SOLID STATE PHYSICS
FYSC13: Powder X-ray Diffraction Lab

Mehran Sedrpooshan
Andrea Grespi

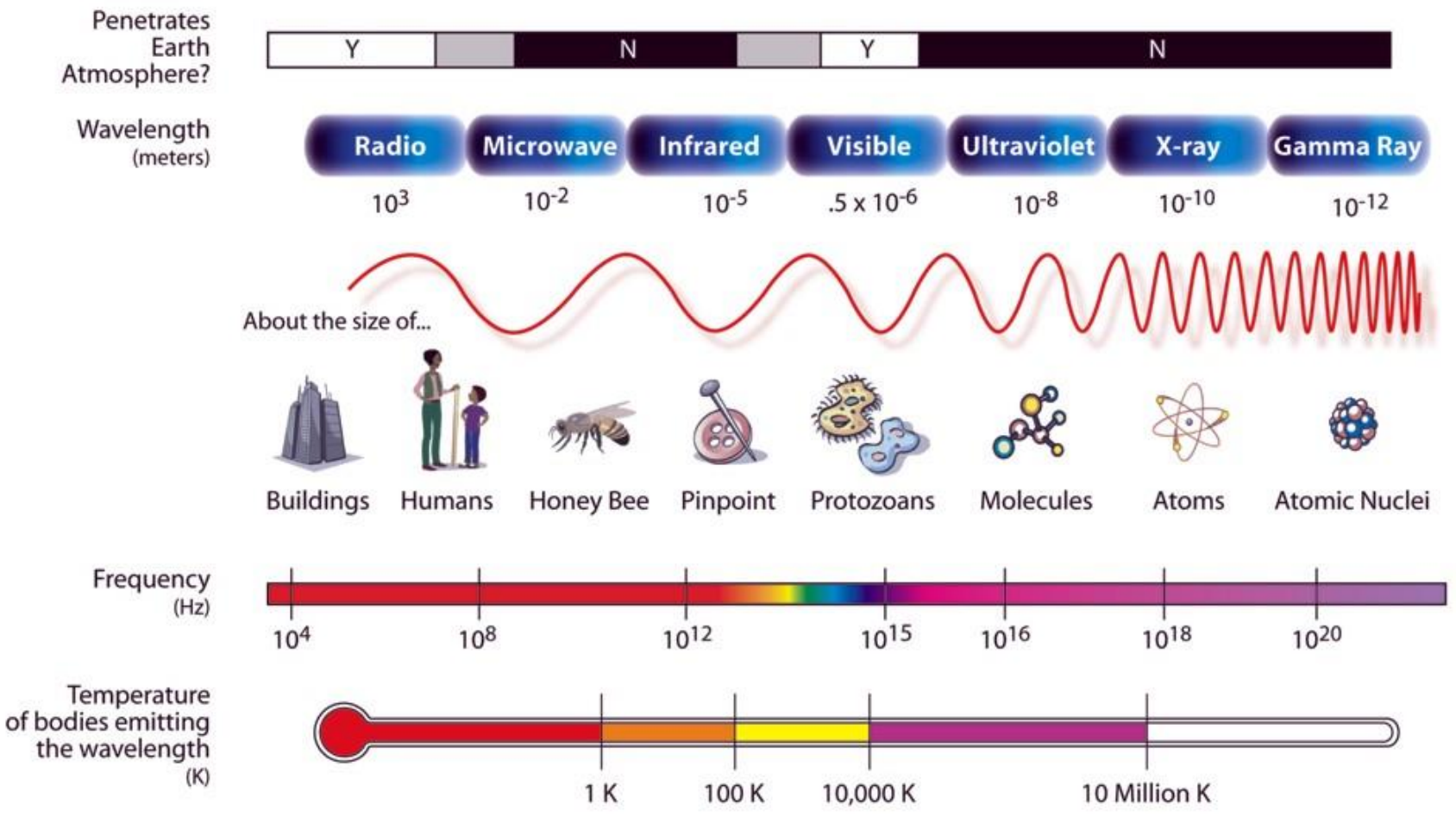
Division of Synchrotron Radiation Research, Fysicum
e-mail: mehran.sedrpooshan@sljus.lu.se

LUND
UNIVERSITY

OUTLINE

- Short introduction to X-ray Diffraction
- Crystal structure
- Diffraction patterns analysis
- Experiments

THE ELECTROMAGNETIC SPECTRUM



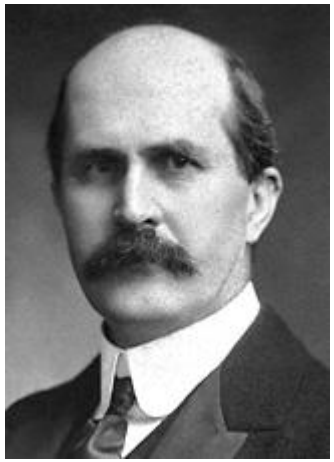
www.mynasadata.larc.nasa.gov

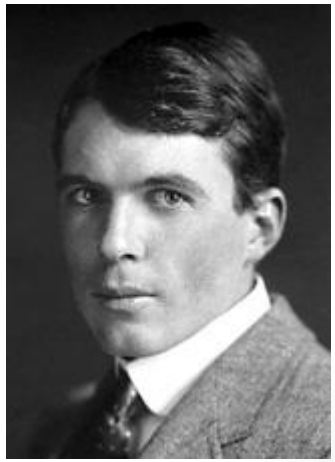
DISCOVERY OF X-RAY DIFFRACTION

1895 Wilhelm Conrad Röntgen
discovers X-rays

1912 Max Theodor Felix von Laue
invents X-ray diffraction

1912 Sir William Henry Bragg
William Lawrence Bragg
determine the crystal structure of NaCl, ZnS, and diamond; Bragg's law is derived

