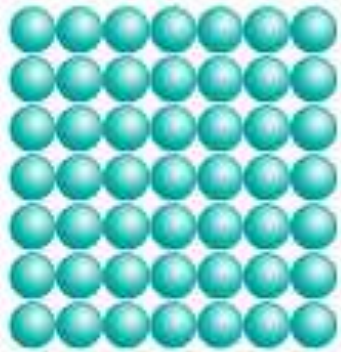
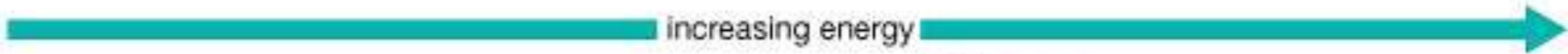


Module II

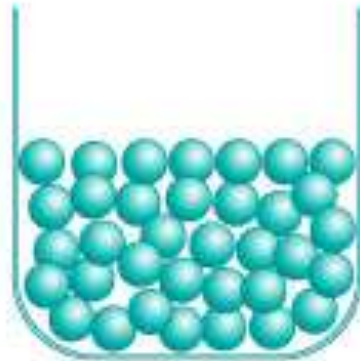
Solidification of crystalline materials

Physical states



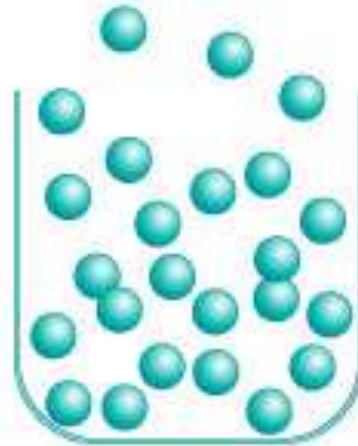
Solid

The molecules that make up a solid are arranged in regular, repeating patterns. They are held firmly in place but can vibrate within a limited area.



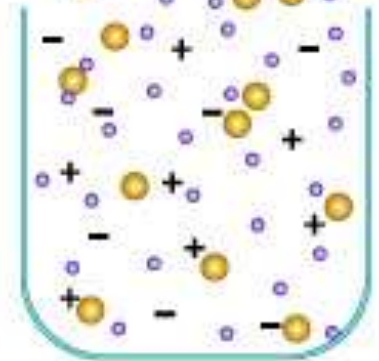
Liquid

The molecules that make up a liquid flow easily around one another. They are kept from flying apart by attractive forces between them. Liquids assume the shape of their containers.



Gas

The molecules that make up a gas fly in all directions at great speeds. They are so far apart that the attractive forces between them are insignificant.



Plasma

At the very high temperatures of stars, atoms lose their electrons. The mixture of electrons and nuclei that results is the plasma state of matter.

Solidification of crystalline materials

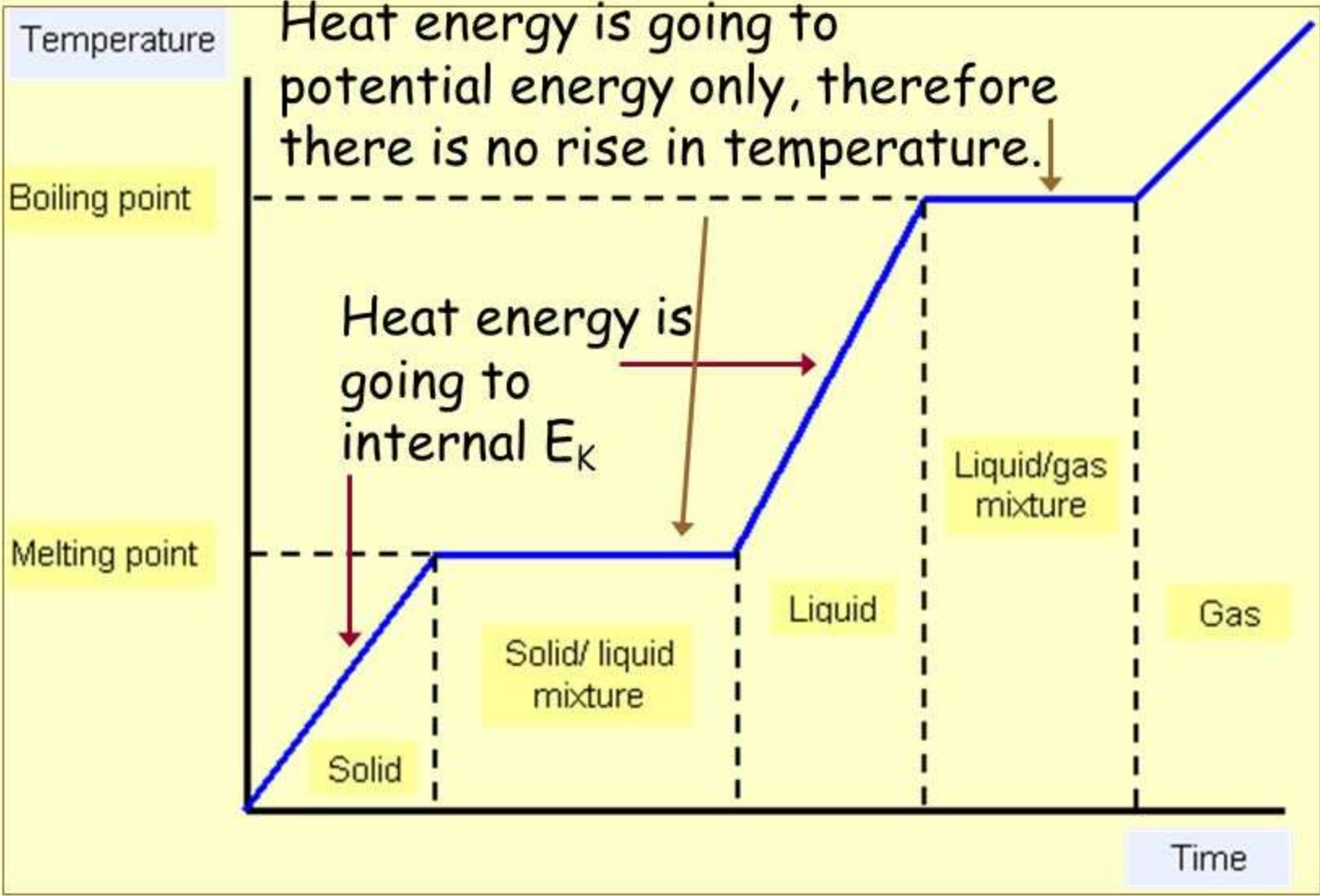
- Solidification (crystallization) – transition from liquid state to solid state
- In liquid state atoms will be in constant motion – high KE due to high temperature
- Atoms don't have any definite arrangement
- When temp: brought down some atoms group together
- These groups are not permanent, break up continually and regroup at other points
- Life determined by temp: and size of group
- At sufficiently decreased temp: atomic movement also decreases
- This lengthens life of group and promotes formation of more groups –

Embryos (more like solid)...

Undercooling

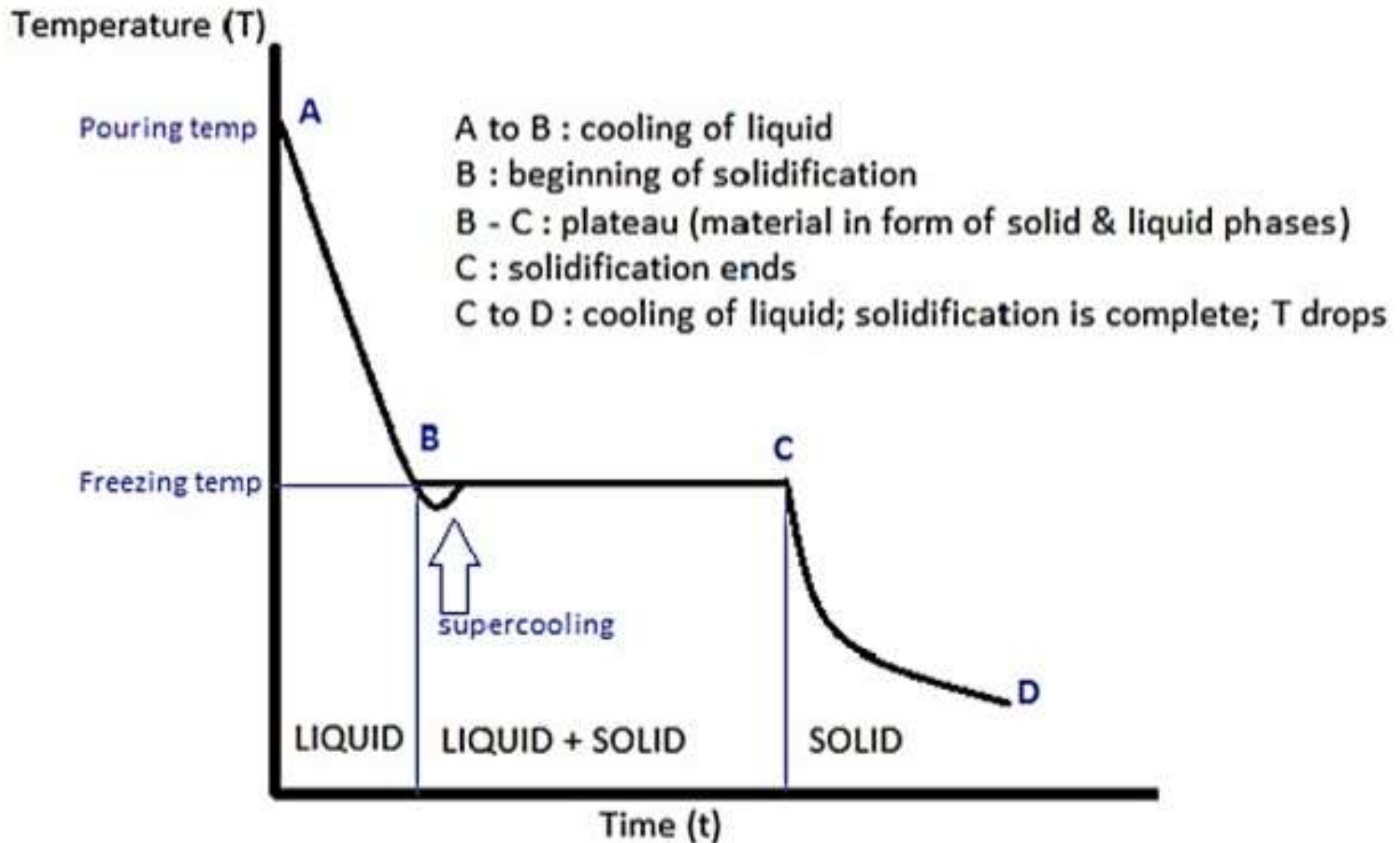
- As temperature of liquid metal drops, a stage is reached where embryos and surrounding liquid co-exist – solidification point OR freezing point
- at this point both liquid and solid states are at the same temperature – same KE for respective atoms
- but significant difference in PE – ie, in solid, low PE
- Thus, solidification occurs with release of energy
- The difference in PE b/w liquid and solid states – latent heat (heat of fusion OR heat of solidification)

Change of state (Melting and boiling)



- At freezing point energy is required to establish a surface b/w the solid and the liquid
- In pure materials at freezing point, insufficient energy is released by heat of fusion to create a stable boundary surface
- Hence some undercooling (supercooling) is necessary to form a stable solid region

