
MATERIAL SCIENCE (MEE102B)

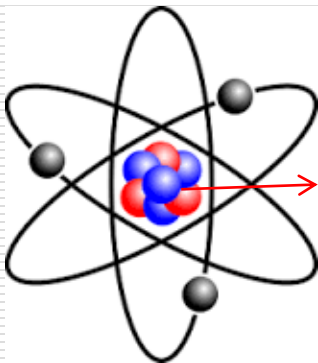
Module-I

Nature of metal and characterization

Introduction

- Different properties of metals are examined by their structures.
- The crystal structure is a atoms arranged in a systematic and repeating manner.
- Crystalline solids are generally built up of number of crystals which may be metals or non-metals.
- The examples of crystalline materials are iron, copper and aluminium etc.

Starts with Atom..



nucleus

Diameter: 1×10^{-14} m

Sub-atomic particles of the atom weighs:

Proton : 1.673×10^{-24} gm

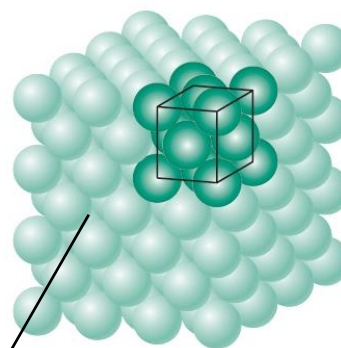
Neutron: 1.675×10^{-24} gm

Electron: 9.109×10^{-28} gm

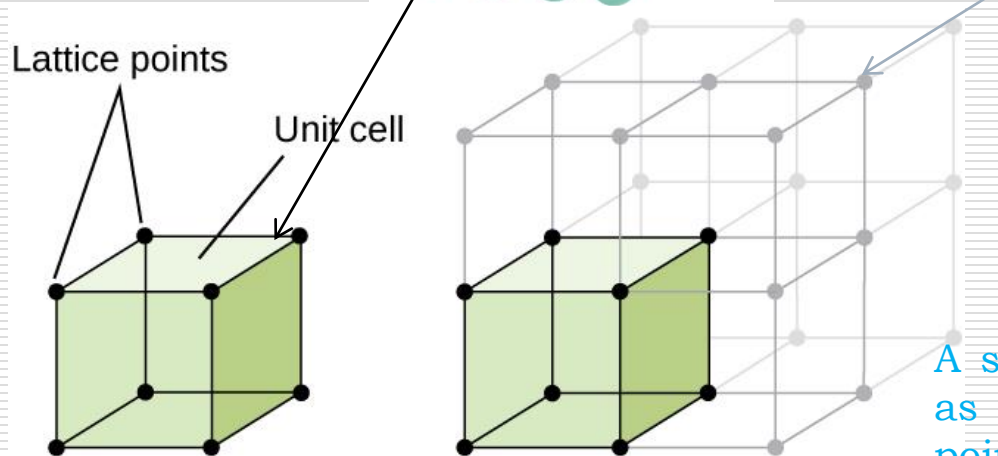
❑ An atom is the smallest constituent unit of ordinary matter.

❑ NOTE: The electrons particularly outer ones determines most of the electrical, Mechanical, Chemical and thermal properties.

The crystal structure is, atoms arranged in a systematic and repeating manner.



Crystal structure may be defined as a when many unit cells repeat in a three-dimensional space.



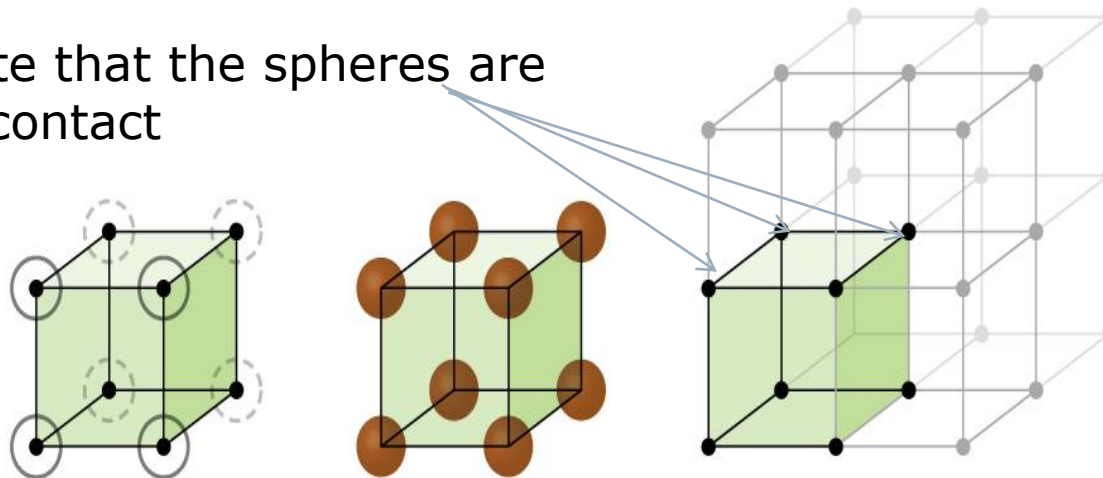
Space Lattice

Simplest repeating structural unit, which is referred to as its **unit cell**.

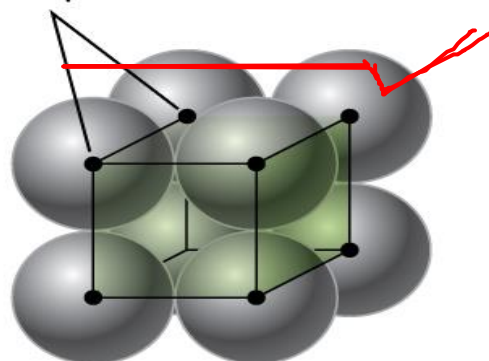
A space lattice is defined as an infinite array of points in three-dimensional space in which every point is symmetrically located with respect to the other.

Crystal Structure

Note that the spheres are in contact

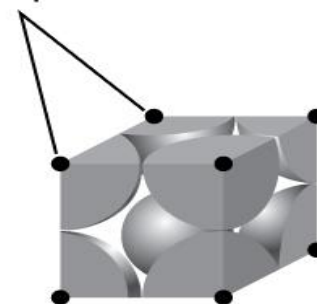


Lattice points



Simple cubic lattice cell

Lattice points



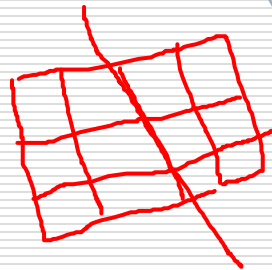
8 corners

Crystalline and non crystalline materials

Physical structure of solid material

Atoms, ions or molecules **are arranged** in pattern that are periodic and repeats itself in 3D
↓
Forms

A Solid
-has Long Range Order
↓
Crystalline material



Atoms, ions or molecules are not arranged in pattern that are periodic and repeats itself in 3D
↓
Forms

A liquid / amorphous
-has Shorter Range Order
↓
Non-crystalline material

- **Crystalline** materials have atoms situated in a periodic array over large atomic distances

- Metals and most ceramics

- Some properties depend on the crystal structure of the material (e.g. density and ductility)



Amorphous materials have no long range order (non-crystalline).

- Glasses – Silica
- Plastics

Crystal Structure

- The units of length in the space lattice are the distance between the space lattice points are define by a , b and c .
- The angles between the axes are called lattice angles. These are represented by α , β and γ .

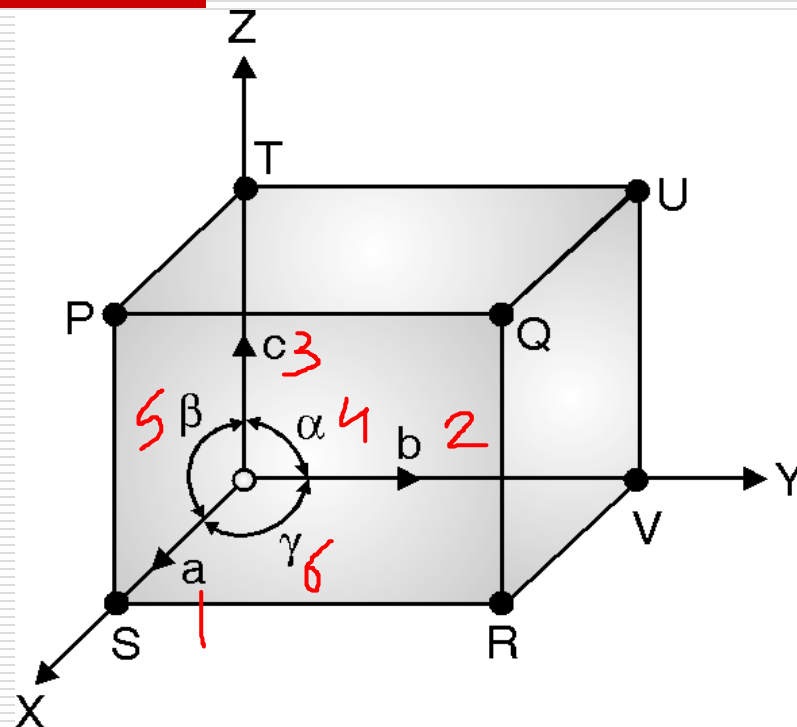
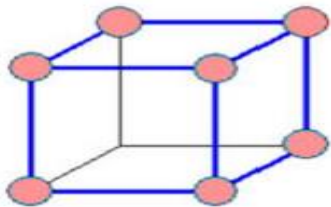


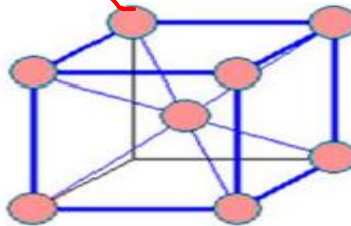
Fig. Lattice parameter of a unit cell

Crystal Systems

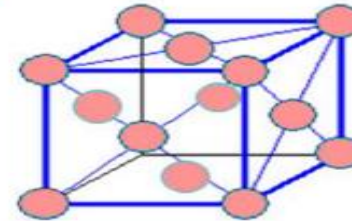
Cubic: $a = b = c$, $\alpha = \beta = \gamma = 90^\circ$



Simple
cubic

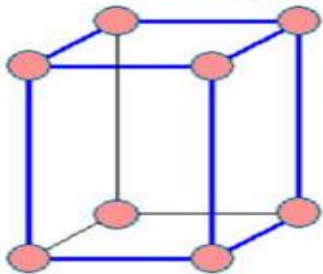


Body-centered
cubic (BCC)

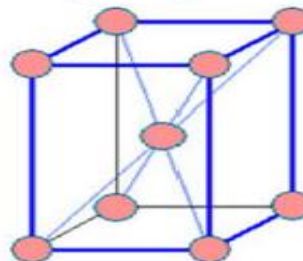


Face-centered
cubic (FCC)

Tetragonal: $a = b \neq c$, $\alpha = \beta = \gamma = 90^\circ$



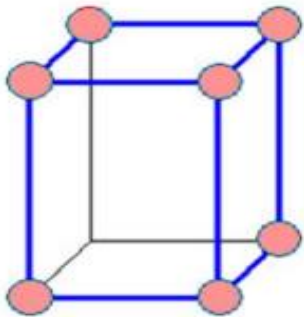
Simple
Tetragonal



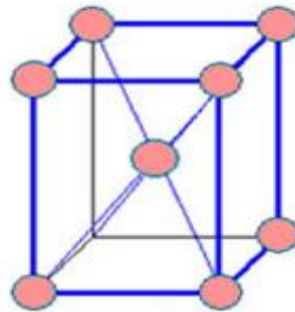
Body-centered
Tetragonal (BCT)

Crystal System

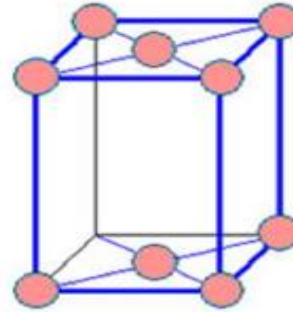
Orthorhombic: $a \neq b \neq c$, $\alpha = \beta = \gamma = 90^\circ$



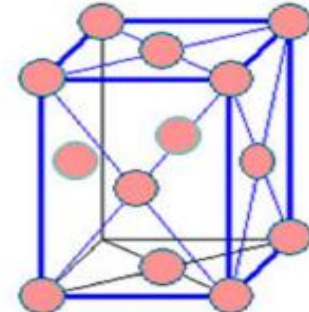
Simple



Body-centered

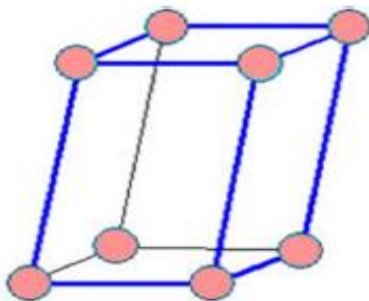


Base-centered

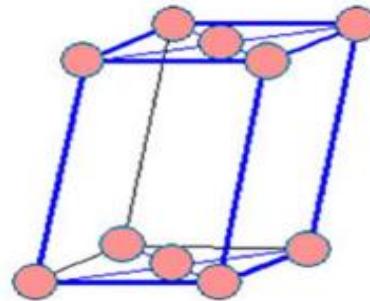


Face-centered

Monoclinic: $a \neq b \neq c$, $\alpha = \gamma = 90^\circ \neq \beta$

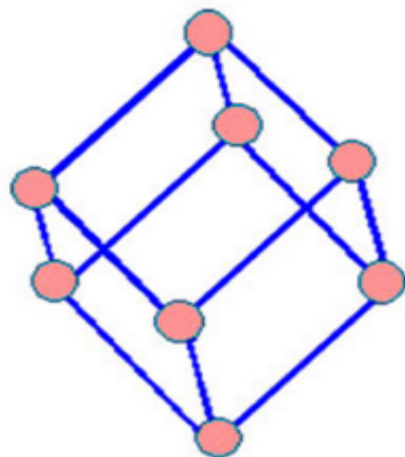


Simple
monoclinic

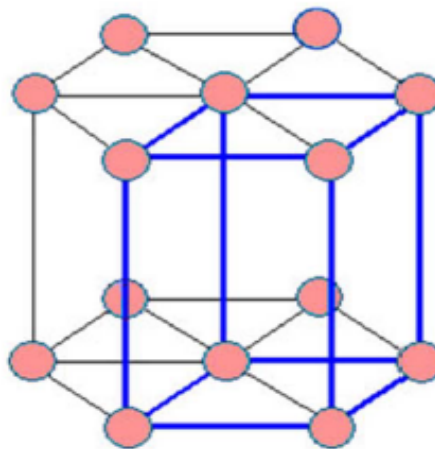


Base-centered
monoclinic

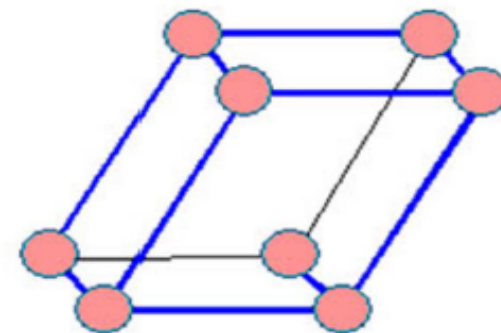
Crystal Systems



Rhombohedral
 $a = b = c$
 $\alpha = \beta = \gamma \neq 90^\circ$



Hexagonal
 $a = b \neq c$
 $\alpha = \beta = 90^\circ \gamma = 120^\circ$



Triclinic
 $a \neq b \neq c$
 $\alpha \neq \beta \neq \gamma \neq 90^\circ$

Types of Crystal Structures

The followings are the types of crystal structures :

1. Simple Cubic Crystal structure (S.C.) (polonium) ✓
2. Body Centered Crystal structure (B.C.C.) (Fe, Cr, V) ✓
3. Face Centered Crystal structure (F.C.C.) (aluminum, copper, nickel, gamma iron, gold, silver.) ✓
4. Hexagonal Close Packed structure (H.C.P.) (cobalt, cadmium, zinc, α phase of titanium) ✓

Simple Cubic Structure (S.C.)

- In this structure there are 8 atoms representing to 8 corners and there are no atoms in the interior of the unit cell or no atoms on the faces

- Average number of atoms per unit cell ✓

$$N_{av} = \frac{N_c}{8} + \frac{N_f}{2} + \frac{N_i}{1} \dots (1)$$

- N_c = Total number of corner atoms in a unit cell

- N_{av} = Average number of atoms per unitcell

- N_f = Total number of face atoms in an unit cell

- N_i = Centre or interior atom

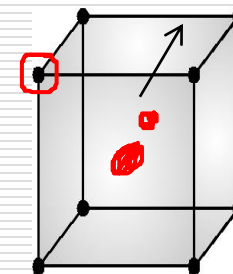
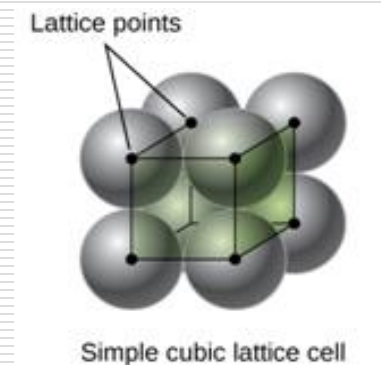


Fig. Simple cubic structure

Simple Cubic Structure (S.C.)

□ In simple cubic structure

□ $N_c = 8$, $N_f = 0$ and $N_i = 0$

From equation (1)

□
$$\begin{aligned} N_{av} &= N_c/8 + N_f/2 + N_i/1 = 1 \\ &= 8/8 + 0/2 + 0/1 \\ &= 1 \end{aligned}$$

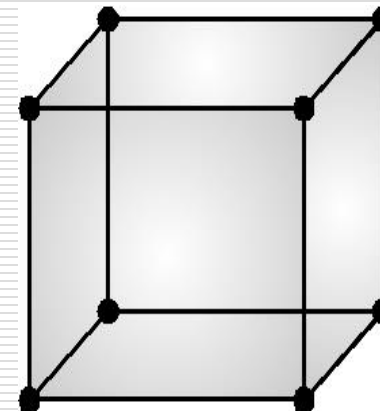


Fig. Simple cubic structure

Body Centered Structure (B.C.C.)

- Average number of atoms per unit cell

$$N_{av} = N_c/8 + N_f/2 + N_i/1 \dots (1)$$

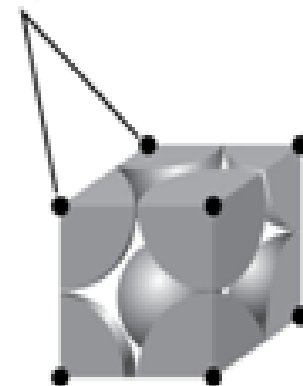
In body centered cubic structure. There are 8 corner atoms and one atom **in the interior** means in the centre of the unit cell with no atoms on the faces.

$$N_c = 8, N_f = 0 \text{ and } N_i = 1$$

From Equation (1)

$$\begin{aligned} \square N_{av} &= N_c/8 + N_f/2 + N_i/1 = 1 \\ &= 8/8 + 0/2 + 1/1 \\ &= 2 \end{aligned}$$

Lattice points



8 corners

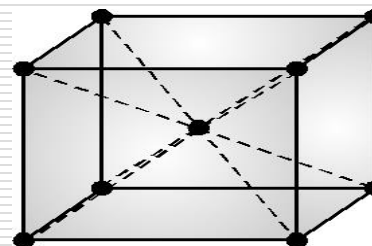


Fig. Body centered cubic structure

Face Centered Structure (F.C.C.) :

- In this structure, there are eight atoms at the eight corners and six atoms at the centres of six faces and there is no interior atom.

- $\therefore N_c = 8, N_f = 6, N_i = 0$

- From Equation (1)

- $$\begin{aligned} N_{av} &= N_c/8 + N_f/2 + N_i/1 = 1 \\ &= 8/8 + 6/2 + 0/1 \\ &= 4 \end{aligned}$$

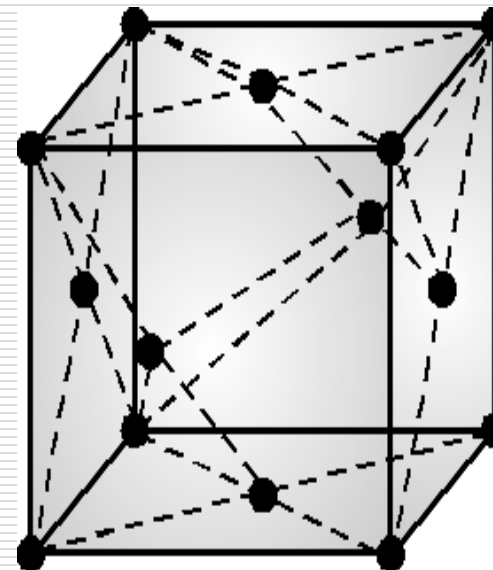


Fig. Face centered cubic structure

Hexagonal Close Packed Structure (H.C.P.) :

- ❑ In this structure, the corner atoms are shared by six cells means that three from below and three from above, face atoms are shared by adjacent two cells and atoms in the interior are shared by only one cell.
- ❑ In general, the number of atoms per unit cell will be as follows :
- ❑ $N_{av} = N_c/6 + N_f/2 + N_i/1$
- ❑ In HCP structures, there are **twelve** corners atoms means that six at the bottom face and six at the top face.

Hexagonal Close Packed Structure (H.C.P.)

Two atoms at the centres of the above two faces and three atoms in the interior of the unit cell.