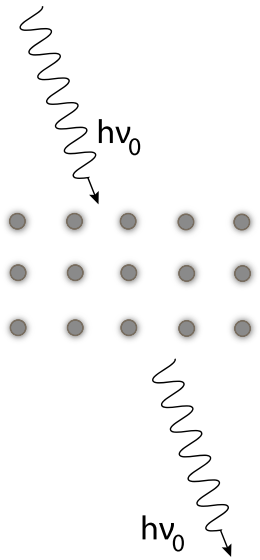




NOBEL PRIZES

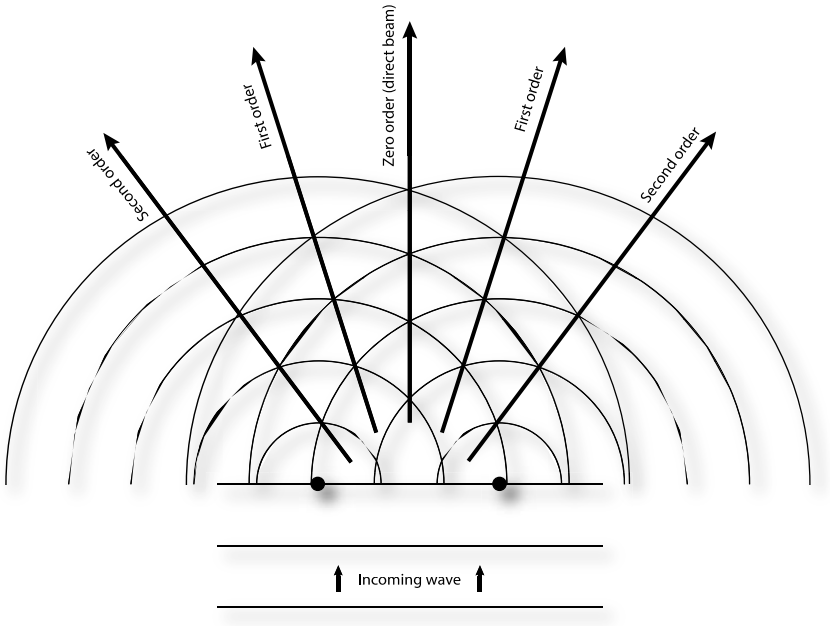
Year	Prize	Awardee	Topic
1901	Physics	W. C. Röntgen	Discovery of X-rays
1914	Physics	M. Von Laue	Discovery of XRD
1915	Physics	W. H. Bragg W. L. Bragg	Analysis of crystal structure using XRD
1962	Chemistry	J. C. Kendrew M. F. Perutz	Structural determination of globular proteins (myoglobin and hemoglobin)
1964	Chemistry	D. C. Hodgkin	Structure determinations of important biochemical molecules
1962	Medicine	F. H. Crick J. D. Watson M. H. Wilkins	Structure of DNA
1988	Chemistry	J. Deisenhofer R. Huber H. Michel	Structure of photosynthetic reaction center

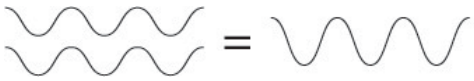
BASIC INTERACTION PROCESSES



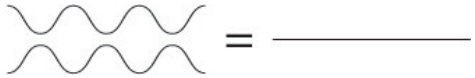
Transmission

INTERFERENCE PATTERN



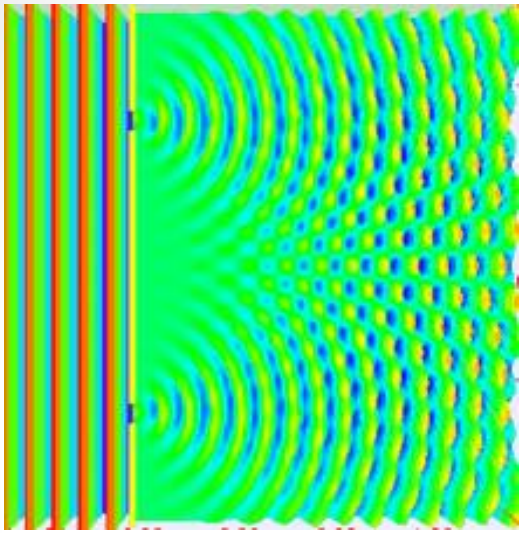


Constructive interference
(increase of amplitude)



Destructive interference
(decrease of amplitude)

LASER THROUGH DOUBLE SLIT



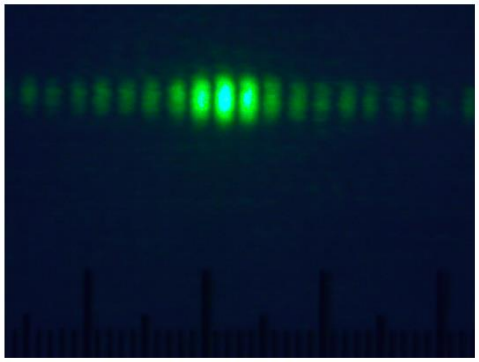


Figure 4.13: Interference pattern with green light

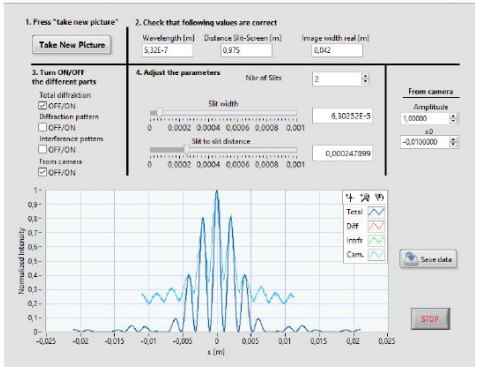
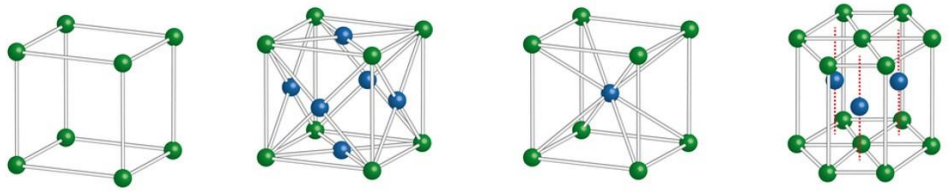


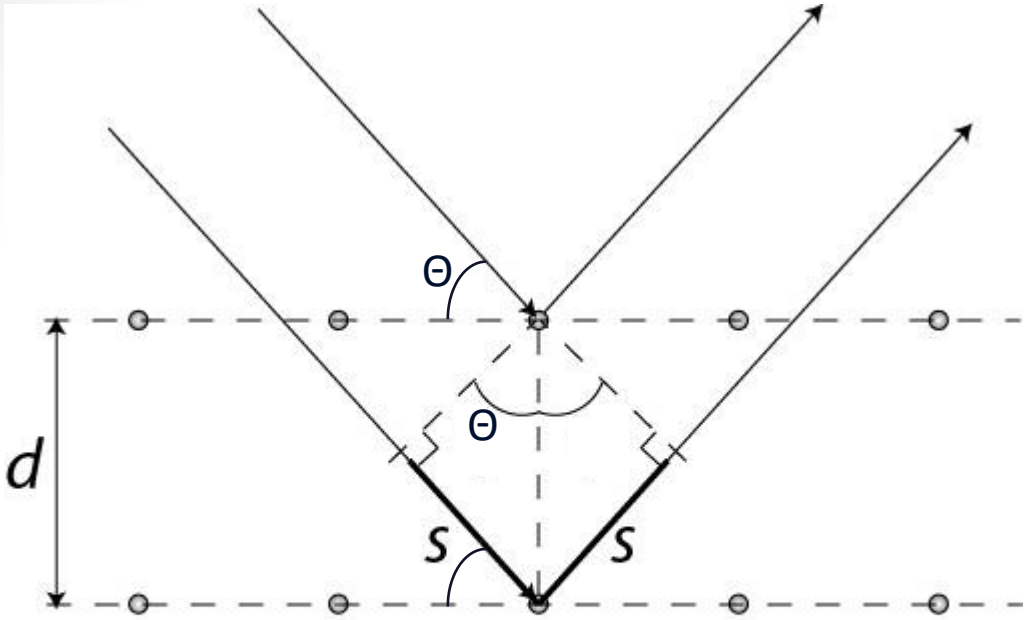
Figure 4.14: Computer modelling (green light)

