Interaction between X-ray and Matter
Peak Position

Hammond Chapter 8, 9, 10

Pecharsky Chapter 2

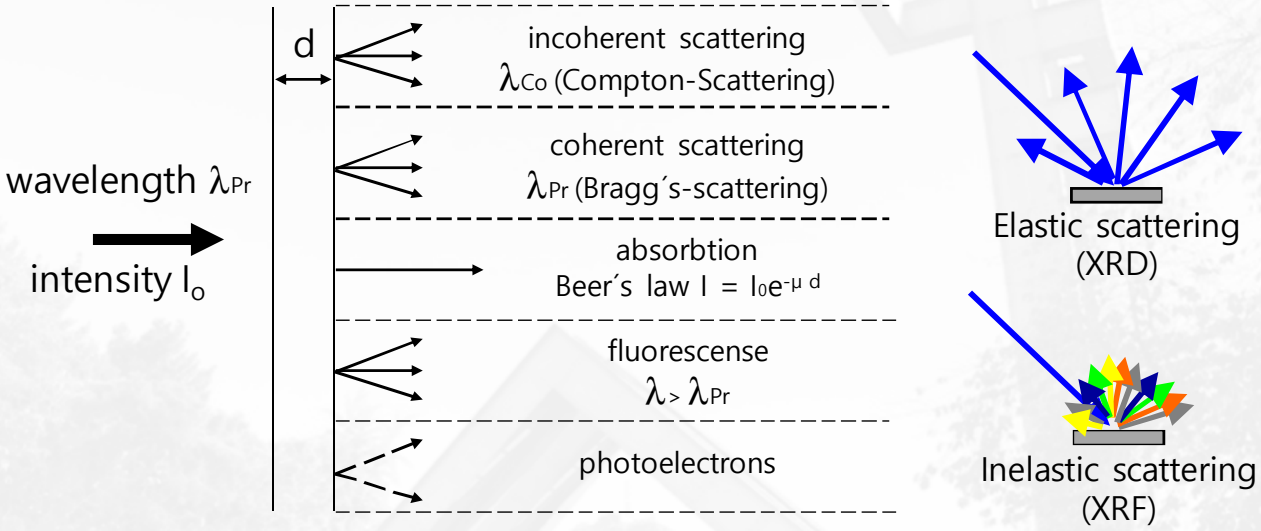
Sherwood & Cooper Chap 4.13; 6.1~6.3; 8.9~8.16

Krawitz Chap 5, p119~128

Birkholz Chapter 1

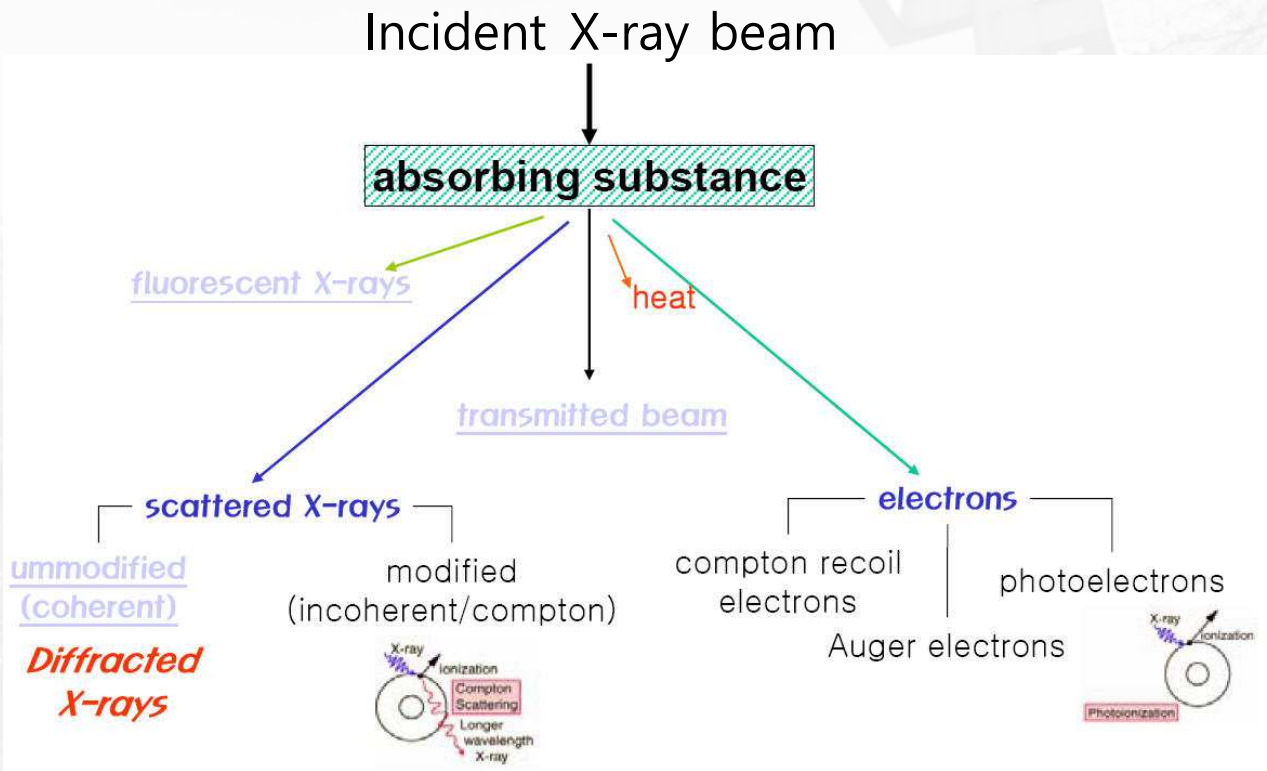
Cullity Chap 3-1~3-6

Interaction between X-ray and Matter



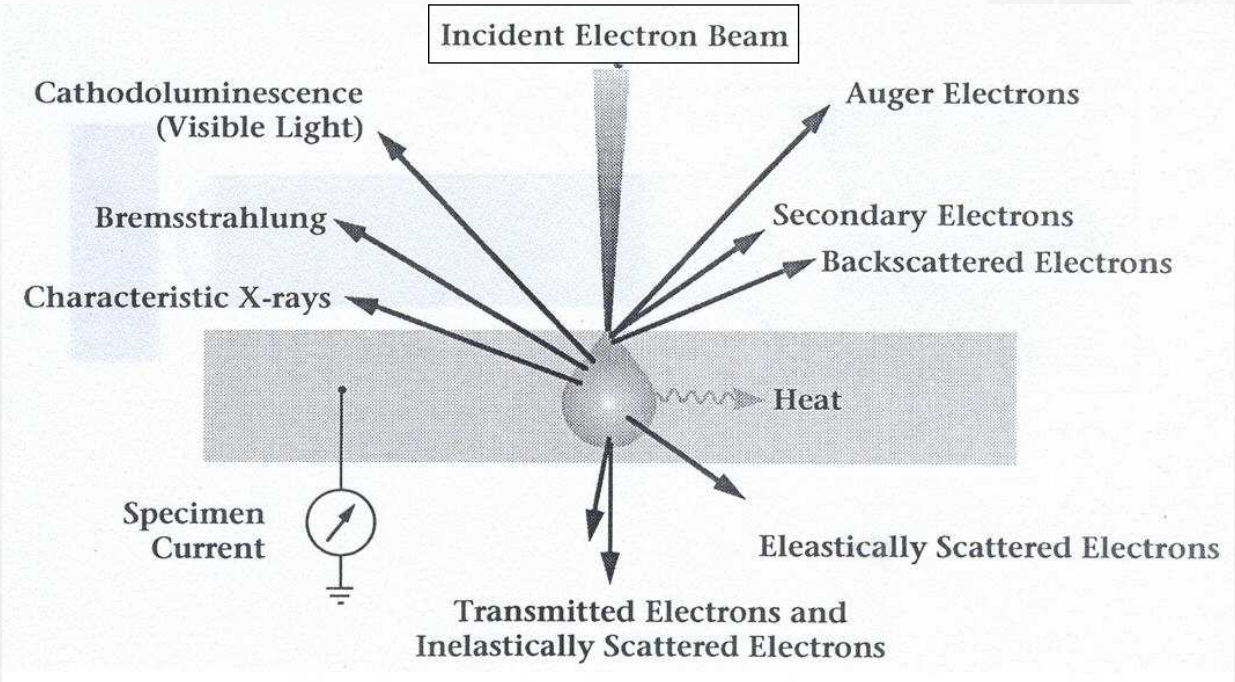
- Incoherent (Compton) scattering – λ of scattered beam increases due to partial loss of photon energy in collision with the core electrons (Compton effect).
- Coherent scattering – scattered beam has the same λ as the primary beam.

X-ray - matter interaction



e' beam - matter interaction

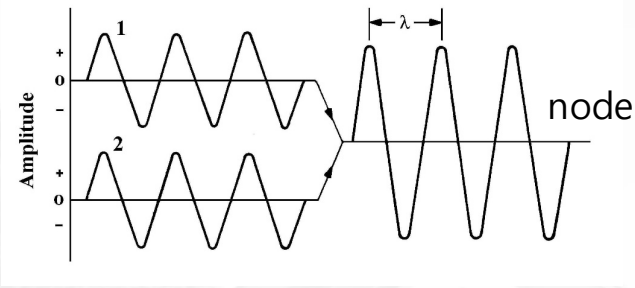
When an electron beam strikes a sample,



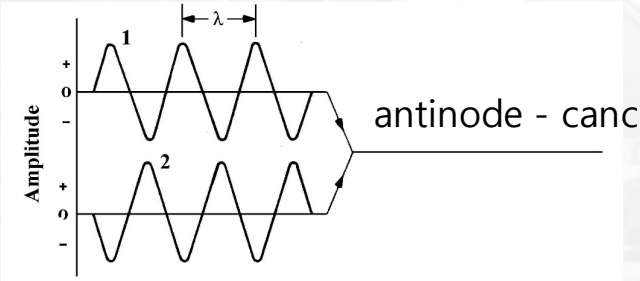
Interference

Interaction between two or more trains of waves of the same frequency emitted from coherent sources.

A series of stationary nodes and antinodes is established, known as interference.

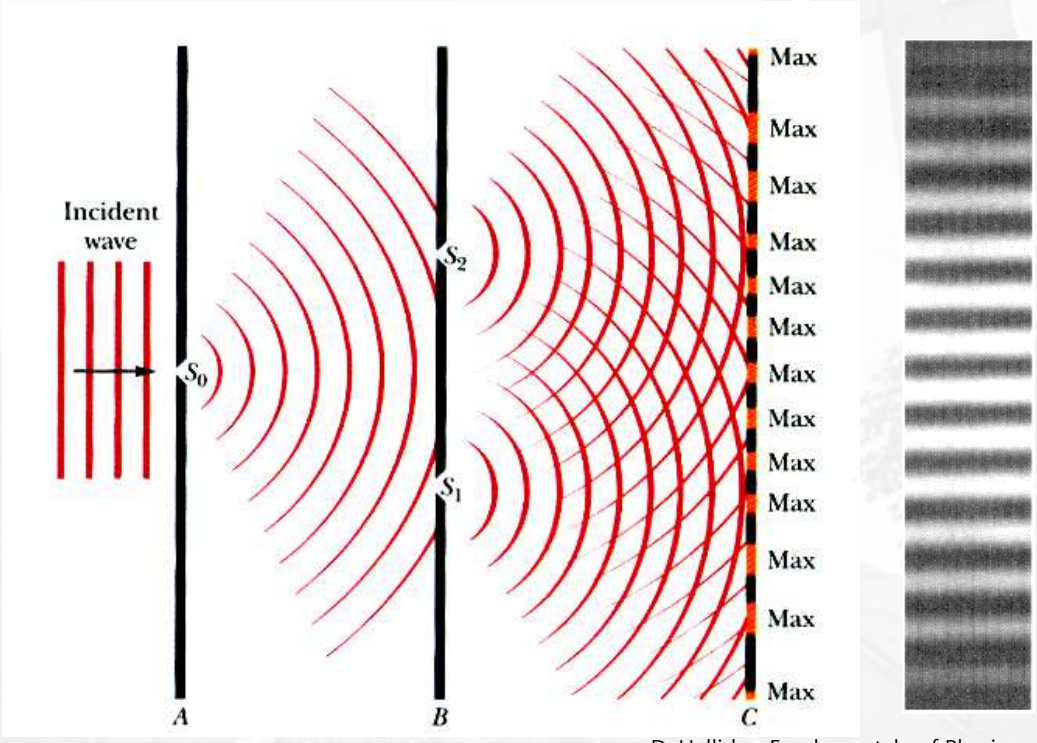


node - reinforcement



antinode - cancellation

Diffraction of Light



D. Halliday, Fundamentals of Physics

Diffraction, X-ray diffraction (XRD)

- Read
  - ✓ Pecharsky Chap 2, Hammond Chap 7, 8; Cullity Chap 2, Appendix 1; Krawitz Chap 3, 5
- Diffraction: reinforced coherent scattering
- Diffraction: coherent and elastic scattering of radiation

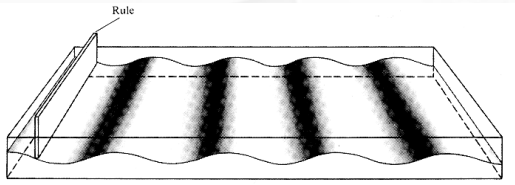
  by **periodic arrays of objects**

  resulting in **concerted constructive interference at specific angles**
- Diffraction occurs whenever wave motion encounters **a set of regularly spaced scattering objects**, provided the **wavelength of the wave motion is the same order of magnitude as the repeat distance** between the scattering centers.