Undercooling (or) Supercooling in pure metals



Hari Prasad-Assistant Professor
Diagram showing cooling curve of a pure metal

Nucleation

- Homogeneous
- heterogeneous

Homogeneous Nucleation

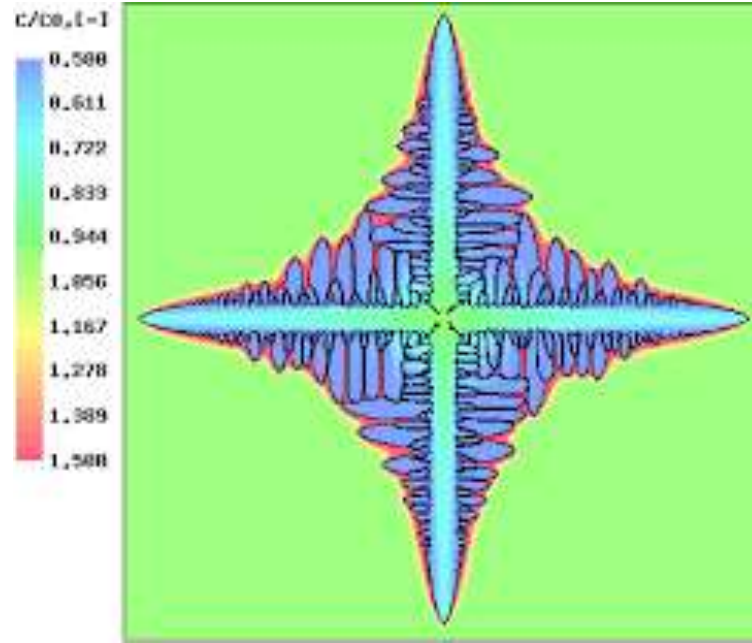
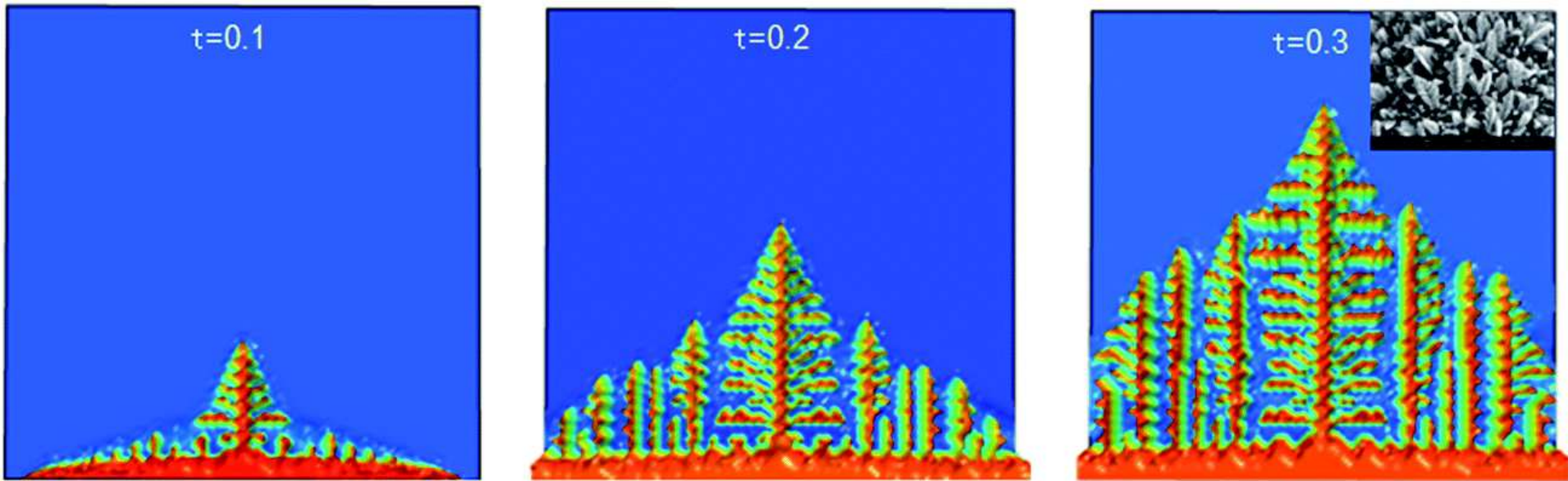
- At sufficiently low temperatures, nucleation occurs by grouping of a few atoms from the liquid
- Nucleation of this type originated with the support of undercooling – Homogeneous
- Occurs uniformly throughout the liquid
- Nucleus grows further by adding more and more atoms to it which leads to small solid region → Nuclei

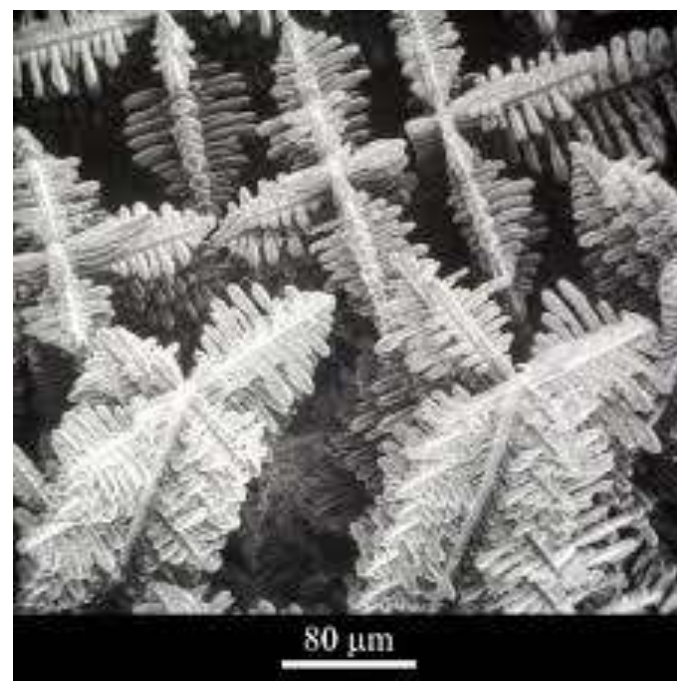
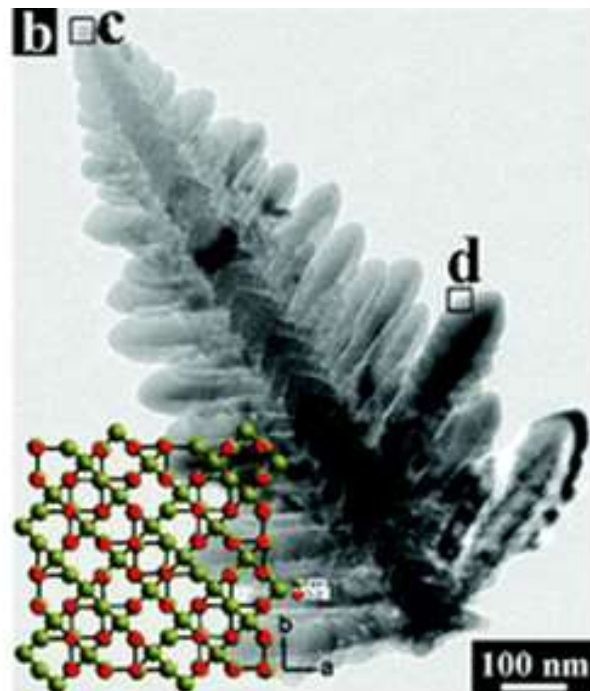
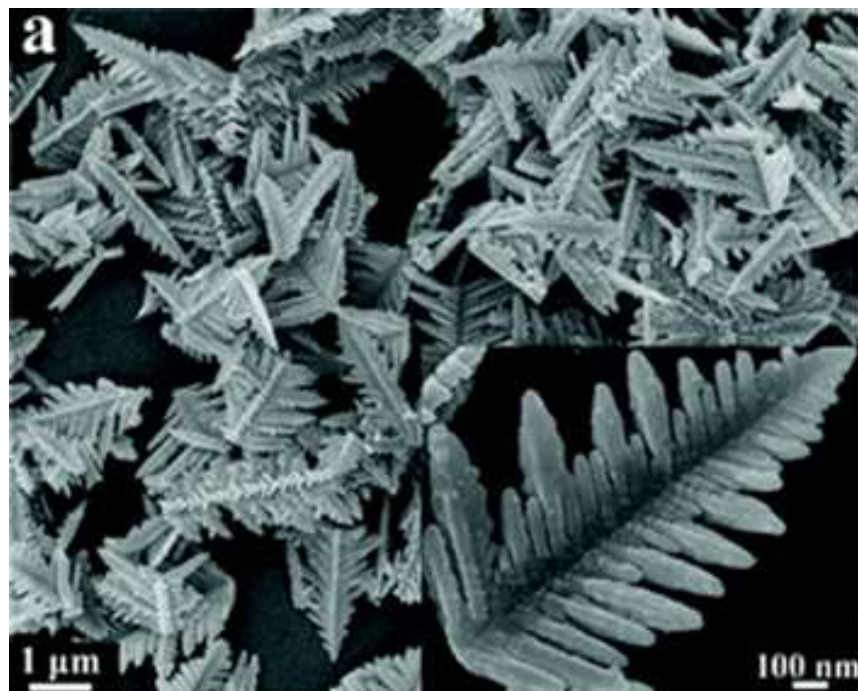
- Once nuclei is formed heat of fusion is released and temperature will be raised to the freezing point again
- More atoms start to freeze, theses may attach to already existing nuclei or form new nuclei of their own
- Thus the process of solidification continues at constant temperature till entire liquid is transformed to solid

Heterogeneous Nucleation

- Nucleation can be facilitated by the presence of impurity atoms, grain refiners or imperfections
- This type of preferential nucleation -
Heterogeneous Nucleation
- A much lower level of undercooling is sufficient
- Even the wall of the container can promote heterogeneous nucleation
- Addition of grain refiner produce fine grains.....