λ (visible light) ≈ a (slits gap)

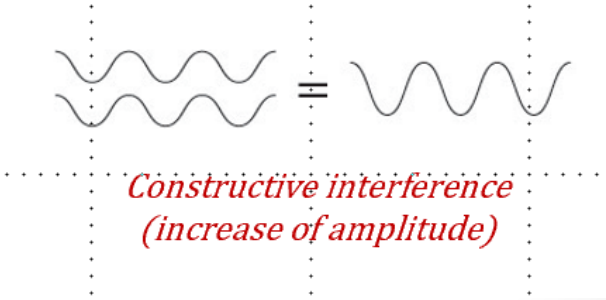


λ (X-ray) ≈ a (lattice parameter)

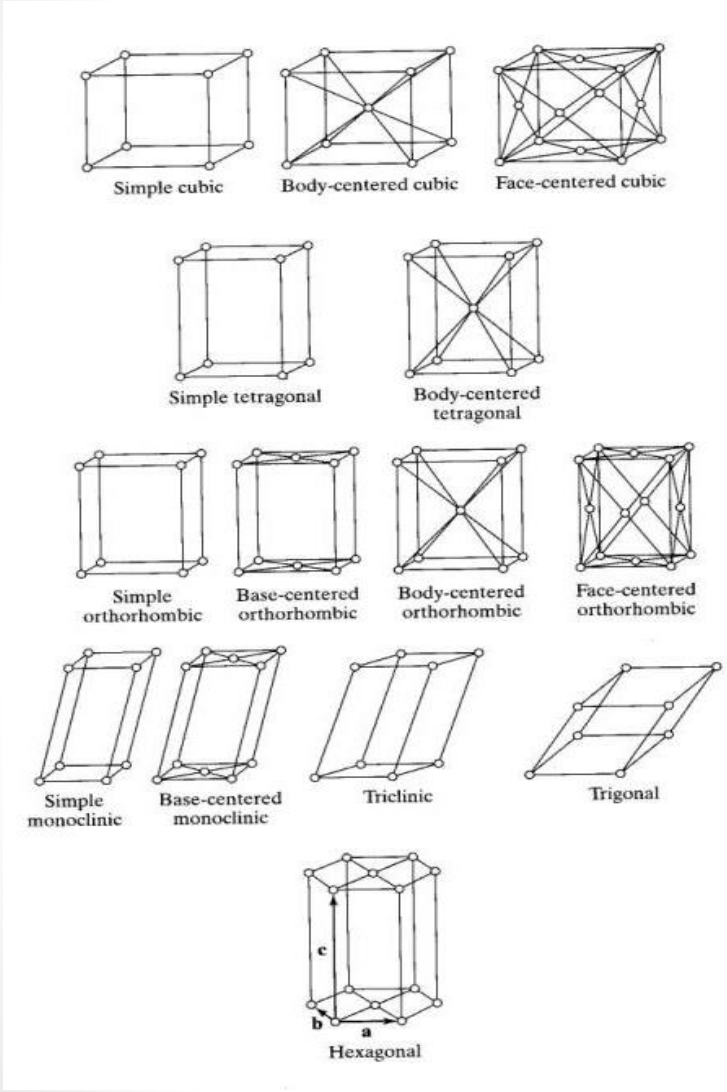
BRAGG'S LAW



$$n\lambda = PD = 2s = 2d\ \mathbf{sin\Theta}$$



CRYSTAL LATTICE



Auguste Bravais

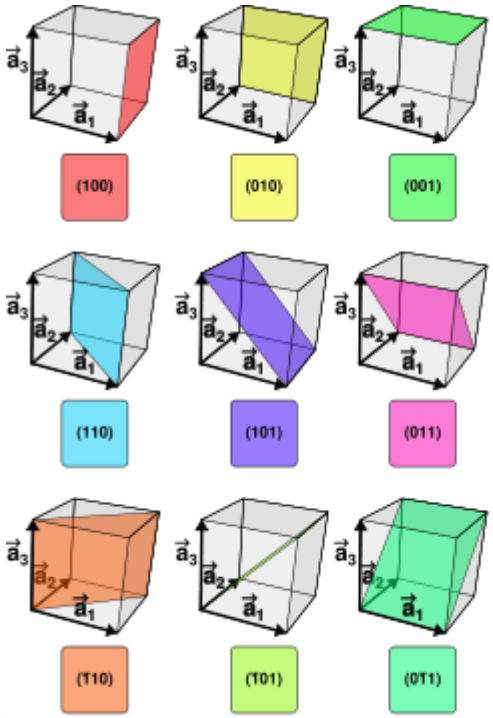
A ***crystal lattice*** is an array of lattice points.

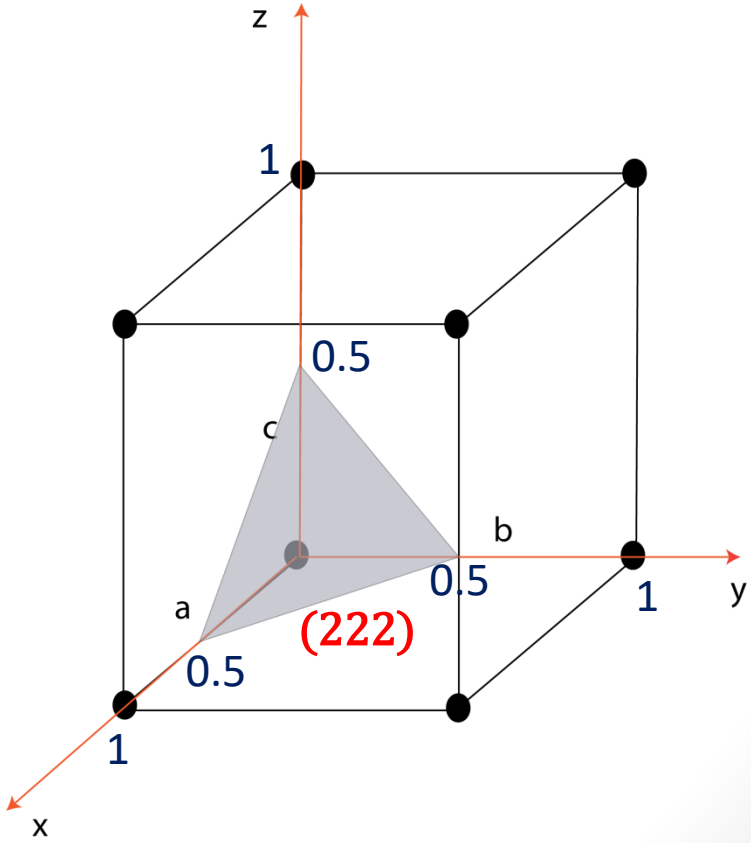
The ***unit cell*** is unit of the translationally repeating pattern.