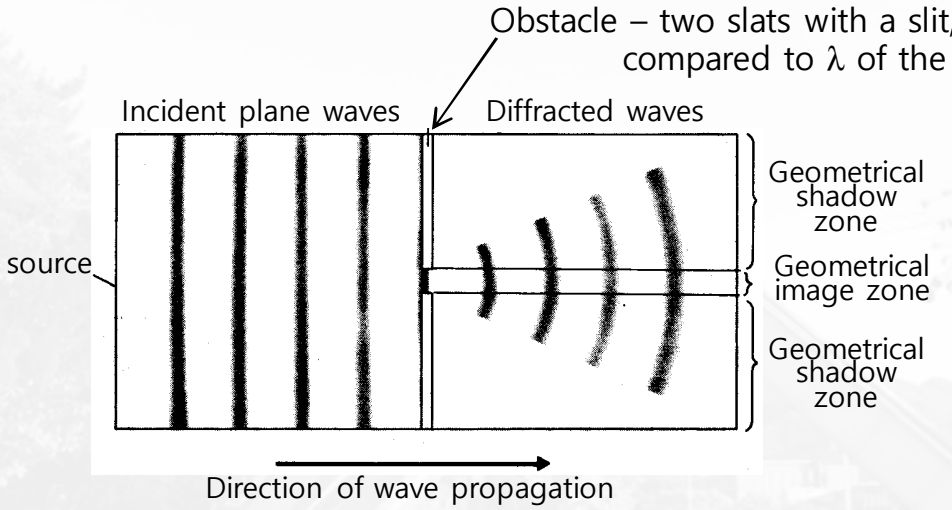
Atoms/ions/molecules in crystals	Lattice parameter of crystals	λ of X-ray (few Å)

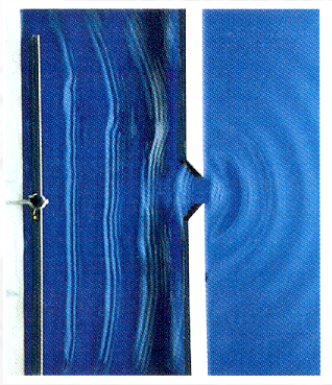
Diffraction of Water Waves

ripple tank; periodic dipping of a ruler
→ plane water wave



Sherwood page 94, 186

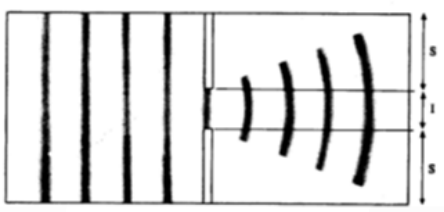




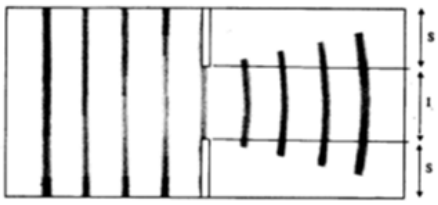
D. Halliday, Fundamentals of Physics

plane wave → obstacle → curved wave

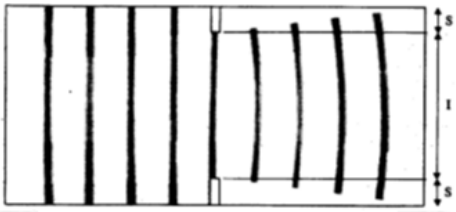
Diffraction of Water Waves



Slit width = λ



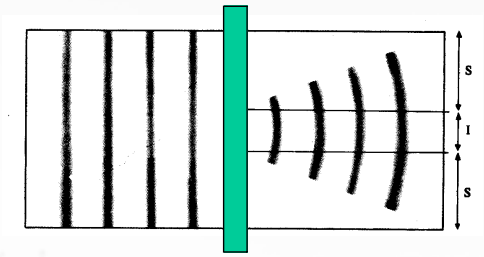
Slit width > λ

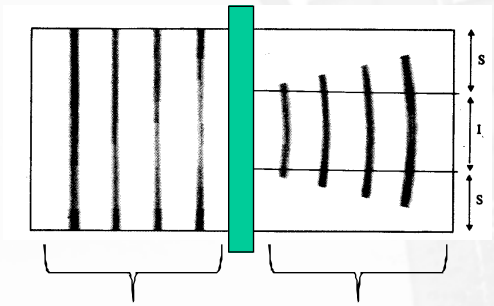


Slit width >>> λ

- When slit width is wide compared to the λ of the wave motion, the diffraction effects are masked.
- Info on the obstacle can be obtained from the diffraction effect.

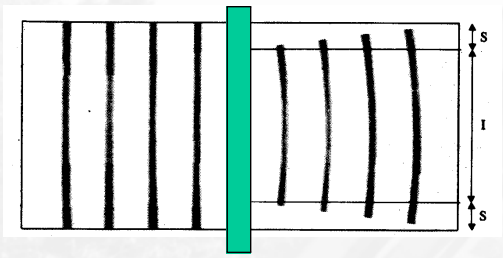
Diffraction of water waves





Waves do not know the presence of the obstacle.

Waves do know the presence of the obstacle.

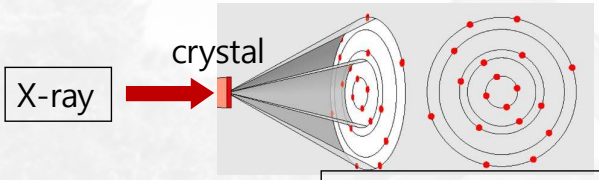


Diffracted wave contains additional information about the obstacle.

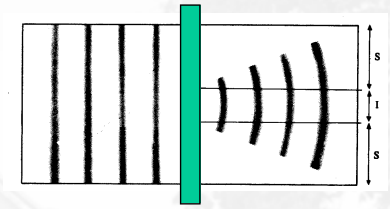
Diffraction pattern of an obstacle contains information on the structure of the obstacle.

Diffraction and information

- When a wave interacts with an obstacle, diffraction occurs.
- Diffracted wave contains additional information about the obstacle.
- The detailed behavior depends solely on the diffracting obstacles. → The diffracted waves can be regarded as containing information on the structure of the obstacles.

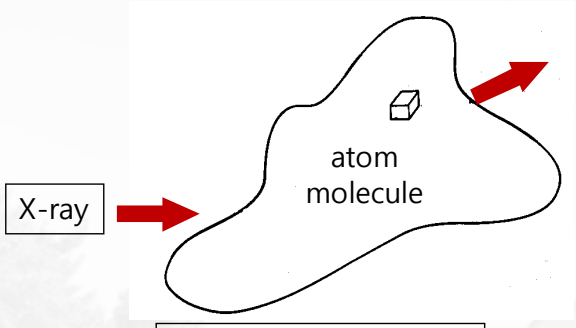


diffraction pattern



information about nature of molecules and the way they are packed → can reconstruct crystal structure

Diffraction of X-rays



crystal: well-defined, 3D ordered array

well-defined phase relationship between scattered waves

clear and precise diffraction pattern

- structure & physical properties of a diffracting obstacle
- mathematical expression for the diffraction pattern

We can predict diffraction pattern of any obstacle.

Diffraction pattern → Nature of the obstacle

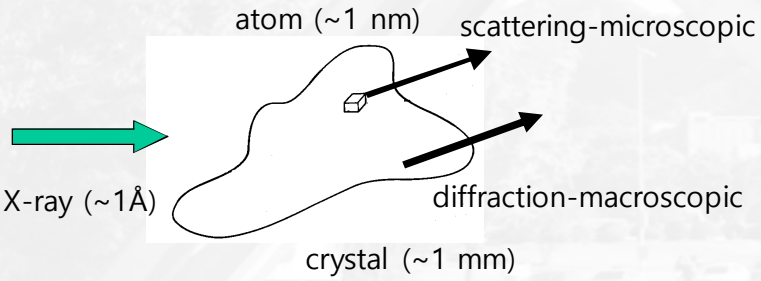
chap 6.6 & 6.7 of Sherwood & Cooper

Diffraction

Infinite plane wave with wave vector **k** and frequency w; $\psi = \psi_0 \, ei\,(\mathbf{k} \bullet \mathbf{r} - wt)$

- What happens when a wave motion interacts with an obstacle placed in its path?
- How is the wave equation modified to take account of the interaction of the wave with the obstacle?

- Scattering; wave-obstacle interaction
  when the dimensions of obstacles ≈ wavelength
- Diffraction; wave-obstacle interaction
  when the dimensions of obstacles ≫ wavelength



What is X-ray Diffraction?

- X-rays are an ideal probe of electromagnetic radiation for the study of crystals as the wavelength λ is of the same order as the distances between the atoms in crystals (Å, nm).
- Elastic scattering → no energy transfer & no wavelength change
- When the periodic array consists of crystalline matter of three dimensional (3-d) arrangement of atoms, monochromatic X-ray radiation diffracts in a number of different directions in 3-d space.

Why do we use x-rays ?
read Sherwood & Cooper, Chap 6.1~6.5

X-ray diffraction

- Diffraction occurs when each object in a periodic array scatters radiation coherently, producing concerted constructive interference at specific angles.
- The **electrons** in an atom **coherently** scatter light.
  - ✓ The electrons interact with oscillating electric field of light wave.
- Atoms in a crystal form a **periodic array** of coherent scatterers.
  - ✓ The wavelength of X rays are similar to the distance between atoms.
  - ✓ Diffraction from different planes of atoms produces a diffraction pattern, which contains information about the atomic arrangement within the crystal.
- X rays are also reflected, scattered incoherently, absorbed, refracted, and transmitted when they interact with matter.

Interaction of electromagnetic wave & matter

- Electric field (**E**) & magnetic field (**H**) propagate through the matter.
- Effect of **E** >>> effect of **H**
- When a particle of charge q is placed in **E,** $F = q\mathbf{E} = ma \quad a = \frac{q}{m}\mathbf{E}$
- **E** oscillates w/ very high ν. → make the charged particle oscillate. → particle radiates E-M wave (scattering).

  intensity of scattered wave $I \propto |a|^2 \propto (\frac{q}{m})^2\mathbf{E}^2$
- Neutron (no charge), proton, electron vs. **E**

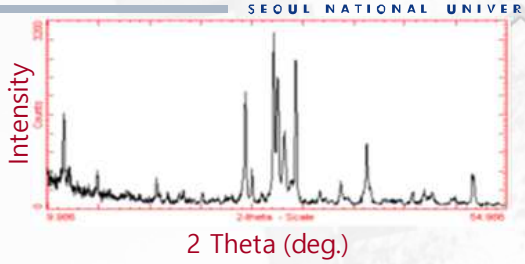
$m_p = 1800\, m_e$ $\quad I_p \propto (\frac{e}{m_p})^2$ $\quad \frac{I_e}{I_p} = (\frac{e}{m_e})^2 (\frac{m_p}{e})^2 = 1800^2$

I of radiation scattered by e′ $\quad I_e \propto (\frac{e}{m_e})^2$

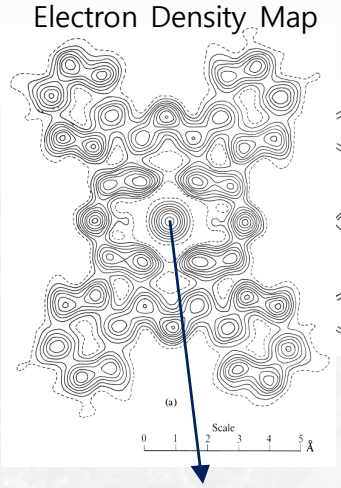
- XRD looks at the electron distribution in a crystal.

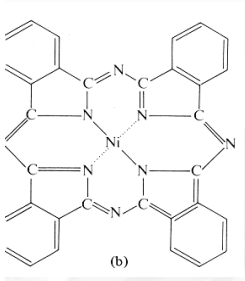
X-ray can see electrons.

In XRD, intensities of diffracted X-rays are measured.
→ can determine the location of electrons in the unit cell of the crystal.

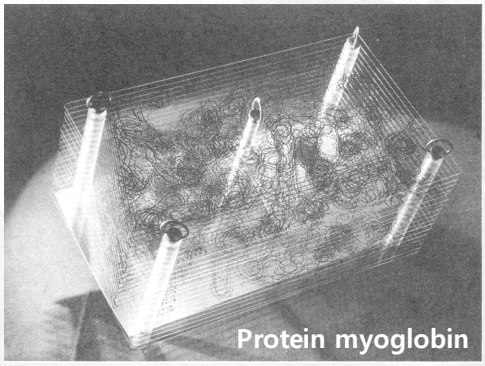


Electron Density Map





Nickel phthalocyanine



high e' density → location of a nucleus of a heavy atom

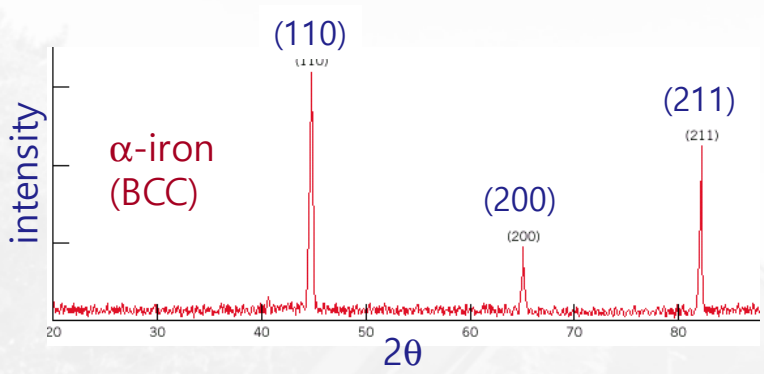
- Where the e' density is high, the presence of nucleus can be inferred.
  ← Heavy atoms scatter X-rays more effectively than light ones.
- Large e' density area can be the position of nuclei of heavy atoms.