- $N_c = 12, \quad N_f = 2 \quad \text{and} \quad N_i = 3$
- $N_{av} = N_c/6 + N_f/2 + N_i/1$
 $= 12/6 + 2/2 + 3/1$
 $= 2 + 1 + 3$
 $= 6$

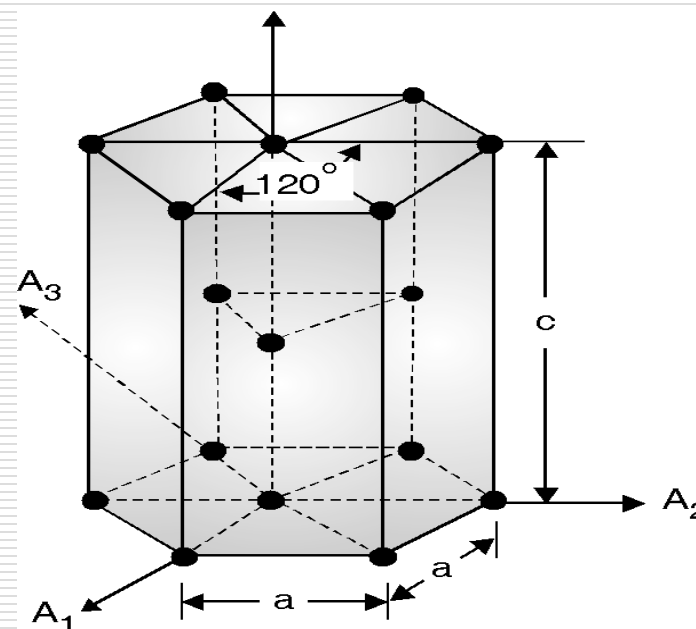


Fig. Hexagonal closed packed structure

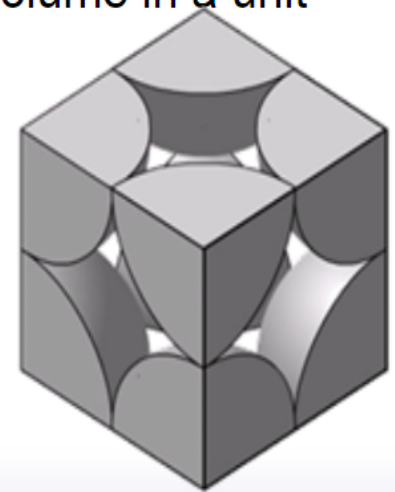
Atomic Packing Factor

■ In crystalline materials:

- Atomic packing factor = total volume of atoms in unit cell / volume of unit cell
- A value <1 (APF is the fraction of solid sphere volume in a unit cell)

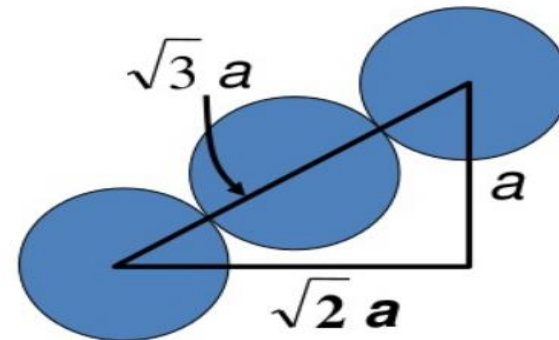
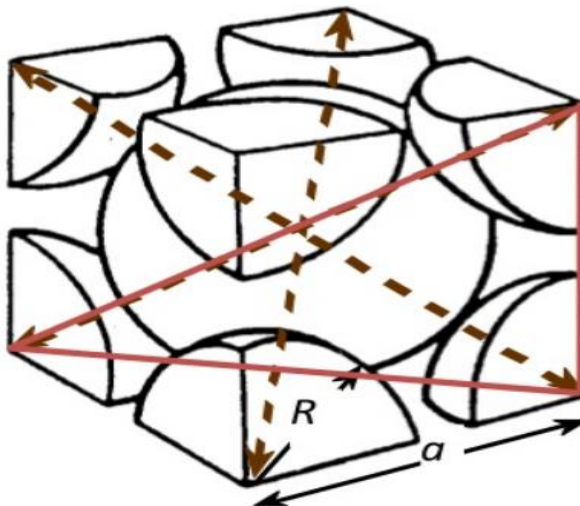
$$\text{APF} = \frac{\text{Volume of atoms in unit cell}^*}{\text{Volume of unit cell}}$$

*assume hard spheres



Atomic Packing Factor: BCC

- APF for a body-centered cubic structure = 0.68



Close-packed directions:

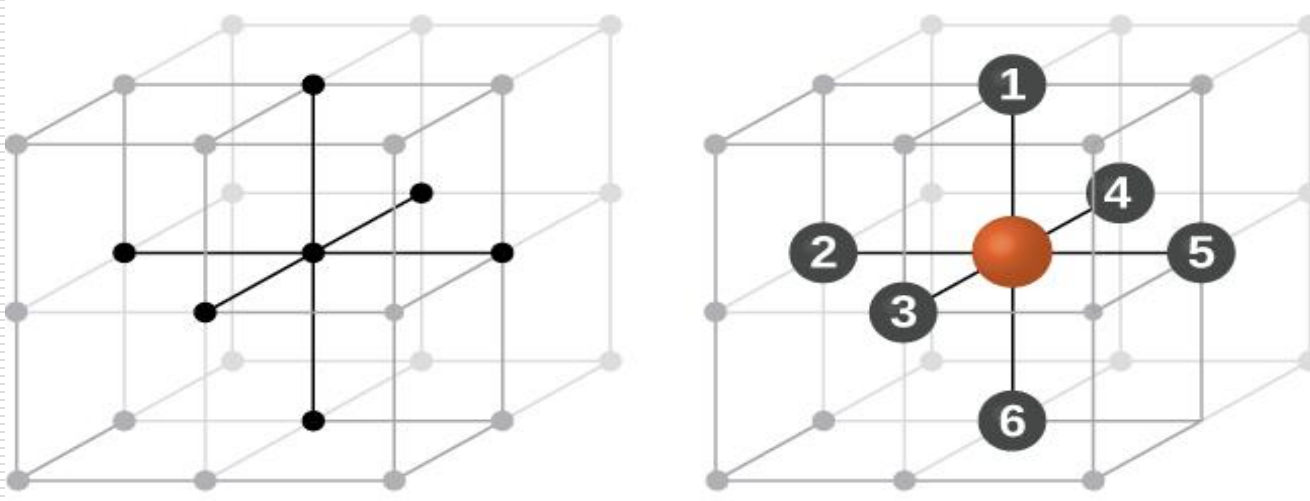
$$\text{length} = 4R = \sqrt{3}a$$

$$\text{APF} = \frac{\text{atoms unit cell} \times \text{volume atom}}{\text{volume unit cell}}$$

$$\text{APF} = \frac{2 \times \frac{4}{3} \pi \left(\frac{\sqrt{3}a}{4} \right)^3}{a^3}$$

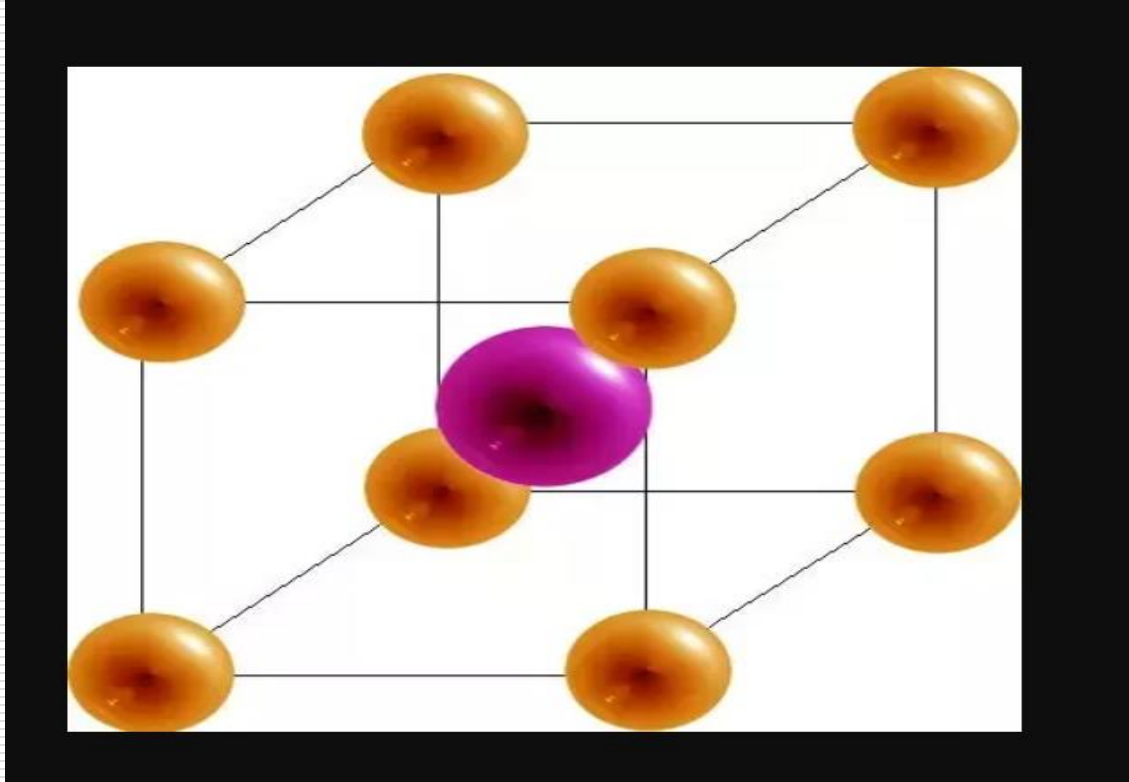
Coordination number

Coordination number: The **number** of atoms, ions, or molecules that a atom or ion holds as its nearest neighbours in a complex or **coordination** compound or in a crystal.



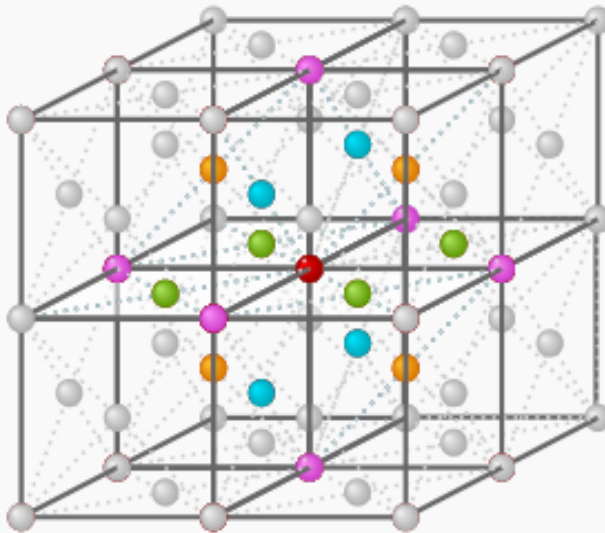
Simple Cubic

Coordination number



BCC

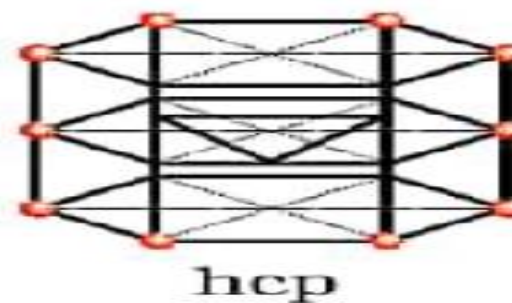
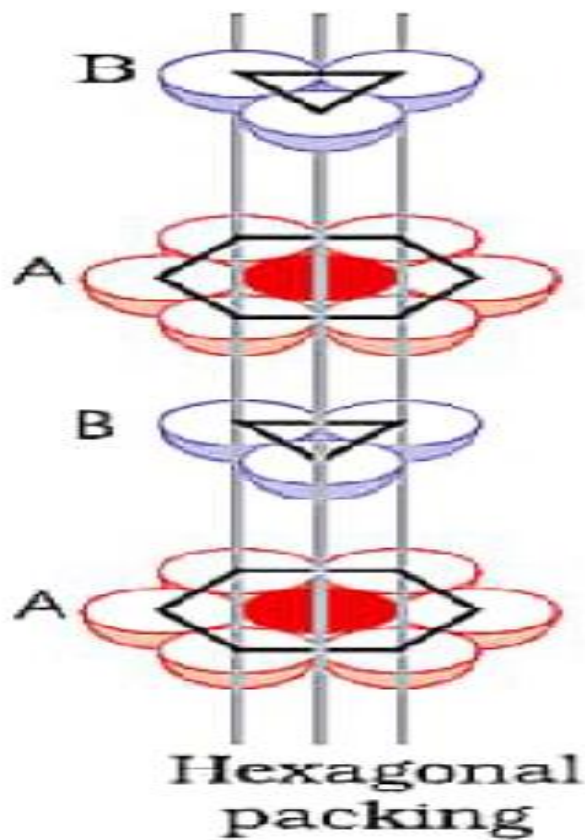
Coordination number



- reference point
- ● ● 12 nearest neighbours
- 6 next-nearest neighbours

FCC

Coordination number



HCP

Comparison of crystal structures

Crystal structure	CN	APF
■ Simple Cubic (SC)	6	0.52
■ Body Centered Cubic (BCC)	8	0.68
■ Face Centered Cubic (FCC)	12	0.74
■ Hexagonal Close Pack (HCP)	12	0.74

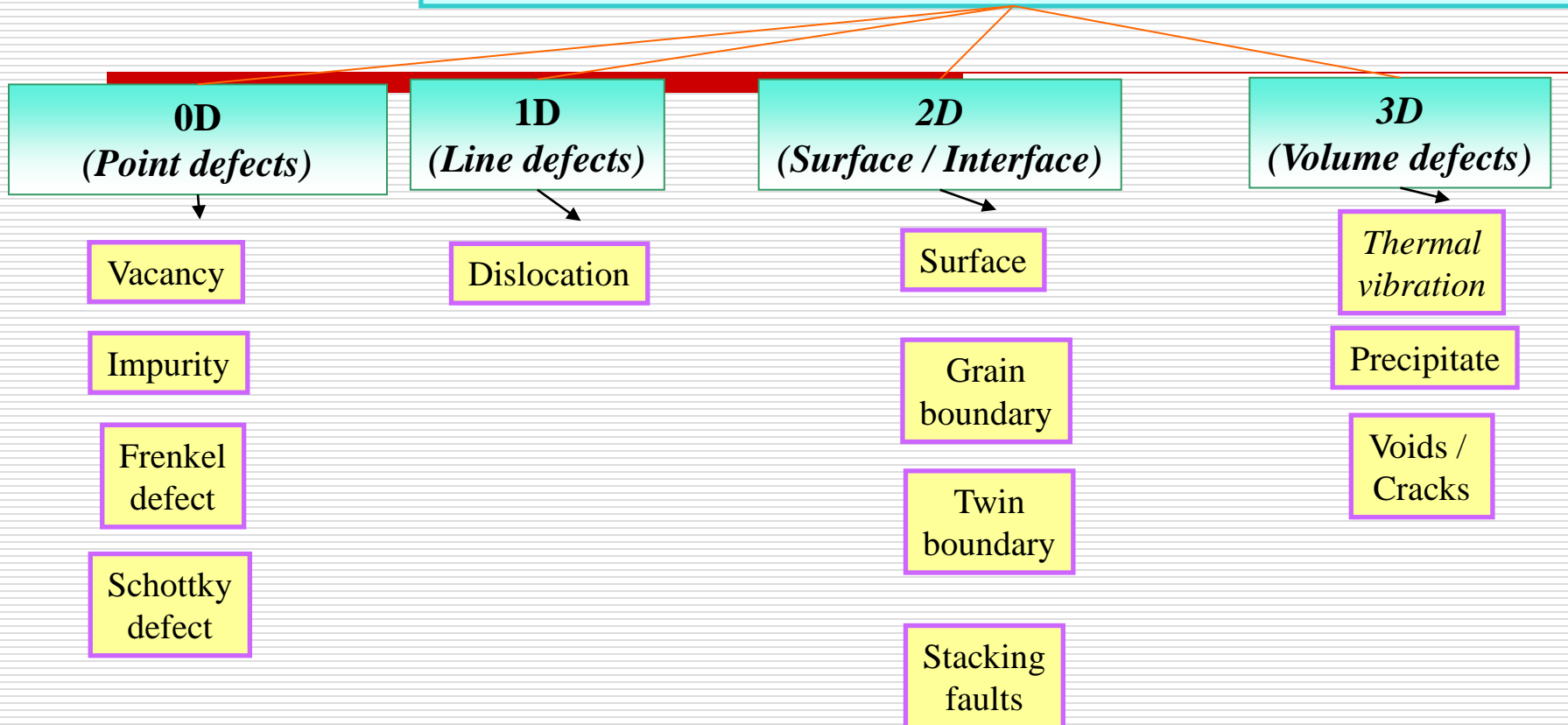
Imperfections In crystal Structures

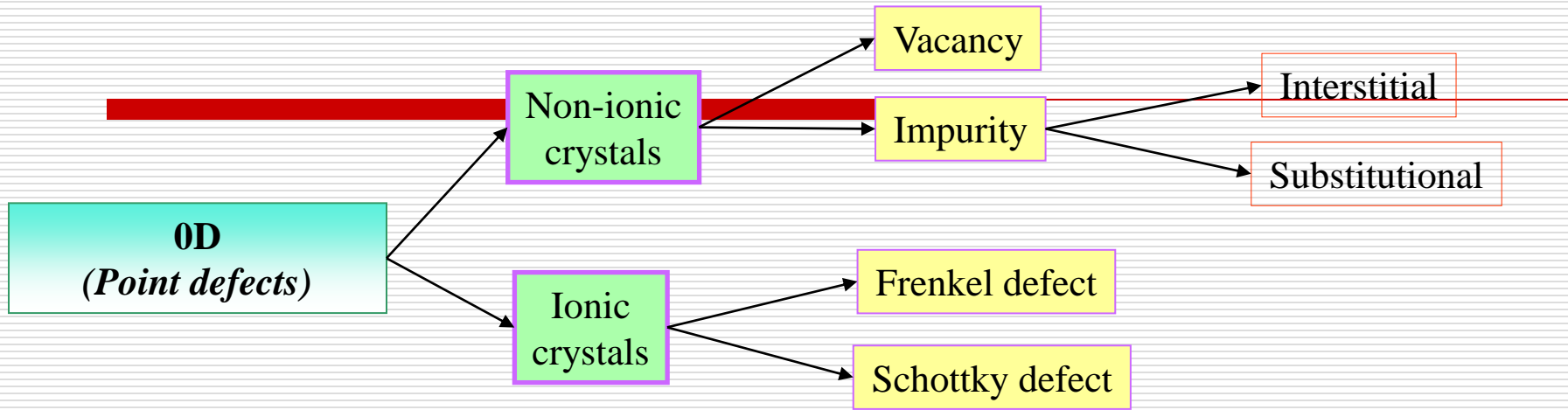
- In crystal imperfections we can say that the crystal are rarely observed to be perfect. The defects in atomic structures and their arrangement are called as imperfections.
- Dislocation follow an extended and sometimes complex path through the crystal structure.

Imperfections in Solids

- The **properties** of materials are profoundly **influenced** by the presence of **imperfections**.
- It is important to have knowledge about the **types of imperfections** that exist and the roles they play in **affecting** the behavior of materials.

CLASSIFICATION OF DEFECTS BASED ON DIMENSIONALITY





❑ Imperfect point-like regions in the crystal about the size of 1-2 atomic diameters

Methods of producing point defects

❑ Growth and synthesis

Impurities may be added to the material during synthesis

❑ Thermal & thermochemical treatments and other stimuli

- Heating to high temperature and quench
- Heating in reactive atmosphere

❑ Plastic Deformation

Vacancy defect

- In vacancy defect one or more atoms are missing from occupied position as shown in Fig. No. (a) the defect caused is known as vacancy.
- Such type of defects can be result of imperfect packing during the formation of crystals. They are also arise from thermal vibration of the atoms at high temperatures.