Dendritic Growth



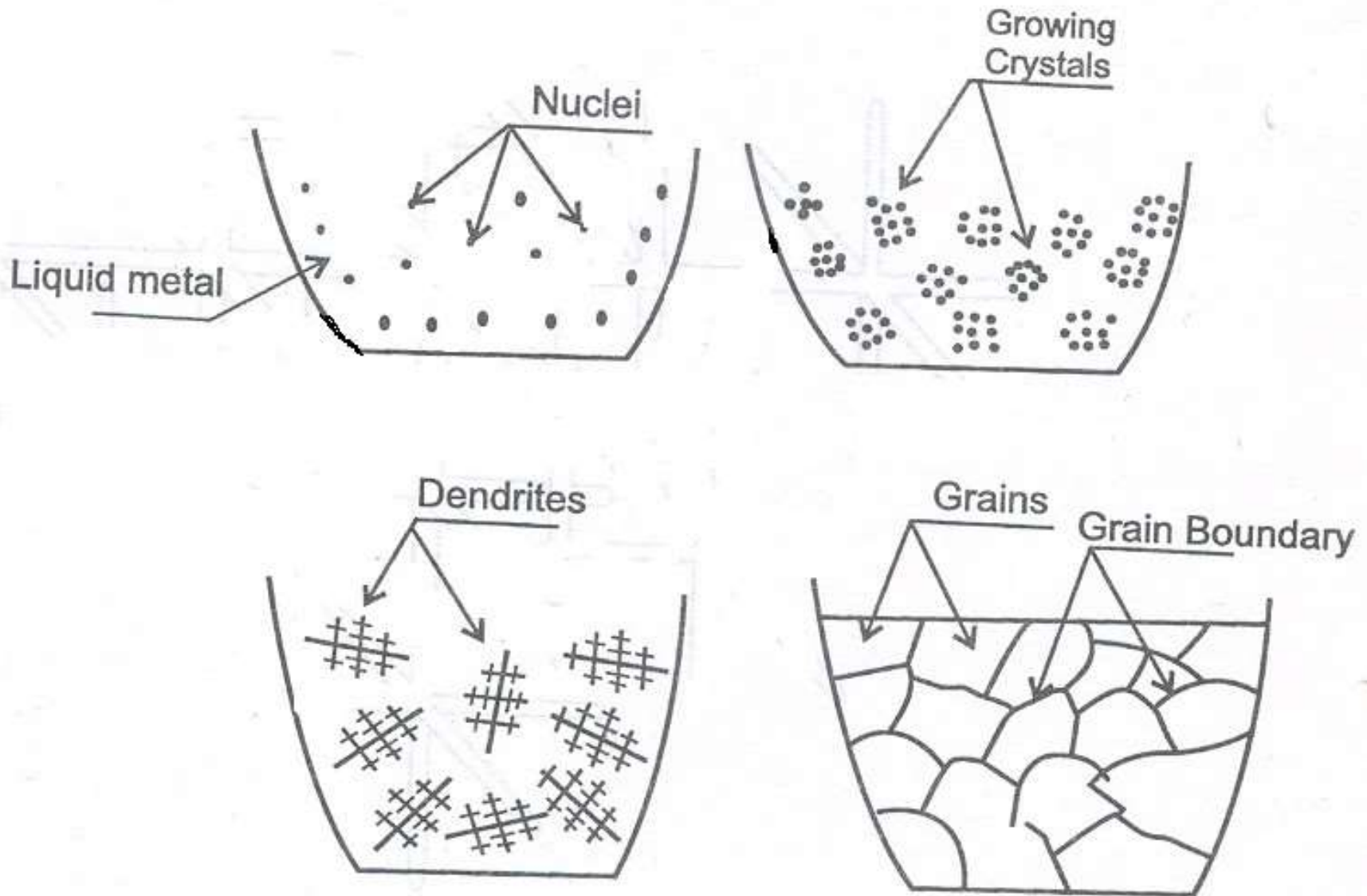


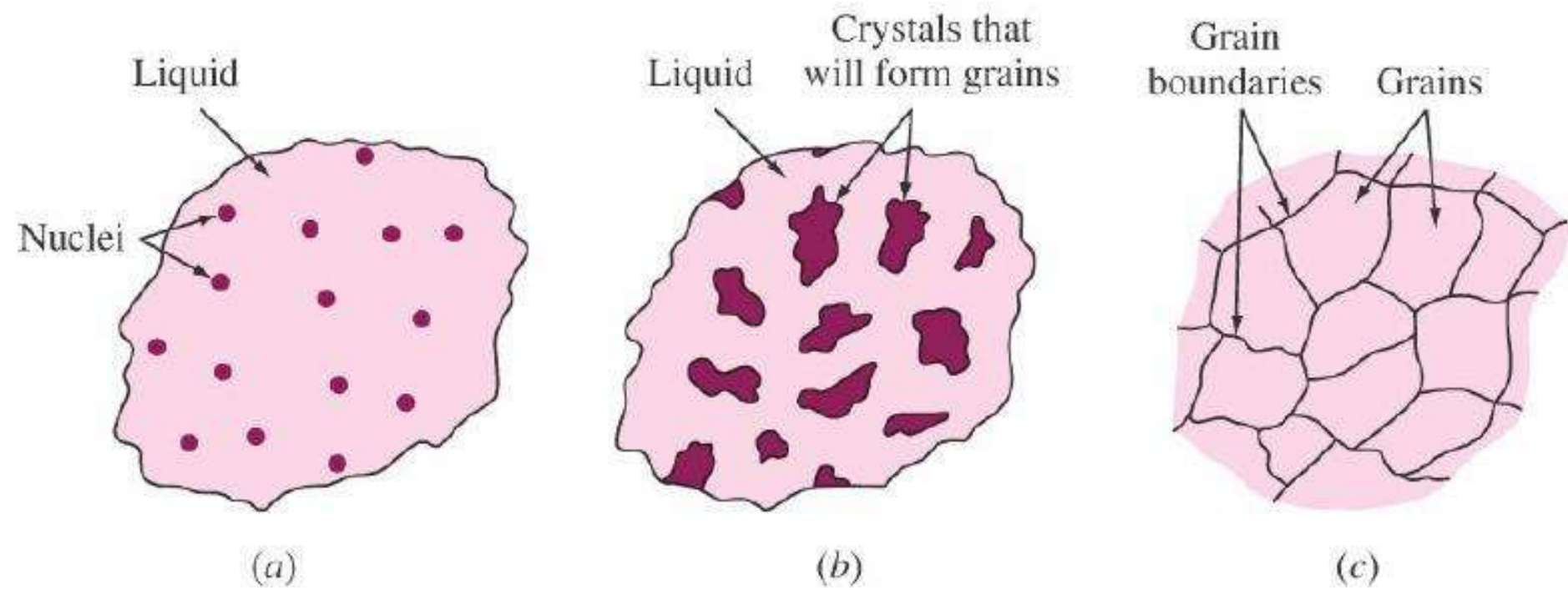
Dendritic Growth

- During solidification, each nucleus grows by attracting atoms from the liquid
- Crystal growth continues in 3-dimensions
- This results in a characteristic tree like structure
 - Dendrite
- In each crystal dendrites grow in different directions
- Dendrites can be considered as crystal skeleton from which arms begin to grow in all directions

- From the primary arms, secondary, tertiary etc arms starts to grow similar to branches of a tree, leading to the formation of an elongated skeleton
- The dendritic arm continue to grow and thicken at the same time until the space b/w them will be filled with solid
- In pure material, no evidence of dendritic growth left when solidification is complete, since all atoms are identical
- If the material is impure dissolved impurities will remain in the spaces b/w dendrites

Grains

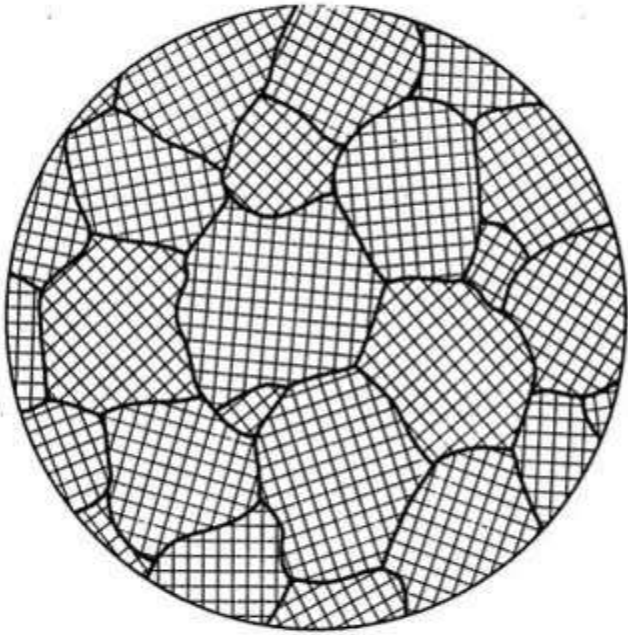




Grains

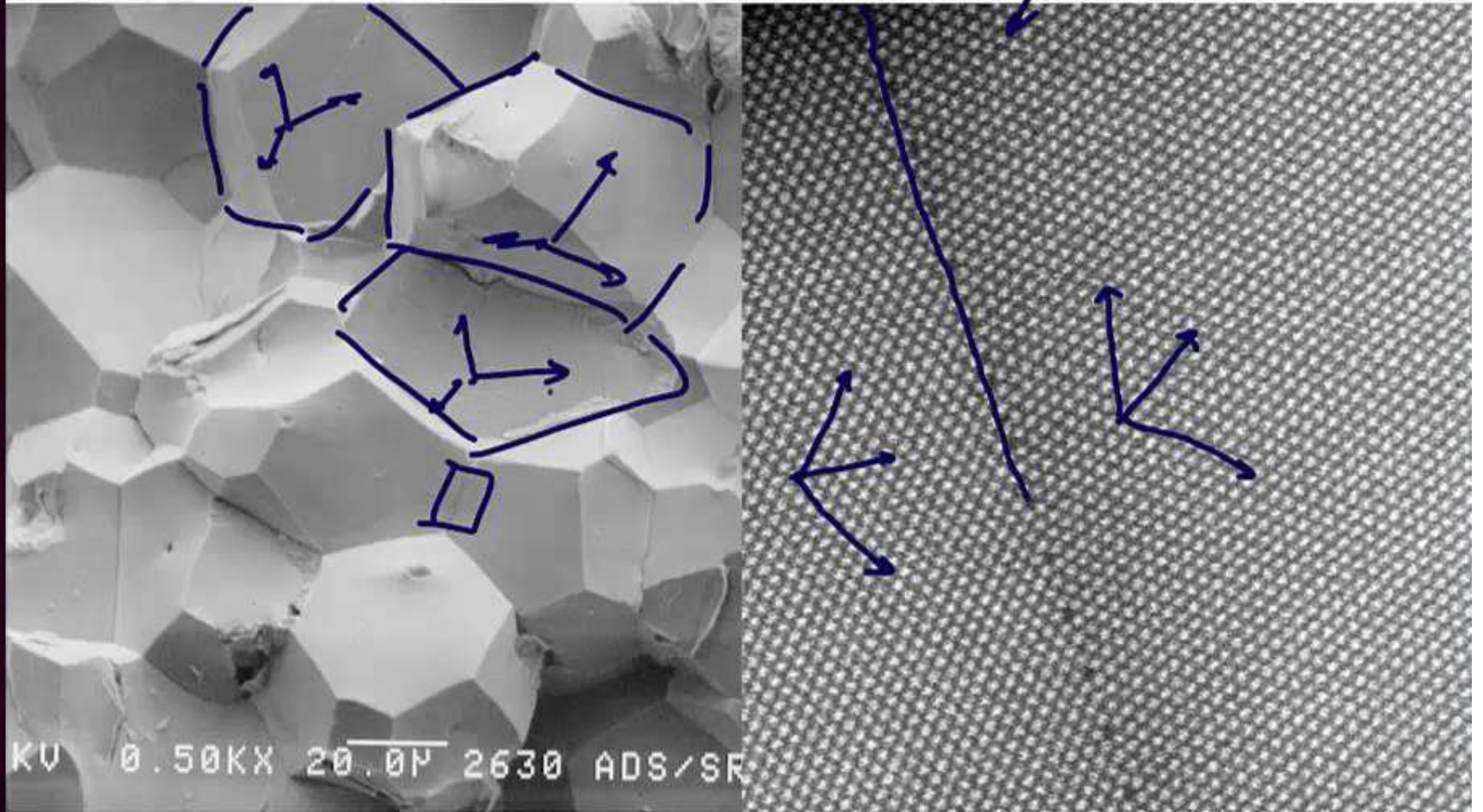
- A grain is a portion of the material within which the atomic arrangement is nearly identical
- But the orientation of atomic arrangement may be different for adjoining grains
- The area along which grains meet – grain boundary...

Grains



Brass Grains

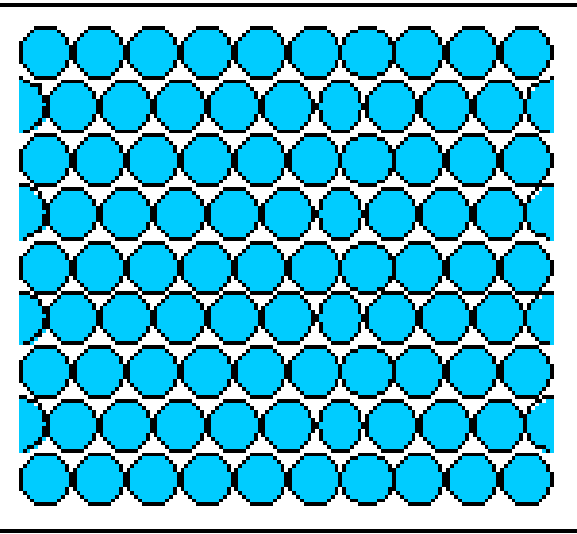
GRAN BOUNDARY



Single crystal and poly crystals

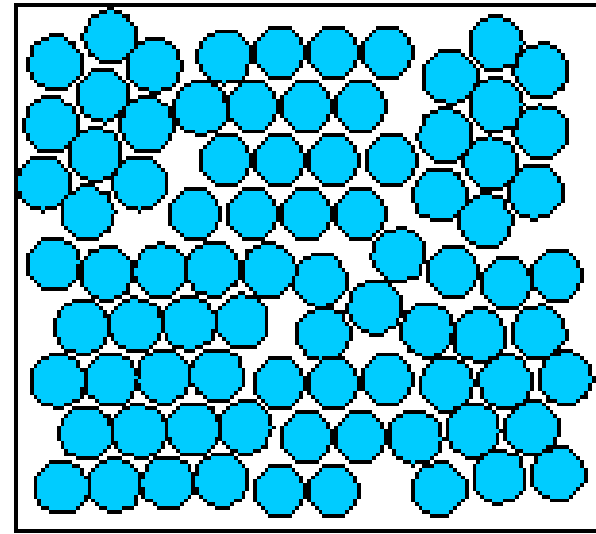
- The no: of grains formed during solidification depends on no: of nuclei formed
- If all nucleation sites except one are suppressed, the liquid solidifies in to a single grain – single crystal material
- These show excellent high temperature properties – absence of grain boundaries

- When a liquid is normally cooled under equilibrium condition, there will be large no of nucleation sites hence solidifies in to large no of small grains – polycrystalline
- Although these grains look alike, the crystallographic orientations are different
- The size and shape of the grains in a solid material constitute its microstructure



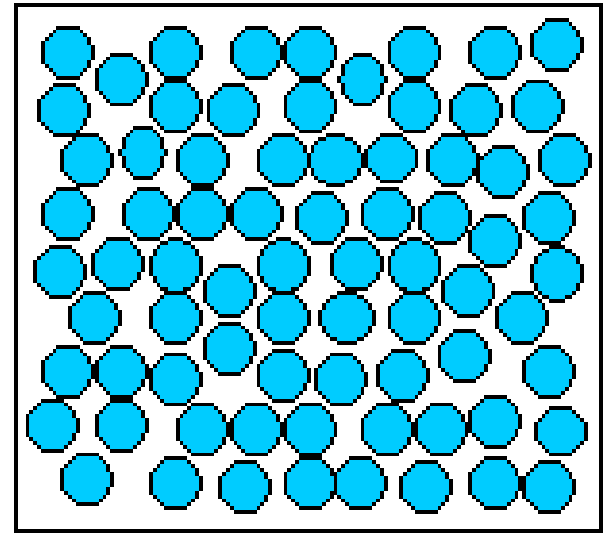
Single crystal

Periodic across the whole volume.



Polycrystal

Periodic across each grain.



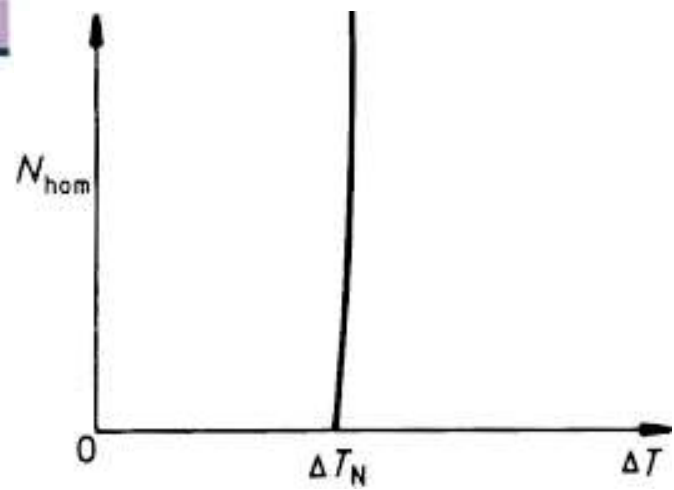
Amorphous solid

Not periodic.