NOTATION OF PLANES

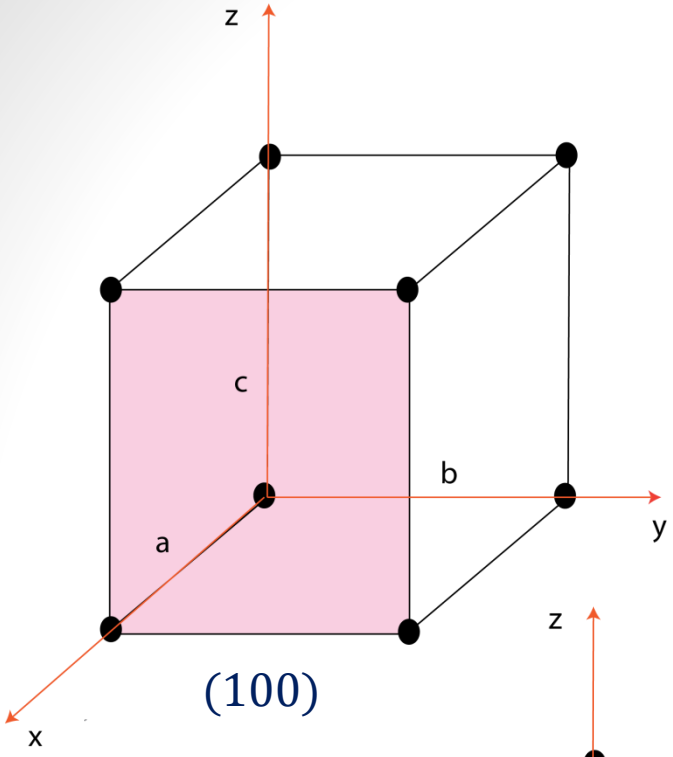
Miller indices describe the orientation of a plane or set of planes within a lattice in relation to the unit cell.

Miller indices are the reciprocals of intersection distances.

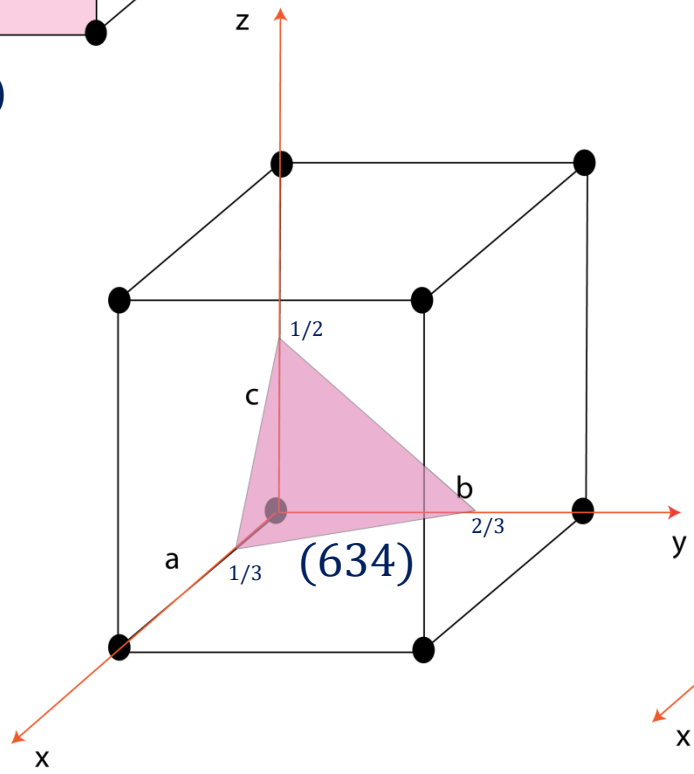




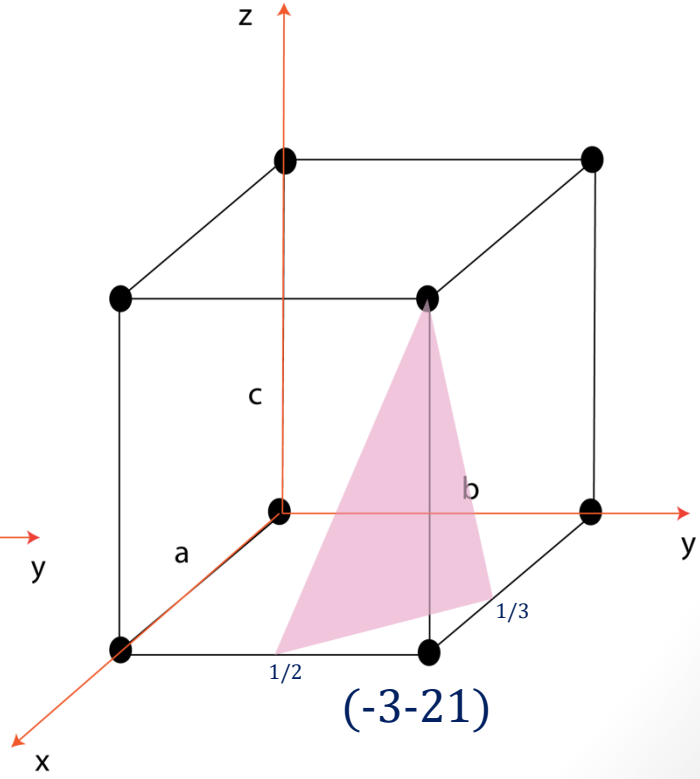
Miller indices should be integer numbers, thus if we are getting fractions for h, k, l values, we need to reduce them to lowest common denominator.