Vacancy defect

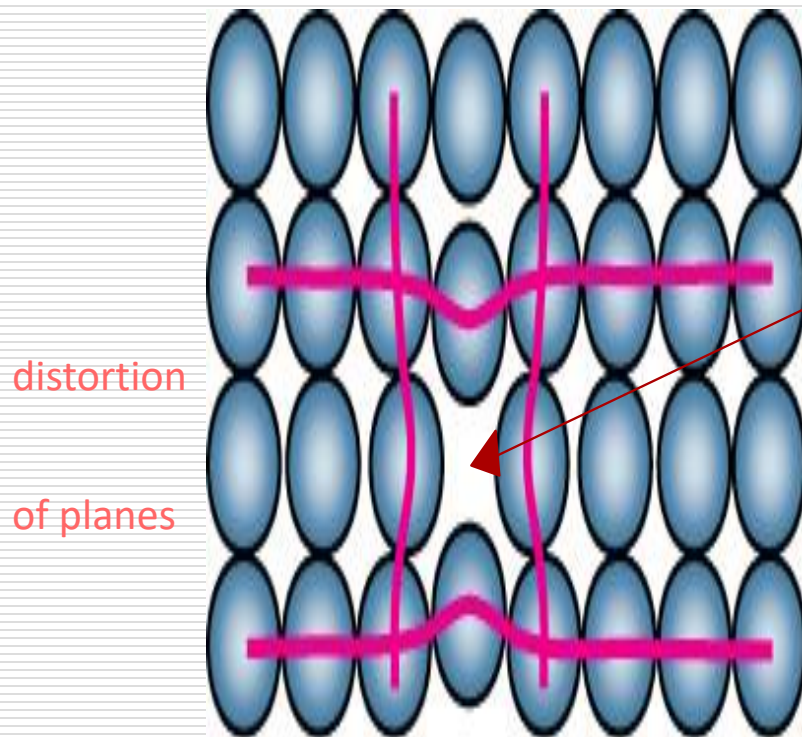
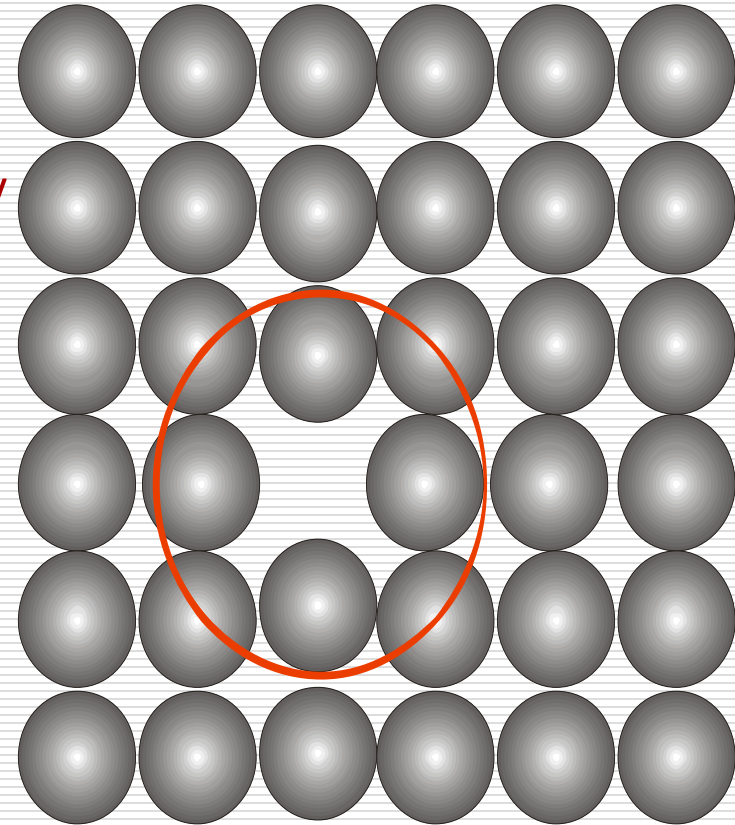
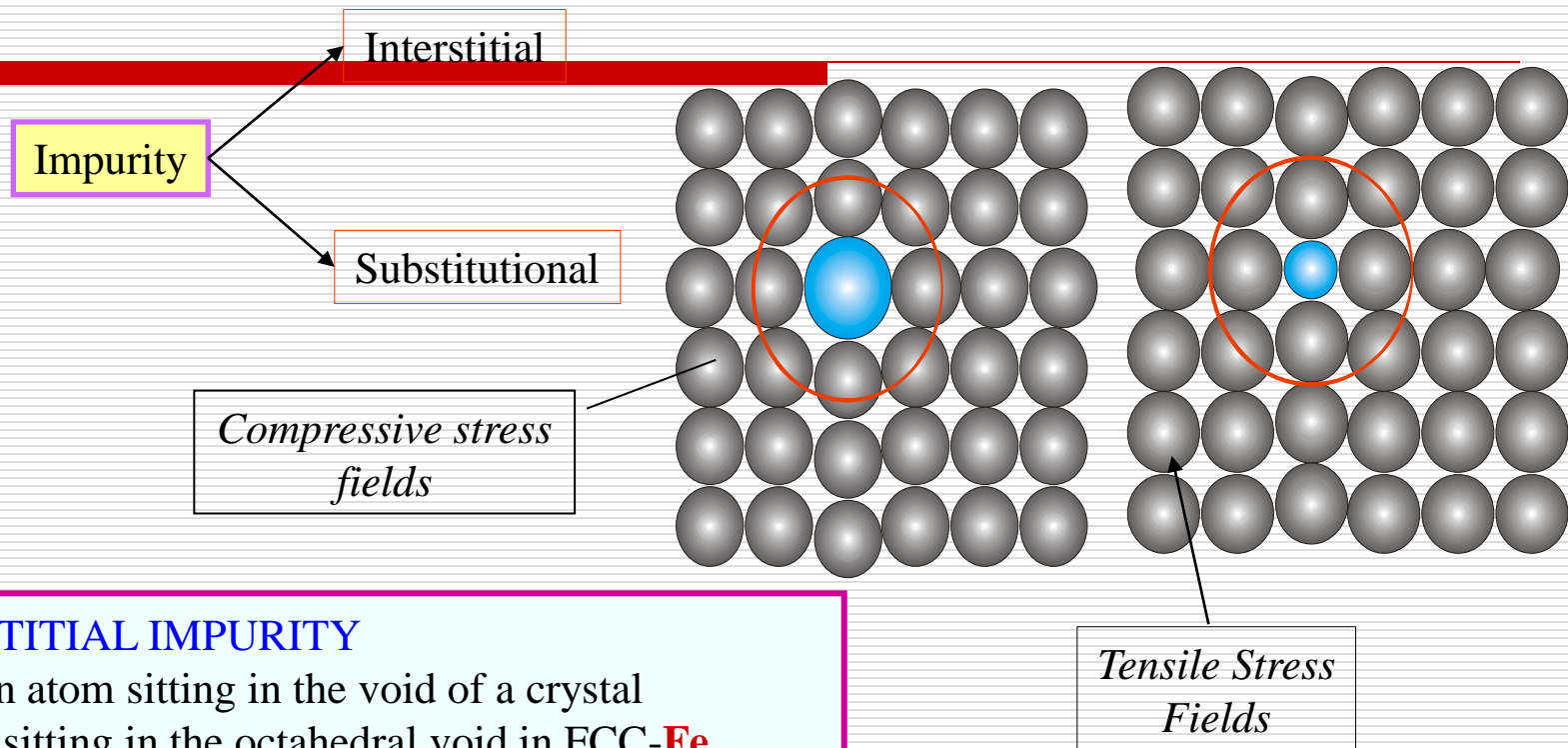


Fig: Vacancy Defect





❑ INTERSTITIAL IMPURITY

- Foreign atom sitting in the void of a crystal
- E.g. **C** sitting in the octahedral void in FCC-**Fe**

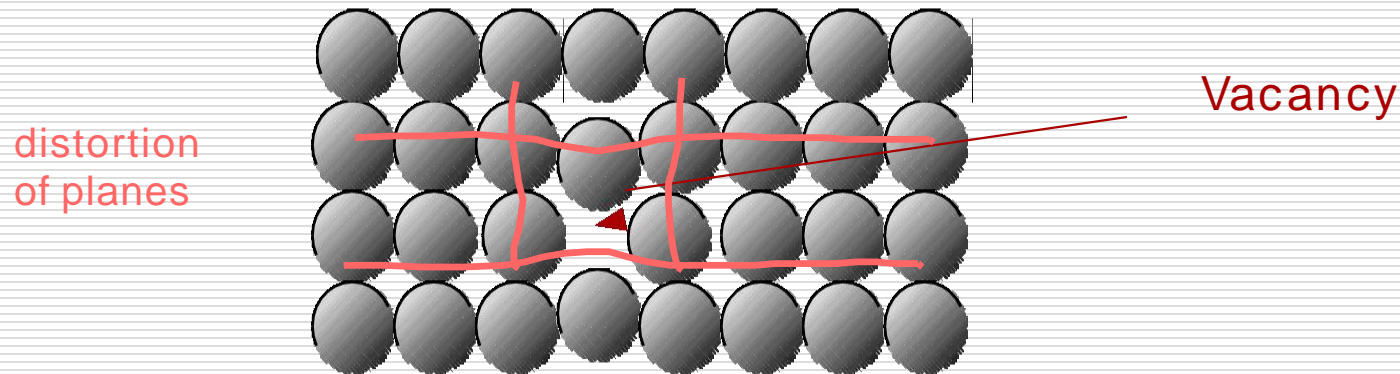
❑ SUBSTITUTIONAL IMPURITY

- Foreign atom replacing the parent atom in the crystal
- E.g. **Cu** sitting in the lattice site of FCC-**Ni**

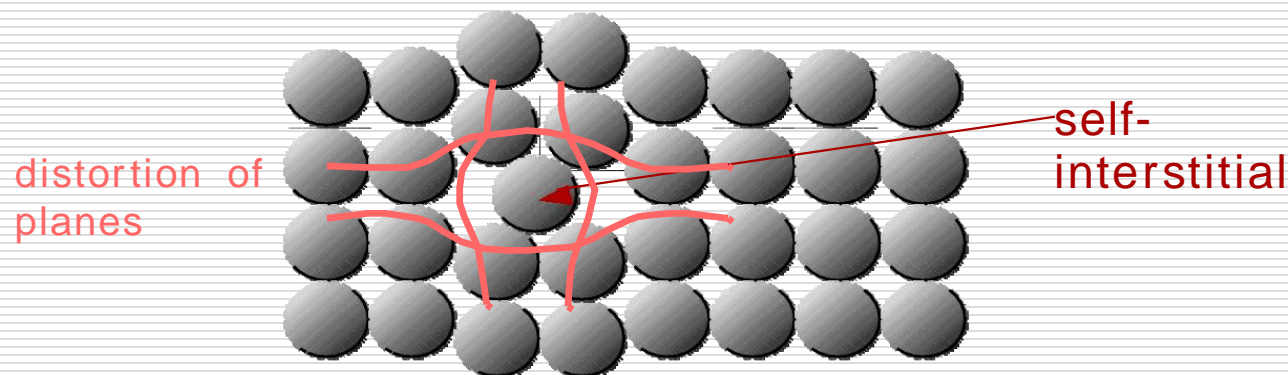
Fig: Interstitial & Substitutional Defect

Point Defects

Vacancies: vacant atomic sites in a structure.



Self-Interstitials: "extra" atoms positioned between atomic sites



Point Defects

Interstitial defect :

- In interstitial defect an extra atom occupies interstitial position in the crystal system, without disordering the parent atom as shown in Fig. The defect caused is known as interstitial defect.

Substitutional defect:

- In substitutional defect a foreign atom occupies a position, which was initially meant for a parent or replaced a parent atom as shown in Fig. the defect caused is known as substitutional defect.

Frenkel defect

Ionic Crystals

❑ Overall electrical neutrality has to be maintained

Frenkel defect

- Cation (being smaller) get displaced to interstitial voids
- E.g. AgI, CaF_2

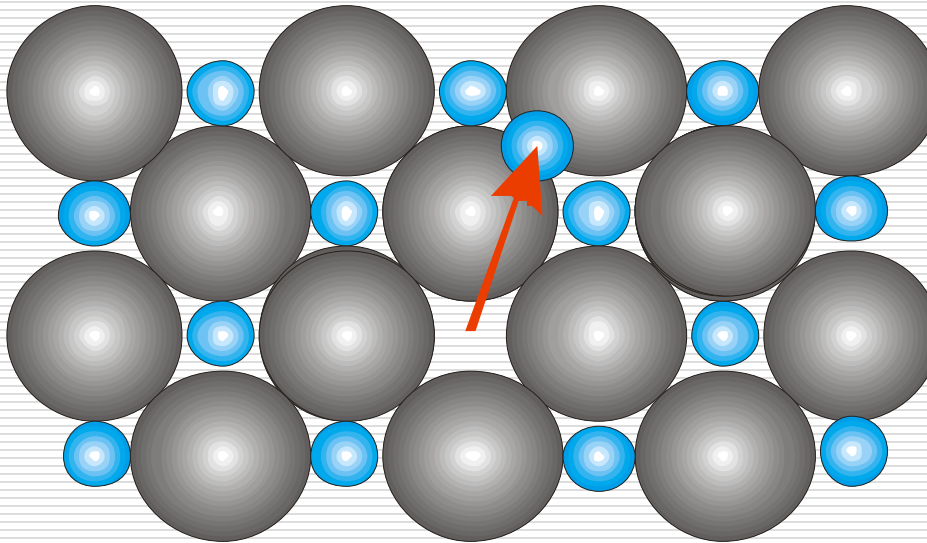


Fig: Frenkel defect

Schottky defect

- Pair of anion and cation vacancies
- E.g. Alkali halides

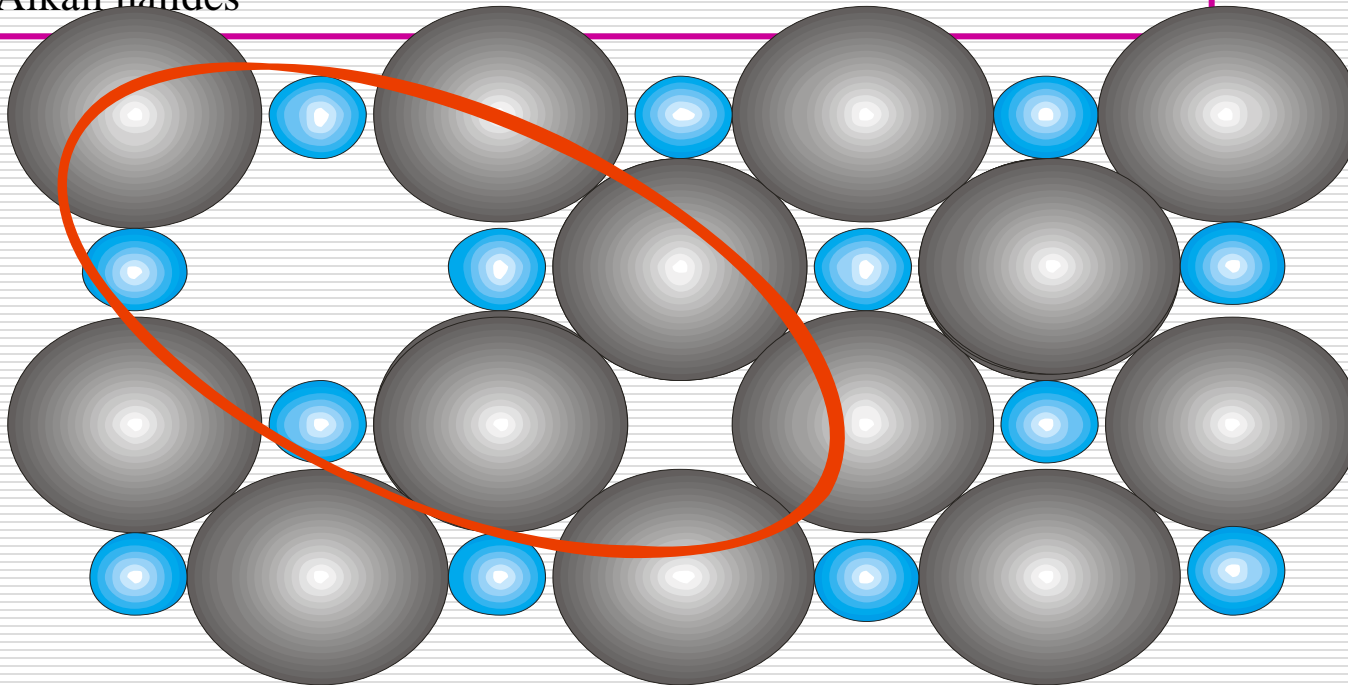


Fig: Schottky defect

Difference Between Frenkel & Schottky Defect

- Frenkel defect does not have any effect on the density of the lattice. This is because the leaving atoms or ions occupy a nearby position instead of leaving the lattice completely.
- Frenkel defect is found in lattices composed of atoms or ions that have considerable differences between their sizes.
- examples for Frenkel defect are AgCl, AgI, CaF₂ and ZnS
- Schottky defect causes the density of the lattice to be reduced.
- Schottky defect occurs in lattices with ions in similar sizes.
- This type of defect is common in lattices like NaCl, KBr, and KCl.

The properties of materials influenced by the presence of imperfections

- An **alloy** is a combination, either in solution or compound, of two or more elements, at least one of which is a metal.
- An alloy with two components is called a binary alloy; one with three is a ternary alloy; one with four is a quaternary alloy.
- The result of alloying is a metallic substance with properties different from those of its components.

Example: **Steel** is a metallic alloy whose major constituent is iron. One classical definition is that steels are iron-carbon alloys with up to 2.1% carbon with the increased carbon, steel is harder and has a much higher tensile strength than iron, but is also more brittle.

Line defects

□ The line defects, which take place due to dislocation of atoms along with a line in some direction, are called as line defects. There are two types of line defects which are as follows :

- (i) Edge dislocation
- (ii) Screw dislocation

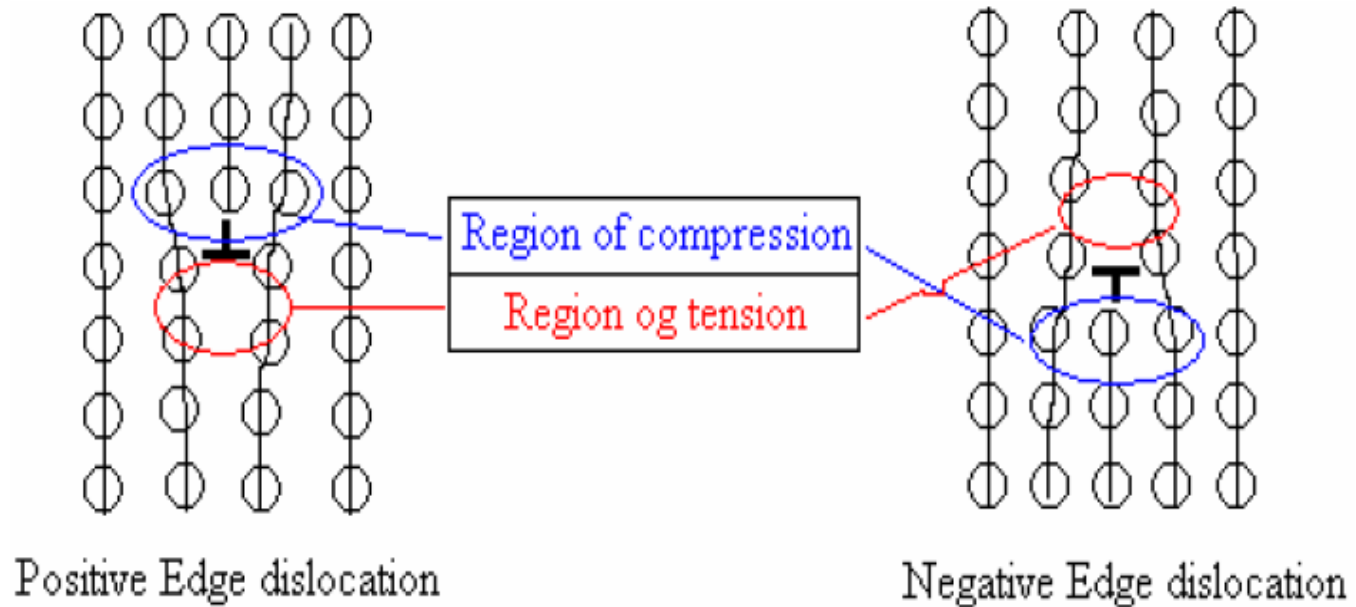
Edge Dislocation

- In perfect crystal, atoms are arranged in both vertical and horizontal planes parallel to the side faces.
- If one of these vertical planes does not extend to the full length, but ends in between within the crystal it is called '**edge dislocation**'.
- In the perfect crystal, just above the edge of the incomplete plane the atoms are squeezed and are in a state of compression.
- Just below the edge of the incomplete plane, the atoms are pulled apart and are in a state of tension.

Edge Dislocation

- Edge dislocations are represented by ' \perp ' or 'T' depending on whether the incomplete plane starts from the top or from the bottom of the crystal.
- These two configurations are referred to as positive and negative edge dislocations respectively.

Edge Dislocation



Edge Dislocation

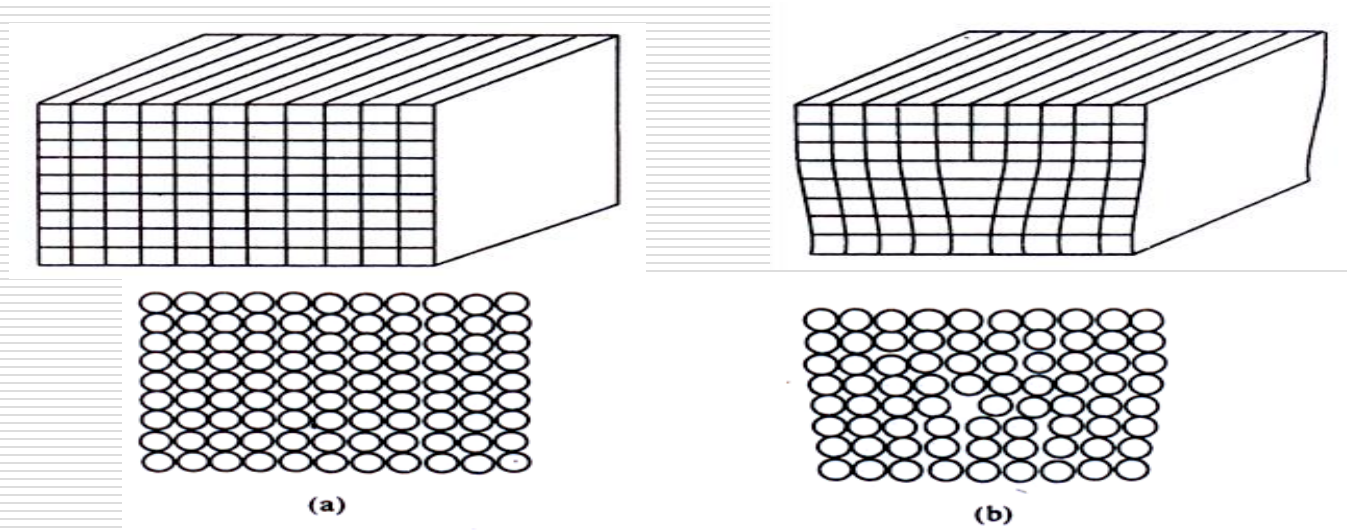
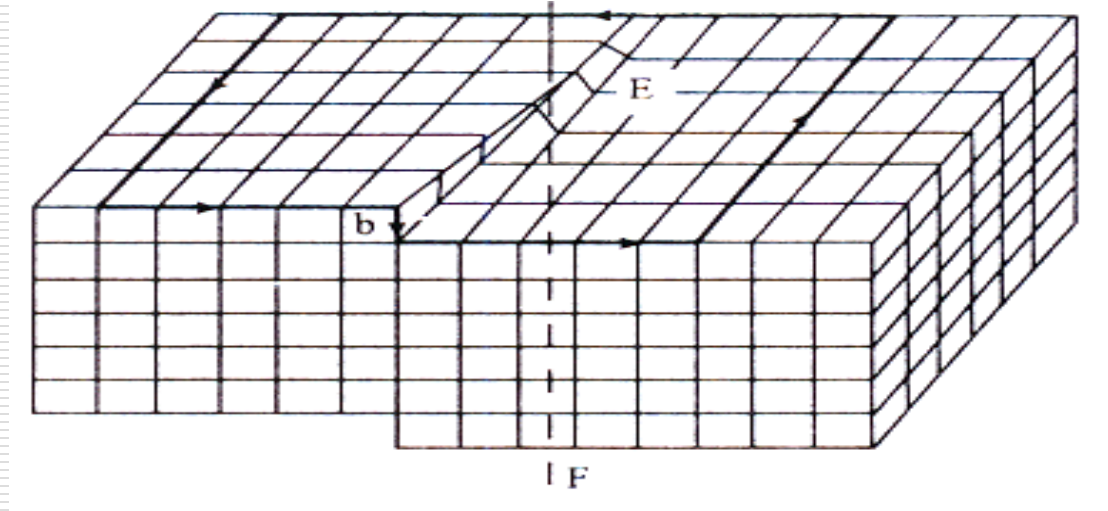


Fig: Edge Dislocation

Screw Dislocation

-
- In this dislocation, the atoms are displaced in two separate planes perpendicular to each other.
 - Speed of movement of a screw dislocation is lesser compared to edge dislocation.
 - Normally, the real dislocations in the crystals are the mixtures of edge and screw dislocation.