Grain size

- Size and shape of grains are related to the conditions during grain growth
- Cooling rate of molten metal affects the size of crystals formed
- Slow cooling leads to small degree of under cooling – forms relatively few nuclei – large grain size
- Rapid cooling – high degree of under cooling – large no: of nuclei – small grain size

Material	Freezing Temperature (T_m)	Heat of Fusion (ΔH_f)	Solid-Liquid Interfacial Energy (σ_{sl})	Typical Undercooling for Homogeneous Nucleation (ΔT)
	(°C)	(J/cm ³)	(J/cm ²)	(°C)
Ga	30	488	56×10^{-7}	76
Bi	271	543	54×10^{-7}	90
Pb	327	237	33×10^{-7}	80
Ag	962	965	126×10^{-7}	250
Cu	1085	1628	177×10^{-7}	236
Ni	1453	2756	255×10^{-7}	480
Fe	1538	1737	204×10^{-7}	420
NaCl	801			169
CsCl	645			152
H ₂ O	0			40



- Control of nucleation process to control material properties
- Thermo mechanical treatments – combination of controlled deformation and thermal treatments
- Gives a microstructure with a preferred orientation of grains and tailored material properties

Grain size determination

- Grain size and no: of grains are quantified using standards put forward by ASTM
- Grain size is specified by an index called ASTM grain size number
- A specimen is prepared to reveal the grain structure, and photographed at a magnification of 100X
- This is compared with standard comparison charts having different grain sizes
- Grain size is expressed as Grain size number that matches Grain size of the specimen

Table 2.7. ASTM Grain Size Numbers

<i>ASTM Grain Size Number</i> <i>N</i>	<i>Average Number of grains per square inch. at 100 X</i> <i>n</i>	<i>Average Diameter of Grain (Assumed) as sphere at 1 X</i> <i>mm</i>	<i>Grains per millimeter square at 1 X</i>
00	1/4	0.51	3.9
0	1/2	0.36	7.8
1	1	0.25	15.5
2	2	0.18	31.0
3	4	0.125	62.0
4	8	0.090	124.0
5	16	0.065	248.0
6	32	0.045	496.0
7	64	0.032	992.0
8	128	0.022	1980.0
9	256	0.016	3970.0
10	512	0.011	7940.0
11	1024	0.008	15870.0
12	2048	0.006	31,700.0

- If n = Grain size number
 N = Avg no: of grains / sq. inch at 100 X

$$N = 2^{(n-1)}$$

- A large ASTM grain size number indicates large no: of grains of a fine grain size – high strength

Effect of grain size on material properties

- In a fine grained material more grain boundaries
- Grain boundaries are obstructions to movement of dislocations
- Dislocations pile up near grain boundaries
- More stress needed to deform – ie, more strength and hardness
- Poor ductility

Grain shape

1. Fine grains
2. Columnar grains
3. Equi-axed grains

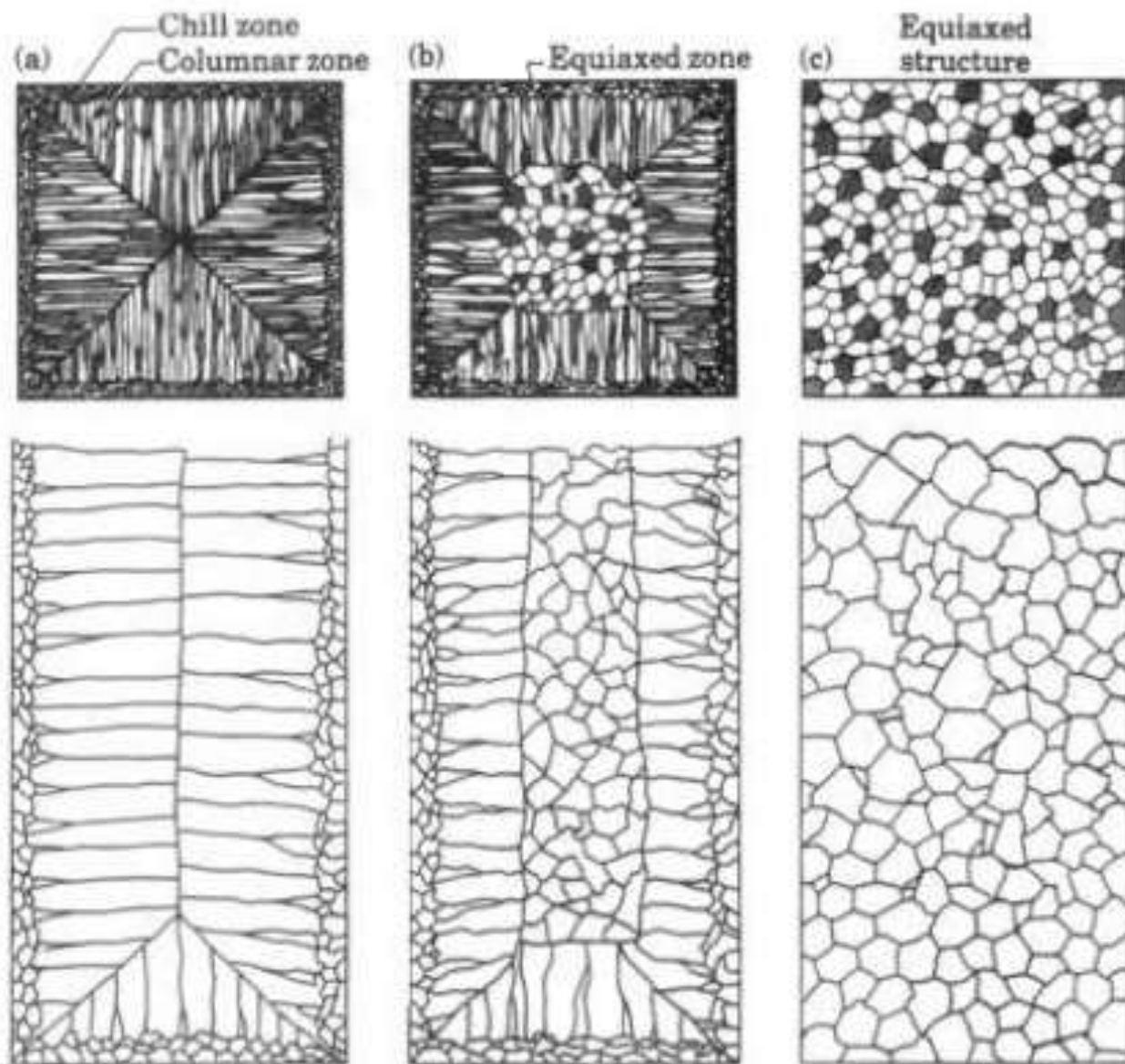
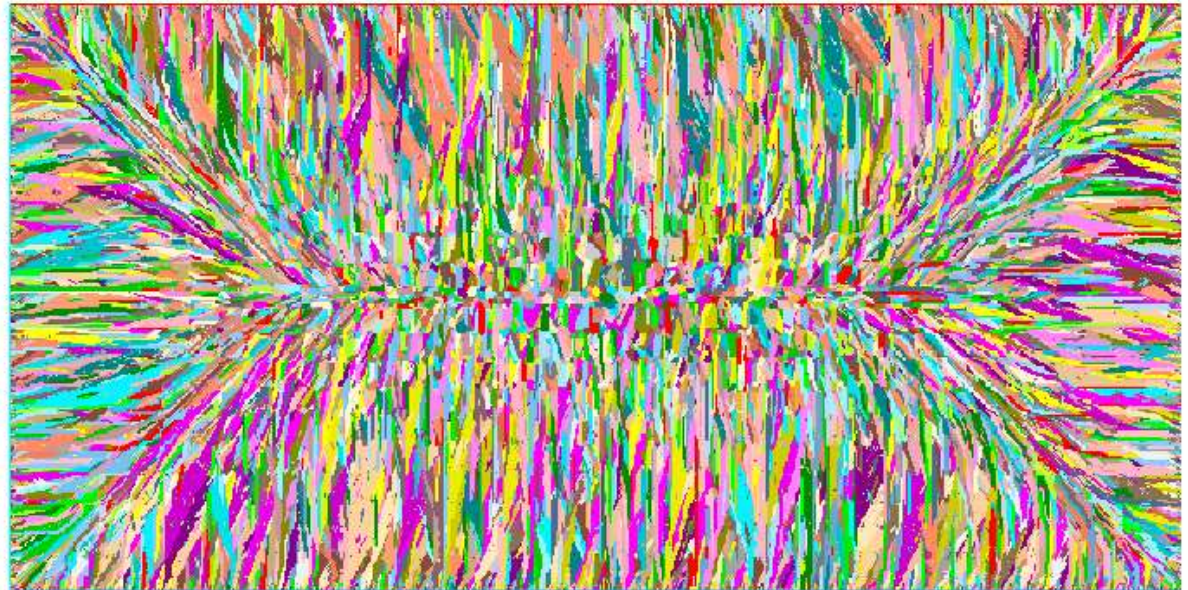
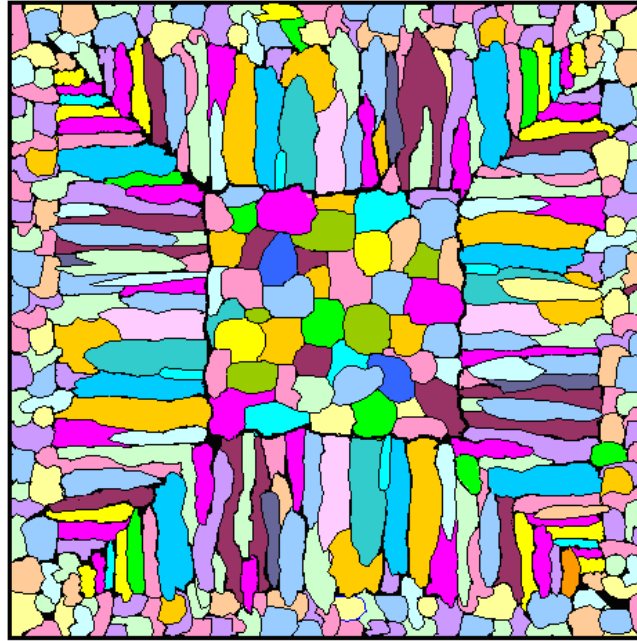


FIGURE 5.8 Schematic illustration of three cast structures of metals solidified in a square mold: (a) pure metals; (b) solid-solution alloys; and (c) structure obtained by heterogeneous nucleation of grains using nucleating agents. *Source: G. W. Form, J. F. Wallace, J. L. Walker, and A. Cibula.*



Grain Boundary

- Grain boundary is a region of disturbed lattice
- On moving from one grain to another the crystallographic orientation changes abruptly
- Misalignment can be quantified by the angle b/w crystallographic orientations
- Up to 10° - low angle grain boundary
- 10° to 30° - High angle grain boundary

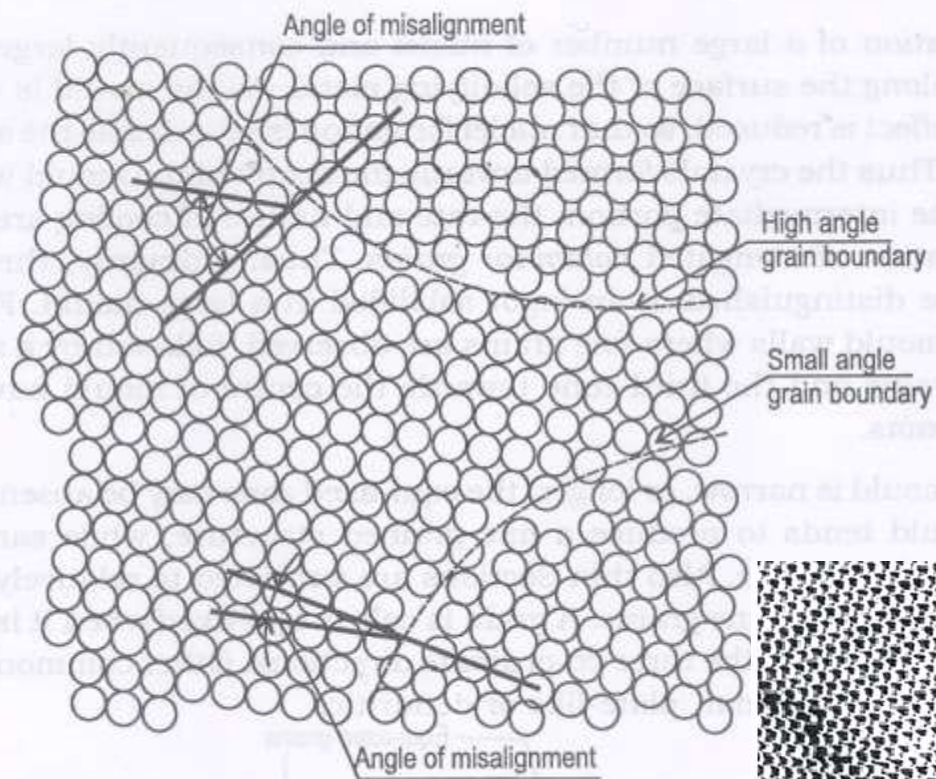
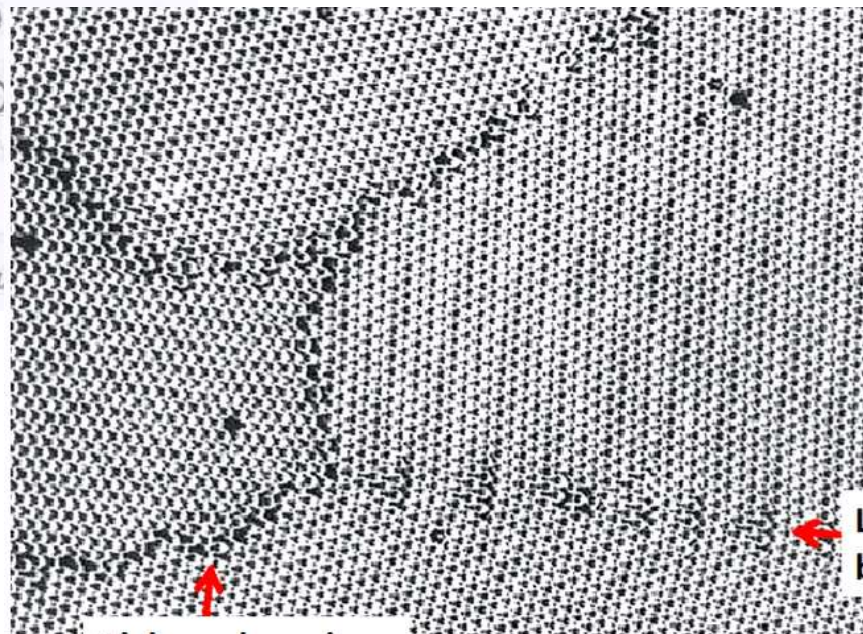