(100)

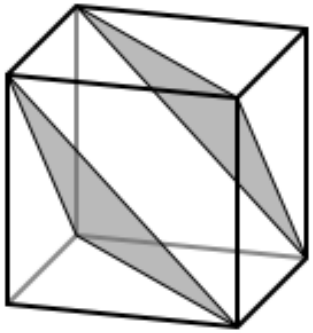


(634)



(-3-21)

SEPARATION OF PLANES



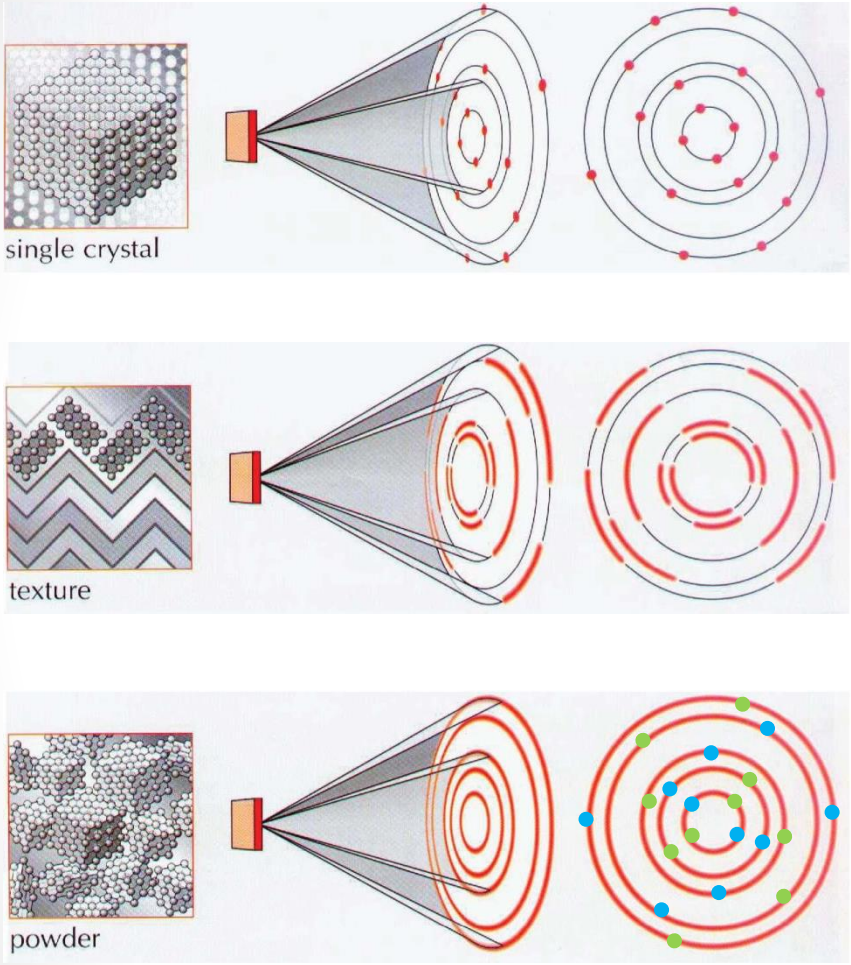
The distance between planes of atoms can be calculated as

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

For cubic cell:

$$\frac{1}{d^2} = \frac{h^2+k^2+l^2}{a^2} \quad \text{or} \quad d = \frac{a}{\sqrt{h^2+k^2+l^2}}$$

TYPES OF X-RAY DIFFRACTION PATTERNS



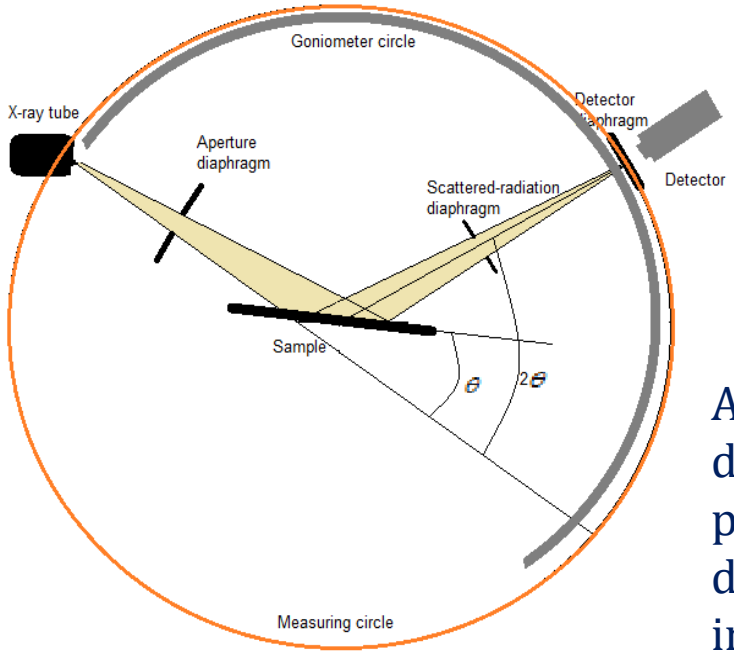
In powder sample we will have all possible orientations of crystallites and some of them will be orientated in a proper way to give rise to diffracted intensity at the glancing angle θ.

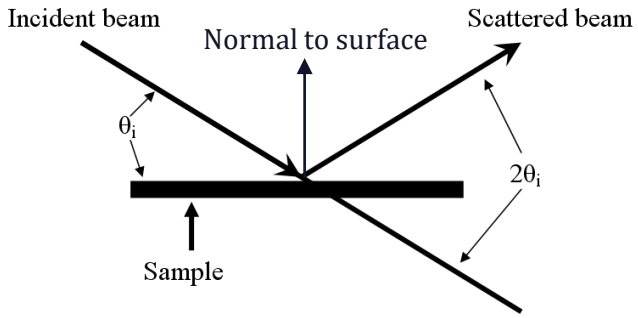
The crystallites with this glancing angle will lie at all possible angles around the incoming beam (azimuthal angles), so the diffracted beams lie on a cone around the incident beam of half-angle 2θ.

http://ap.polyu.edu.hk/apakhwon/

Θ - 2Θ SCAN

For structural characterization of crystalline compounds.