SCREW DISLOCATION



**Fig.No.15: Screw
Dislocation**

Burgers Vector

-
- The magnitude and the direction of the displacement are defined by a vector, called the **Burgers Vector**.
 - In figure 16 (a), starting from the point P, we go up by 6 steps, then move towards right by 5 steps, move down by 6 steps and finally move towards left by 5 steps to reach the starting point P. Now the Burgers circuit gets closed.

Burgers Vector

-
- When the same operation is performed on the defect crystal (figure 16 (b)) we end up at Q instead of the starting point.
 - So, we have to move an extra step to return to P, in order to close the Burgers circuit.
 - The magnitude and the direction of the step defines the Burgers Vector (BV).
 - $BV = \overline{QP} = b$
 - The Burgers Vector is perpendicular to the edge dislocation line.

Burgers Vector

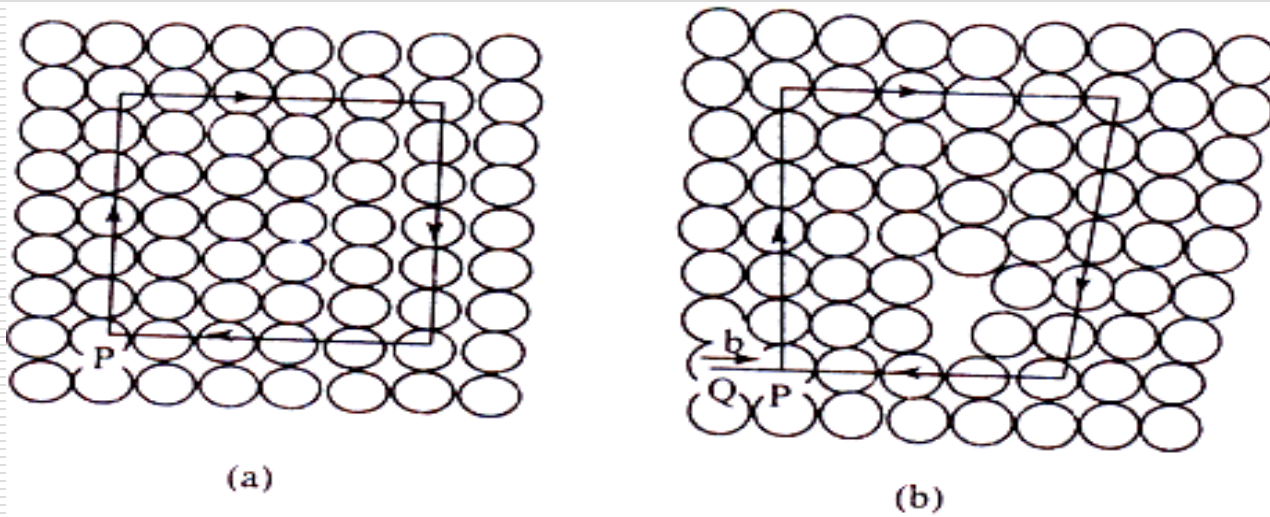


Fig. No.16 : Burger Vector

Surface Imperfections

- Surface imperfections arise from a change in the stacking of atomic planes on or across a boundary.
- The change may be one of the orientations or of the stacking sequence of atomic planes.
- In geometric concept, surface imperfections are two-dimensional.

Surface Defect

- Surface defect is the defects takes place on the surface of a material are known as surface defects.
- The following are the types of surface defects :
 1. Grain boundary
 2. Twin boundary
 3. Stacking fault

Grain Boundary

1. Grain boundary :

- Grains of different orientation of atoms which exhibits a boundary as shown in Fig.No. 17.
- The defect caused is called as grain boundary. When a liquid metal gets solidified the defect takes place.

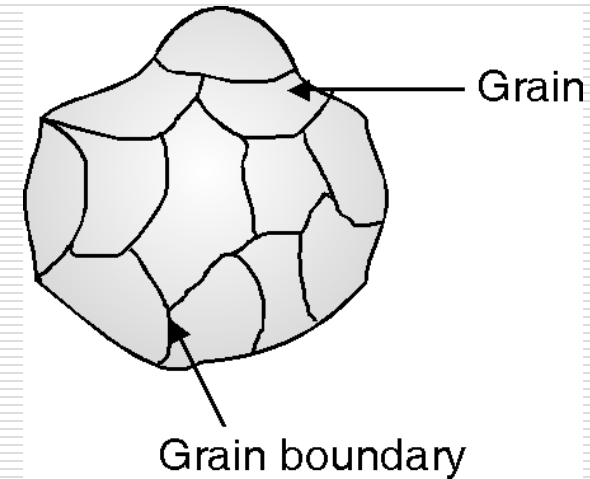


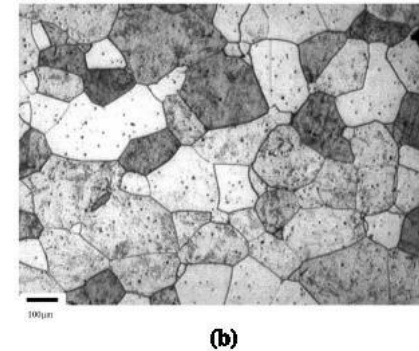
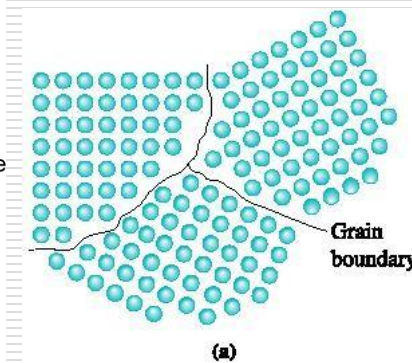
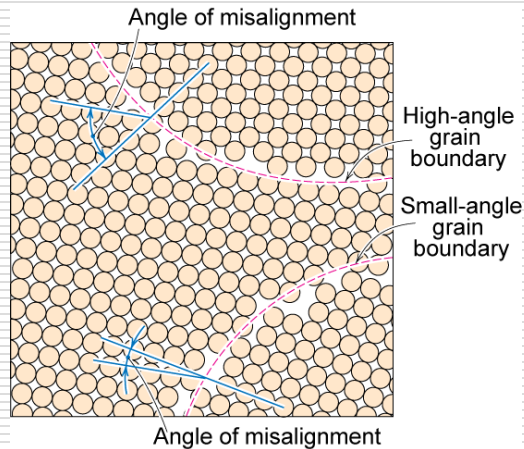
Fig.No. 17 : Grain boundary

Grain Boundary

Grain boundaries:

- boundaries between crystals.
- have a change in crystal orientation across them.
- impede dislocation motion.
- produced by the solidification process

Schematic

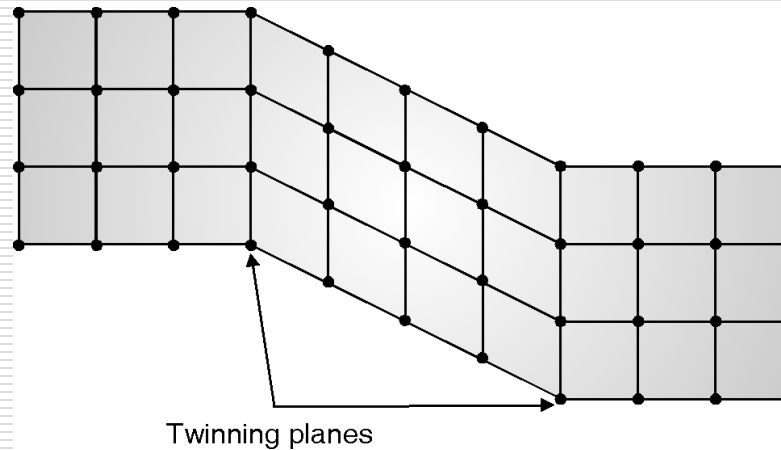


Grain Boundary

- ❑ A grain boundary is the interface between two grains, or crystallites, in a polycrystalline material.
- ❑ Grain boundaries are 2D defects in the crystal structure, and tend to decrease the electrical and thermal conductivity of the material.
- ❑ Most grain boundaries are preferred sites for the onset of corrosion and for the precipitation of new phases from the solid.
- ❑ They are also important to the mechanisms of creep.
- ❑ On the other hand, grain boundaries disrupt the motion of dislocations through a material, so reducing crystallite size is a common way to improve mechanical strength,

Planar Defects in Solids - Twinning

- A shear force that causes atomic displacements such that the atoms on one side of a plane (twin boundary) mirror the atoms on the other side. A reflection of atom positions across the **twin plane**.
- Twins may form during solidification, deformation or during deformation and annealing



Twin boundary

- When the boundaries in which the arrangement of atoms on one side of the boundary is a mirror image of the arrangement of atoms of the another side as shown in Fig. No.18

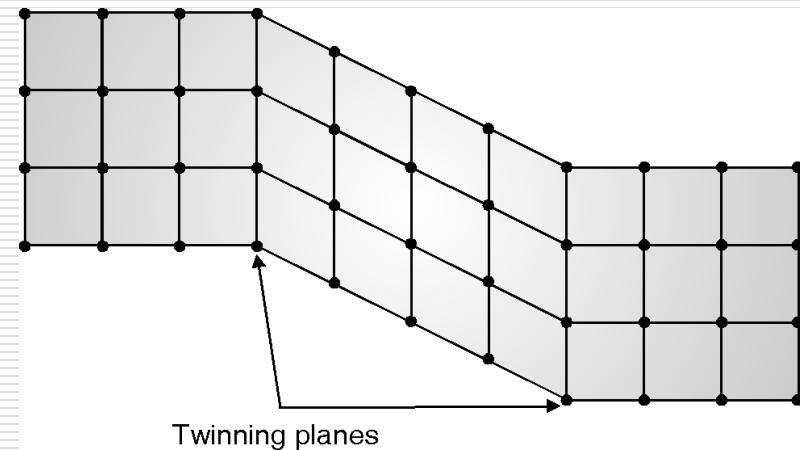


Fig. No.18 : Twin bounda

Stacking fault

- When the stacking of atoms is not in a proper sequence throughout the crystal, the fault caused is called as stacking fault.
- Fig.No.19(a) shows that the proper sequence of atomic planes we can read from lower end to upper end end is A-B-C-A-B-C-A-B-C.
- The region in which stacking fault obtain (A-B-A-B) form a thin region of hexagonal close pack in a F.C.C. crystals.

Stacking fault

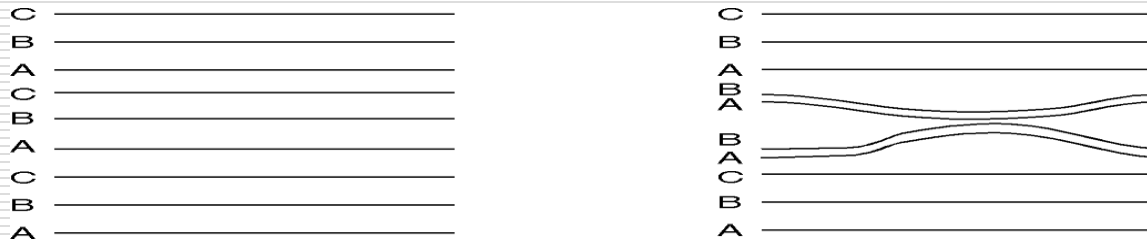


Fig. No.19 : Stacking fault

Volume or 3D Defect:

- **Volume Defect:** Voids or local regions featuring different phase (e.g. precipitates or amorphous phase) in crystalline materials.

Volume or 3D Defect:

-
- **Pores** - can greatly affect thermal, mechanical properties
 - **Cracks** - can greatly affect mechanical properties
 - **Foreign inclusions** - can greatly affect electrical, mechanical properties

MATERIAL SCIENCE

Miller Indices

Definition

- Miller indices are used to specify directions and planes.
- These directions and planes could be in lattices or in crystals.
- The number of indices will match with the dimension of the lattice or the crystal.
- E.g. in 1D there will be 1 index and 2D there will be two indices etc.

Notation Summary

- $[hkl]$ represents a direction
- $\langle hkl \rangle$ represents a family of directions
- (hkl) represents a plane
- $\{hkl\}$ represents a family of planes

Miller Indices for Directions

- A vector \mathbf{r} passing from the origin to a lattice point can be written as:

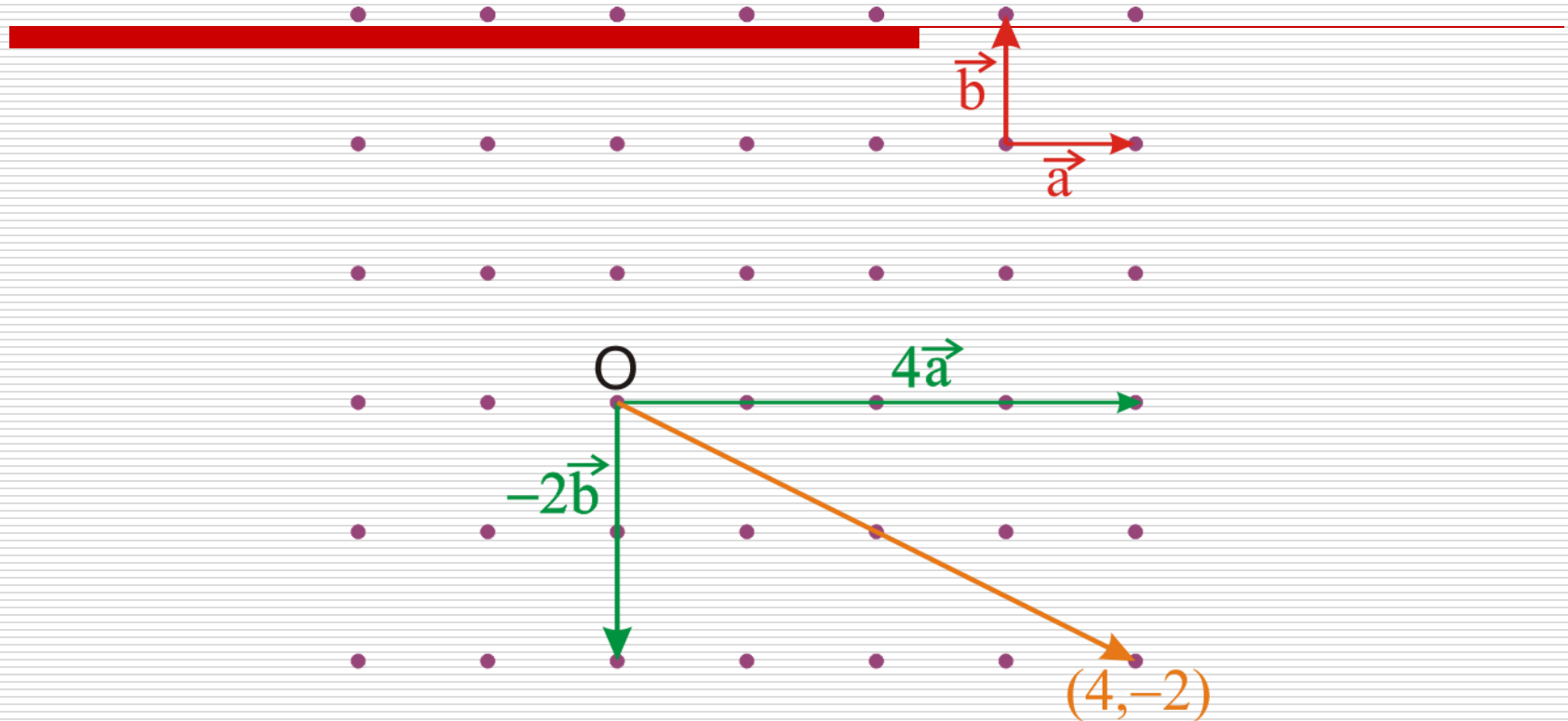
$$\mathbf{r} = r_1 \mathbf{a} + r_2 \mathbf{b} + r_3 \mathbf{c}$$

where, $\mathbf{a}, \mathbf{b}, \mathbf{c} \rightarrow$ basic vectors and

millar indices $\rightarrow (r_1 r_2 r_3)$

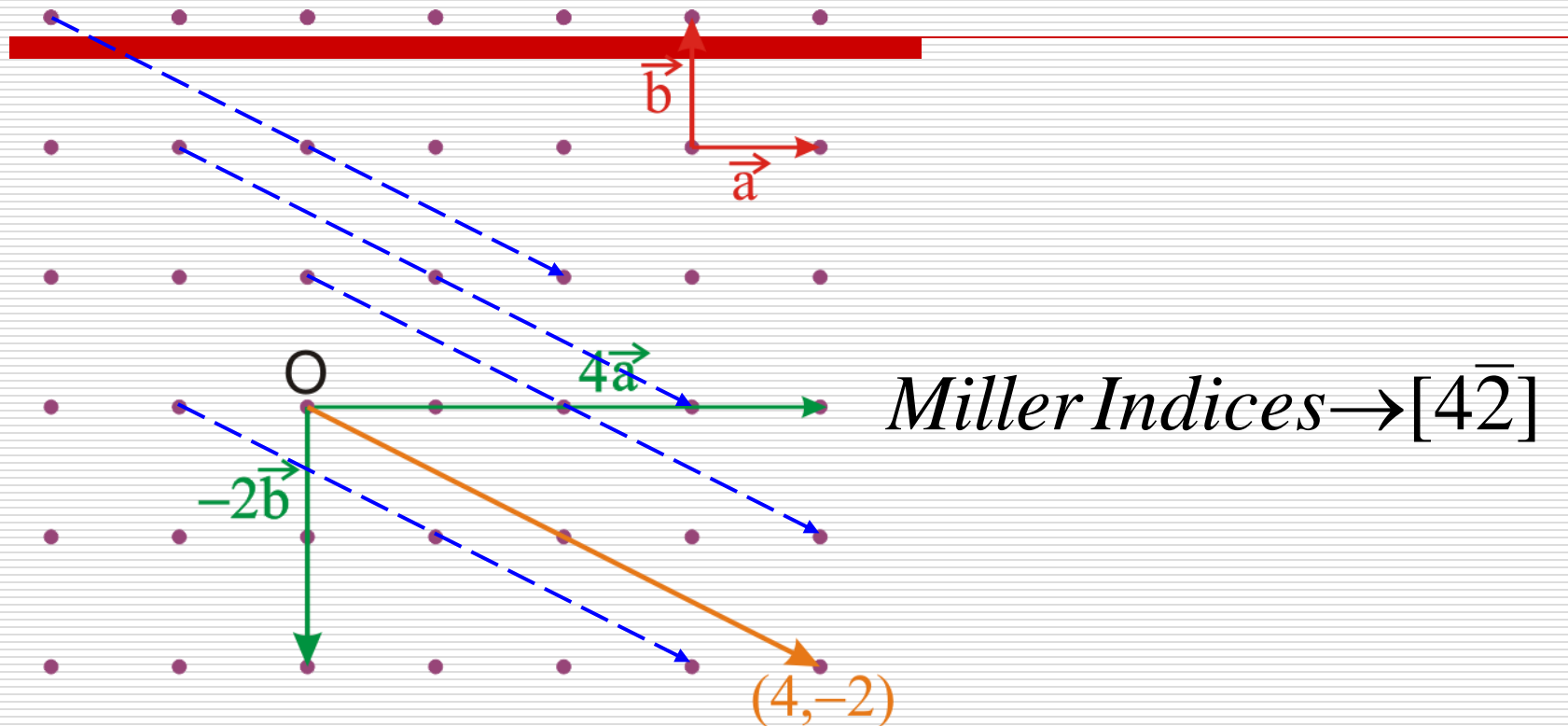
- Fractions in $(r_1 r_2 r_3)$ are eliminated by multiplying all components by their common denominator.
- [e.g. $(1, \frac{3}{4}, \frac{1}{2})$ will be expressed as (432)]

Example



Miller Indices $\rightarrow [4\bar{2}]$

Example (cont'd)