Figure 2.14 *Low and high-angle grain bo*

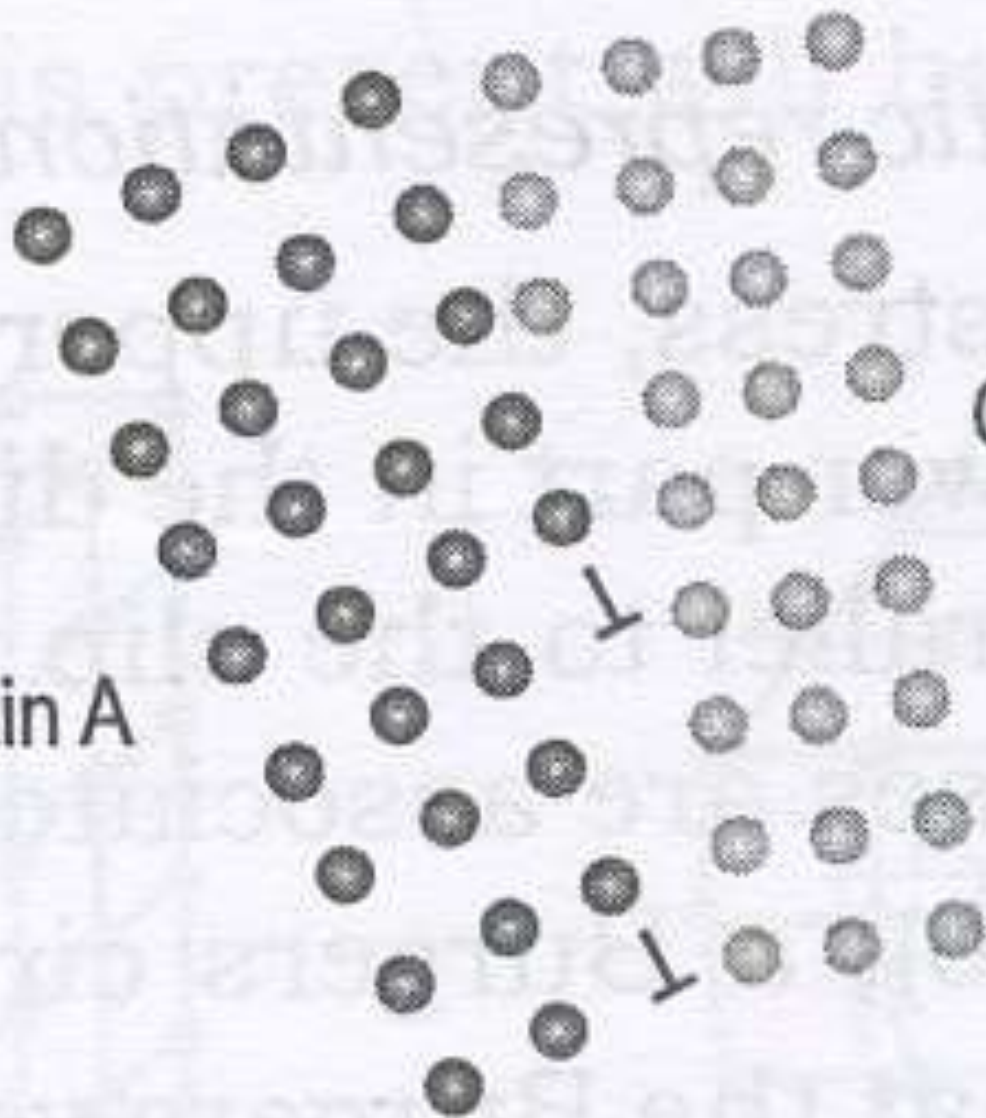


**High-angle grain
boundary**

**Low-angle grain
boundary**

Grain A

Grain B



Hall-Petch Equation

- A general relationship b/w yield stress and grain size of a material

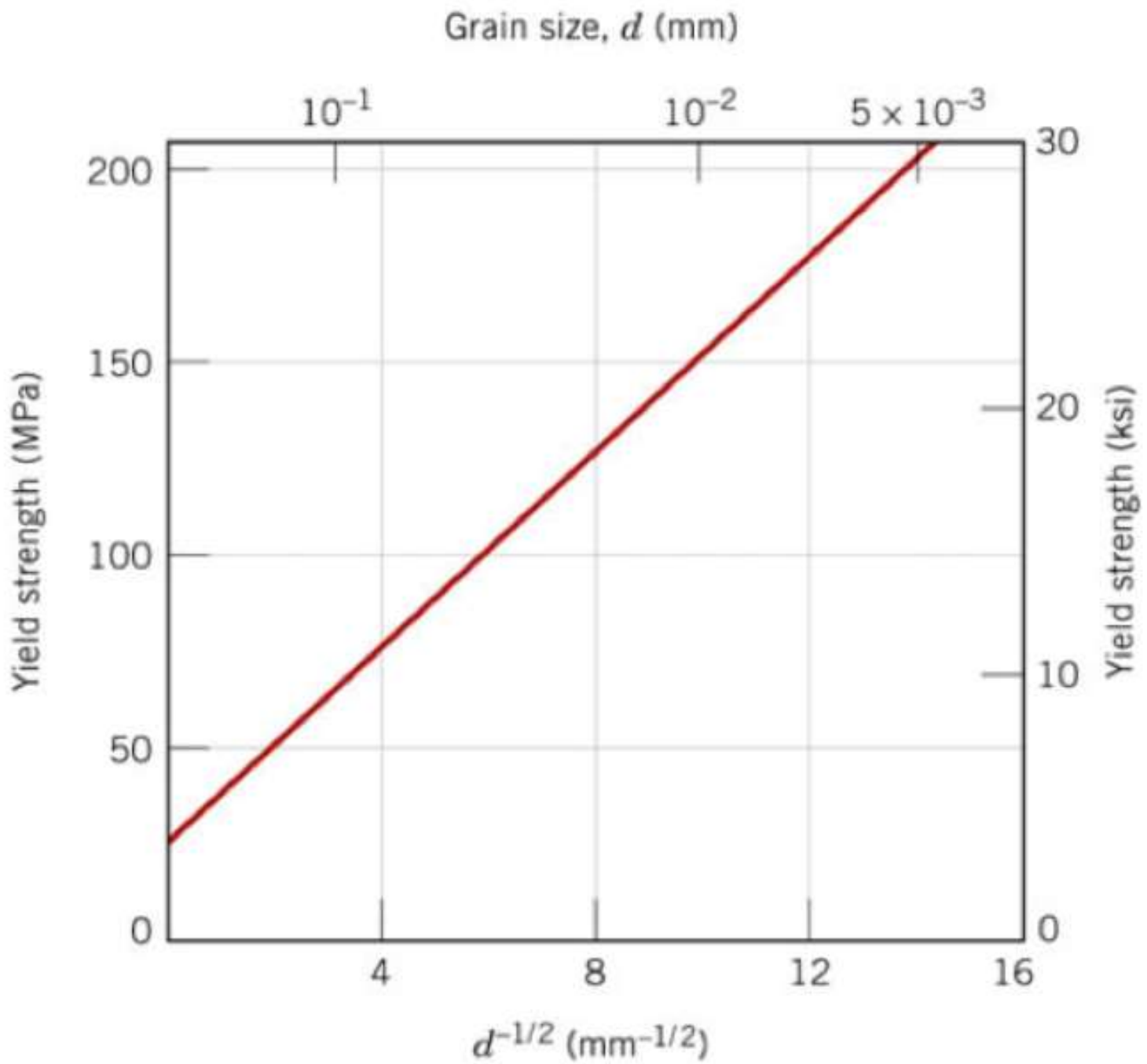
$$\sigma_0 = \sigma_i + (k/\sqrt{D})$$

σ_0 = yield strength

D = average diameter of grains

σ_i and k are material constants

- Not valid for both very large and extremely fine grain materials



Imperfections in solids

- When the repeated arrangement of atoms is perfect and extends throughout the material without any interruptions – single crystal
- Single crystals exists in nature; rarely
- Can be grown artificially
- All crystalline solids are made of many small crystals known as grains – Polycrystalline
- Individual grains may not be perfect in the arrangement of atoms in the crystal lattice
- Many interruptions may exist – *crystal imperfections* or *crystal defects*

Types of Imperfections

- In actual cases regular arrangement of atoms are disrupted – crystal imperfections
- Volume wise these disruptions may be as small as 0.01% of total volume
- Crucial in determining many of the mechanical, chemical and electrical properties (structure sensitive)
- Properties like density, colour, elasticity etc. do not depend on the structure of materials (structure insensitive)

Classification based on the geometry

1. Point defects or zero dimensional defects
 - a) vacancy
 - b) self interstitial or interstitialcy
 - c) substitutional impurity
 - d) interstitial impurity
 - e) Frenkel defect
 - f) Schottky defect

2. Line defects or 1-D defects
 - a) Edge dislocation
 - b) Screw dislocation

3. Surface defects or 2-D defects

- a) Grain boundaries
- b) Tilt boundaries
- c) Twin boundaries
- d) Stacking fault

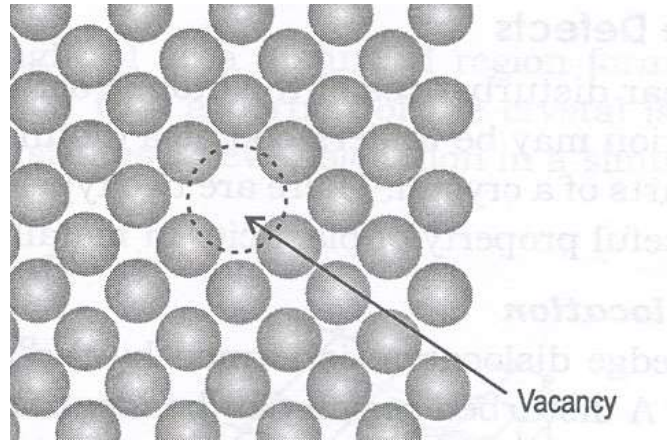
4. Volume defects or 3D defects

- Voids
- Cracks
- Pores
- Precipitate

Point Defects

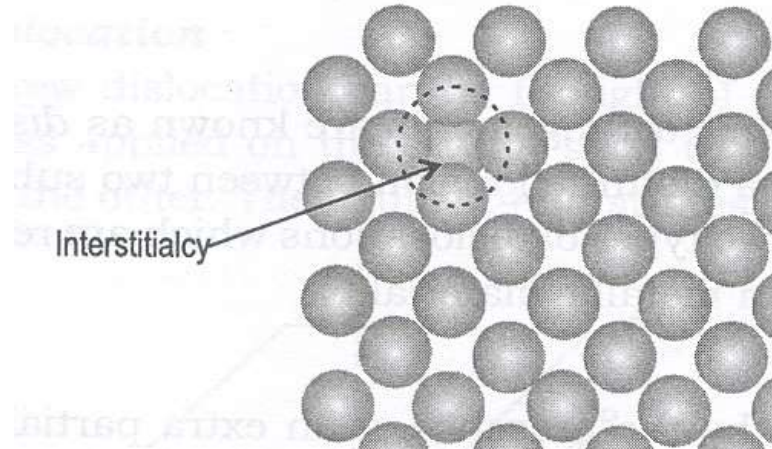
- Imperfect point like regions in a crystal
- Size – one or two atomic diameters
- Affect a region involving several atoms or ions