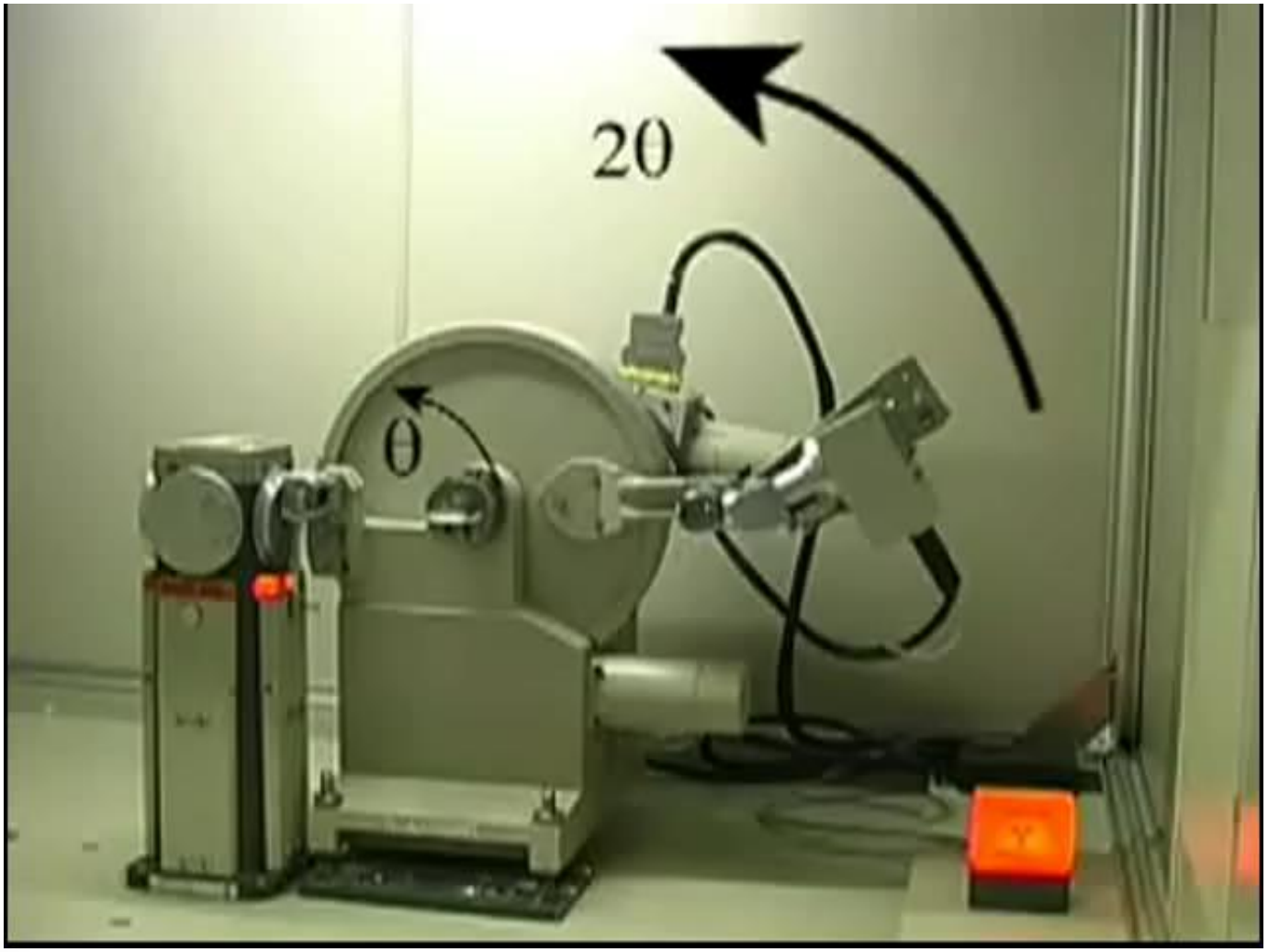


A diffraction peak is observed in a certain direction from a sample if there are crystal planes which can reflect X-rays in that direction, and if the reflected X-rays interfere constructively.

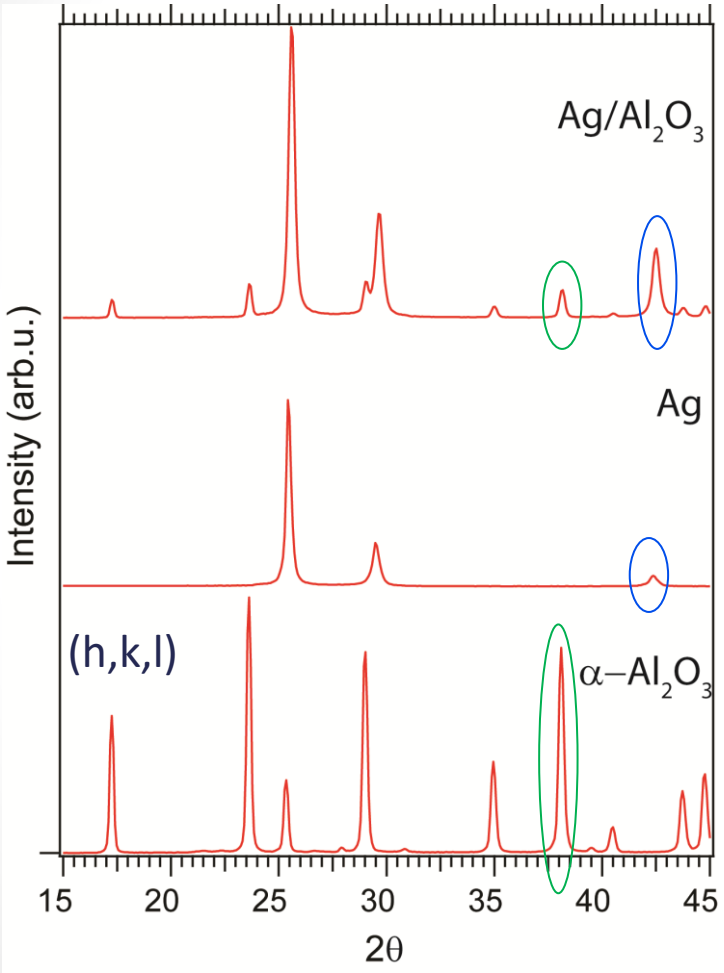
When X-ray beam hits a sample and is diffracted we can measure the distances between the planes of the atoms that constitute the sample