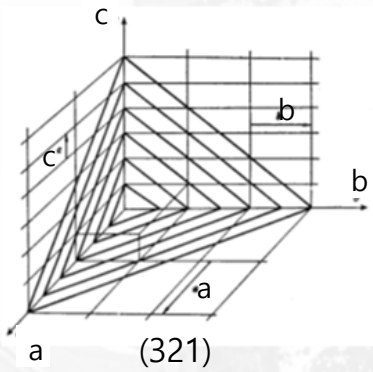
Interaction of X-ray & crystal

- XRD looks at the **electron distribution** in a crystal.
- XRD does not directly look at the positions of the nuclei of atoms.
- Atom of atomic number Z → intensity of scattered wave $\propto (\frac{Ze}{m_e})^2$
- High Z atoms (heavy atoms) are much more effective scattering centers. → **XRD cannot give much info on light elements.**
- Intensities of scattered X-ray → locations of electrons in one unit cell → electron density distribution (e' density map)

Diffraction pattern

- Each reflection (diffraction peak) has an (hkl) index and a measured intensity.
- Each reflection index defines a set of parallel planes that slice thru the crystal.





reflection ≠ diffraction
(see Cullity chapter 3.2)

Calculation of electron density; Fourier transform

- Electron density at (xyz) = the sum of contributions to the point (xyz), of waves scattered from the plane (hkl) whose amplitude depends on the number of electrons in the plane, **added with the correct relative phase relationship.**

1/Volume of unit cell

Phase associated with the structure factor amplitude

Can't measure φ_{hkl}

$$\rho_{xyz} = \frac{1}{V}\sum |F_{hkl}| \exp(i\varphi_{hkl}) \exp[-2\pi i(hx+ky+lz)]$$

Density @ (xyz)

Structure Factor Amplitude (hkl)

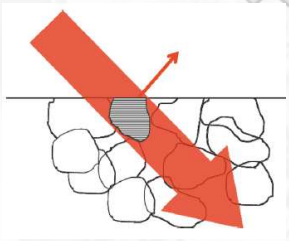
Wave scattered from the plane hkl

Can measure $|F_{hkl}|$

- Given all F(hkl) (amplitude & phase for each reflection), ρ(xyz) can be calculated.
- Given ρ(xyz) (i.e., the structure), F(hkl) can be calculated.
- **F, φ ← FT → ρ(xyz)**

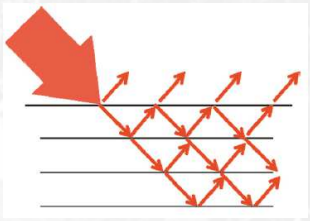
Kinematical vs. Dynamical theories of diffraction

- **Kinematical theory**



  - ✓ A beam scattered once is not scattered again.
  - ✓ Interaction of diffracted beam with crystal is negligibly small.
    - Crystal consists of individual mosaic blocks.
    - Size of the crystallites is small.
    - Misalignment of crystallites is large enough, so that interaction of X-ray with matter at length scale larger than the size of the mosaics is negligible.

- **Dynamical theory**



  - ✓ Accounts for scattering of diffracted beam & other interactions of waves inside the crystal.
  - ✓ Needed when crystals are nearly perfect or when there is a strong interaction of the radiation with the material (electron diffraction).

- Many dynamical effects (primary & 2ndary extinction, simultaneous diffraction, thermal diffuse scattering, etc.) are accounted for as corrections to the kinematical diffraction model.

Rigaku Journal, 25(2), 2009, X-ray thin film measurement techniques

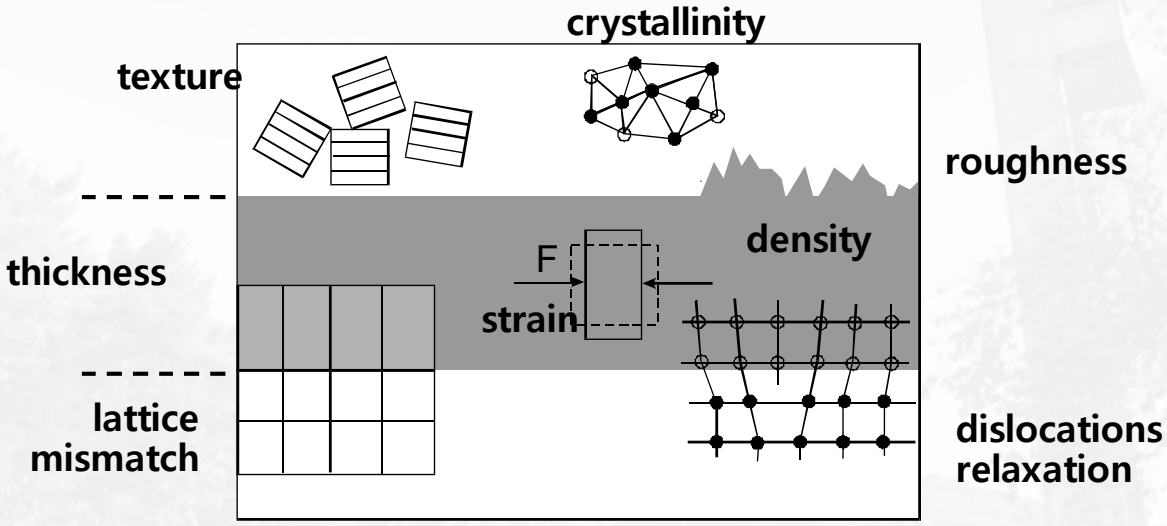
Range of Applications of X-Ray Analytical Methods

- Qualitative and quantitative element analysis (XRF; X-ray fluorescence)
- Qualitative and quantitative phase analysis (XRD)
- % crystallinity
- Micro-strain and crystallite size determination
- Residual stress and texture analysis
- Grazing incidence diffraction (GID)
- X-ray reflectometry (XRR; X-ray reflection)
- High Resolution X-ray Diffraction (HRXRD)
- Structure solution and refinement
- Micro-diffraction (phase identification, texture, stress…)
- Nano-structure investigations by small angle X-ray scattering (SAXS)

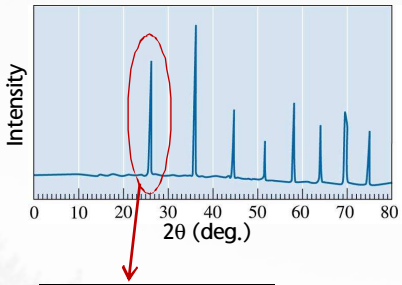
What can we do with XRPD?

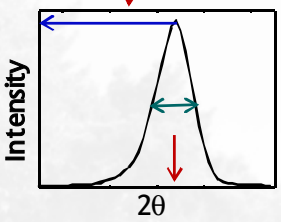
- **Qualitative phase analysis (Identification of unknown phases)**
- **Quantitative phase analysis**
- Accurate lattice parameter measurement
- % crystallinity
- Measurement of crystal size
- Measurement of internal elastic strains
- Preferred orientation measurement
- Cation site disorder
- Micro-diffraction (phase identification, texture, stress…)
- Structure refinement (vs. single crystal)

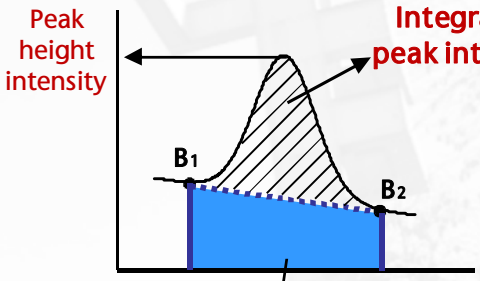
What can be measured by X-ray analysis of Thin Film ???



θ-2θ scan > 2θ, intensity, peak breadth







XRD를 이용한 구조 분석

position
intensity
shape

① →

← ②

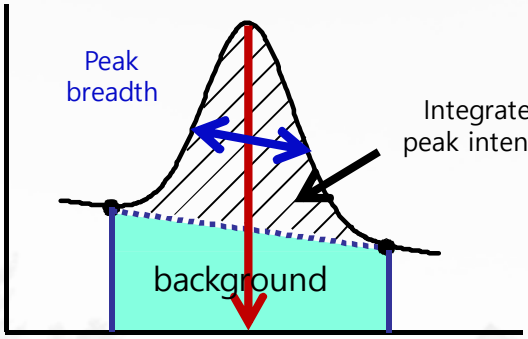
crystal structure
physical state

Peak position ← lattice parameter and crystal system (size & shape of unit cell)

Intensity ← info on the atoms in the unit cell (kind, position and occupancy of atoms in the unit cell)

Peak width ← size & strain, defects

Peak Position



Peak position

Direction of diffracted beam

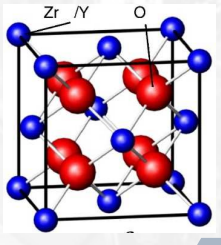
unit cell의 crystal system
lattice parameter

Size & Shape of unit cell

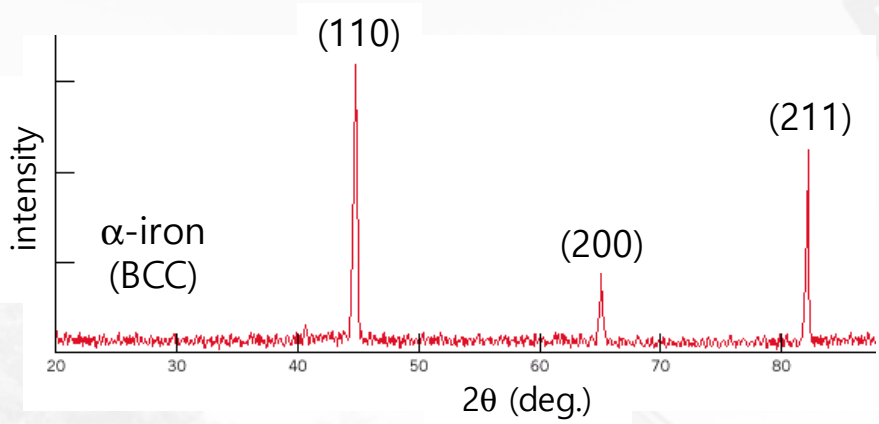
Crystal Structure of cubic "ZrO_2"

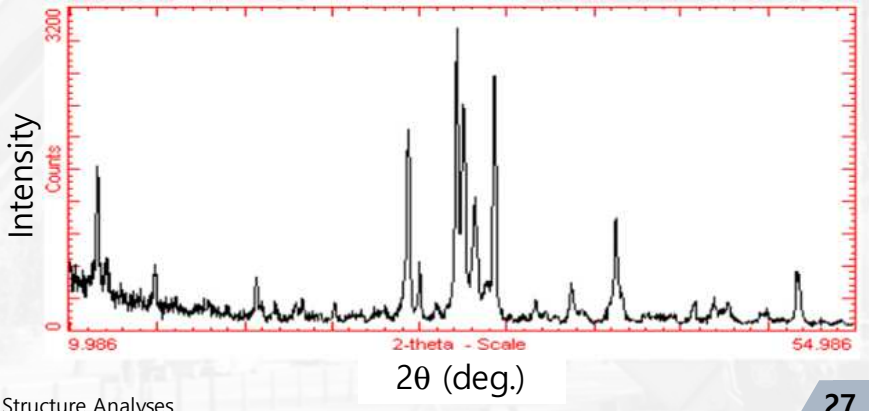
Space Group $Fm\bar{3}m$ (225)
→ cubic
Lattice Parameter a=5.11Å

Atom	x	y	z	B_{iso}	occupancy
Zr	0	0	0	1.14	1
O	0.25	0.25	0.25	2.4	1



XRD θ -2θ pattern



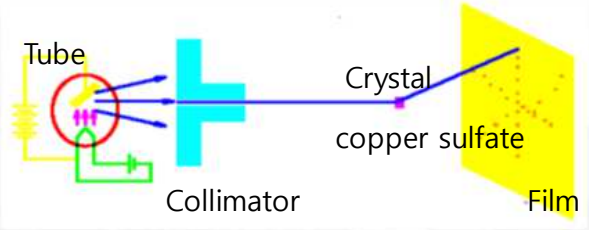


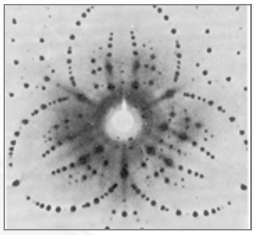
Max von Laue

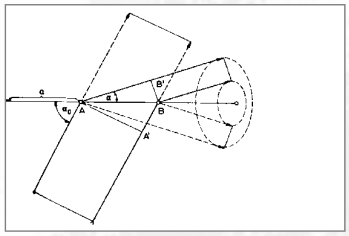
Laue's Experiment in 1912

Univ. of Munich

single crystal X-ray diffraction







Max von Laue put forward the conditions for scattering maxima, the Laue equations →

$$a(\cos\alpha-\cos\alpha_0) = h\lambda$$
$$b(\cos\beta-\cos\beta_0) = k\lambda$$
$$c(\cos\gamma-\cos\gamma_0) = l\lambda$$

