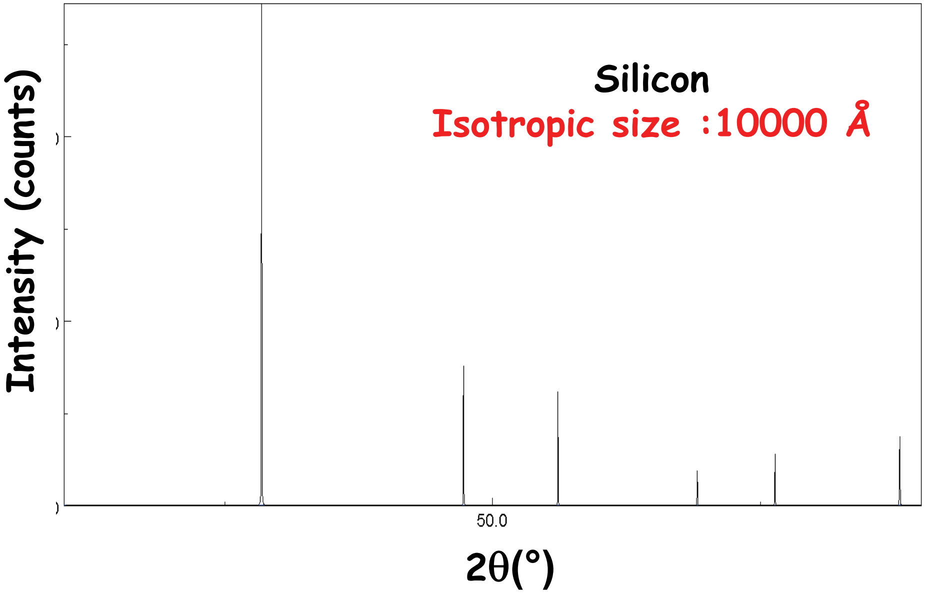


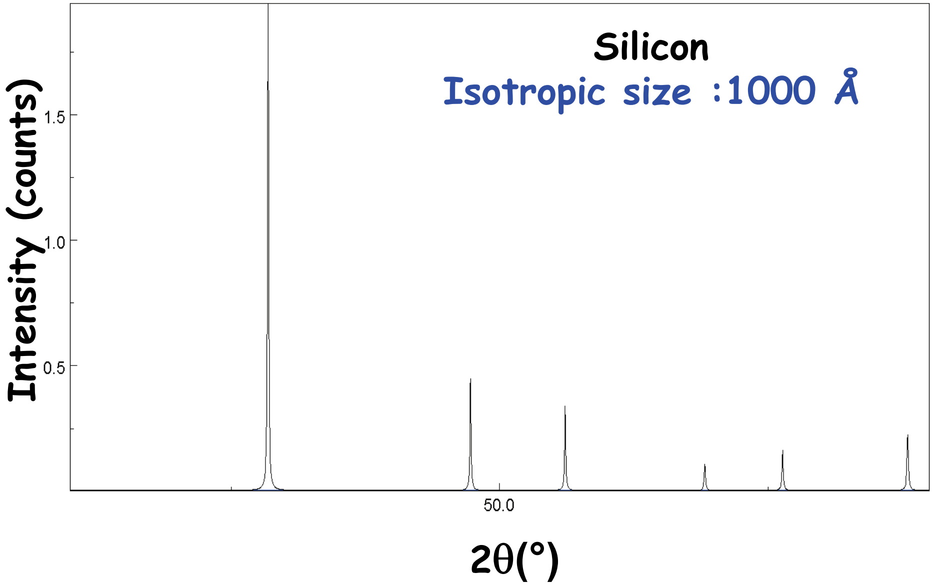


Peak width (B) is inversely proportional to crystallite size (L) ➔

$$B(2\theta)=\frac{K\lambda}{L\cos\theta}$$

Debye Scherrer formula : estimation of crystallite size





The constant of proportionality, K (Scherrer constant) depends on how the width is determined, the shape of the crystal, and the size distribution.