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

POWDER X-RAY DIFFRACTOMETER



DATA ANALYSIS



1. Calculation of d and/or a for particular planes

$$n\lambda = 2dsin\theta$$

2. Calculation of relative concentration of components in case of multicomponent sample

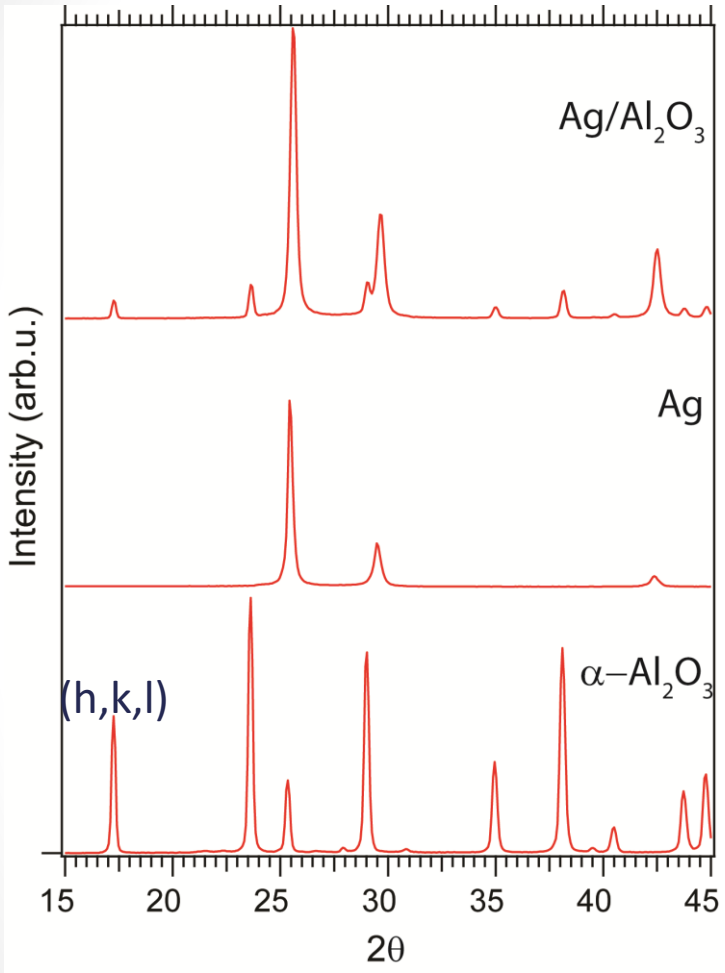
Measuring relative intensity of strong non-overlapping maxima belonging to different species.

3. Calculation of size of crystallites

$t = \frac{k\lambda}{\beta cos\theta}$ - Scherrer's formula

t –mean size of ordered domains(crystallites), k – shape factor(=0.94 for our case), β-FWHM(full width at the half of maximum) in radians.

DATA ANALYSIS



4. Calculation of allowed *hkl* values, that will give Bragg points on the diffraction pattern

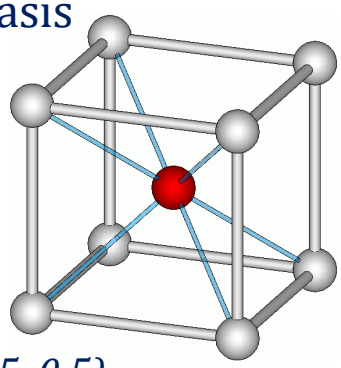
Structure factor (mathematical description of how a material scatters incident radiation)

$$F(hkl) = \sum_m f_m \exp(2\pi i(a_1 h + a_2 k + a_3 l))$$

$$I_{peak} \sim F^2$$

a_i - real space coordinates of unit cell atoms taken in terms of unit cell basis

f_m - atomic scattering factor

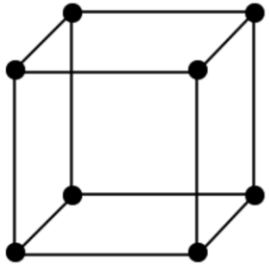


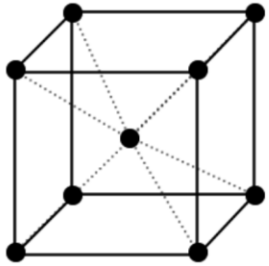
Example:

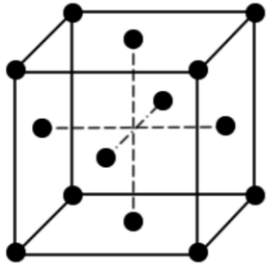
BCC unit cell contains *atoms with coordinates (0, 0, 0) and (0.5, 0.5, 0.5)*

Then if we do the calculation for "l",
For BCC, for all (hkl) planes, x-ray intensity is non-zero except when h+k+l is odd

Different lattice types







Body centered

$$\sum_n e^{i\mathbf{Q}\cdot\mathbf{r}_n} = e^{i\mathbf{Q}\cdot 0} + e^{i\mathbf{Q}\cdot(\frac{1}{2}\mathbf{a}_1+\frac{1}{2}\mathbf{a}_2+\frac{1}{2}\mathbf{a}_3)} = 1 + e^{i(h\mathbf{b}_1+k\mathbf{b}_2+l\mathbf{b}_3)\cdot(\frac{1}{2}\mathbf{a}_1+\frac{1}{2}\mathbf{a}_2+\frac{1}{2}\mathbf{a}_3)}$$

$$= 1 + e^{i2\pi\frac{1}{2}(h+k+l)} = \begin{cases} \text{Solve this yourself!} \end{cases}$$

Face centered

$$\sum_n e^{i\mathbf{Q}\cdot\mathbf{r}_n} = e^{i\mathbf{Q}\cdot 0} + e^{i\mathbf{Q}\cdot(\frac{1}{2}\mathbf{a}_1+\frac{1}{2}\mathbf{a}_2)} + e^{i\mathbf{Q}\cdot(\frac{1}{2}\mathbf{a}_1+\frac{1}{2}\mathbf{a}_3)} + e^{i\mathbf{Q}\cdot(\frac{1}{2}\mathbf{a}_2+\frac{1}{2}\mathbf{a}_3)}$$

$$= 1 + e^{i2\pi\frac{1}{2}(h+k)} + e^{i2\pi\frac{1}{2}(h+l)} + e^{i2\pi\frac{1}{2}(k+l)}$$