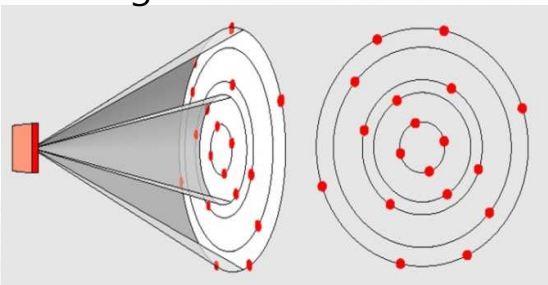
Proved

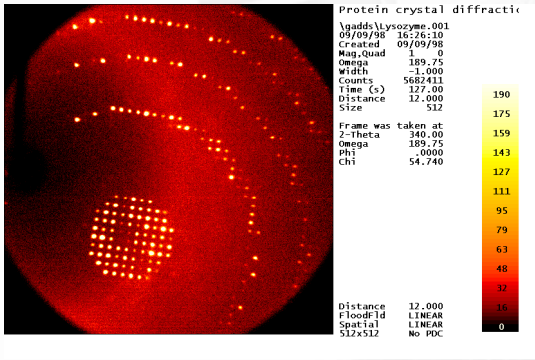
(1) wave nature of X-ray

(2) periodicity of the arrangement of atoms within a crystal

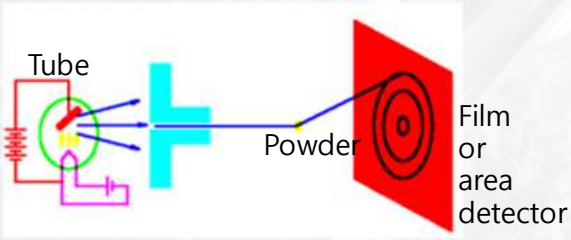
XRD of single crystal & powder

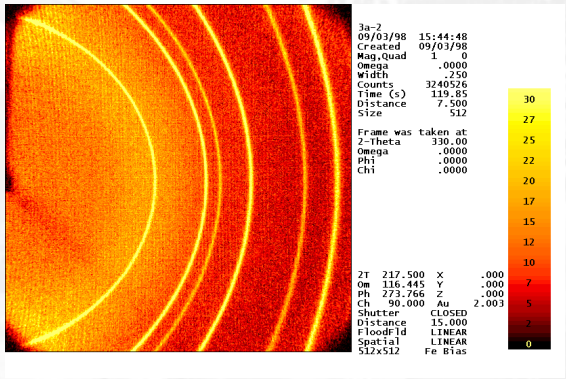
Single xtal diffraction

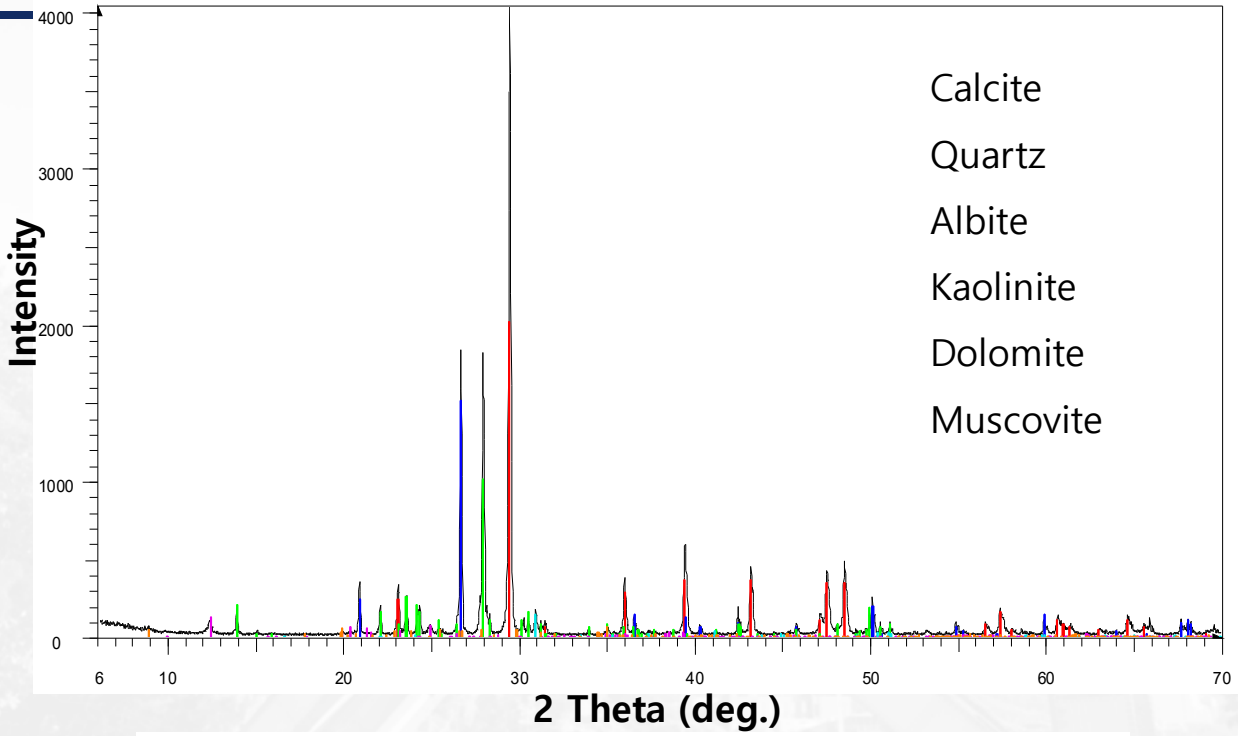




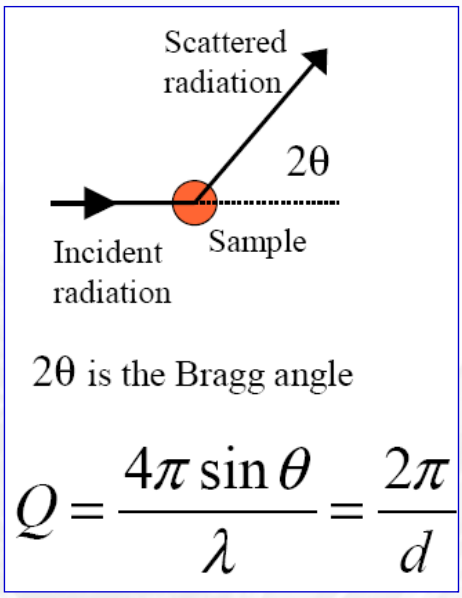
Powder diffraction





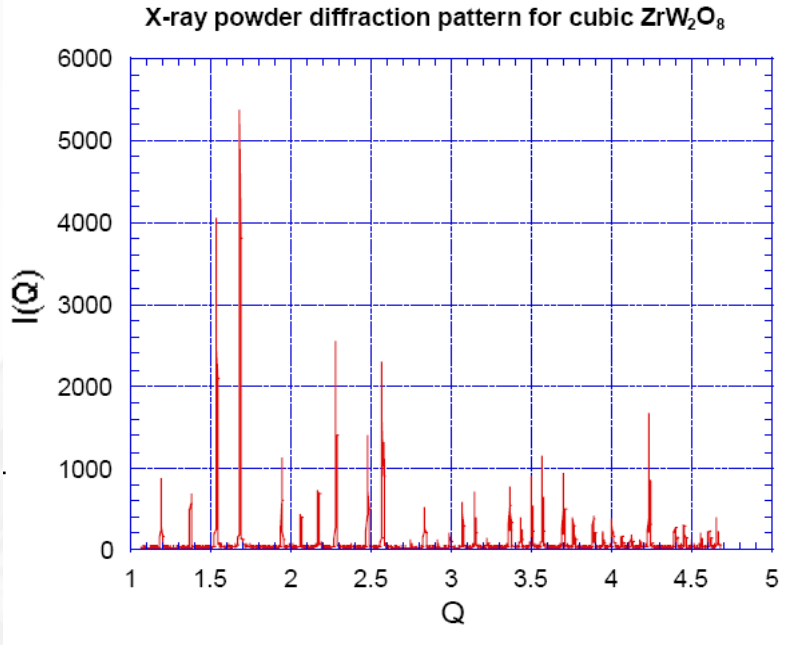


XRPD pattern



2θ is the Bragg angle

$$Q = \frac{4\pi \sin\theta}{\lambda} = \frac{2\pi}{d}$$



Camera vs. Diffractometer

- Diffraction camera
  - ✓ I is measured thru amount of blackening it produces on a film.
  - ✓ All diffraction lines recorded simultaneously. Variation in I of incident beam during exposure has no effect on the relative I.
  - ✓ Quantitative measurements of line position & intensity need at least two steps (recording pattern on the film + microphotometer record of the film).

- Diffractometer
  - ✓ I is measured directly by an electronic X-ray detector.
  - ✓ Diffraction lines recorded one after another → incident beam intensity must be kept constant → voltage & current needs to be stabilized.
  - ✓ Quantitative measurement of line position & intensity is made in one operation.

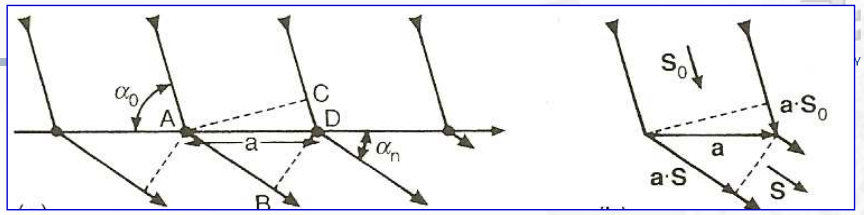
Laue vs. Bragg

➢ Laue

- ✓ Crystals consist of 3-D network of rows of atoms.
- ✓ Crystal behaves as a 3D diffraction grating.
- ✓ Laue equations

➢ Bragg

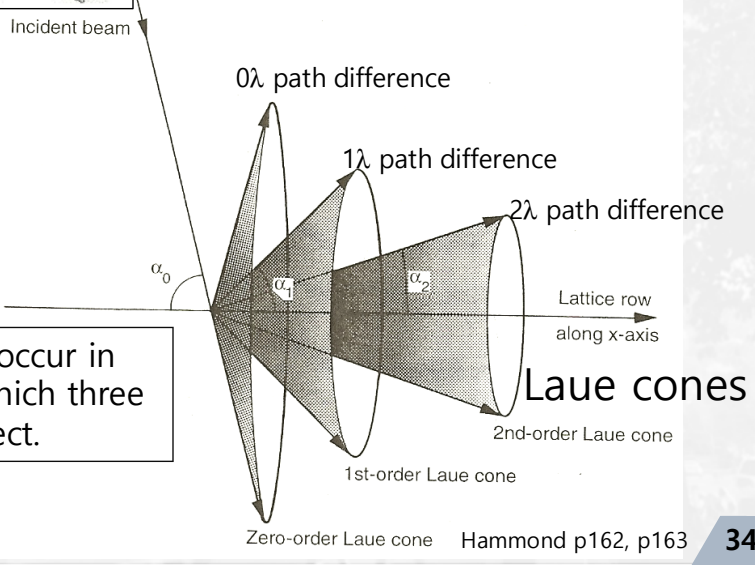
- ✓ Crystals consist of planes of atoms which behaves as reflecting planes.
- ✓ Strong reflected beam is produced when the path difference between reflections from successive planes in a family is equal to whole number of wavelengths.
- ✓ Bragg's law



$$(AB - CD) = a(\cos\alpha_n - \cos\alpha_0) = n_x\lambda$$

$$a(\cos\alpha_n - \cos\alpha_0) = \mathbf{a}\cdot(\mathbf{s} - \mathbf{s}_0) = n_x\lambda$$
$$b(\cos\beta_n - \cos\beta_0) = \mathbf{b}\cdot(\mathbf{s} - \mathbf{s}_0) = n_y\lambda$$
$$c(\cos\gamma_n - \cos\gamma_0) = \mathbf{c}\cdot(\mathbf{s} - \mathbf{s}_0) = n_z\lambda$$

Laue equation



Laue cones

Diffracted beams only occur in those directions along which three Laue cones intersect.