The most common values for K are:

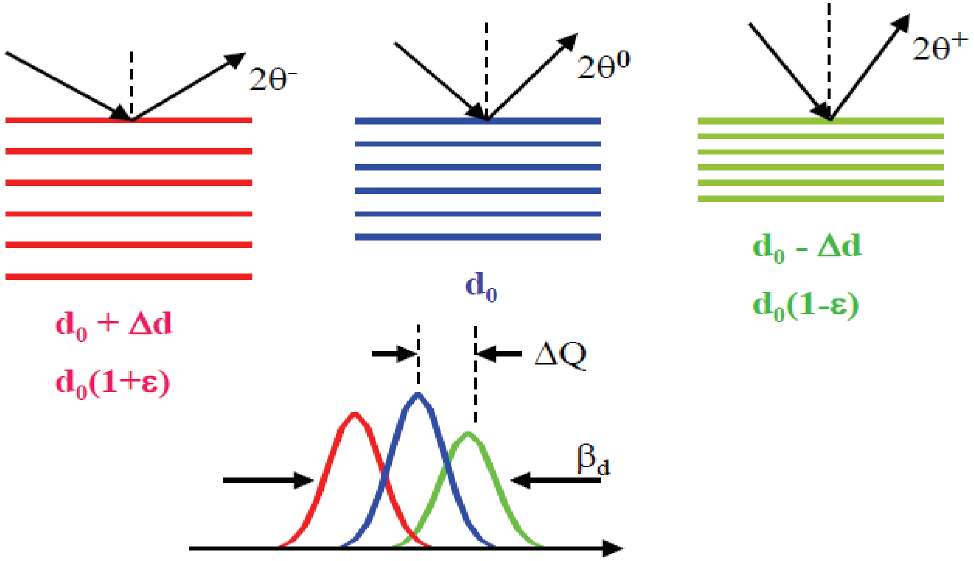
- 0.94 for FWHM of spherical crystals
- 0.89 for integral breadth of spherical crystals

Peak width (B) is inversely proportional to crystallite size (L) ➔
Debye Scherrer formula : estimation of crystallite size

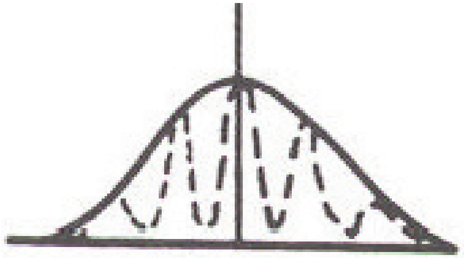
$$B(2\theta)=\frac{K\lambda}{L\cos\theta}$$

Peak broadening by microstrain (varying lattice parameter = non uniform strain)

Microstrain : $\langle \varepsilon \rangle = \dfrac{\Delta d}{d}$; in a same grain (several crystallites) :



Pic width : effect of microstrain = peaks juxtaposition :