a) Vacancy



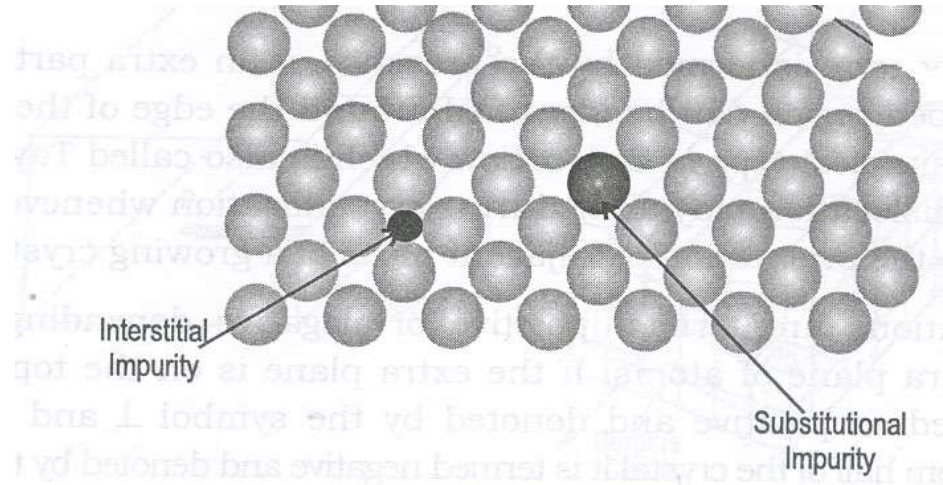
- ✓ Refers to a regular atomic site on the lattice from which an atom is missing
- ✓ A result of imperfect packing during crystallization
- ✓ Or due to thermal vibrations at high temperature
- ✓ Vacancies may be single, double or more...

b) self interstitial or interstitialcy



- Inverse of a vacancy
- An atom squeezes into the space b/w normal lattice positions
- Since such spaces are very small, a self interstitial produces much greater distortions of the surrounding lattice than does a vacancy
- Probability of formation of this defect is less compared to vacancy...

C, d) Impurities



- Atoms of a foreign material are called impurities
- A foreign atom that substitutes or replaces a parent atom at a regular site in the crystal – **substitutional impurity**
- Small sized atom occupying the void space b/w the parent atoms on regular sites – interstitial impurity
- Impurity atoms cause local distortions in the lattice, promote atomic diffusion and phase transformation
- Thermal and electrical conductivities are also affected by the presence of impurities...

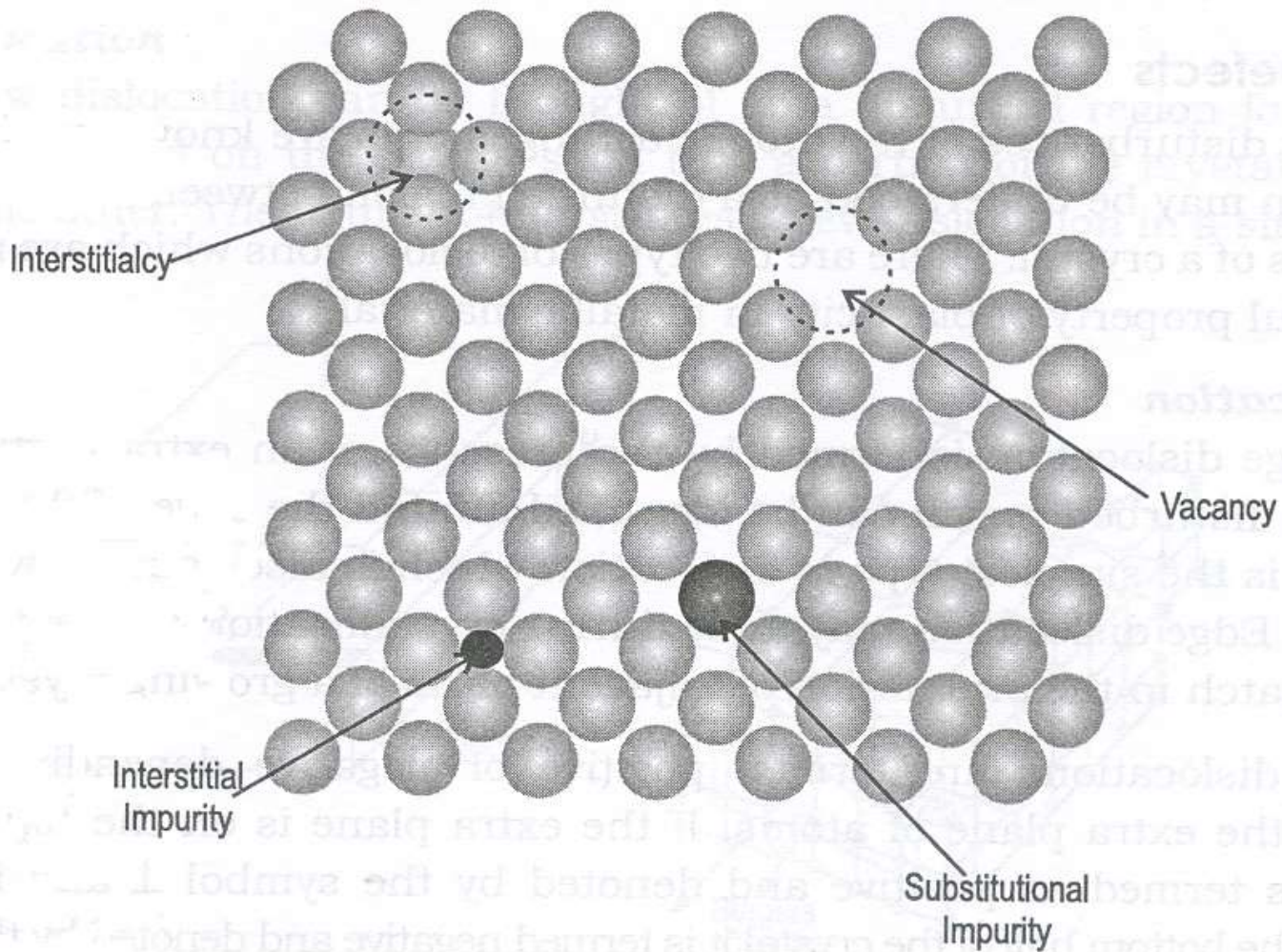
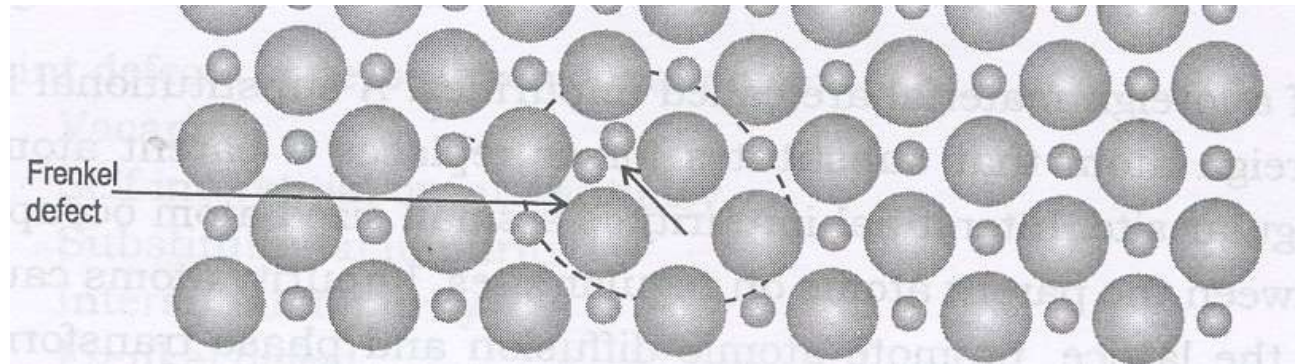


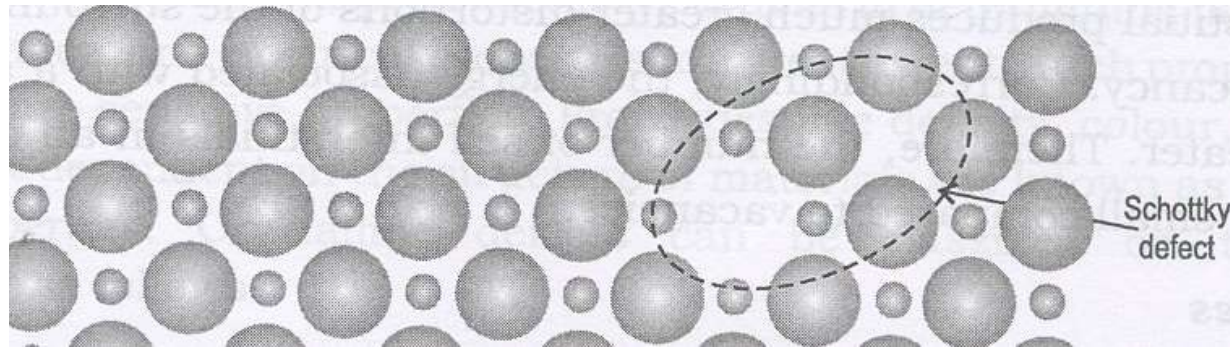
Figure 2.1 Point imperfections

e) Frenkel defect



- Frenkel & Schottky defects are Observed in ionic crystals – (overall electrical neutrality to be maintained)
- When an ion is displaced from a regular site to another site – Frenkel defect – eg: CaF_2
- As cations are smaller it is possible for them to get displaced into the void spaces
- Anions don't get generally displaced like cations as the void space b/w anions is too small

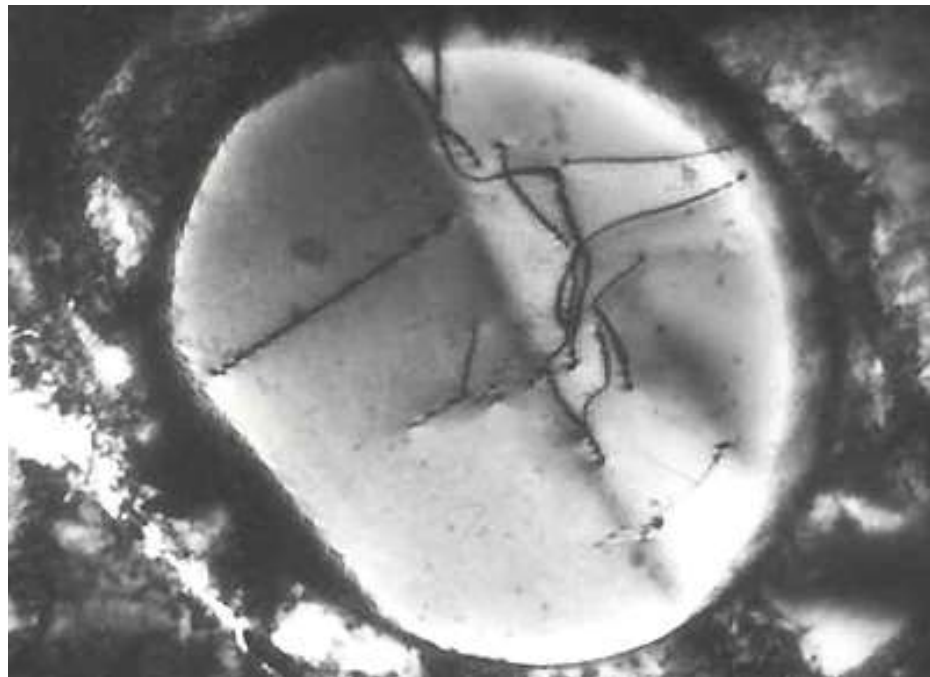
f) Schottky defect



- When a pair of cation and anion is missing from a lattice of an ionic crystal, the defect is known as Schottky defect
- To maintain electrical neutrality, the valency of missing pair of ions should be equal
- Closely related to 'vacancy'
- Facilitates atomic diffusion

Line Defects

- Linear disturbances of atomic arrangements are known as **DISLOCATIONS**
- Can be considered as a disturbed region b/w two perfect parts of a crystal
- Two types of dislocations which are responsible for the useful property of plasticity in metallic materials:
 1. Edge dislocation
 2. Screw dislocation