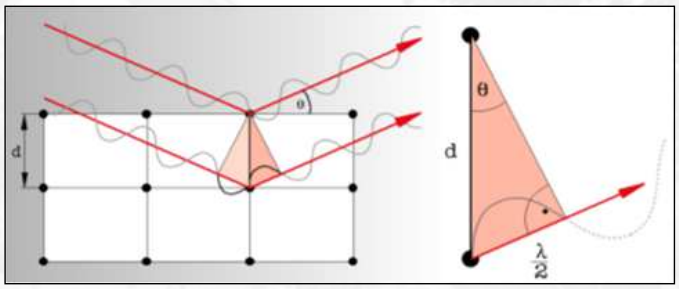
Bragg´s Law

- Braggs – W.H. and W.L. Bragg (father & son)
- 1913 – 1914
- Realized that X-ray scattering could be imagined as reflections from planes.
- Showed how it could be used to determine atomic structure of NaCl.
- Nobel prize 1915

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

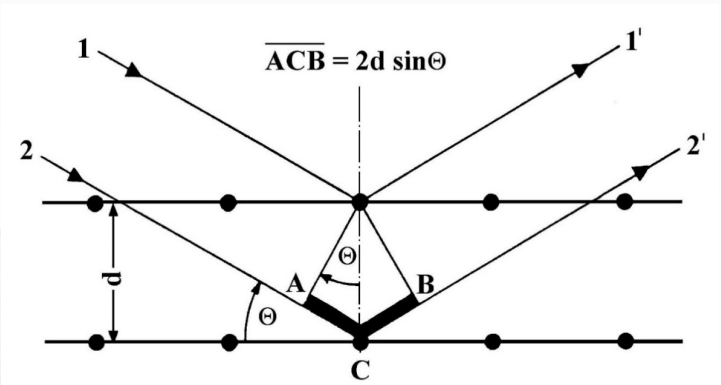
d = distance of lattice planes

λ = x-ray wavelength



reflection ≠ diffraction
(see Cullity chapter 3.2)

Interference: Bragg's Law



$$\frac{'AC'}{d} = \sin\Theta$$

$$'AC' = d\sin\Theta$$

$$'ACB' = 2d\sin\Theta$$

$$'ACB' = n\lambda$$

Constructive interference

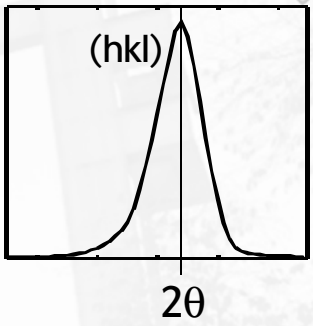
$$\boxed{n\lambda = 2d\sin\Theta}$$

n = 1, 2, 3, ... (Reflection order)

Peak position – d value

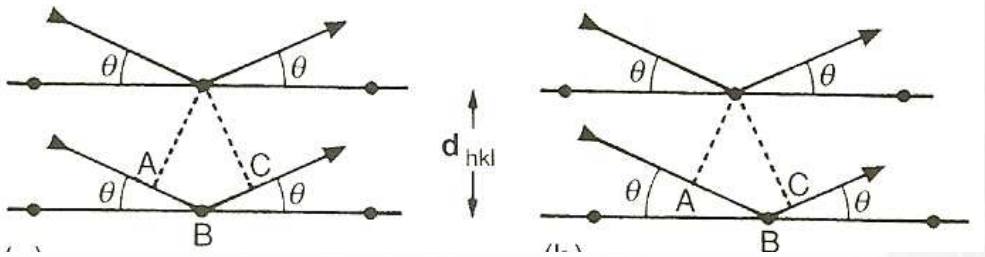
Bragg´s law $\lambda = 2d\sin\theta$

λ, θ → d can be calculated. → lattice parameter can be calculated.



$1/d^2 = (h^2 + k^2)/a^2 + l^2/c^2$ d vs lattice parameter relation in a tetragonal cell

- h, k, l --- Miller indices of the peaks
- a, c --- lattice parameter
- a, c known → can get θ, the peak position.
- θ, peak position known → can get lattice parameters.



The path difference between the waves scattered by atoms from adjacent (hkl) lattice planes of spacings d_{hkl} is given by

$$(AB + BC) = (d_{hkl}\sin\theta + d_{hkl}\sin\theta) = 2d_{hkl}\sin\theta.$$

Hence for constructive interference:

$$n\lambda = 2d_{hkl}\sin\theta,$$

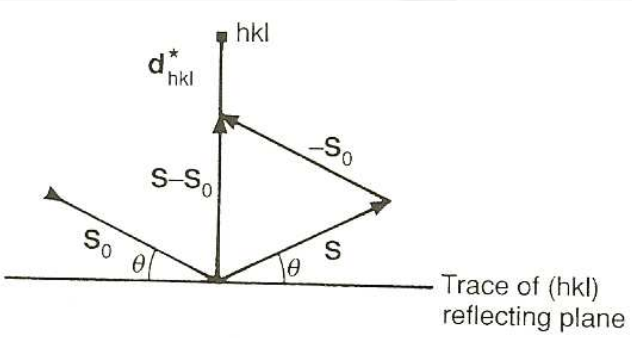
where n is an integer (the order of reflection or diffraction).

$$\lambda = 2\left(\frac{d_{hkl}}{n}\right)\sin\theta = 2d_{nh\,nk\,nl}\sin\theta$$ Bragg's law

Bragg's law

$$|\mathbf{s} - \mathbf{s}_0| = 2\sin\theta$$

$$|\mathbf{d}^*_{hkl}| = 1/d_{hkl}$$



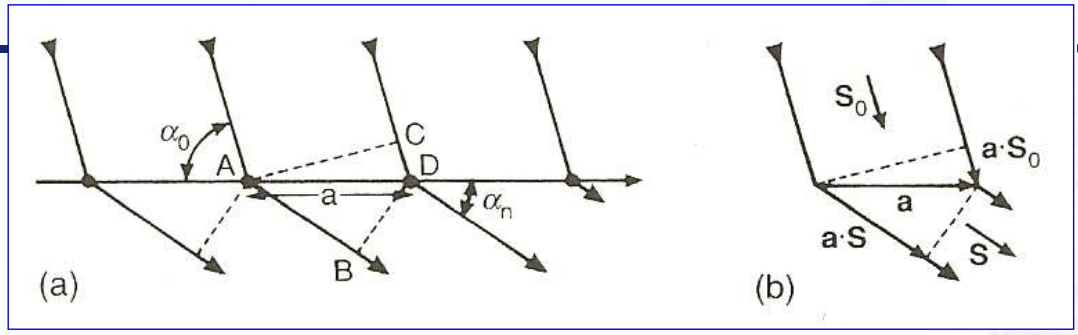
$$\lambda = 2\left(\frac{d_{hkl}}{n}\right)\sin\theta = 2d_{nh\,nk\,nl}\sin\theta$$

$$\frac{(\mathbf{s}-\mathbf{s}_0)}{\lambda} = \mathbf{d}^*_{hkl} = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^*$$

$$\underline{\mathbf{a}\cdot(\mathbf{s}-\mathbf{s}_0) = n_x\lambda} = \mathbf{a}\cdot\mathbf{d}^*_{hkl}\cdot\lambda = \mathbf{a}\cdot(h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^*)\lambda = h\lambda$$

Laue equation

$n_x = h,\ n_y = k,\ n_z = l$



$$(\mathrm{AB} - \mathrm{CD}) = a(\cos\alpha_n - \cos\alpha_0) = n_x\lambda$$

Laue equation Laue indicies

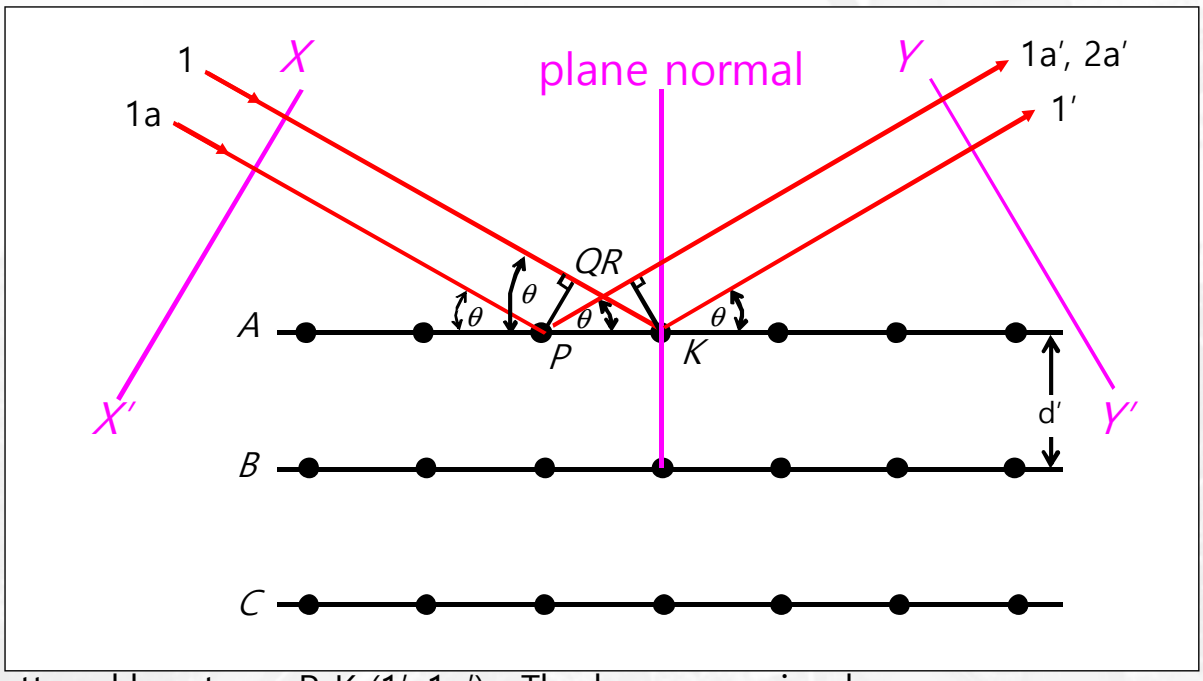
$$a(\cos\alpha_n - \cos\alpha_0) = \mathbf{a}\cdot(\mathbf{s}-\mathbf{s}_0) = n_x\lambda = \mathbf{h\lambda}$$

$$b(\cos\beta_n - \cos\beta_0) = \mathbf{b}\cdot(\mathbf{s}-\mathbf{s}_0) = n_y\lambda = \mathbf{k\lambda}$$

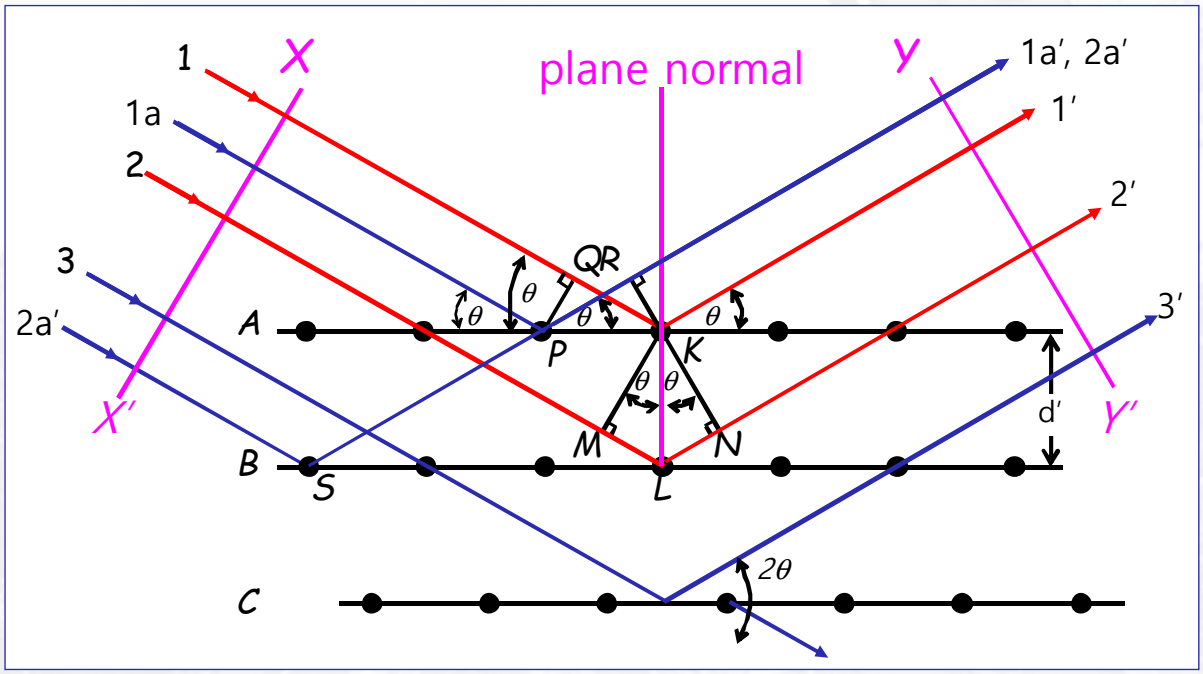
$$c(\cos\gamma_n - \cos\gamma_0) = \mathbf{c}\cdot(\mathbf{s}-\mathbf{s}_0) = n_z\lambda = \mathbf{l\lambda}$$

3rd order diffaction from (111) = 1st order diffraction from 333 (Laue index)
333 planes have 1/3 spacing of (111).

Laue Index; see Hammond p141



Scattered by atoms P, K (1′, 1a′) : The beams are in phase.



Scattered by atoms P, K (1′, 1a′) : The beams are in phase.

Scattered by atoms K and L : ML + LN = 2d′sinΘ = nλ

For fixed value of λ there can be several angles of incidence; Θ_1, Θ_2, Θ_3.