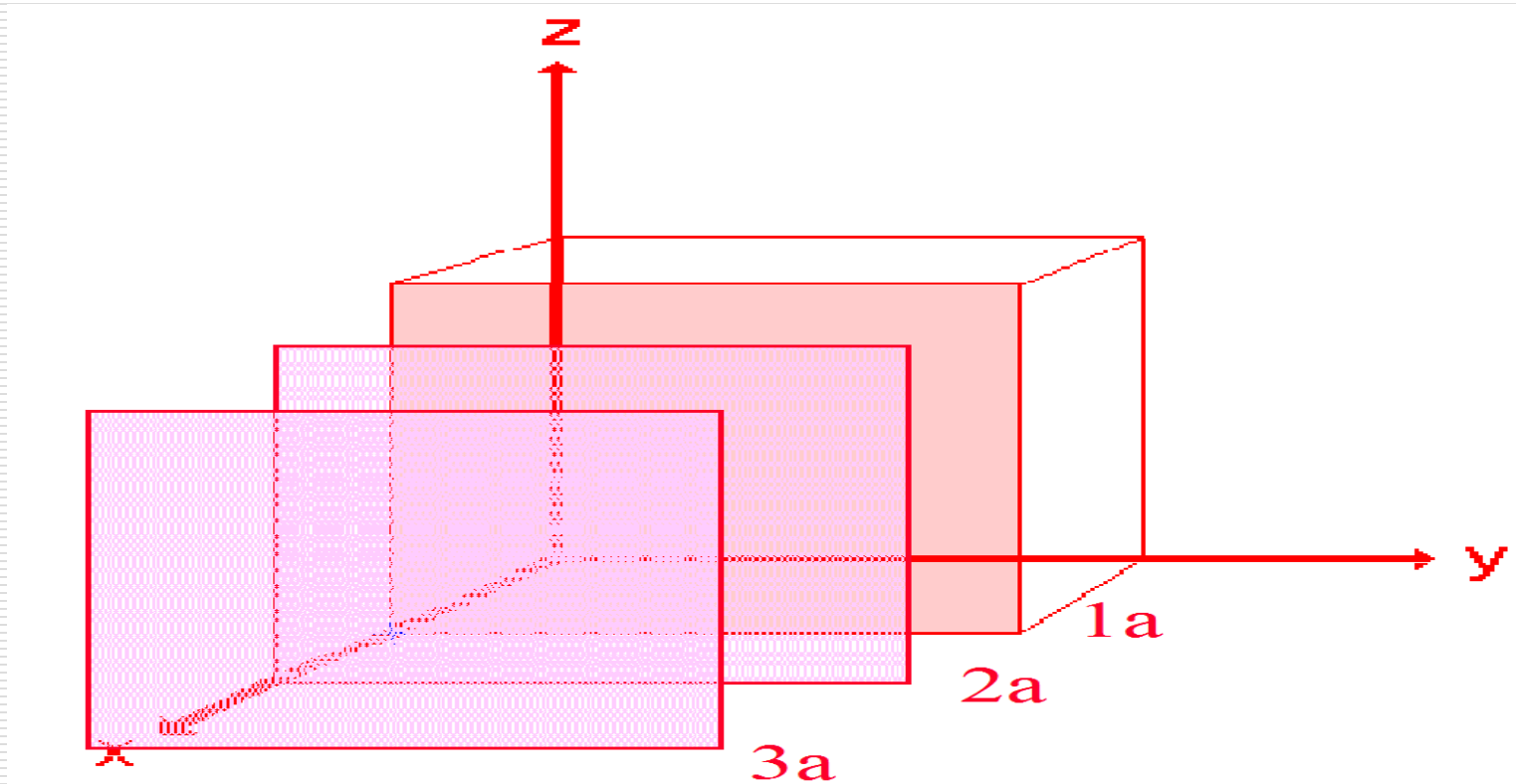
The index represents a set of all such parallel
vectors

Miller Indices for Planes: Procedure



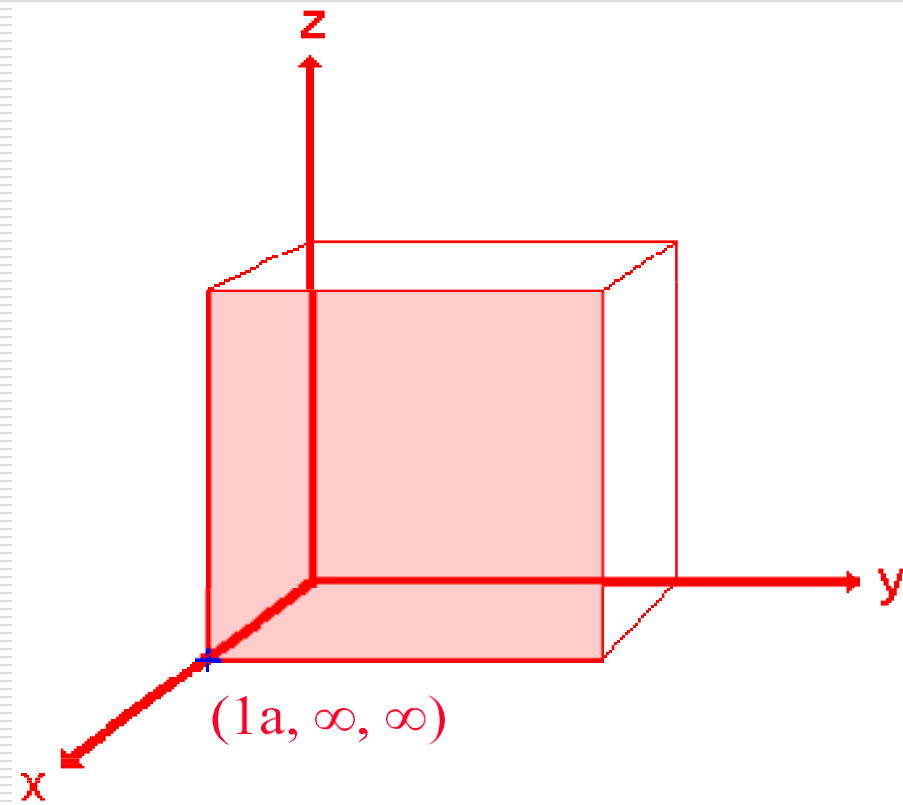
1. Identify the plane intercepts on the x, y and z-axes.
2. Specify intercepts in fractional coordinates.
3. Take the reciprocals of the fractional intercepts.

Miller Indices for Planes: Illustration



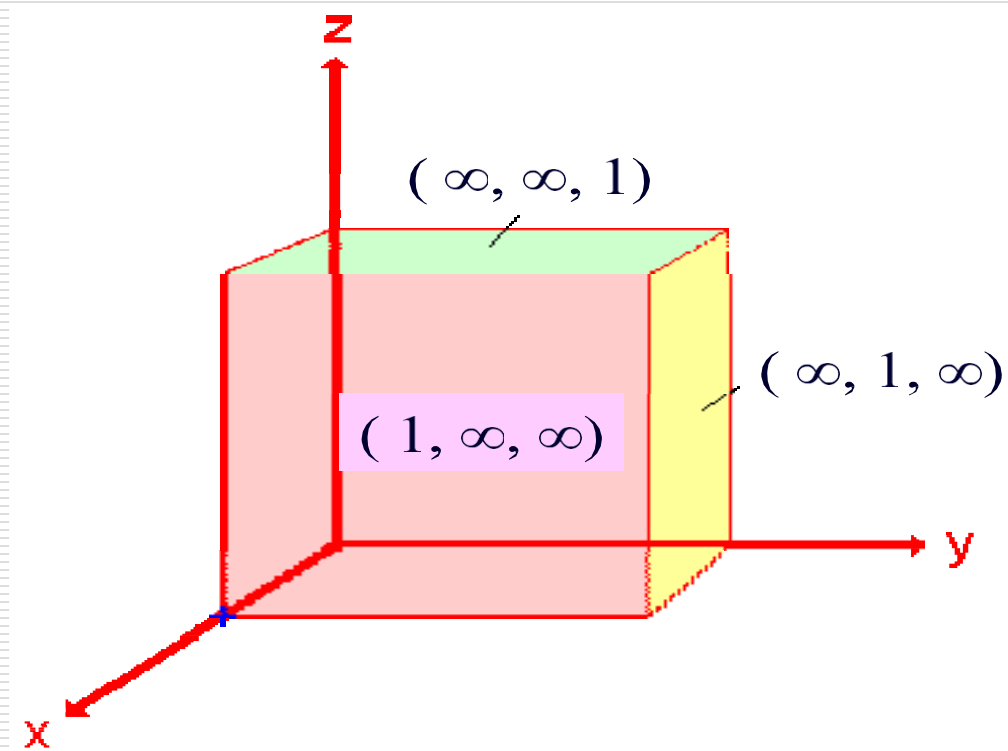
Miller Indices for Planes: Illustration

- The plane intersects the x-axis at point a. It runs parallel along y and z axes.
- Thus, this plane can be designated as $(1, \infty, \infty)$



Miller Indices for Planes: Illustration

- Likewise, the yellow plane can be designated as $(\infty, 1, \infty)$
- And the green plane can be written as $(\infty, \infty, 1)$



Miller Indices for Planes: Illustration

- Miller Indices are the reciprocals of the parameters of each crystal face. Thus:

- Pink Face

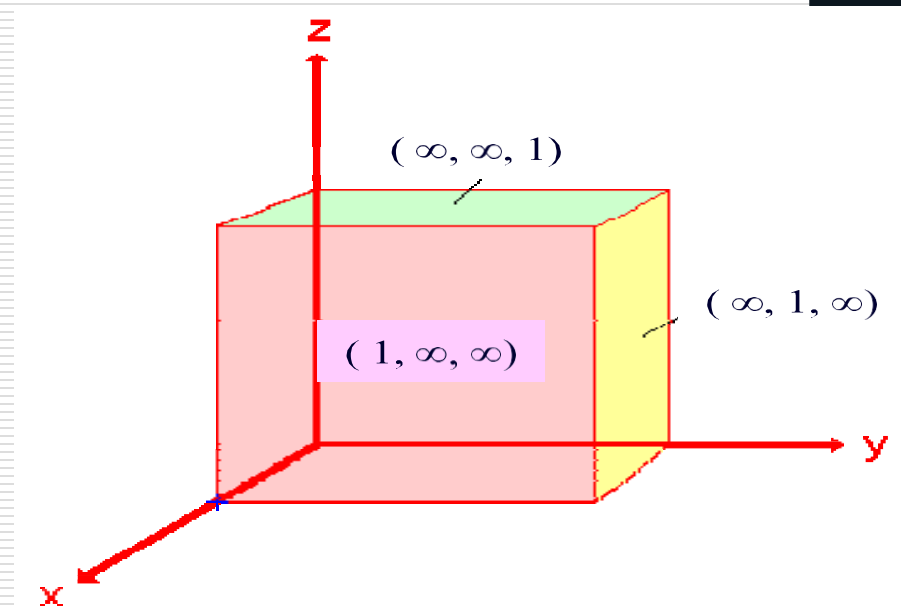
$$= (1/1, 1/\infty, 1/\infty) = (100)$$

- Green Face

$$= (1/\infty, 1/\infty, 1/1) = (001)$$

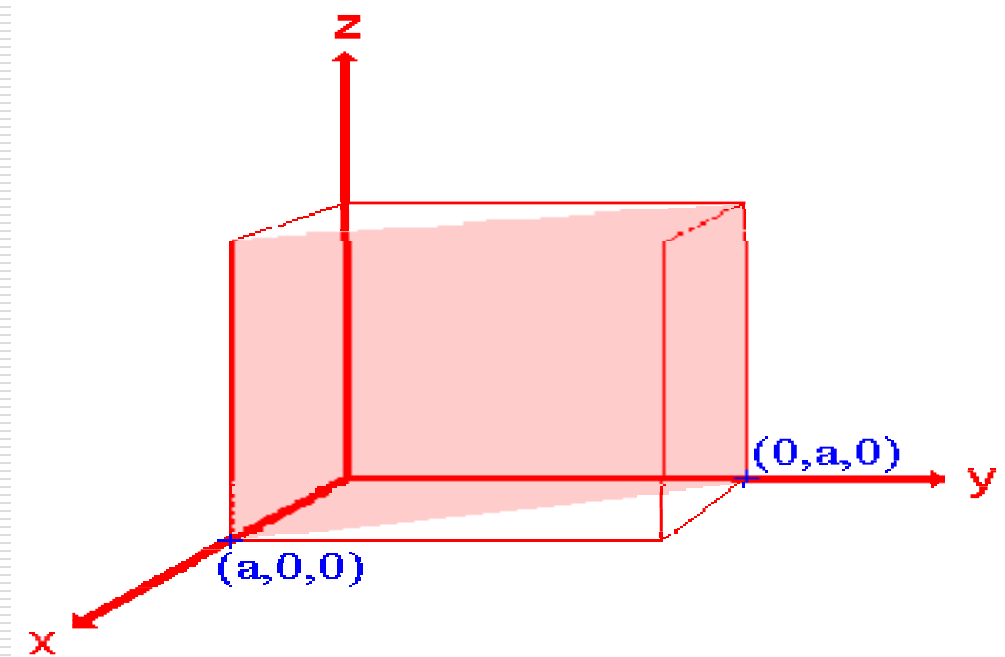
- Yellow Face

$$= (1/\infty, 1/1, 1/\infty) = (010)$$



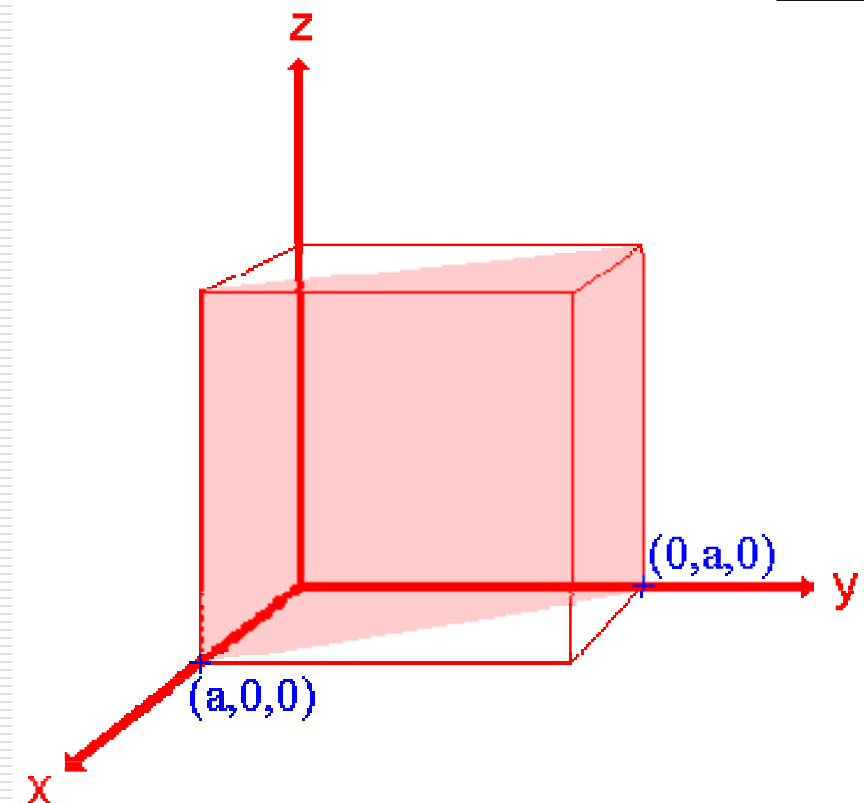
Examples

What's the Miller Index of this plane?



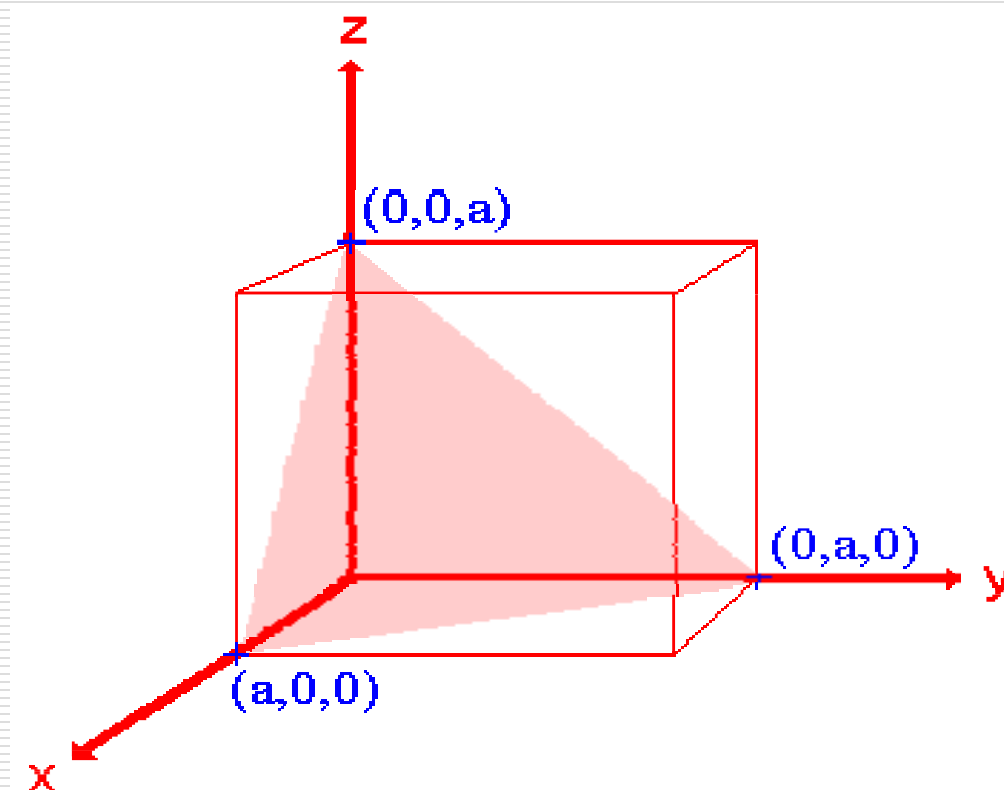
Examples (cont'd)

- The plane of interest cuts two of the crystallographic axes.
- Intercepts: $(1, 1, \infty) \rightarrow (110)$



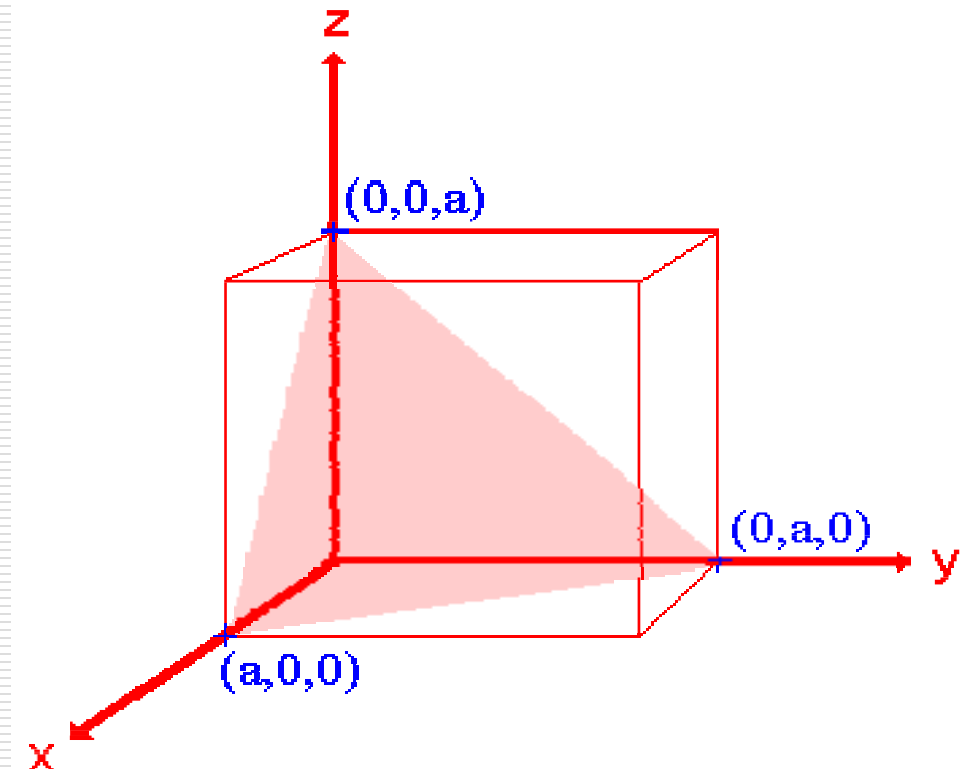
Examples (cont'd)

- Miller Index?