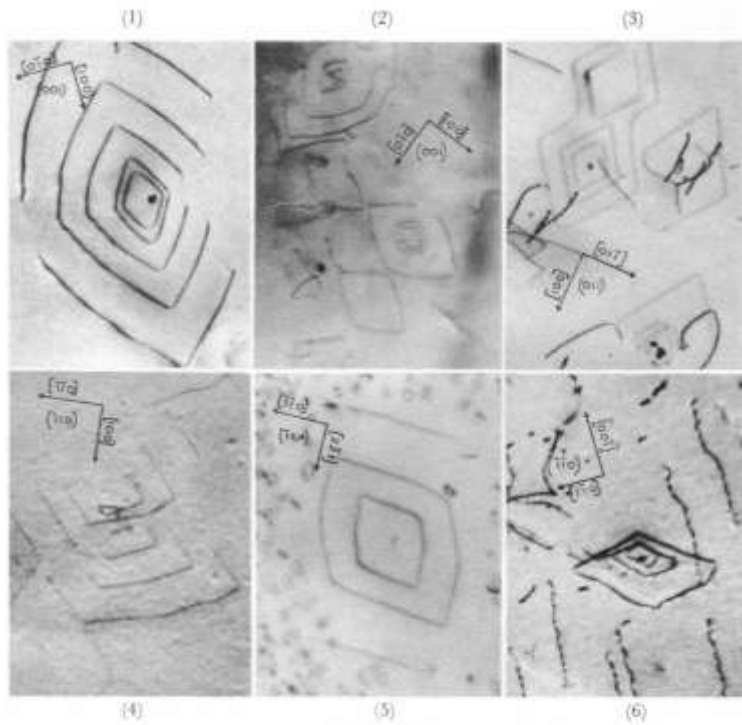
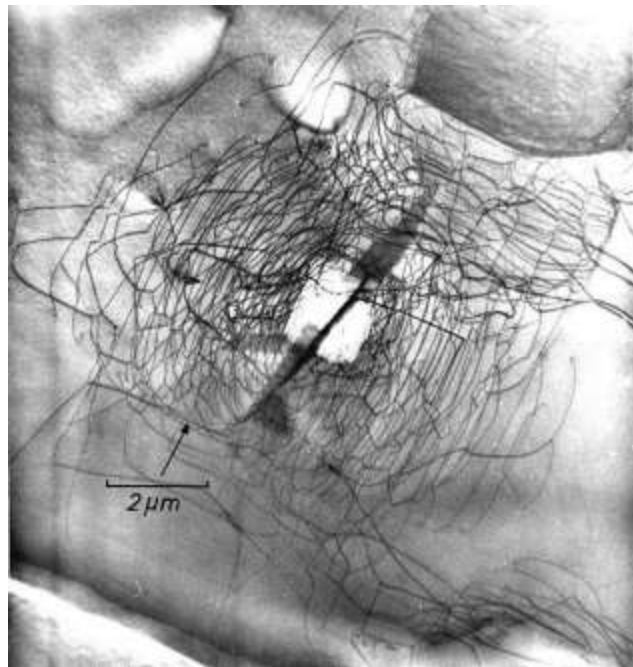
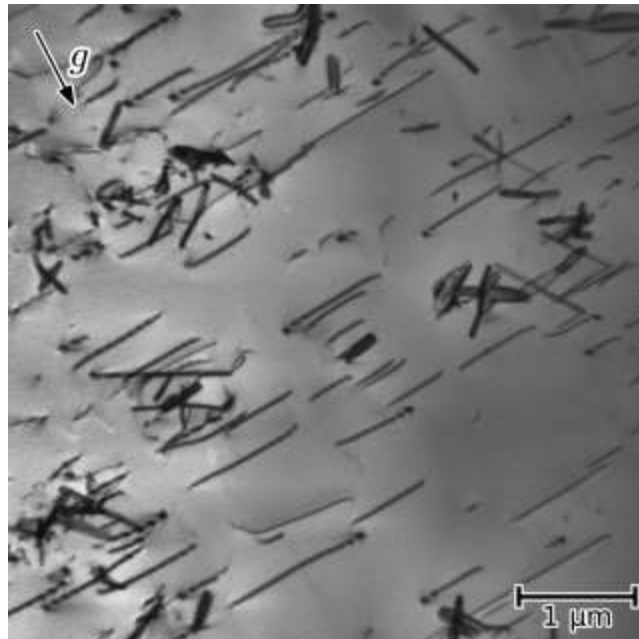
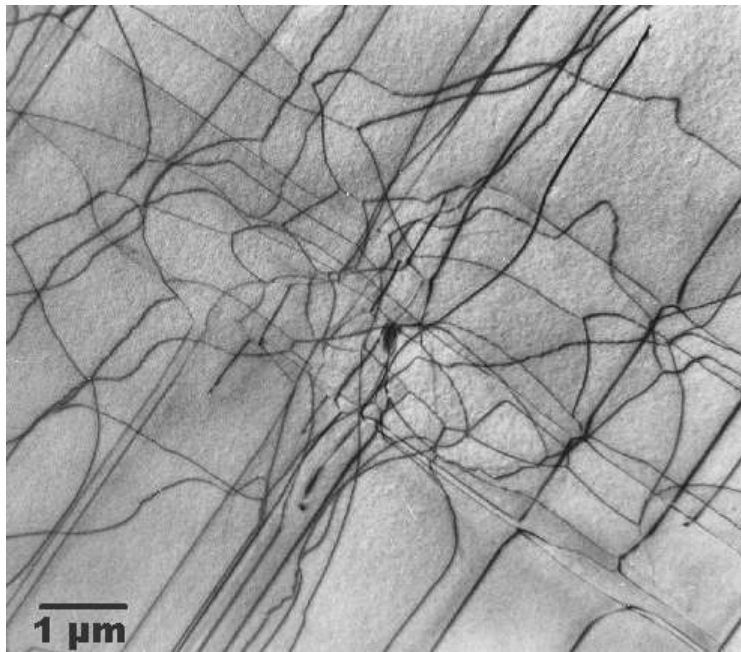
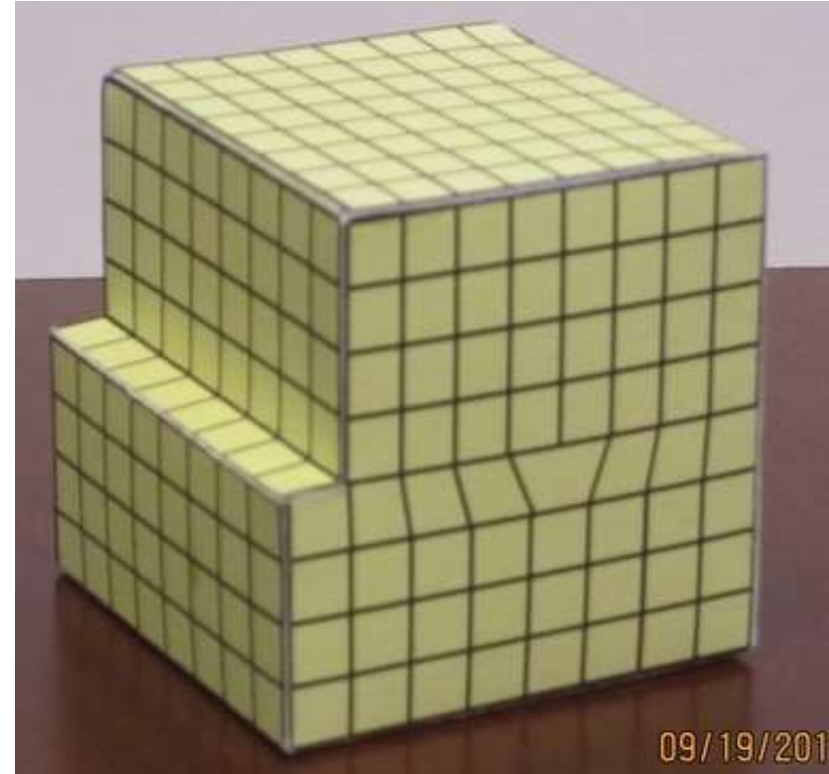
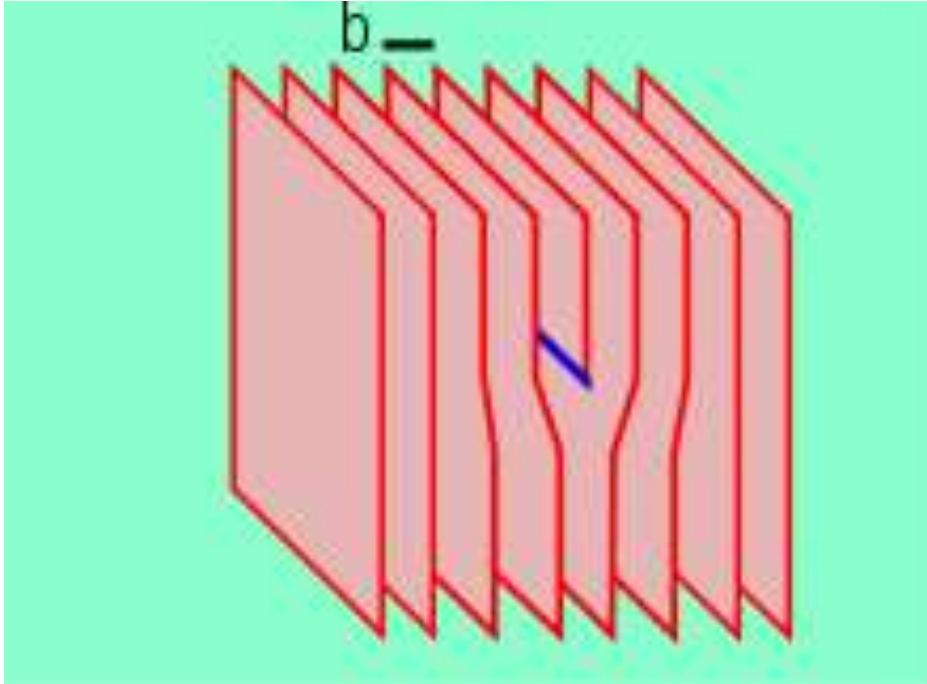
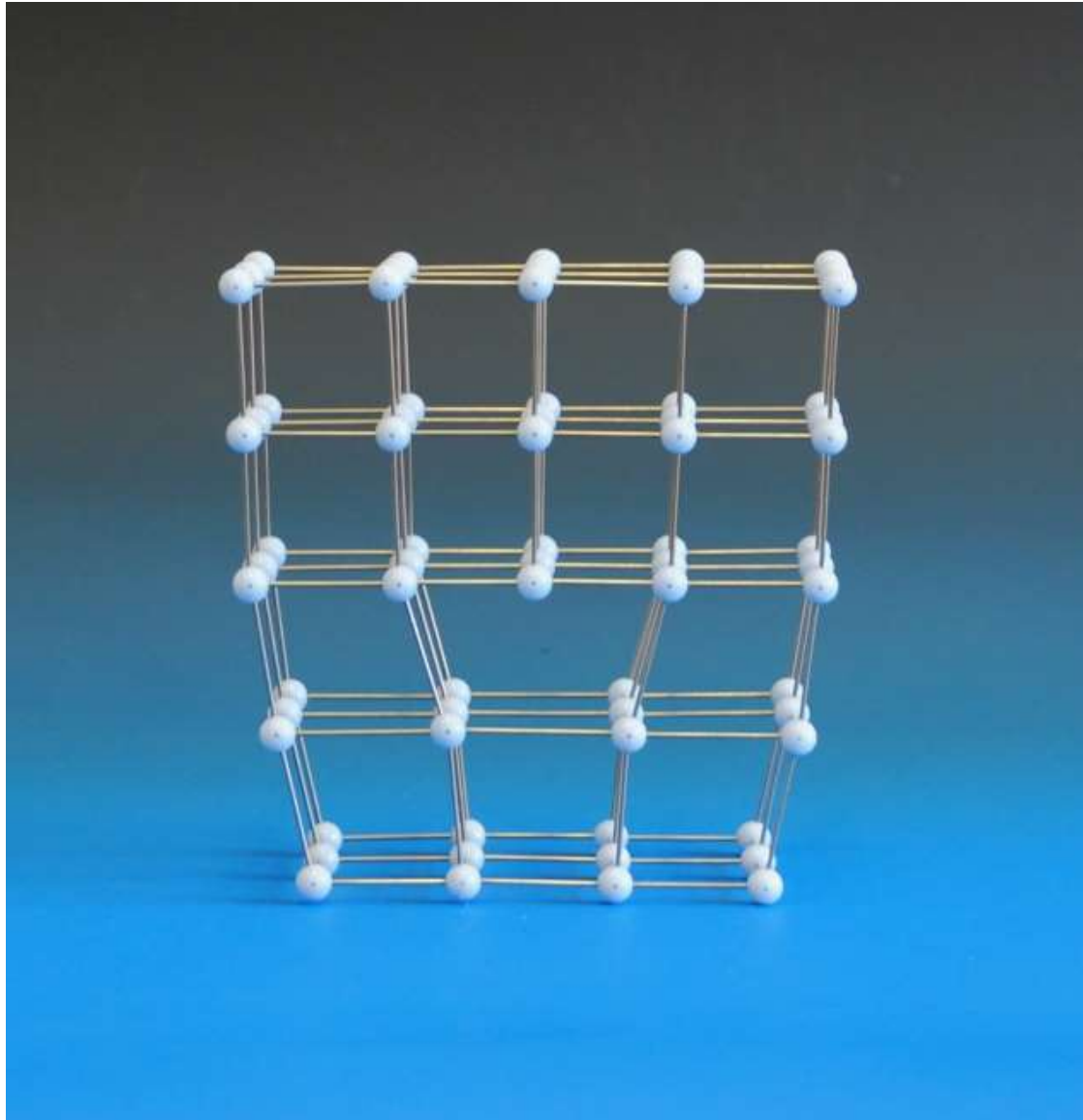