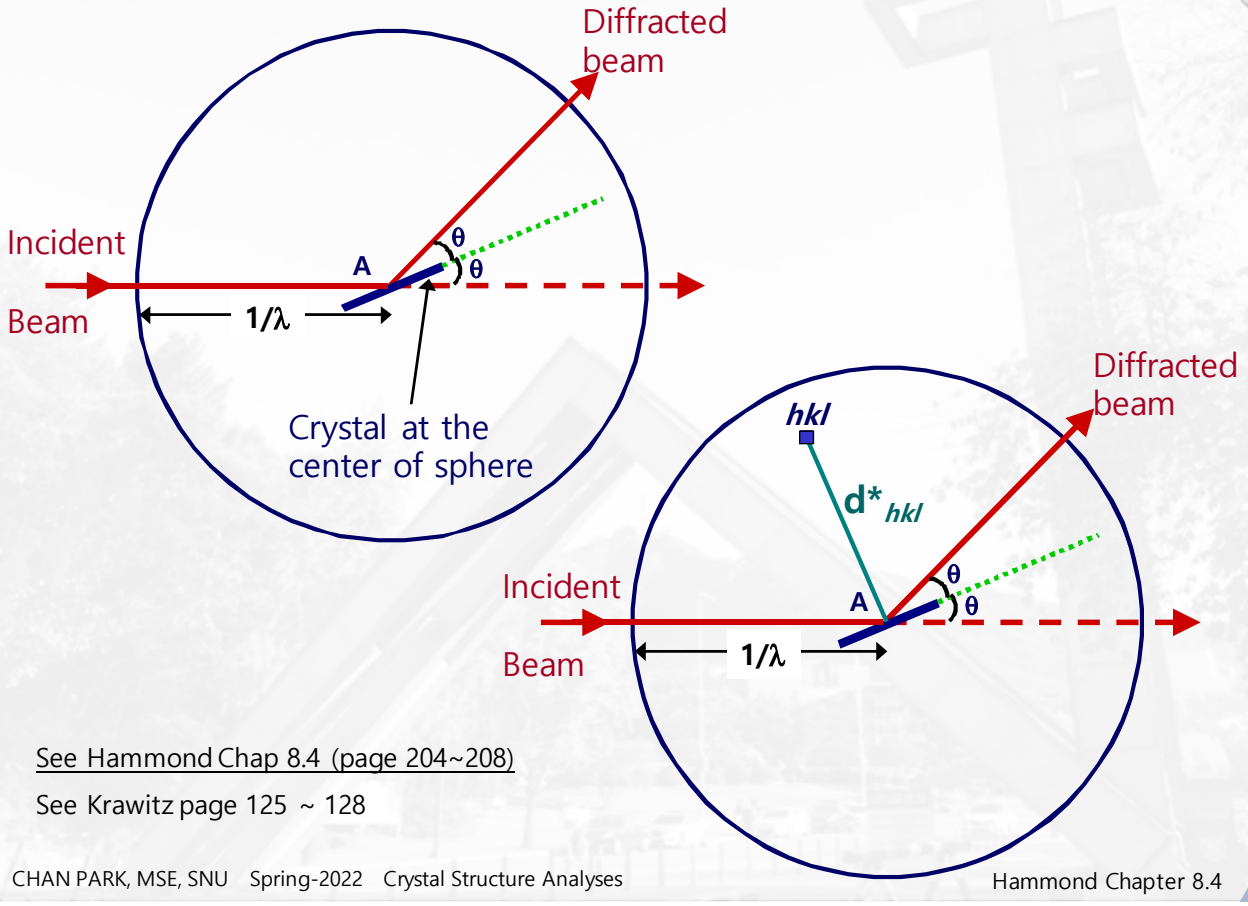
$\lambda = 2d \sin\theta$

- Condition for diffraction
  - ✓ Incident beam
  - ✓ Diffracted beam
  - ✓ Plane normal

  } co-planar

- $\sin\theta = \lambda/2d$ (e.g. d = 4 Å)
  - ✓ If λ = 1000 Å, $\sin\theta$ = 125 → The crystal could not possibly diffract. (No θ can meet the diffraction condition.)
  - ✓ If λ = 0.01 Å, $\sin\theta$ = 0.00125 → Diffraction angle too small to be measured
  - ✓ If λ = 2 Å, $\sin\theta$ = 0.25, θ = 14.5° → Diffraction angle can be easily measured

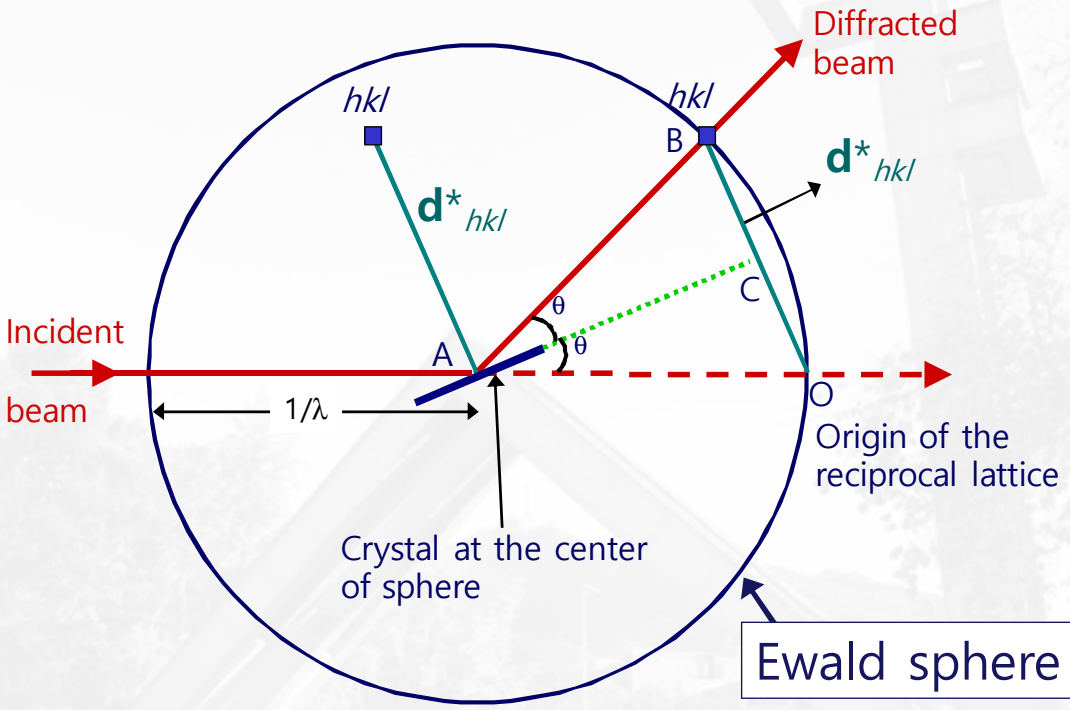
Ewald reflecting sphere



See Hammond Chap 8.4 (page 204~208)

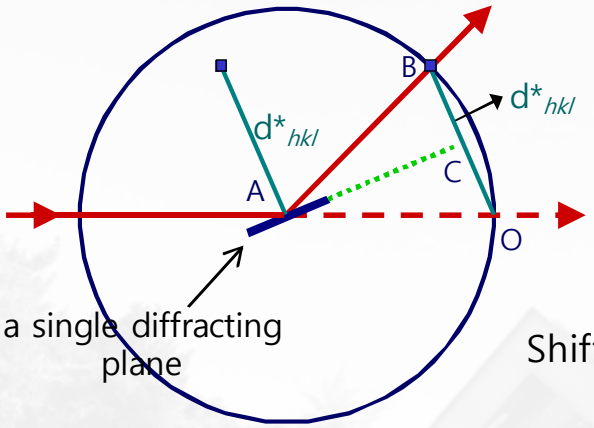
See Krawitz page 125 ~ 128

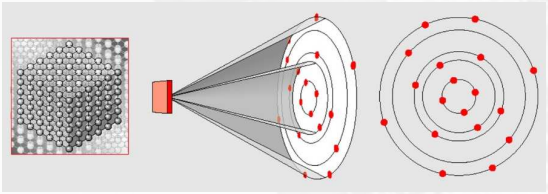
Ewald reflecting sphere



$|\mathbf{OC}| = (1/\lambda)\sin\theta = \frac{1}{2}|\mathbf{d}^*_{hkl}| = \frac{1}{2}(1/d_{hkl}) \rightarrow \lambda = 2d_{hkl}\sin\theta$

Ewald reflecting sphere



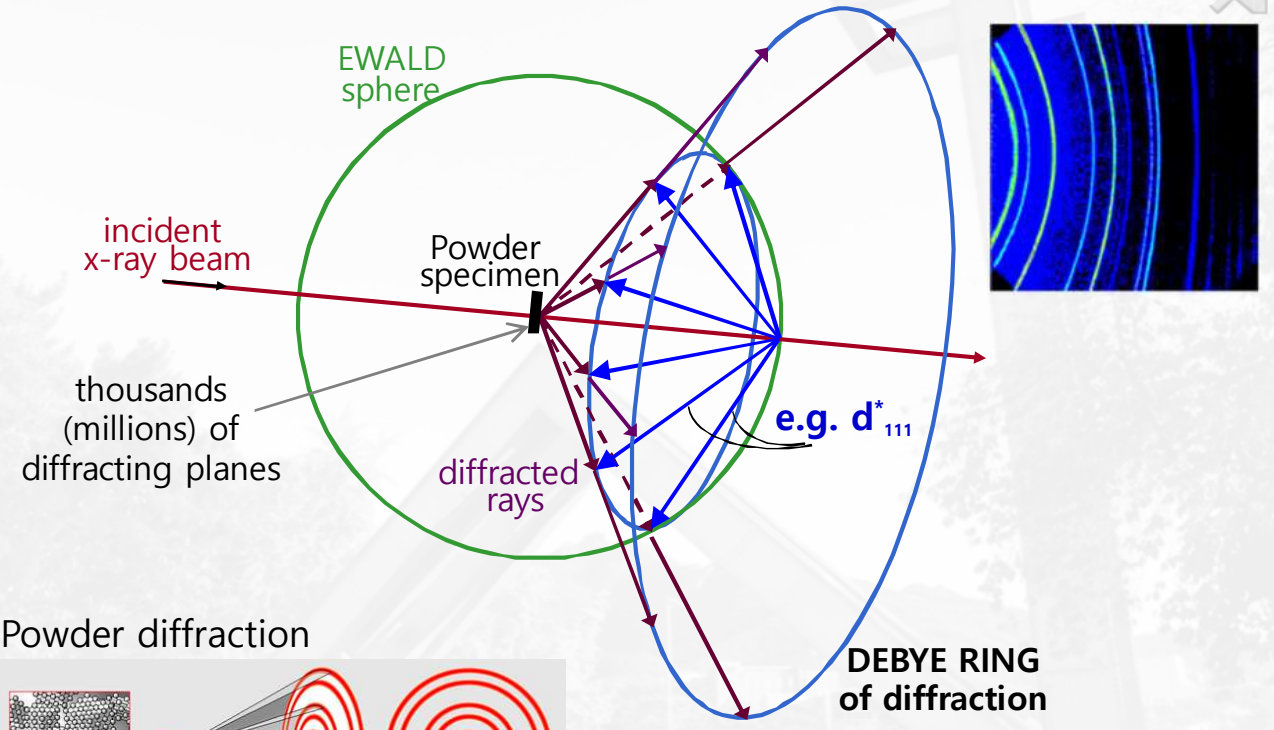


Single Crystal Diffraction

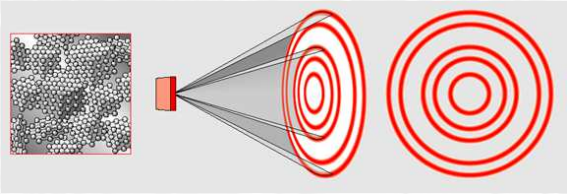
Shift origin from A to O → **OB** = $\mathbf{d}^*_{hkl}$

- Bragg's law ≡ reciprocal lattice point for reflecting plane (hkl) should intersect the sphere.
- If the reciprocal lattice point does not intersect the sphere, then the Bragg's law is not satisfied. → no diffracted beam