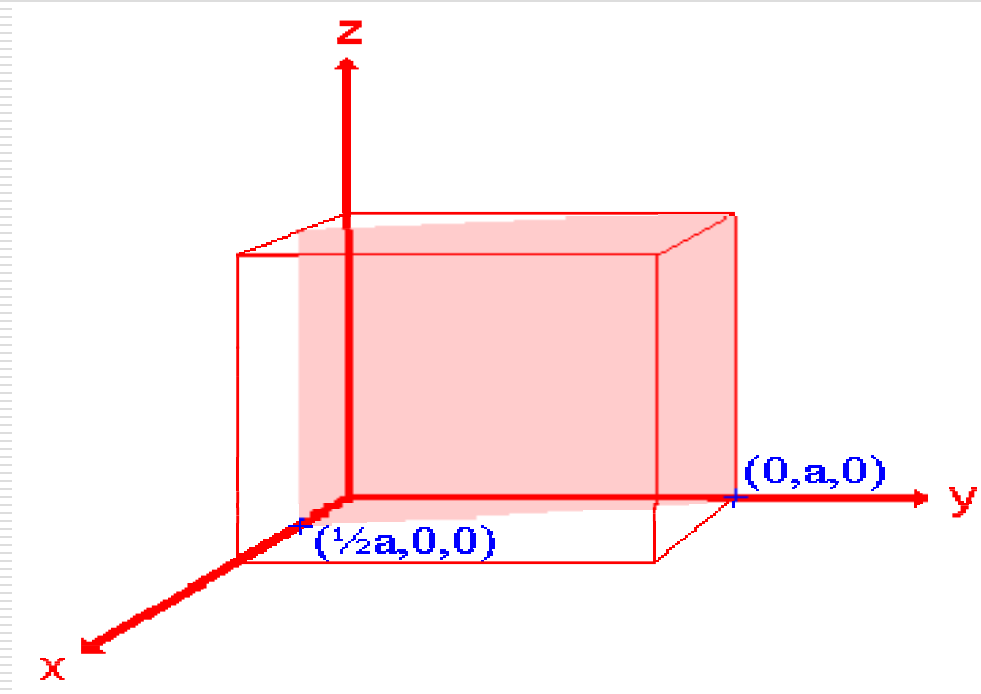
Examples (cont'd)

- This plane cuts all three crystallographic axes.
- Intercepts = $(1,1,1) \rightarrow (111)$



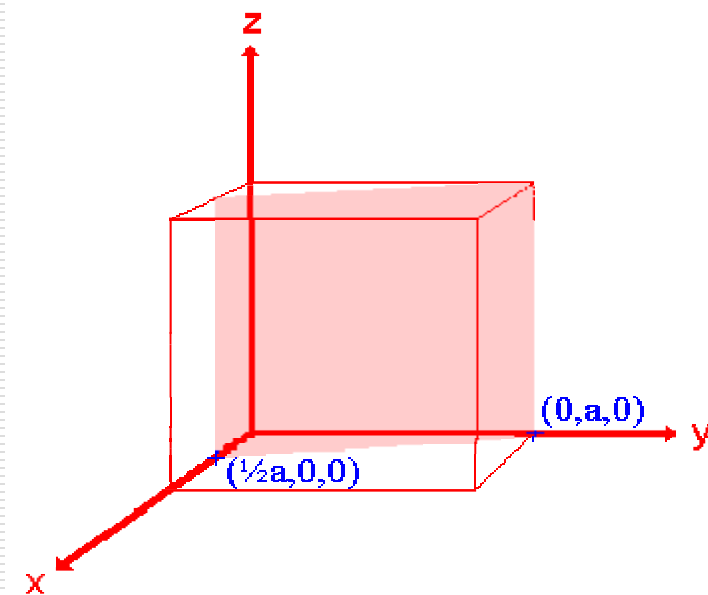
Examples (cont'd)

- Miller Index? (Difficult one)

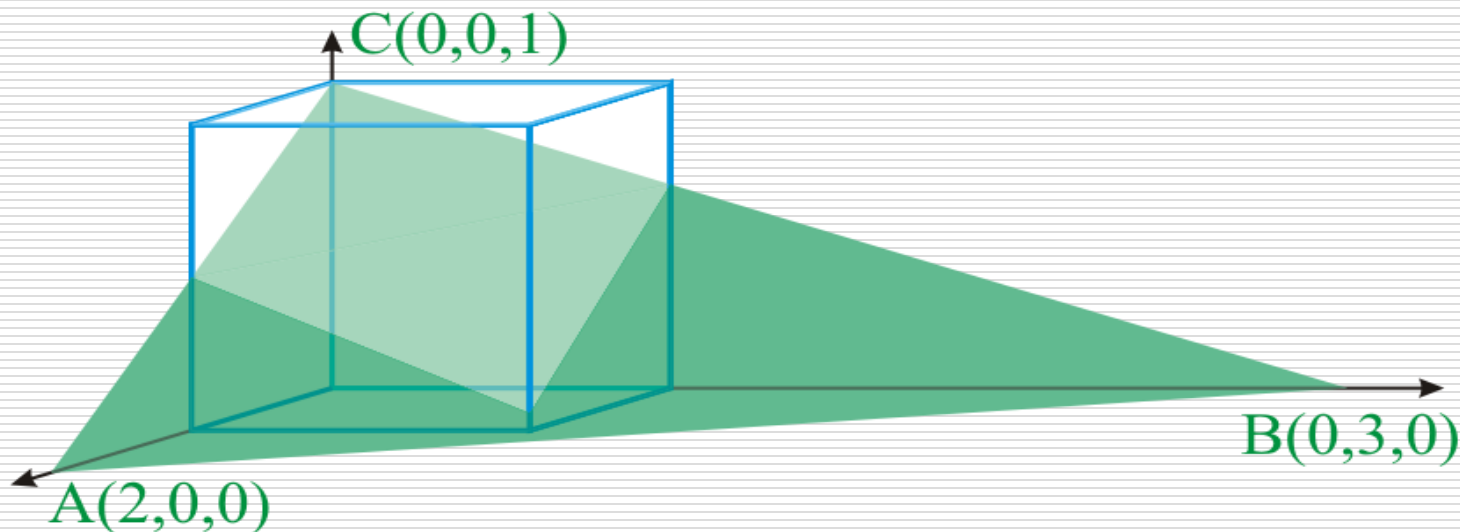


Examples (cont'd)

- This plane cuts two of the reference axes, but not equidimensionally.
- Intercepts: $(\frac{1}{2}, 1, 0) \rightarrow (210)$



Examples (cont'd)



- ❑ Find intercepts along axes $\rightarrow 2 \ 3 \ 1$
- ❑ Take reciprocal $\rightarrow 1/2 \ 1/3 \ 1^*$
- ❑ Convert to smallest integers in the same ratio $\rightarrow 3 \ 2 \ 6$
- ❑ Enclose in parenthesis $\rightarrow (326)$
- ❑ Note: (326) does **NOT** represent one plane but *an infinite set of parallel planes* passing through lattice points.

$$\frac{1}{2} \ \frac{1}{3} \ 1$$

Family of Directions

It's a set of directions related by symmetry operations of the lattice.

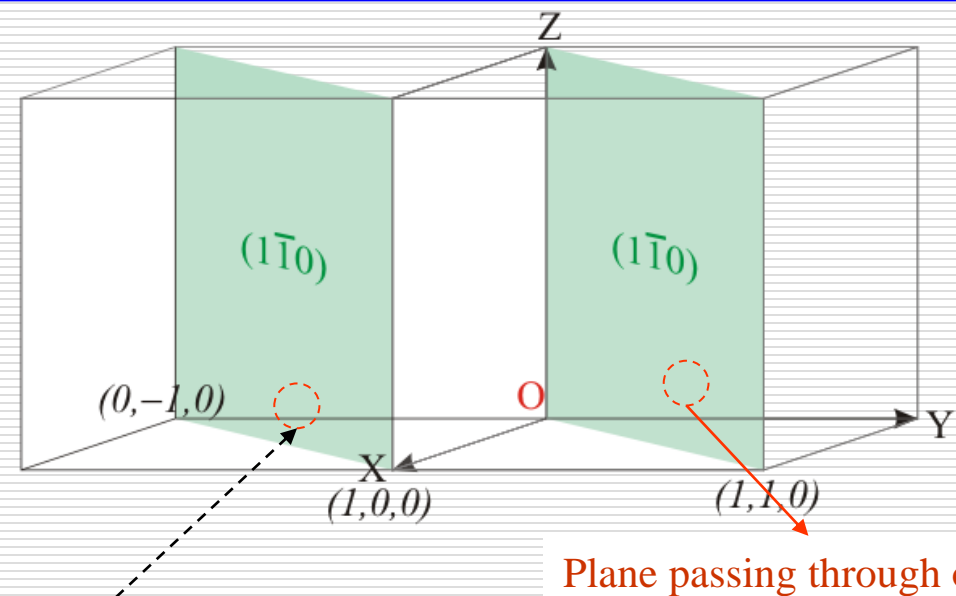
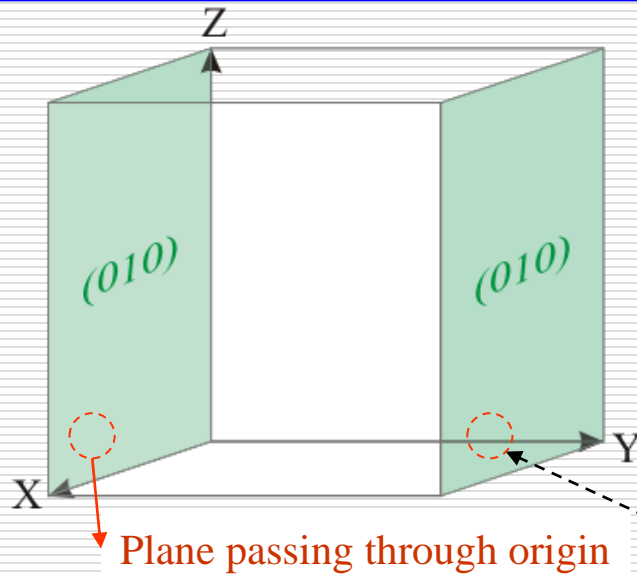
Index	Members in family for cubic lattice
$\langle 100 \rangle$	$[100], [\bar{1}00], [010], [0\bar{1}0], [001], [00\bar{1}]$
$\langle 110 \rangle$	$[110], [\bar{1}\bar{1}0], [1\bar{1}0], [\bar{1}10], [101], [\bar{1}01], [10\bar{1}], [\bar{1}0\bar{1}], [011], [0\bar{1}1], [01\bar{1}], [0\bar{1}\bar{1}]$
$\langle 111 \rangle$	$[111], [\bar{1}\bar{1}\bar{1}], [1\bar{1}\bar{1}], [\bar{1}1\bar{1}], [1\bar{1}1], [\bar{1}\bar{1}1], [11\bar{1}], [\bar{1}11]$

More Examples

❑ What about the plane passing through the origin?



- ❑ The Miller indices refer to a whole set of parallel planes passing through lattice points. This implies that one of the planes of the infinite set actually passes through the origin.
- ❑ As shown below, usually we do not take that particular plane for the determination of Miller indices; but a parallel one, which does not pass through the origin.



Intercepts $\rightarrow \infty 0 \infty$
Plane $\rightarrow (0 \infty 0)$

Hence use this plane

Intercepts $\rightarrow 0 0 \infty$
Plane $\rightarrow (\infty \infty 0)$

We want to avoid infinities in Miller indices
In such cases the plane is translated by a unit distance along the non zero axis/axes
and the Miller indices are computed

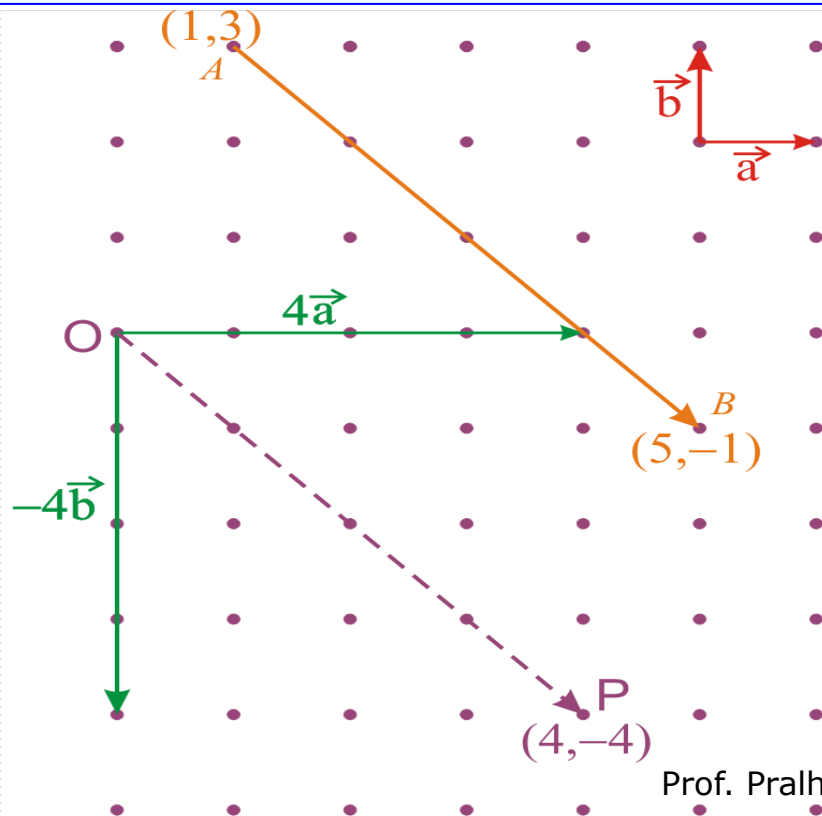
Prof. Pralhad Pesode, MITWPU Pune

Intercepts $\rightarrow \infty 1 \infty$
Plane $\rightarrow (0 1 0)$

Intercepts $\rightarrow 1 -1 \infty$
Plane $\rightarrow (1 -1 0)$

How to find the Miller Indices for an arbitrary direction? → Procedure

- ❑ Consider the example below.
- ❑ Subtract the coordinates of the end point from the starting point of the vector denoting the direction → If the starting point is A(1,3) and the final point is B(5,-1) → the difference (B-A) would be (4, -4).



- ❑ Enclose in square brackets, remove comma and write negative numbers with a bar → $[4\bar{4}]$
- ❑ Factor out the common factor → $4[1\bar{1}]$
- ❑ If we are worried about the direction and magnitude then we write → $4[1\bar{1}]$
- ❑ If we consider only the direction then we write → $[1\bar{1}]$

Materials characterization

- **Characterization**, when used in materials science, refers to the broad and general process by which a material's structure and properties are probed and measured.
- It is a fundamental process in the field of materials science, without which no scientific understanding of engineering materials could be ascertained.
- While many characterization techniques have been practiced for centuries, such as basic optical microscopy, new techniques and methodologies are constantly emerging

Materials characterization....

- Materials testing and characterization is carried out to understand the fundamental properties of a material when subjected to service and environmental loading and operating conditions.
- Testing helps us to understand and quantify whether a specific material is suitable to a particular application.
- Material not properly tested and used in a automotive, aerospace or biomedical product can be very dangerous as it has the potential of putting an end user's life in danger.

Materials characterization....

Analysis and Characterization Technology to Support the Development of New Steels

- Materials characterization based on analysis techniques on the nanometer scale is playing an increasingly important role in the development of high-grade steels.
- While advanced devices such as transmission electron microscopes (TEM), scanning electron microscopes (SEM), and electron probe micro-analyzers (EPMA) have been effectively employed for characterizing micro-metallographic structures, textures, and non-metallic inclusions that govern the material properties of steel products,

Types characterization....

- Microscopy is a category of characterization techniques which probe and map the surface and sub-surface structure of a material.
- Some common examples of microscopy instruments include:
 1. Optical Microscope
 2. Scanning Electron Microscope (SEM)
 3. Transmission Electron Microscope (TEM)
 4. Field Ion Microscope (FIM)
 5. Scanning Tunneling Microscope (STM)
 6. Scanning probe microscopy (SPM)
 7. Atomic Force Microscope (AFM)
 8. X-ray diffraction topography (XRT)

Micro and Macro Examinations

Micro-examination

- Involves the study of the metal structure & their alloys under microscope at magnification from 20X to 2000X & the observed structure is called microstructure.

Macro-examination

- Involves the study of the metal structure & their alloys by naked eye or by low power magnification up to 15X. & the observed structure is called macrostructure.

Macroscopic testing

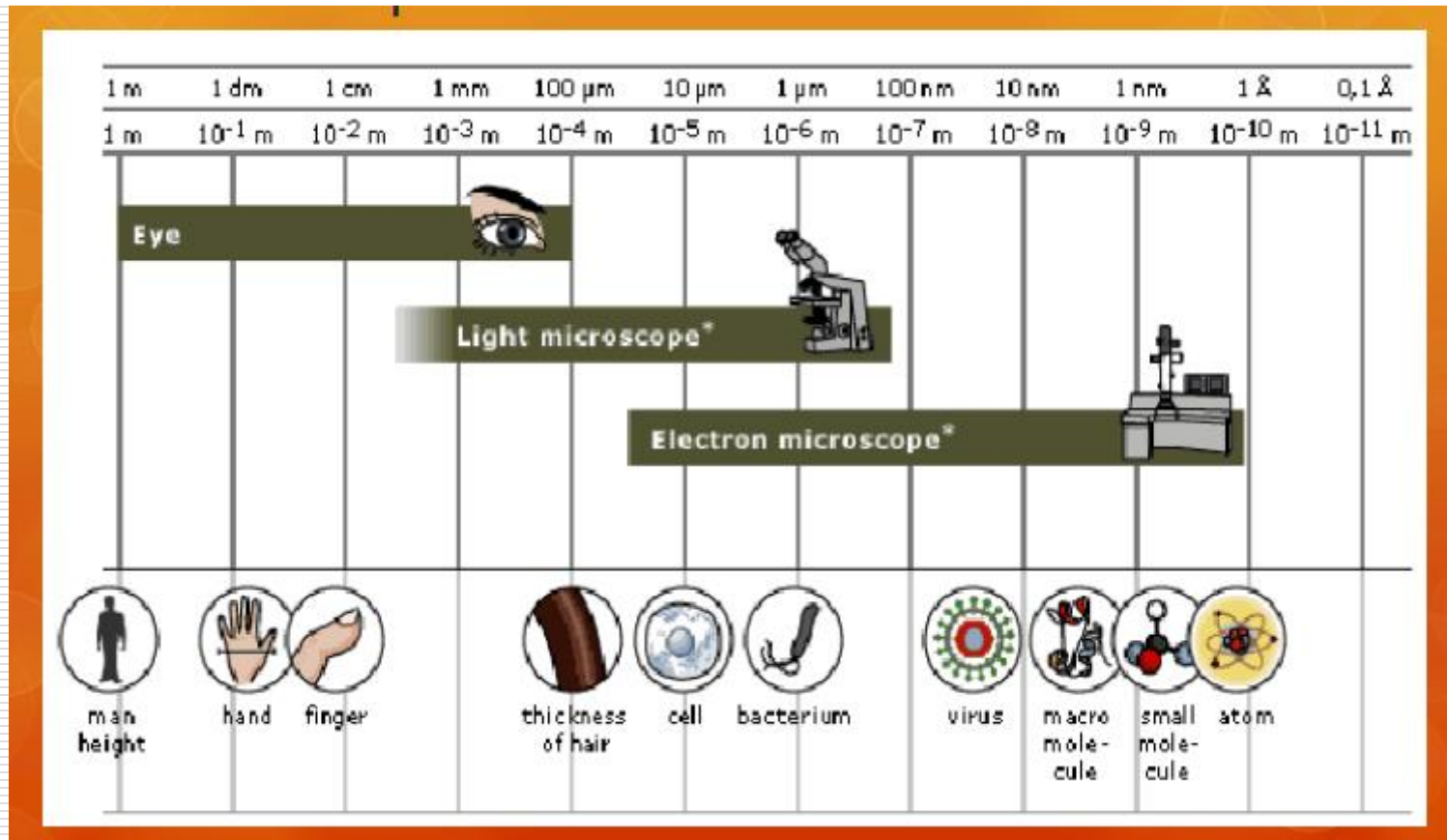
Macroscopic testing:-

- A huge range of techniques are used to characterize various macroscopic properties of materials, including:
- Mechanical testing, including tensile, compressive, torsional, creep, fatigue, toughness and hardness testing

Why We Need Electron Microscope?

- Light Microscopes are limited by the physics of light to 500x or 1000x magnification and a resolution of 0.2 micrometers.
- In the early 1930's there was a scientific desire to see the fine details of the interior structures of organic cells (nucleus)
- This required 10,000x plus magnification which was just not possible using Light Microscopes.

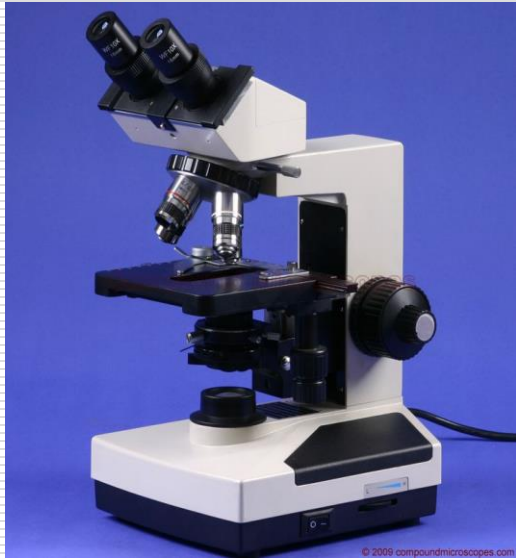
Comparison Between Light & Electron Microscope



Comparison Between Light & Electron Microscope

FEATURE	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
Electromagnetic spectrum used	Visible light 760nm (red) – 390nm Colours visible	Electrons app. 4nm Monochrome
Maximum resolving power	app. 200nm	0.2nm Fine detail
Maximum magnification	x1000 – x1500	↓ x500 000
Radiation source	Tungsten or quartz halogen lamp	High voltage (50kV) tungsten lamp
Lenses	Glass	Magnets
Interior	Air-filled	Vacuum
Focussing screen	Human eye (retina), photographic film	fluorescent (TV) screen, photographic film

Comparison Between Light & Electron Microscope



Compound light microscope

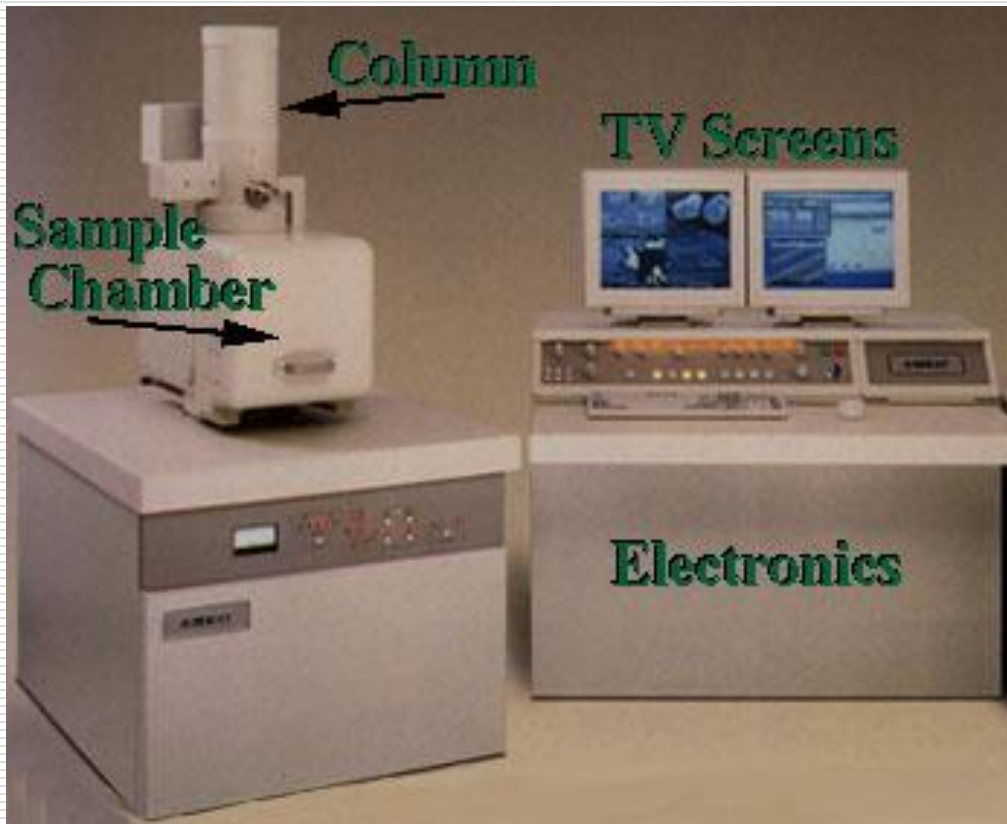


Transmission electron microscope

Electron Microscope

- ❑ An electron microscope is a microscope that uses a beam of accelerated electrons as a source of illumination.
- ❑ As the wavelength of an electron can be up to 100,000 times shorter than that of visible light photons,
- ❑ electron microscopes have a higher resolving power than light microscopes and can reveal the structure of smaller objects
- ❑ Electron microscopes are used to investigate the ultra structure of a wide range of biological and inorganic specimens including micro organism, cells, large molecules, metals, and crystal.
- ❑ Industrially, electron microscopes are often used for quality control and failure analysis. Modern electron microscopes produce electron micrograph using specialized digital cameras and frame to capture the image.