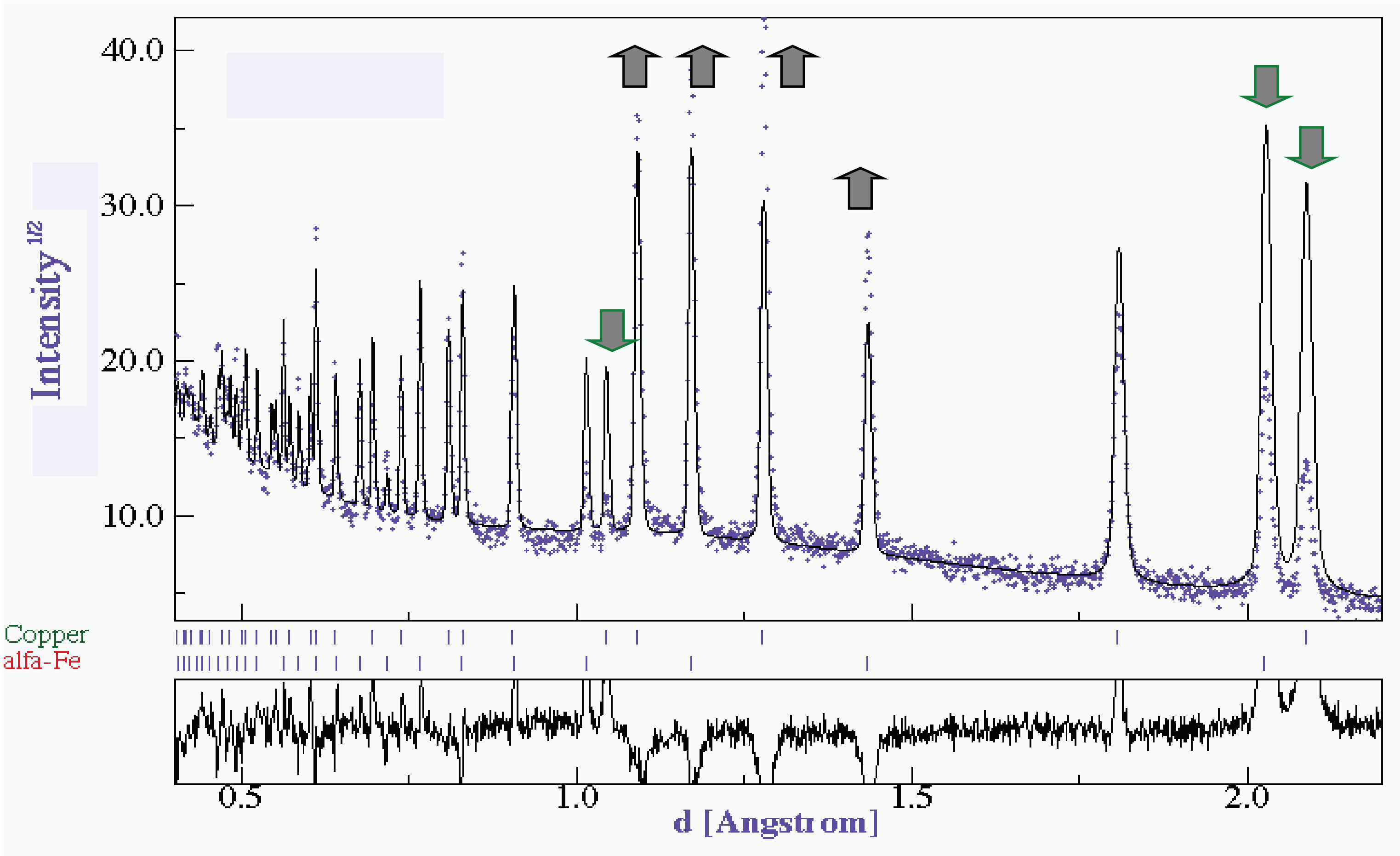


Increased microstrain results in broadening of diffraction peaks

If n single crystals with specific orientation = textured sample

↓