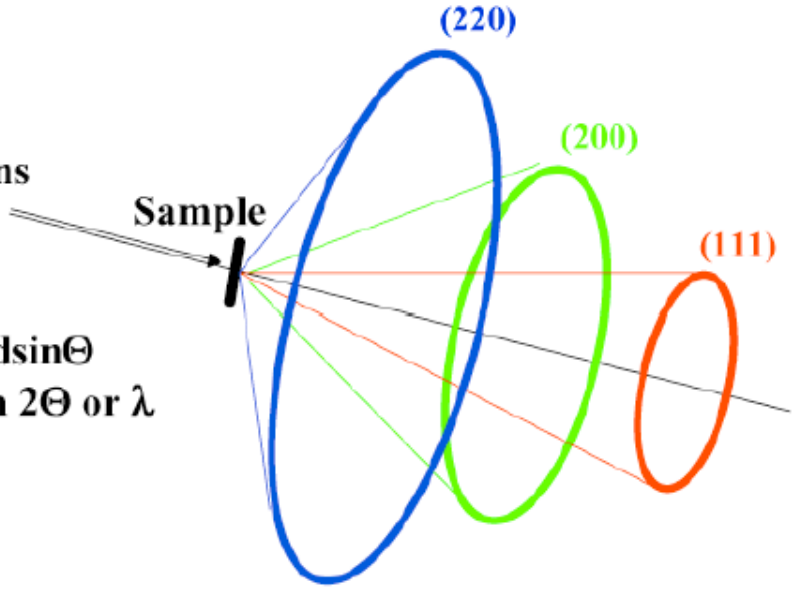
Origin of powder diffraction pattern



Powder diffraction



Powder Diffraction gives Scattering on Debye-Scherrer Cones

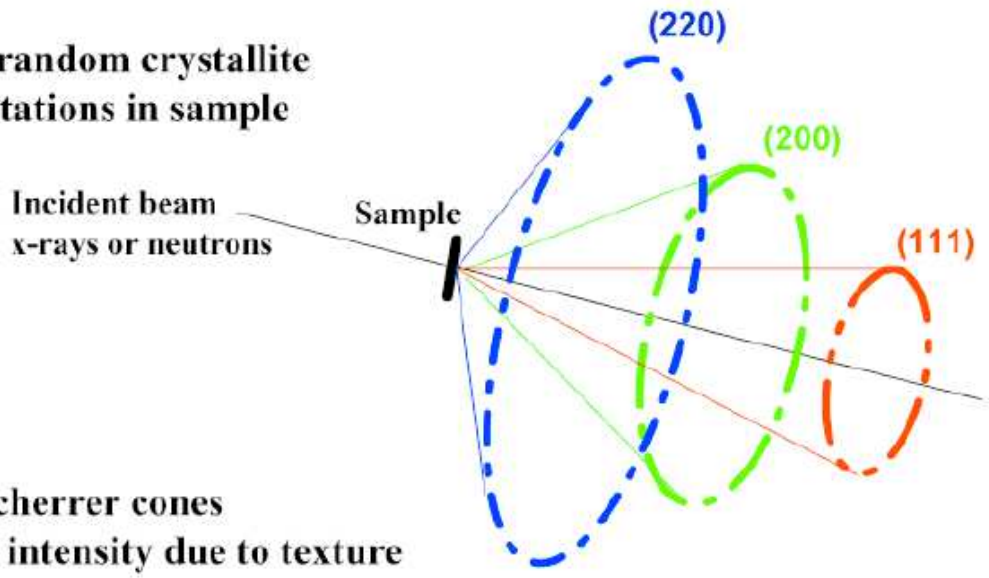


Bragg's Law $\lambda = 2d\sin\Theta$

Powder pattern – scan 2Θ or λ

Texture Measurement by Diffraction

Non-random crystallite orientations in sample

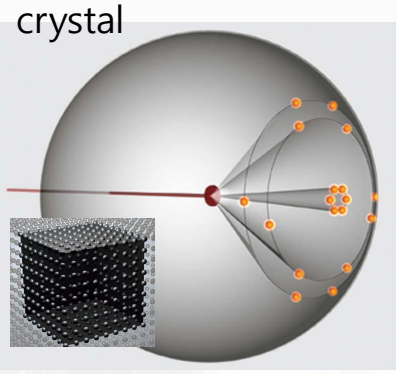


Debye-Scherrer cones

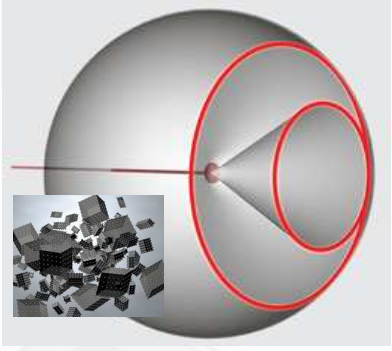
- uneven intensity due to texture
- different pattern of unevenness for different hkl's
- intensity pattern changes as sample is turned

Debye rings from ----

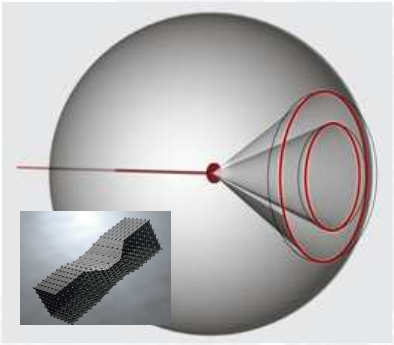
single crystal



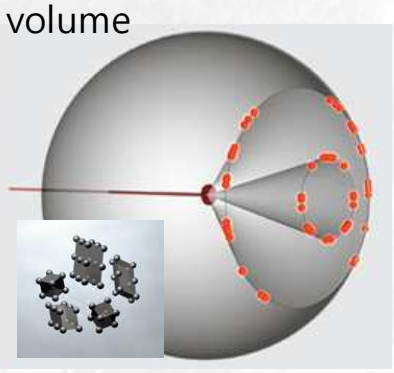
powder



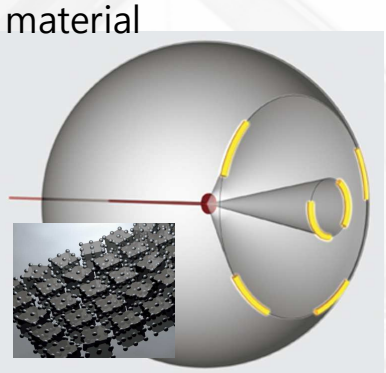
strained material



small volume

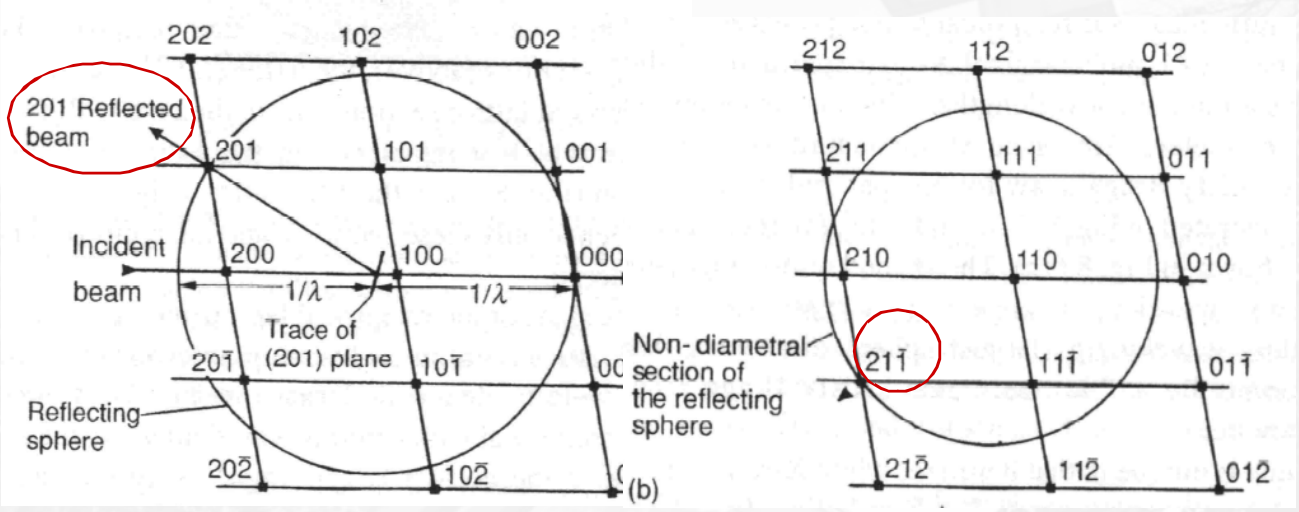


textured material



Ewald reflecting sphere

Section of reciprocal lattice of a monoclinic crystal ⊥ b*

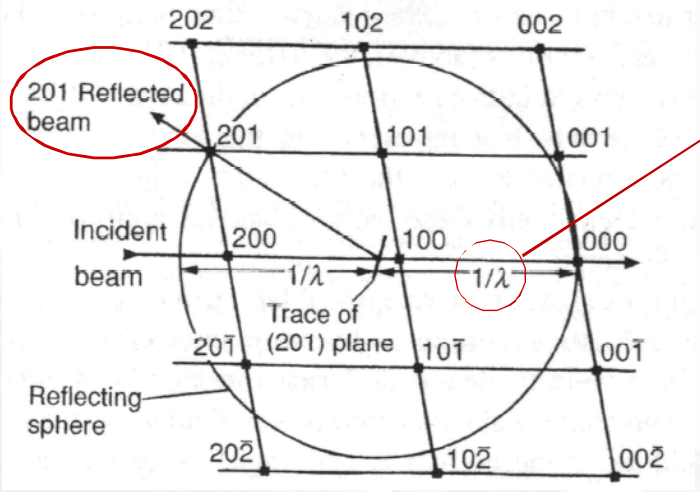


h0l reciprocal lattice section

h1l reciprocal lattice section

Origin of the reciprocal lattice is not at the center of the sphere, but is at the point where the direct beam exits the sphere.

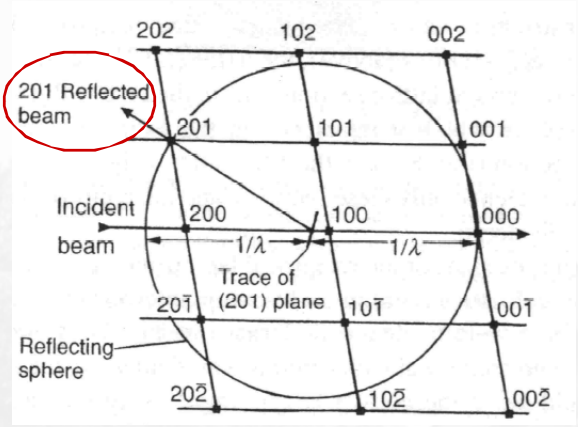
Ewald reflecting sphere



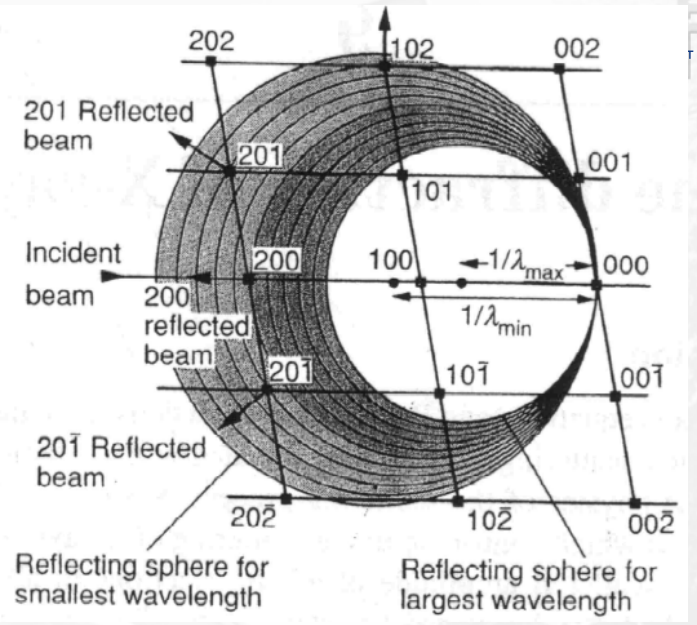
Change λ → radius of sphere changes → other points can intersect sphere

If λ can change continuously → other planes can reflect as their reciprocal lattice points successively intersect the sphere. → Laue's original X-ray experiment using white radiation

Ewald reflecting sphere



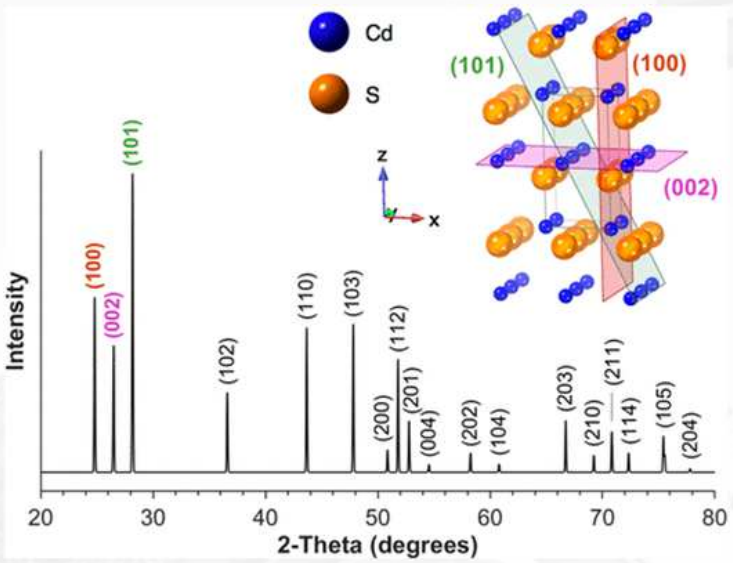
Hammond page 200



Hammond page 201

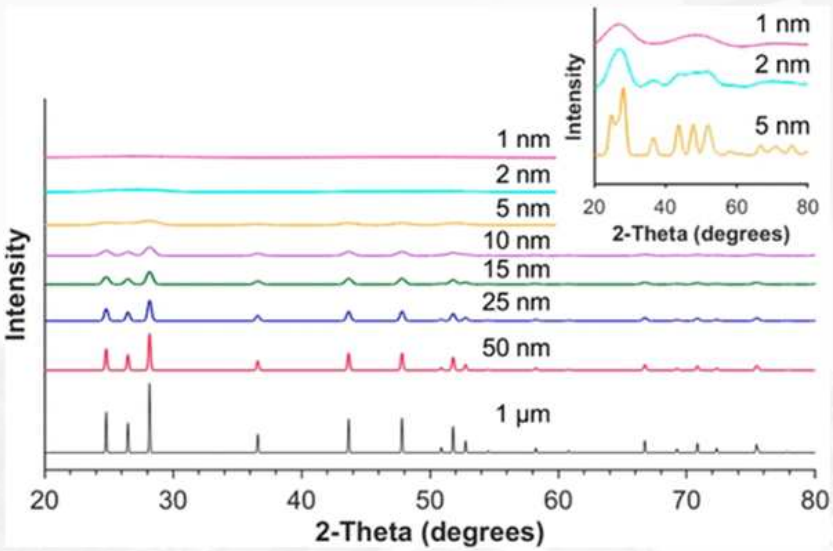
- All the planes in the shaded region satisfy Bragg's law for the particular sphere on which they lie (for that particular λ).
- Monochromatic radiation → crystal and the sphere should move to have more intersection (to have diffracted beams from more planes).