SEM



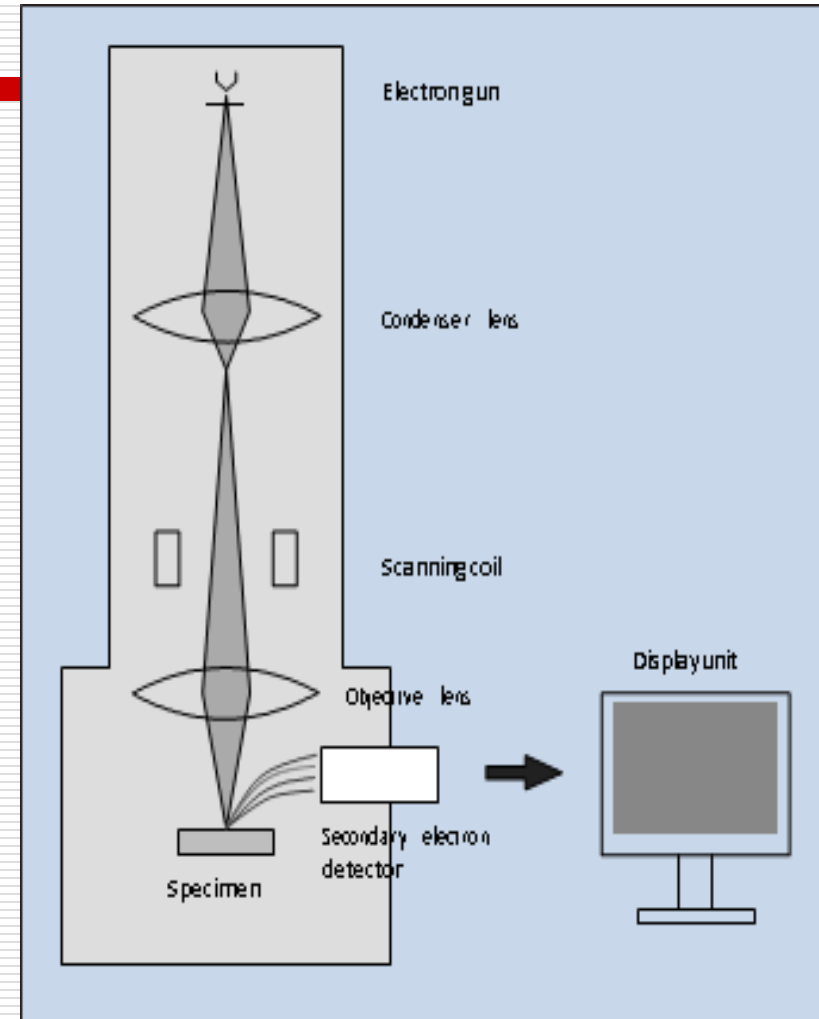
The SEM is designed for direct studying of the surfaces of solid objects

Scanning electron microscope (SEM) is a microscope that uses electrons rather than light to form an image.

SEM

Construction of Instrument

The SEM requires an electron optical system to produce an electron probe, a specimen stage to place the specimen, a secondary-electron detector to collect secondary electrons, an image display unit, and an operation system to perform various operations. The electron optical system consists of an electron gun, a condenser lens and an objective lens to produce an electron probe, a scanning coil to scan the electron probe, and other components.



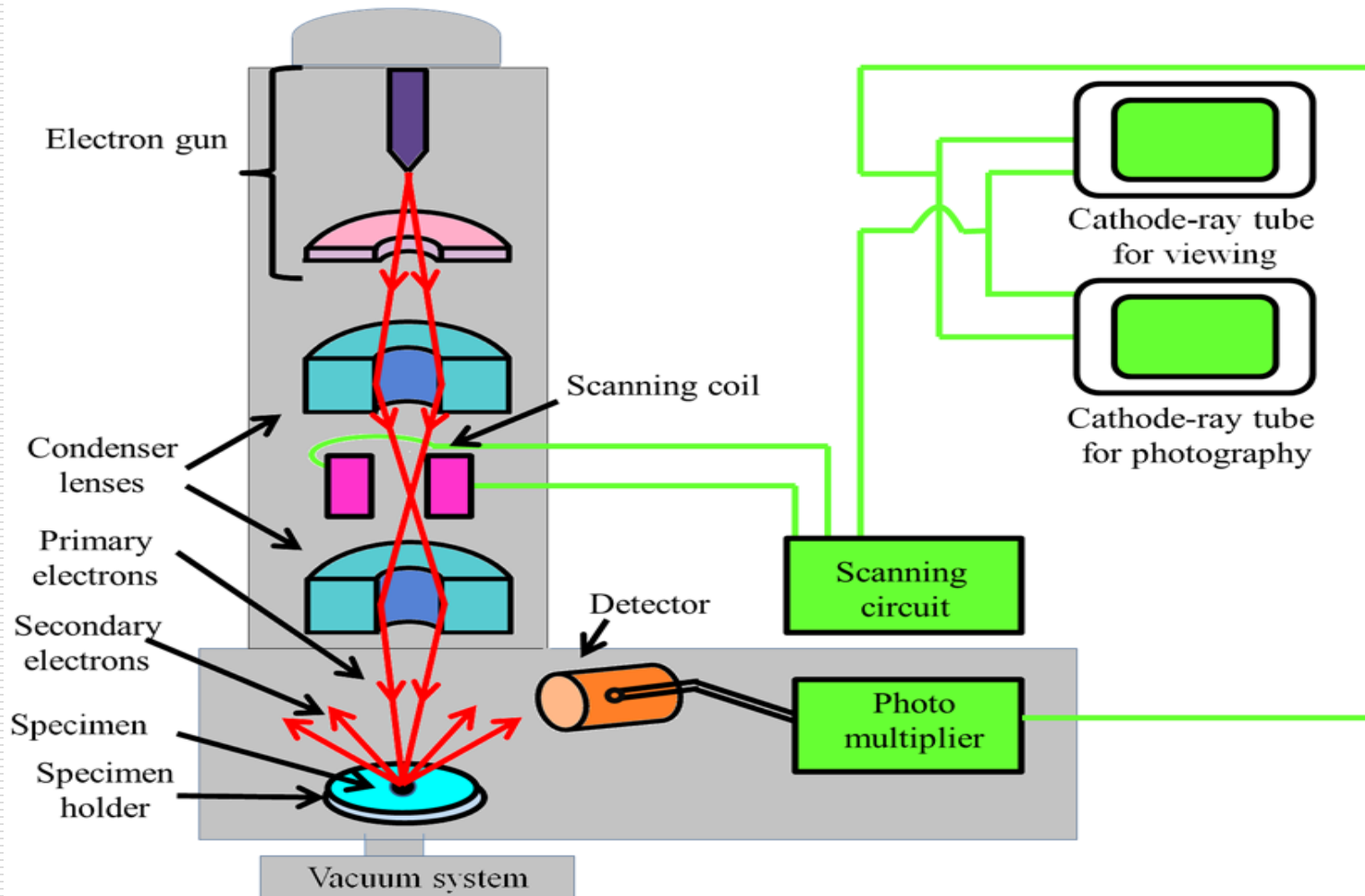


Figure: A simplified schematic diagram of a scanning electron microscope.

SEM

Electron Gun:

The electron gun produces an electron beam. Thermo electrons are emitted from a filament (cathode) made of a thin tungsten wire (about 0.1 mm) by heating the filament at high temperature (about 2800K). (MP 3422 C)

Condensing Lens:

- All the electrons are negatively charged and hence the electrons in a beam will repel each other.
- This will increase the beam diameter and will adversely affect the resolution of the image. Hence electrical coils are used to squeeze the beam to a diameter of 5 nm or less. These are called condensing lens coils.

SEM

Objective lens

- The objective lens is used for focusing, and this lens is a very important lens that determines the final diameter of the electron probe.
- If the performance of the objective lens is not good, an optimally-fine electron probe cannot be produced despite all of the efforts before the action of the objective lens. Thus, it is crucial to make the objective lens with the best performance.

SEM

Secondary Electron Detector :

- When the electrons strike the atoms, sometimes the electron in the inner shell will be ejected out.
- Then, an electron from the outer shell will fall into the inner shell and the difference in energy will be emitted as X Rays. The characteristic X Rays are produced in a tear drop shaped region.
- By monitoring the X Ray, one can determine the composition of the surface.

Back Scattered Electron Detector :

- Some of the primary electrons are scattered back and a detector positioned appropriately can be used to collect the information.
- This is called back scattered electron (BSE) detector. The BSE comes mainly from nuclear interaction.
- The BSE signal depends on the composition. Hence BSE can be used to obtain information about the composition

SEM

Image Display and Recording

- The output signals from the secondary electron detector are amplified and then transferred to the display unit.
- Since the scanning on the display unit is synchronized with the electron-probe scan, brightness variation, which depends on the number of the secondary electrons, appears on the monitor screen on the display unit, thus forming a SEM image.

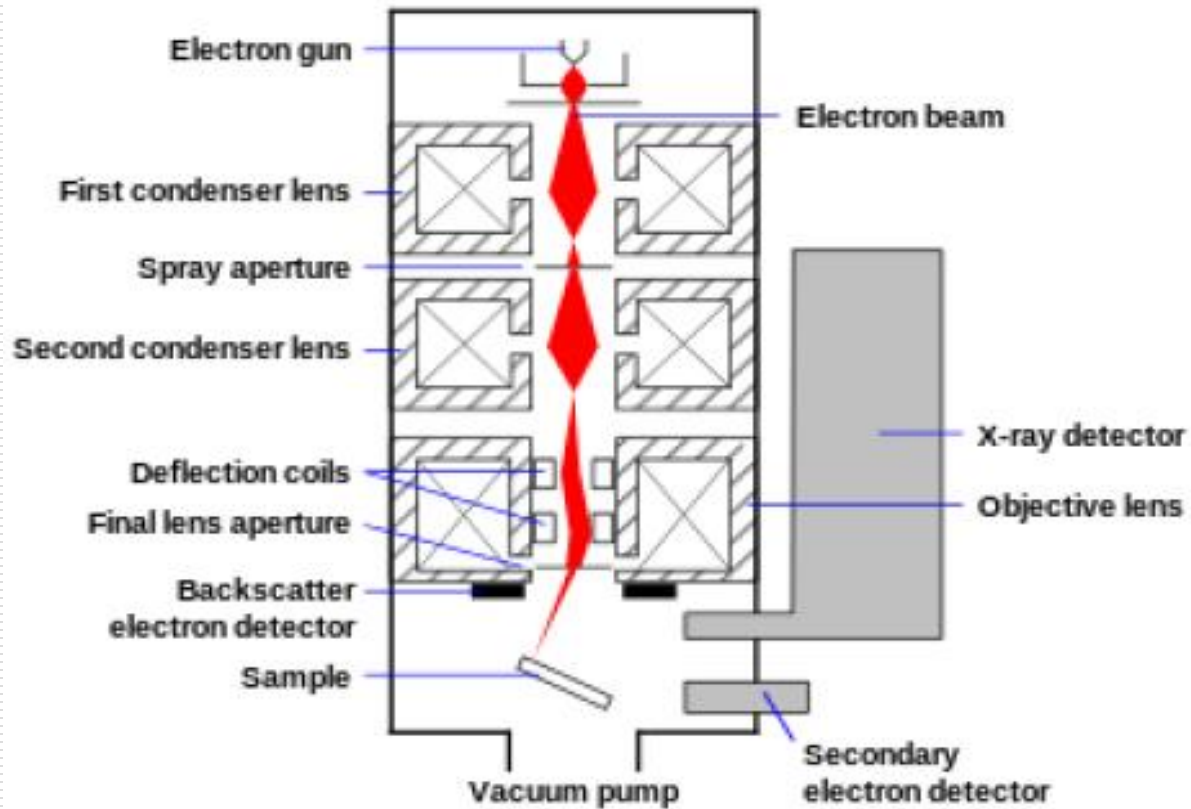
SEM

-
- A **scanning electron microscope (SEM)** is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons.
 - SEM can achieve resolution better than 1 nanometer.
 - Specimens can be observed in high vacuum in conventional SEM,
 - The most common SEM mode is detection of secondary electrons emitted by atoms excited by the electron beam.

SEM Principles

- The signals used by a scanning electron microscope to produce an image result from interactions of the electron beam with atoms at various depths within the sample.
- Various types of signals are produced including secondary electrons (SE), reflected or backscattered electrons.
- Secondary electron detectors are standard equipment in all SEMs.
- In secondary electron imaging, or SEI, the secondary electrons are emitted from very close to the specimen surface.

SEM



Schematic of an SEM

- In addition to topographical, morphological and compositional information, a Scanning Electron Microscope can detect and analyze surface fractures, provide information in microstructures, examine surface contaminations, reveal spatial variations in chemical compositions, provide qualitative chemical analyses and identify crystalline structures.
- SEMs can be as essential research tool in fields such as life science, biology, medical and forensic science, metallurgy.
- In addition, SEMs have practical industrial and technological applications such as semiconductor inspection, production line of miniscule products and assembly of microchips for computers.

SEM Advantages

- Advantages of a Scanning Electron Microscope include its wide-array of applications, the detailed three-dimensional and topographical imaging and the versatile information garnered from different detectors.
- SEMs are also easy to operate with the proper training and advances in computer technology and associated software make operation user-friendly.
- This instrument works fast, often completing analyses in less than five minutes. In addition, the technological advances in modern SEMs allow for the generation of data in digital form.
- Although all samples must be prepared before placed in the vacuum chamber, most SEM samples require minimal preparation actions.

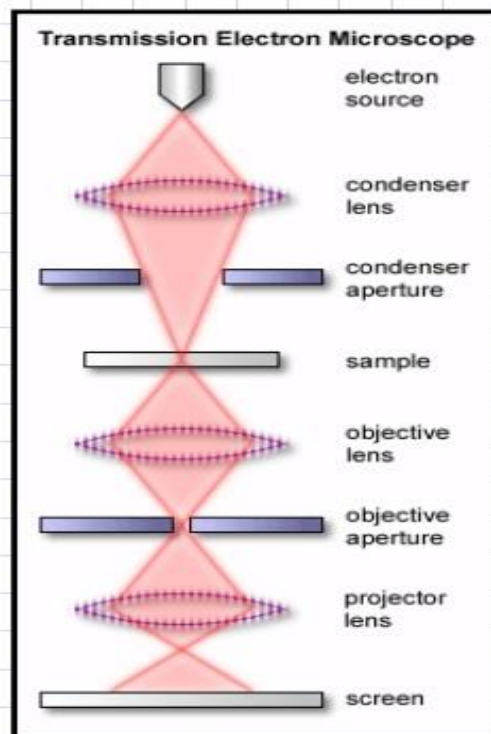
SEM Disadvantages

- The disadvantages of a Scanning Electron Microscope start with the size and cost.
- SEMs are expensive, large and must be housed in an area free of any possible electric, magnetic or vibration interference.
- Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.
- In addition, SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.
- Finally, SEMs carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.

Transmission electron microscopy (TEM)

- **Transmission electron microscopy (TEM, also sometimes conventional transmission electron microscopy or CTEM)**
- It is a microscopy technique in which a beam of electrons is transmitted through a specimen to form an image.
- The specimen is most often an ultrathin section or a suspension on a grid.
- An image is formed from the interaction of the electrons with the sample as the beam is transmitted through the specimen.
- The image is then magnified and focused onto an imaging device, such as a fluorescent screen, a layer of photographic film, or a sensor such as a charge-coupled device.

TEM



DESIGN OF TRANSMISSION ELECTRON MICROSCOPE

A simplified ray diagram of a TEM consists of an electron source, condenser lens with aperture, specimen, objective lens with aperture, projector lens and fluorescent screen.

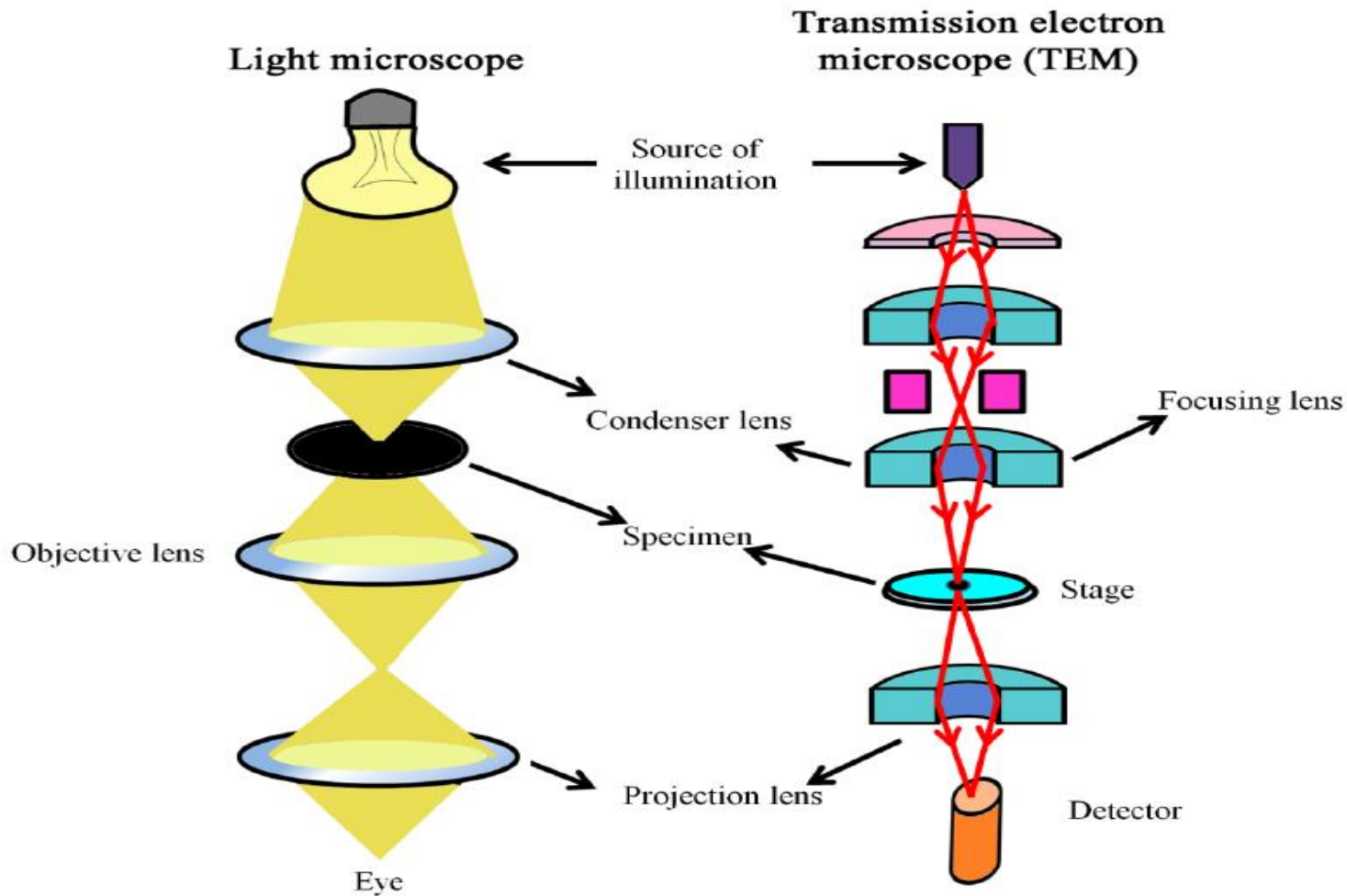


Figure: A simplified comparison of optics in a light microscope with that in a TEM.