Not all possible diffraction peaks are observed or all possible diffraction peaks are observed but with different intensities compared to a powder



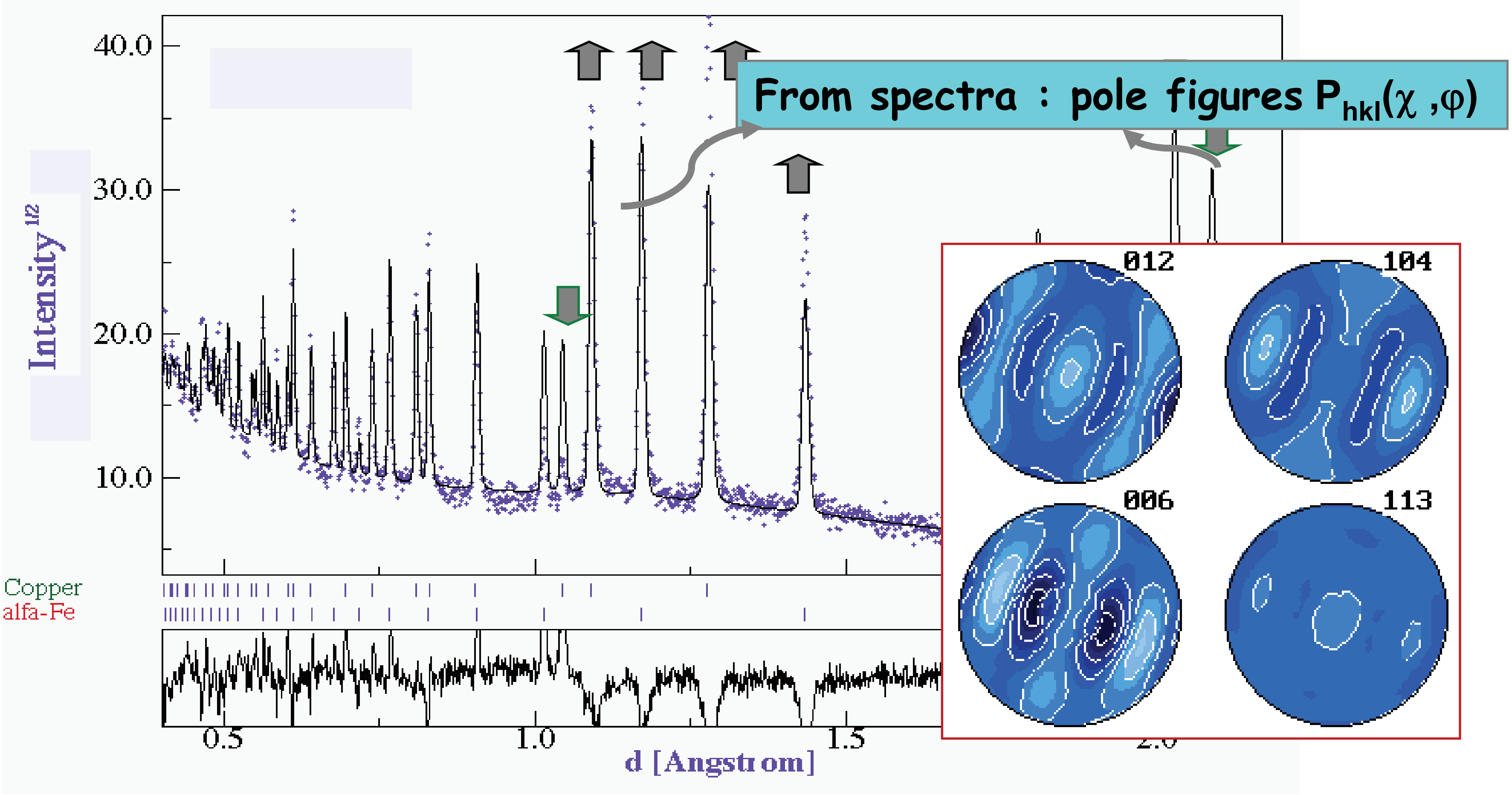
Correction of intensities for textured sample :