X-ray Powder Diffraction



- Simulated and indexed powder XRD pattern for bulk (1 μm) wurtzite CdS.
- The inset shows the crystal structure of wurtzite CdS with the (100), (002), and (101) planes highlighted.

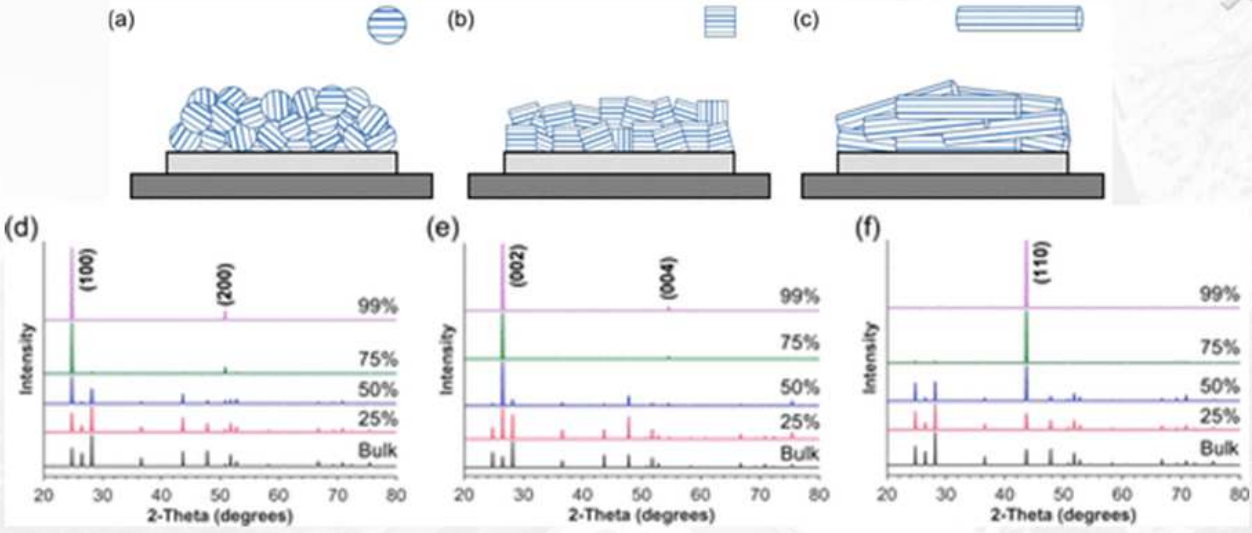
X-ray Powder Diffraction > size broadening



- Simulated powder XRD patterns for wurtzite CdS spherical particles of different sizes that range from 1 μm to 1 nm.
- The inset shows the 1, 2, and 5 nm XRD patterns on an expanded *y*-axis scale for clarity.

ACS Nano 2019, 13, 7, 7359–7365

X-ray Powder Diffraction > texture



- Graphical representation of preferred orientation for nanoparticles having different shapes: (a) spheres, (b) cubes, and (c) rods.
- Simulated XRD patterns for varying degrees of alignment (*i.e.*, preferred orientation) of wurtzite CdS particles along specific crystallographic directions: (d) [100], (e) [001], and (f) [110].

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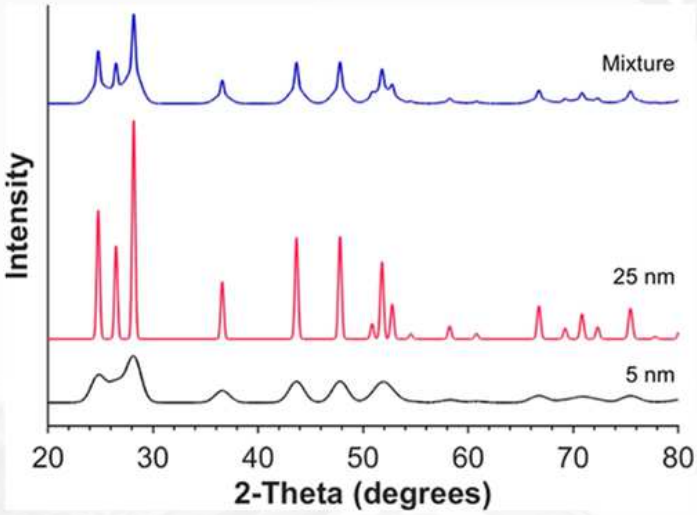
X-ray Powder Diffraction



- Simulated wurtzite CdS powder X-ray diffraction patterns of (bottom) 5 nm particles, (middle) 25 nm particles, and (top) a mixture that contains 75% 5 nm particles and 25% 25 nm particles.

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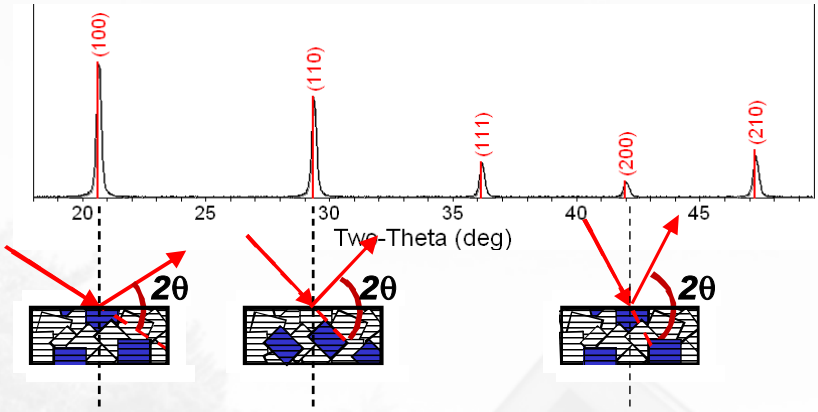
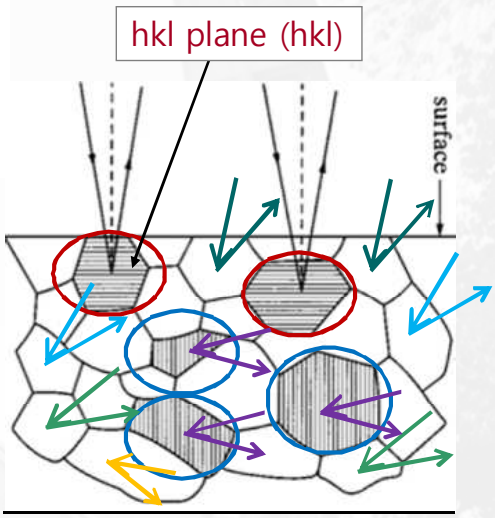
X-ray diffraction





Diffraction occurs only from red grains

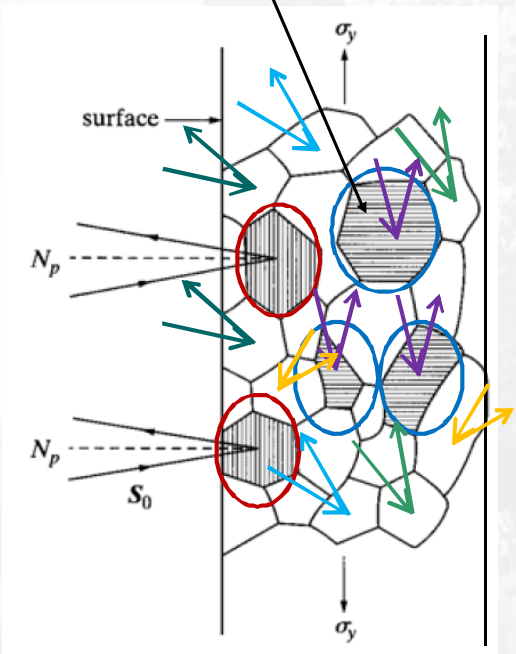
No diffraction from other grains

Scott A Speakman Cullity 3rd ed. p441

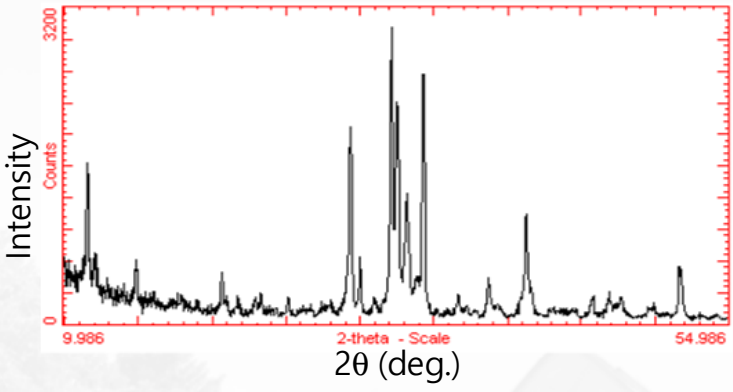
$\lambda = 2d \sin \Theta$

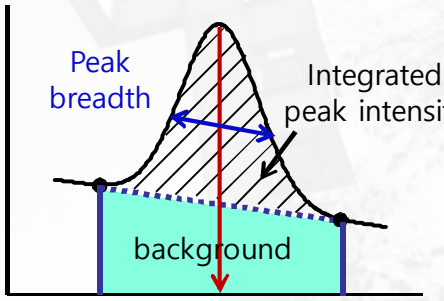
The value of d can be obtained from the peak position (2θ) of the XRD pattern.

The change of d can be obtained from XRD @ many different angles. → info on strain → info on stress

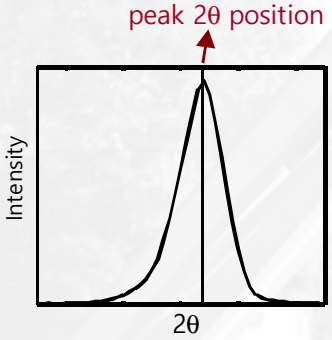


Peak position is determined by ---





Size & Shape of unit cell

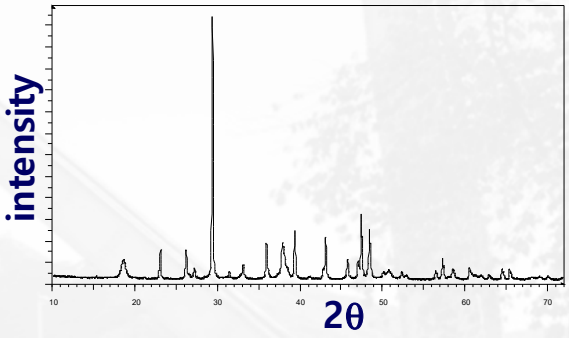


XRD peak
Asymmetric profile
due to axial divergence

Neutron diffraction peak shape is symmetrical.

2θ & intensity of XRD pattern

- 2θ
  - ✓ Size & shape of the unit cell (← λ = 2d sinθ)
- Intensity
  - ✓ Atomic scattering factor
  - ✓ Structure factor (atomic position, occupancy, etc.)
  - ✓ Polarization
  - ✓ Multiplicity
  - ✓ Temperature
  - ✓ Microabsorption
  - ✓ Crystallite size
  - ✓ Residual stress
  - ✓ Preferred orientation (texture)
  - ✓ Degree of crystallinity
  - ✓ Anomalous scattering
  - ✓ Source intensity, voltage drift, take-off angle, slit width, axial divergence, detector dead time, etc.



- 2theta
  - ✓ Geometry (crystal system, lattice parameter) (shape & size)
- Intensity
  - ✓ Atom type
  - ✓ Arrangement
  - ✓ Orientation
- Shape of diffraction lines
  - ✓ Instrument broadening
  - ✓ Particle dimension
  - ✓ Strain

D-spacing accuracy

- Diffractometer misalignment
- Specimen displacement error
- Specimen transparency error
- Problems in establishing true peak position
- Background
- Kα2
- ---

Factor affecting the Relative Intensity of Bragg Reflections

- Structure sensitive
  - ✓ Atomic scattering factor
  - ✓ Structure factor
  - ✓ Polarization
  - ✓ Multiplicity
  - ✓ Temperature
- Sample sensitive
  - ✓ Absorption
  - ✓ Degree of crystallinity
  - ✓ Particle orientation
- Instrument sensitive
  - ✓ Absolute intensities
    - Source intensity
    - Diffractometer efficiency
    - Take-off angle of tube
    - Receiving slit width
    - Axial divergence allowed
  - ✓ Relative intensities
    - Divergence slit aperture
    - Detector dead-time
- Measurement sensitive
  - ✓ Method of peak area measurement
  - ✓ Method of background subtraction
  - ✓ Kα2 stripping or not
  - ✓ Degree of data smoothing employed

Crystal structure determination

- Two step process

(1) Determination of the size & shape of the unit cell ← peak position

(2) Determination of lattice type & distribution of the atoms in the structure ← intensities of the diffraction spots