TEM Components

1. Electron Source

- Produces high energy, large current, and high coherence electron beams necessary for generating diffraction patterns and high spatial resolution images

2. Condenser lenses

- Controls spot size and illumination area on sample (beam intensity)

3. Objective lens

- Images sample and is strongest lens in the system

4. Intermediate and projector lenses

- Controls magnification

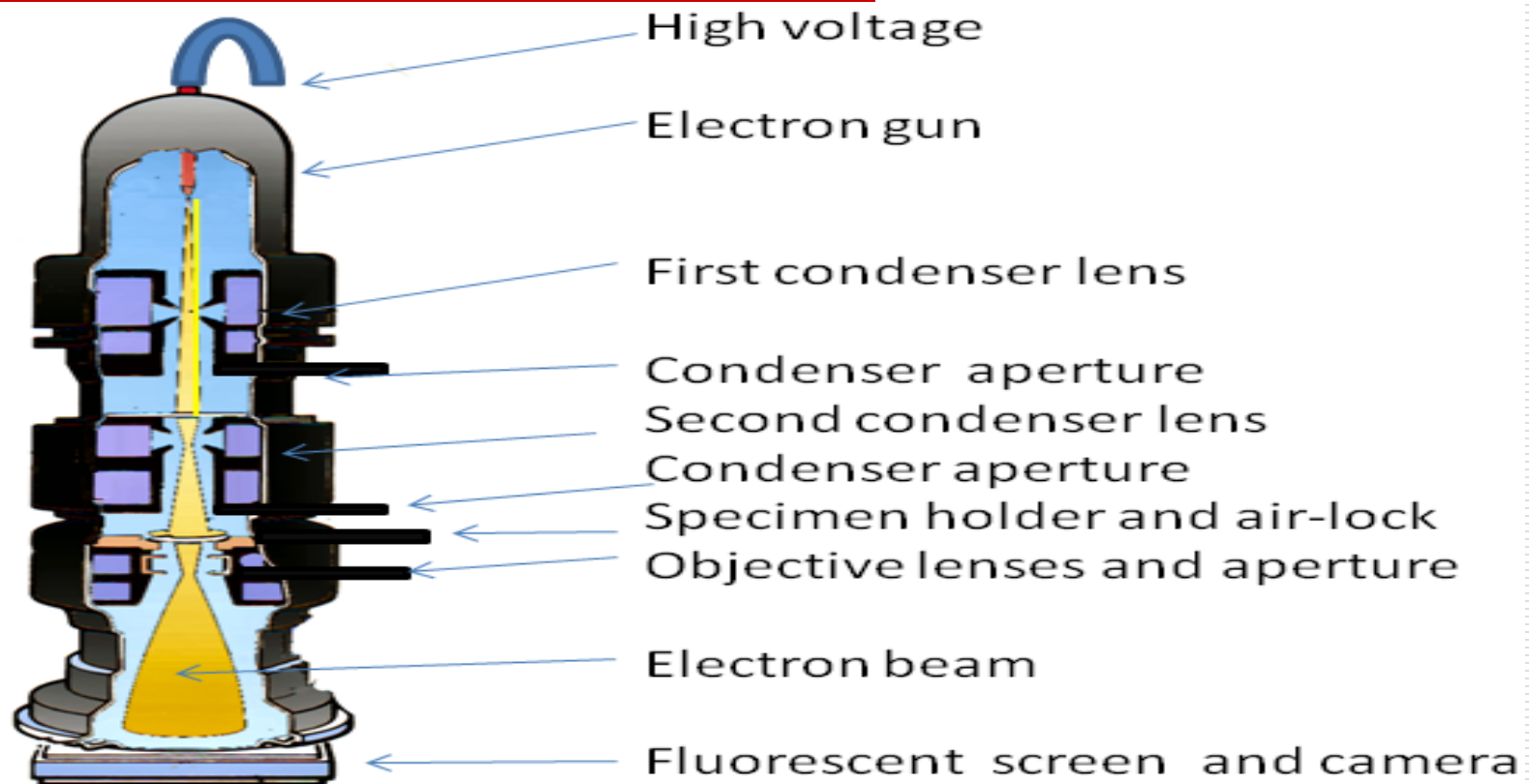
5. Detectors

- Various different configurations designed to collect secondary signals produced by the high-energy electron beam

TEM

- The original form of electron microscope, the transmission electron microscope (TEM) uses a high voltage electron beam to illuminate the specimen and create an image.
- The electron beam is produced by an electron gun, commonly fitted with a tungsten filament cathode as the electron source.
- The electron beam is accelerated by an anode with respect to the cathode, focused by electrostatic and electromagnetic lenses, and transmitted through the specimen that is in part transparent to electrons and in part scatters them out of the beam.

TEM



Transmission Electron Microscope

TEM Principle

- A Transmission Electron Microscope produces a high-resolution, black and white image from the interaction that takes place between prepared samples and energetic electrons in the vacuum chamber.
- Air needs to be pumped out of the vacuum chamber, creating a space where electrons are able to move.
- The electrons then pass through multiple electromagnetic lenses. These solenoids are tubes with coil wrapped around them.
- The beam passes through the solenoids, down the column, makes contact with the screen where the electrons are converted to light and form an image.

TEM Applications

- A Transmission Electron Microscope is ideal for a number of different fields such as life sciences, nanotechnology, medical, biological and material research, forensic analysis, and metallurgy as well as industry and education.
- The images allow researchers to view samples on a molecular level, making it possible to analyze structure and texture.
- TEMs can be used in semiconductor analysis and production and the manufacturing of computer and silicon chips.
- Technology companies use TEMs to identify flaws, fractures and damages to micro-sized objects; this data can help fix problems and/or help to make a more durable, efficient product.

Advantages of TEM

- A Transmission Electron Microscope is an impressive instrument with a number of advantages such as:
- TEMs offer the most powerful magnification, potentially over one million times or more
- TEMs have a wide-range of applications and can be utilized in a variety of different scientific, educational and industrial fields
- TEMs provide information on element and compound structure Images are high-quality and detailed
- TEMs are able to yield information of surface features, shape, size and structure.
- They are easy to operate with proper training.

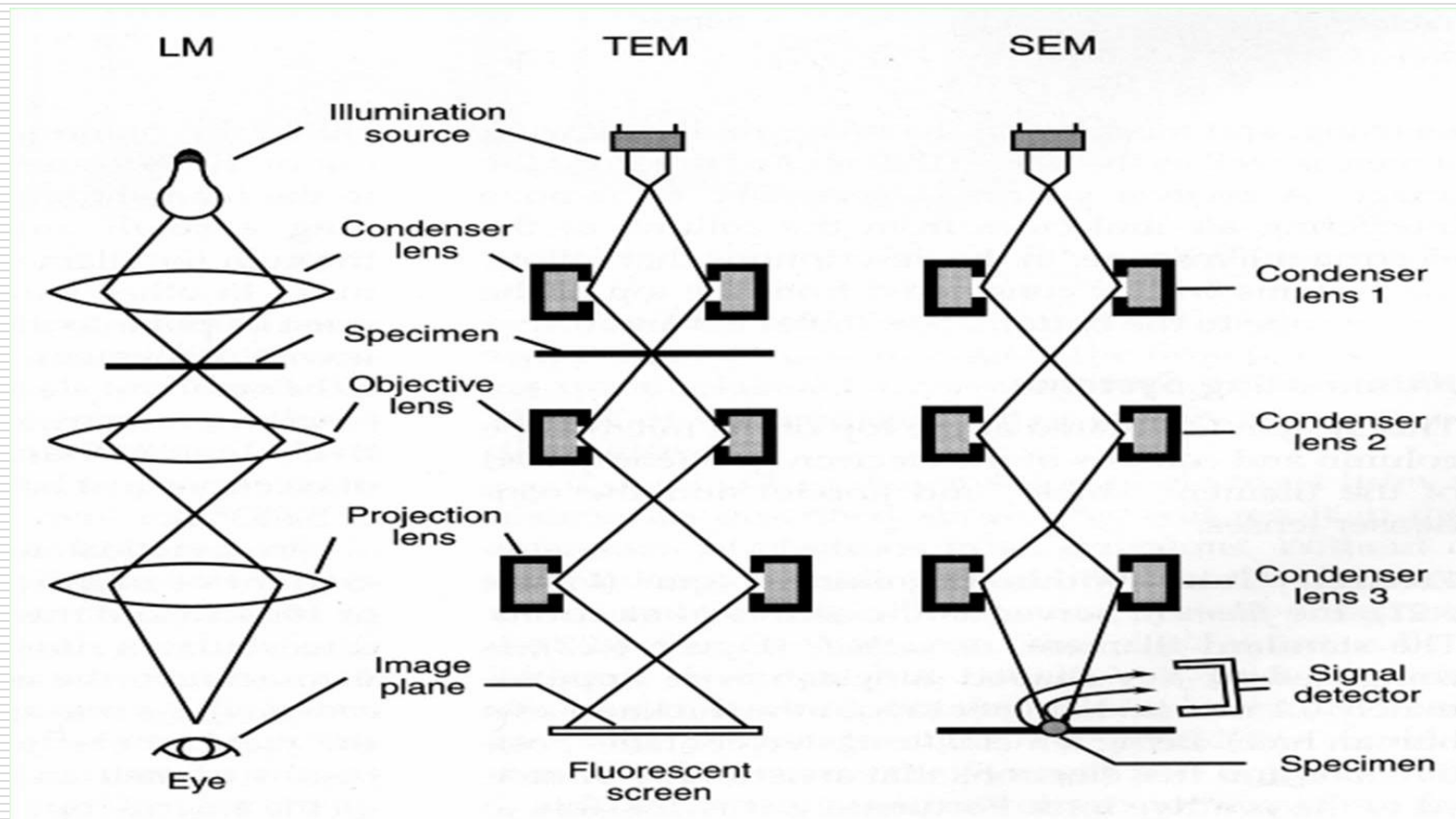
Disadvantages TEM

- TEMs are large and very expensive
- Laborious sample preparation
- Operation and analysis requires special training
- Samples are limited to those that are electron transparent, able to tolerate the vacuum chamber and small enough to fit in the chamber
- TEMs require special housing and maintenance

Difference Between SEM & TEM :

- SEM is based on scattered electrons while TEM is based on transmitted electrons.
- The sample in TEM has to be cut thinner whereas there is no such need with SEM sample.
- SEM allows for large amount of sample to be analyzed at a time whereas with TEM only small amount of sample can be analyzed at a time.
- SEM is used for surfaces, powders, polished & etched microstructures, IC chips, chemical segregation
- whereas TEM is used for imaging of dislocations, tiny precipitates, grain boundaries and other defect structures in solids
- TEM has much higher resolution than SEM.

Comparison of LM and TEM



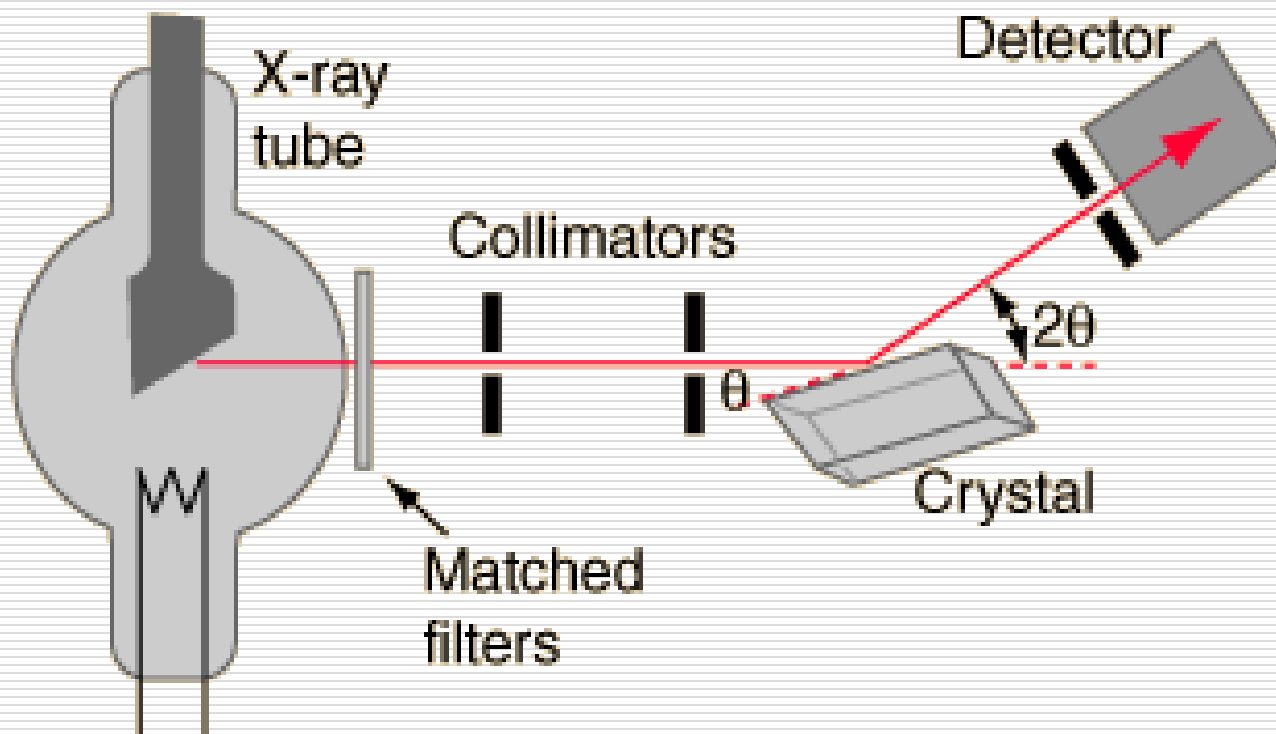
X-Ray Diffraction (XRD)

- It is a novel & non destructive method of chemical analysis and a variety of x –ray techniques are available in practice.
- “Every crystalline substance gives a pattern; the same substance always gives the same pattern; and in a mixture of substances each produces its pattern independently of the others”
- The X-ray diffraction pattern of a pure substance is, therefore, like a fingerprint of the substance. It is based on the scattering of x-rays by crystals.

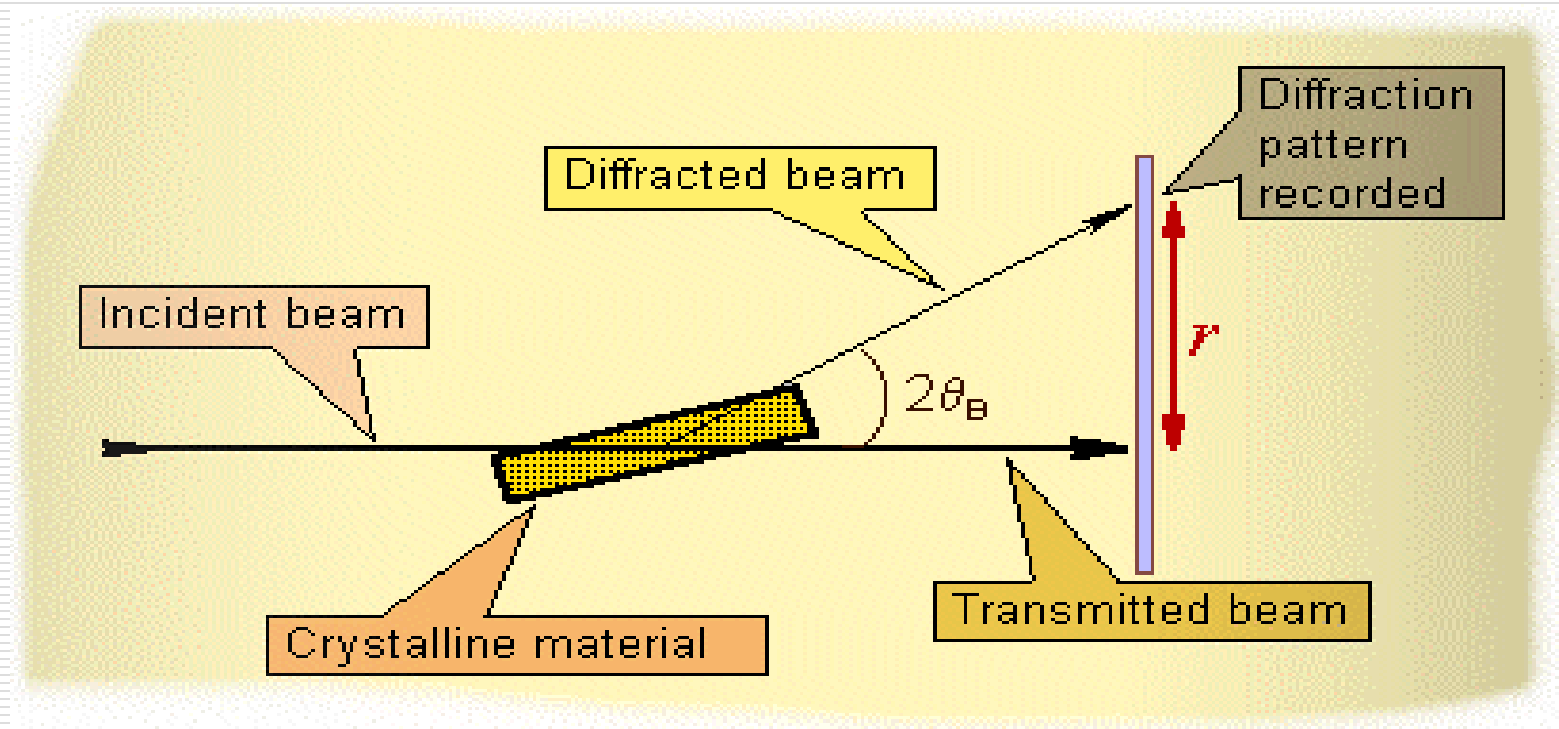
XRD

- X - ray diffraction is one of the earliest methods for studying the structure of solids. In the process of diffraction, electromagnetic waves of a given frequency but different phases interact to produce constructive interference (bright spots on the film exposed to the light) and destructive interference (dark spots). By a careful analysis of the diffraction patterns, very accurate values of the lattice parameters (unit cell dimensions) can be inferred.
- The atomic planes of a crystal cause an incident beam of Xrays to interfere with one another as they leave the crystal. The phenomenon is called X-ray diffraction.

XRD



XRD



XRD Principles

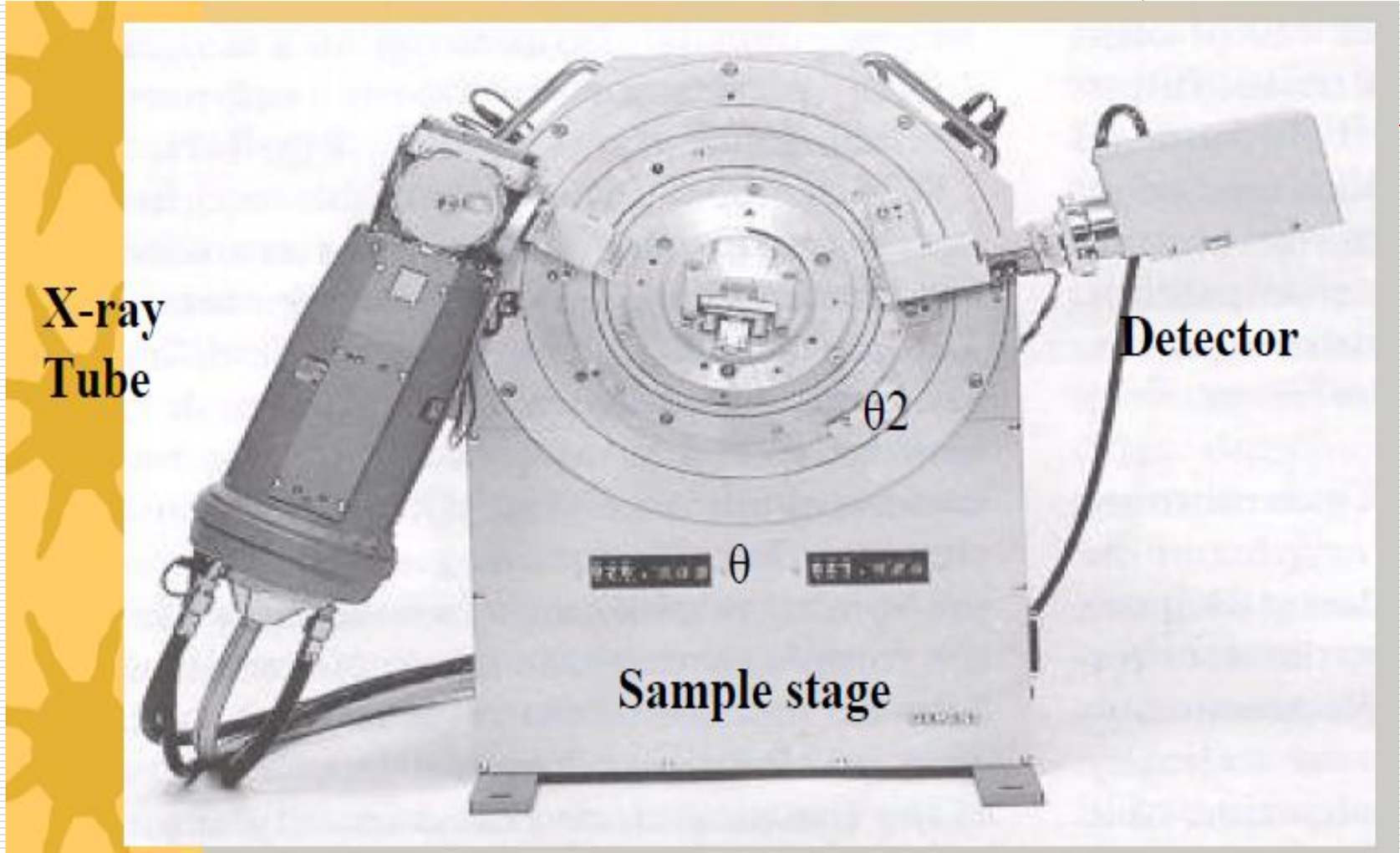
- X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample.
- These X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample.
- The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda = 2d \sin \theta$).
- This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample.
- These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θ angles.
- Conversion of the diffraction peaks to d-spacings allows identification of the mineral because each mineral has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

XRD

- Bragg's law:

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

- Here d is the spacing between diffracting planes, θ is the incident angle, n is any integer, and λ is the wavelength of the beam.
- Thus, X-ray diffraction results from an electromagnetic wave (the X-ray) impinging on a regular array of scatterers (the repeating arrangement of atoms within the crystal).



A Modern Automated X-ray Diffractometer

Applications of XRD

- XRD is a non destructive technique to identify crystalline phases and orientation
- To determine structural properties:
- To measure thickness of thin films and multi-layers.
- To determine atomic arrangement
- Measure the average spacing's between layers or rows of atoms
- Determine the orientation of a single crystal or grain
- Find the crystal structure of an unknown material
- Measure the size, shape and internal stress of small crystalline regions

Advantages of XRD

- Powerful and rapid (< 20 min) technique for identification of an unknown mineral
- In most cases, it provides an unambiguous mineral determination
- Minimal sample preparation is required
- XRD units are widely available
- Data interpretation is relatively straight forward

THANK YOU