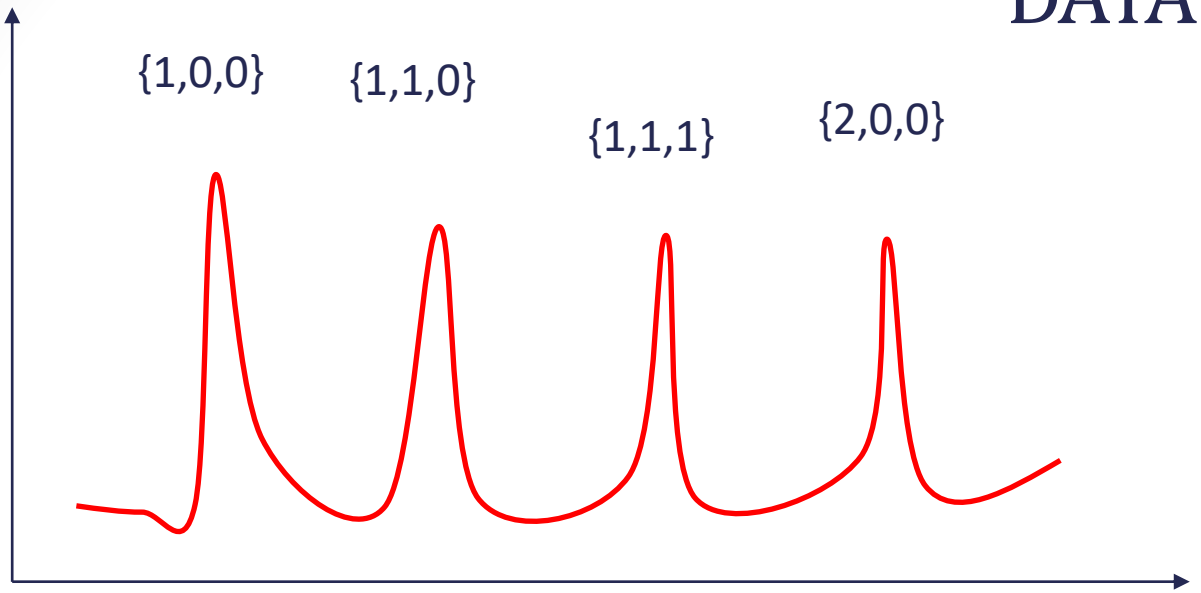
$$= \begin{cases} \text{Solve this yourself!} \end{cases}$$

LUND UNIVERSITY

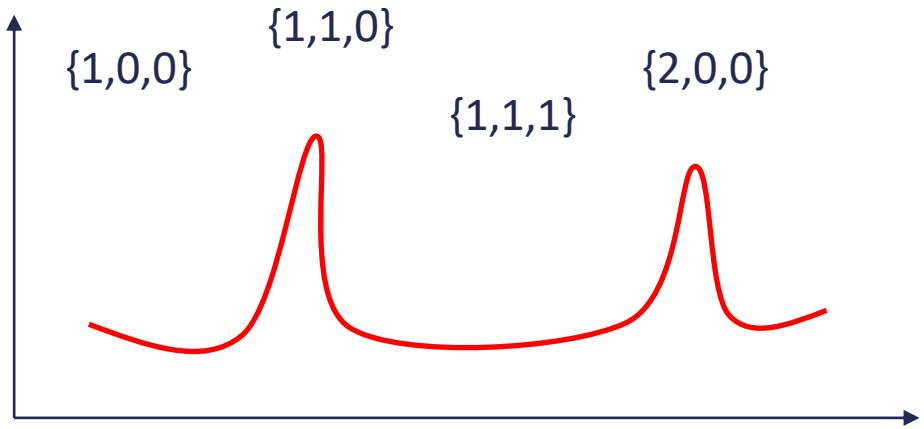
Courtesy of Johan Gustafson

DATA ANALYSIS

SC structure



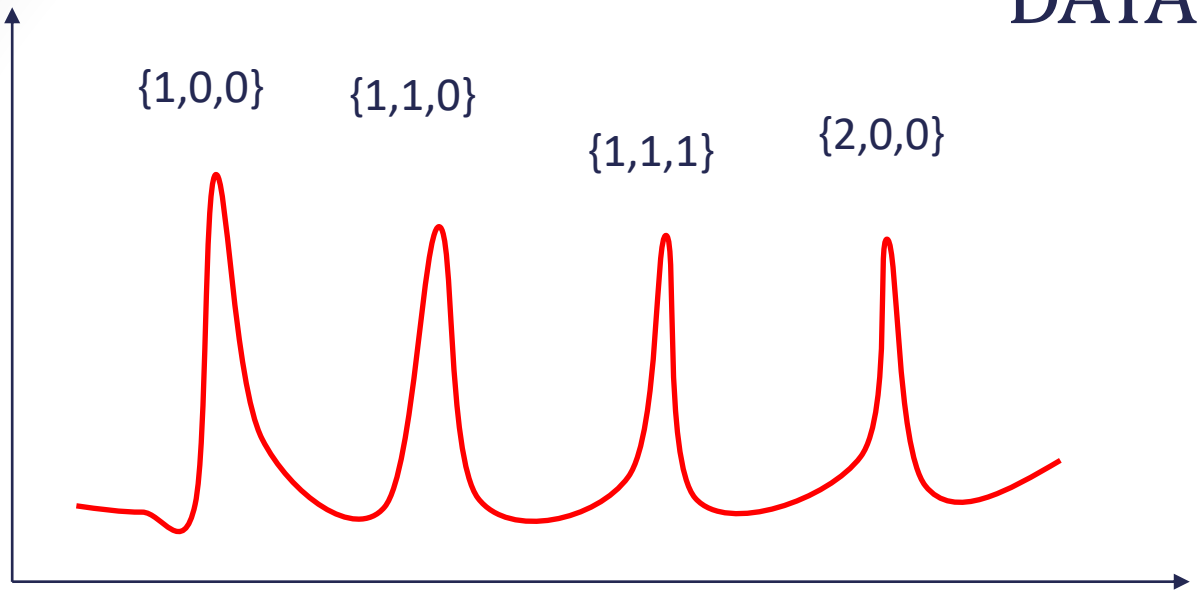
BCC structure



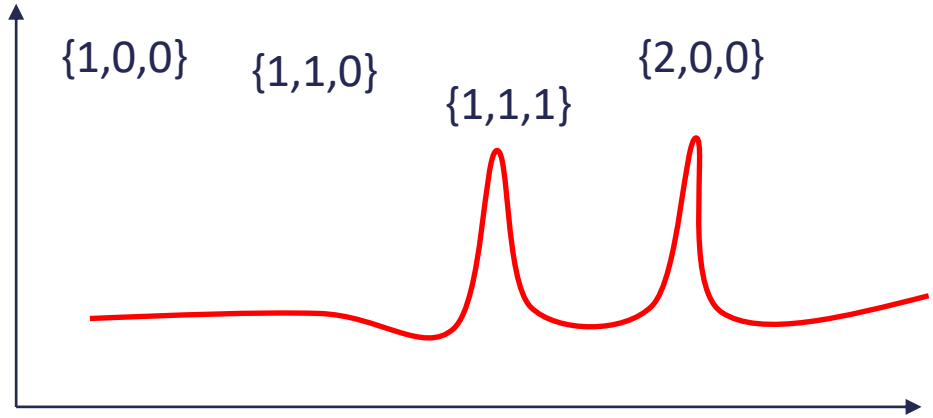
The order of peaks is by values obtained by: $\sqrt{(h)^2 + (k)^2 + (l)^2}$

DATA ANALYSIS

SC structure



FCC structure



The order of peaks is by values obtained by: $\sqrt{(h)^2 + (k)^2 + (l)^2}$

INTEGRATION AND CALIBRATION