$$I_{hkl}(2\theta, \chi, \varphi) = I_{hkl}(2\theta)\, P_{hkl}(\chi, \varphi)$$

If n single crystals with specific orientation = textured sample

↓

Not all possible diffraction peaks are observed or all possible diffraction peaks are observed but with different intensities compared to a powder

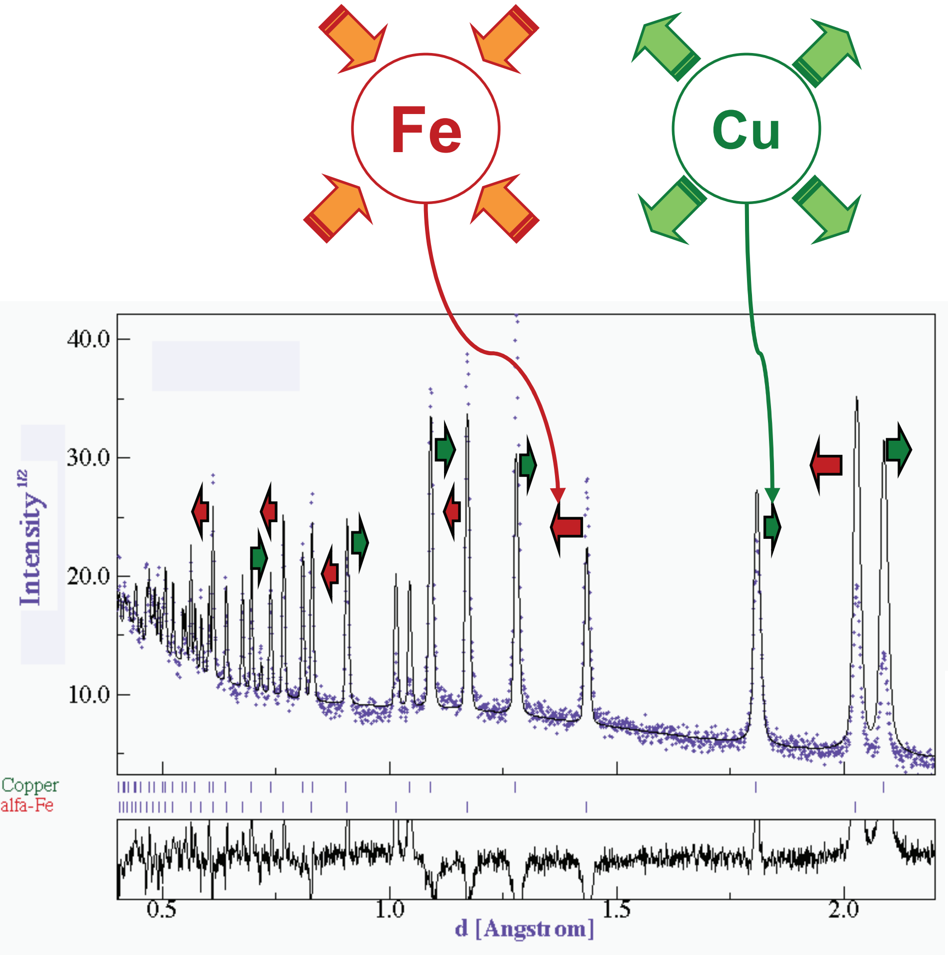


From spectra : pole figures $P_{hkl}(\chi,\varphi)$

Correction of intensities for textured sample :

$$I_{hkl}(2\theta,\chi,\varphi) = I_{hkl}(2\theta)\,P_{hkl}(\chi,\varphi)$$

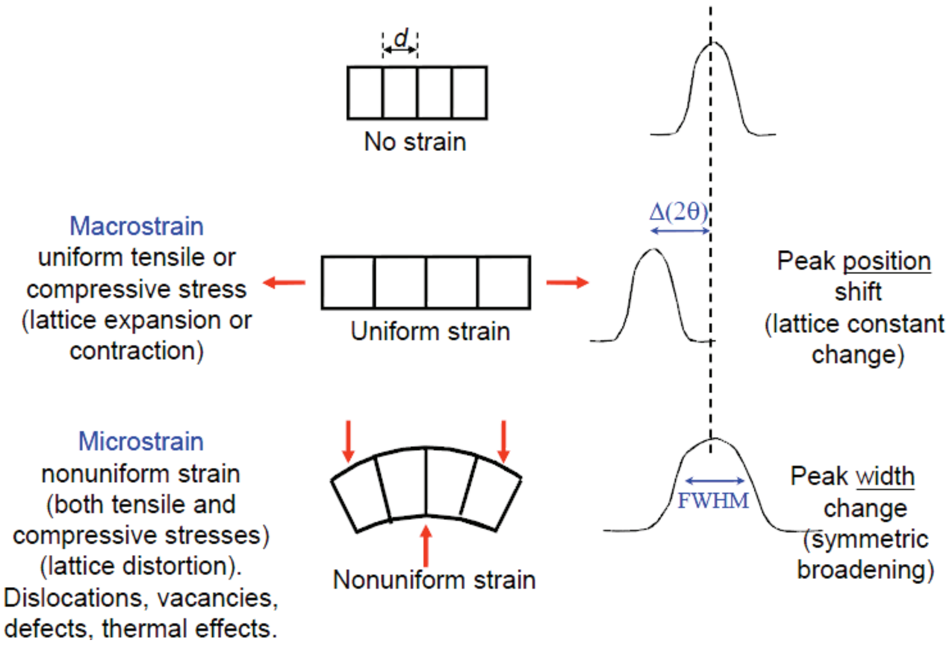
Residual stresses



By changing the orientation of the sample towards the incident beam : peak position moves from a reference position but intensity is the same

peak shifts due to lattice expansion or contraction ➔ tensile or compressive stress

Summary



No strain

Macrostrain
uniform tensile or compressive stress (lattice expansion or contraction)

Uniform strain

Peak position shift (lattice constant change)

Microstrain
nonuniform strain (both tensile and compressive stresses) (lattice distortion). Dislocations, vacancies, defects, thermal effects.

Nonuniform strain

Peak width change (symmetric broadening)

Summary

You can use XRD on powder polycrystalline samples to determine:

- Phase Composition of the sample

– Quantitative Phase Analysis: determine the relative amounts of phases in a mixture by referencing the relative peak intensities

- Unit cell lattice parameters and Bravais lattice symmetry

– Index peak positions
– Lattice parameters can vary as a function of, and therefore give you information about, alloying, doping, solid solutions …

- Residual Strain (macrostrain)

- Texture/Orientation

- Crystal Structure

– By Rietveld refinement of the entire diffraction pattern (not presented here)

- Crystallite Size and microstrain

- Indicated by peak broadening
Other defects (stacking faults, etc.) can be measured by analysis of peak shapes and peak width.