Figure A6.5

Edge dislocation (Taylor-Orowan dislocation)

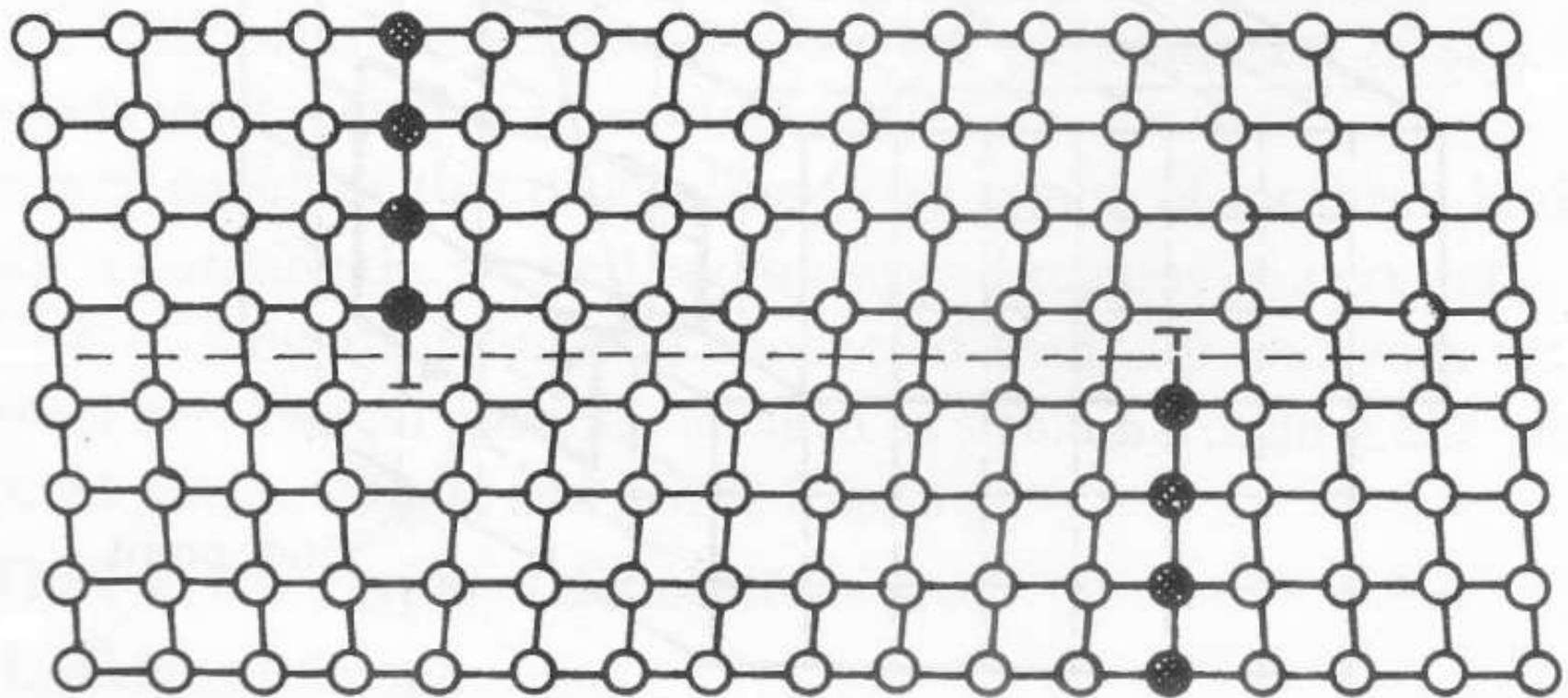


Edge dislocation

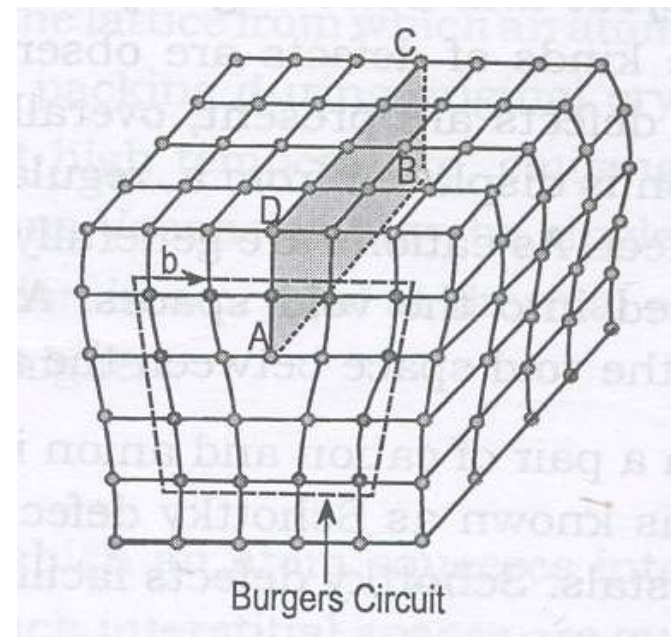
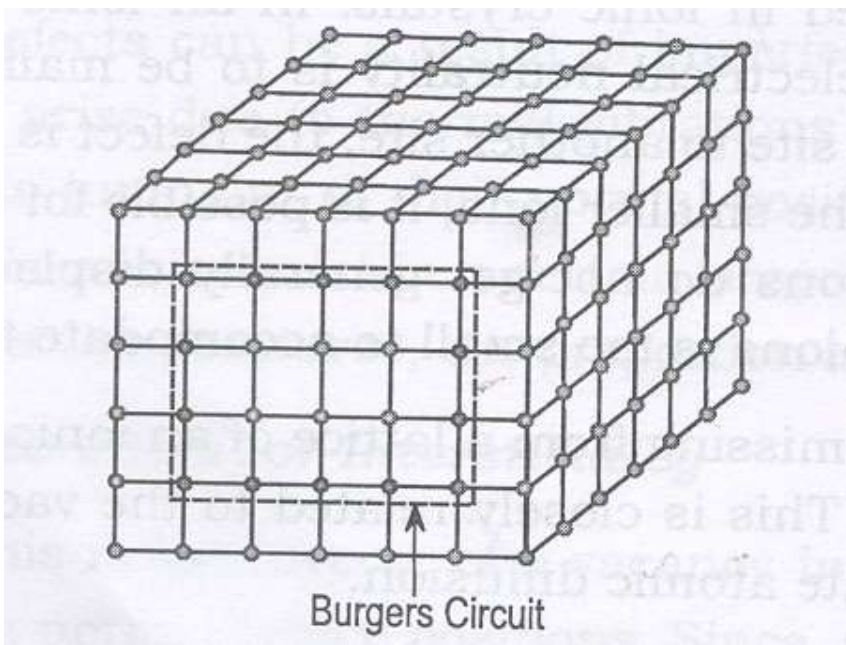


Edge dislocation

- Formed by the presence of an extra partial plane in a crystal
- A disturbed region can be identified around the edge of the incomplete plane
- Arise during crystallization whenever there is a slight mismatch in the orientation of adjacent parts of a growing crystal
- Plastic deformation of materials also causes edge dislocation
- Positive dislocation \perp or negative dislocation \top



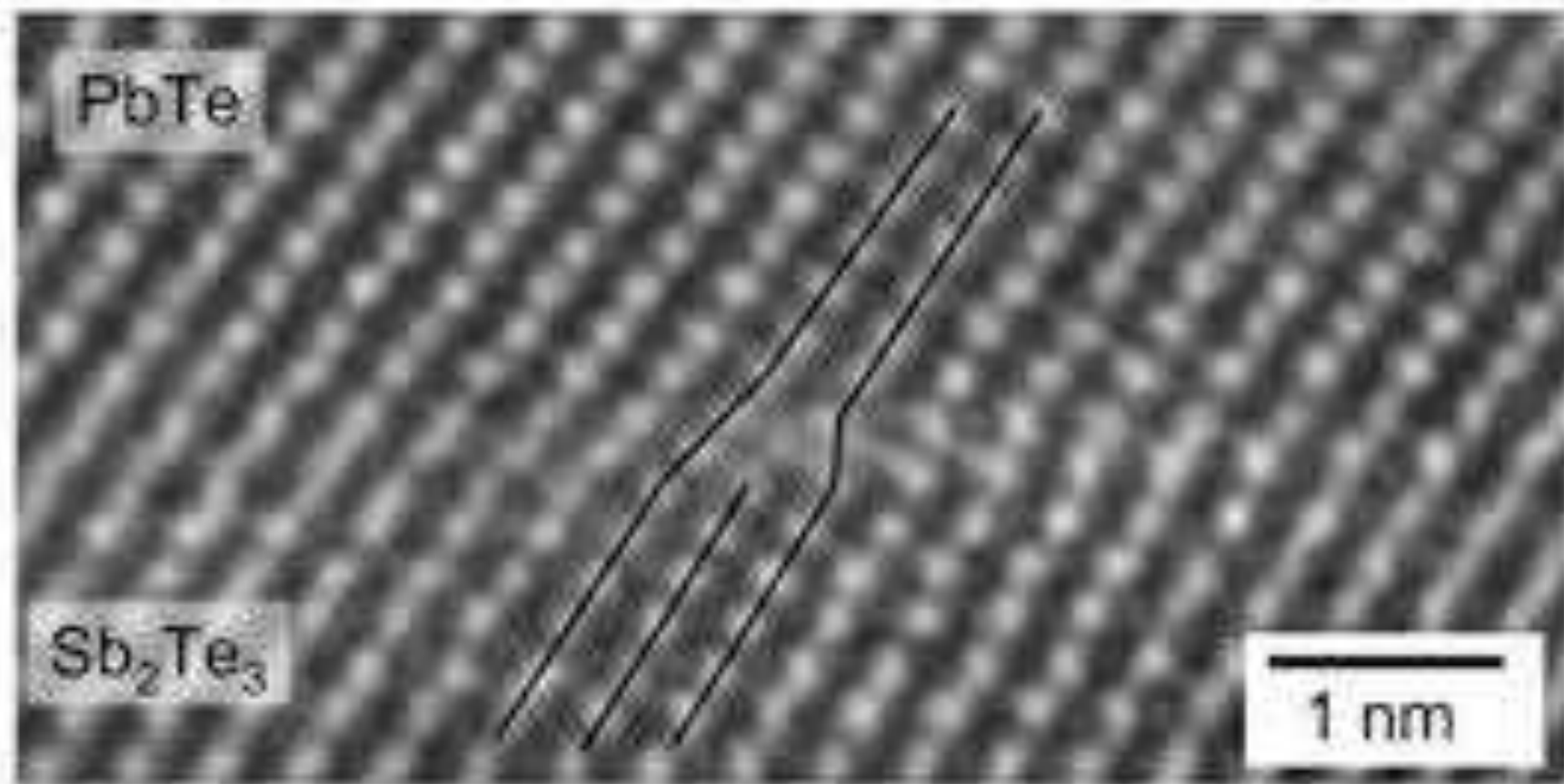
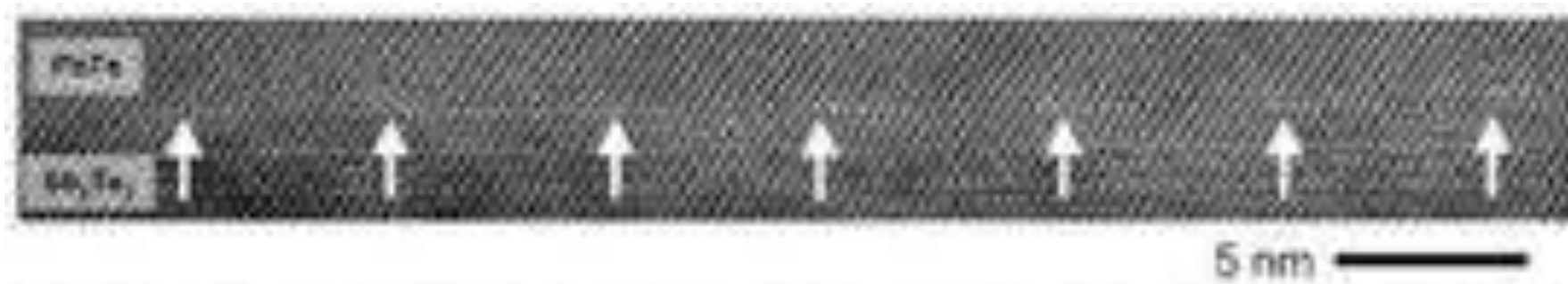
- Presence of extra plane causes distortion within the crystal
- Region of the crystal where extra plane is present will be in compression and the other region in tension



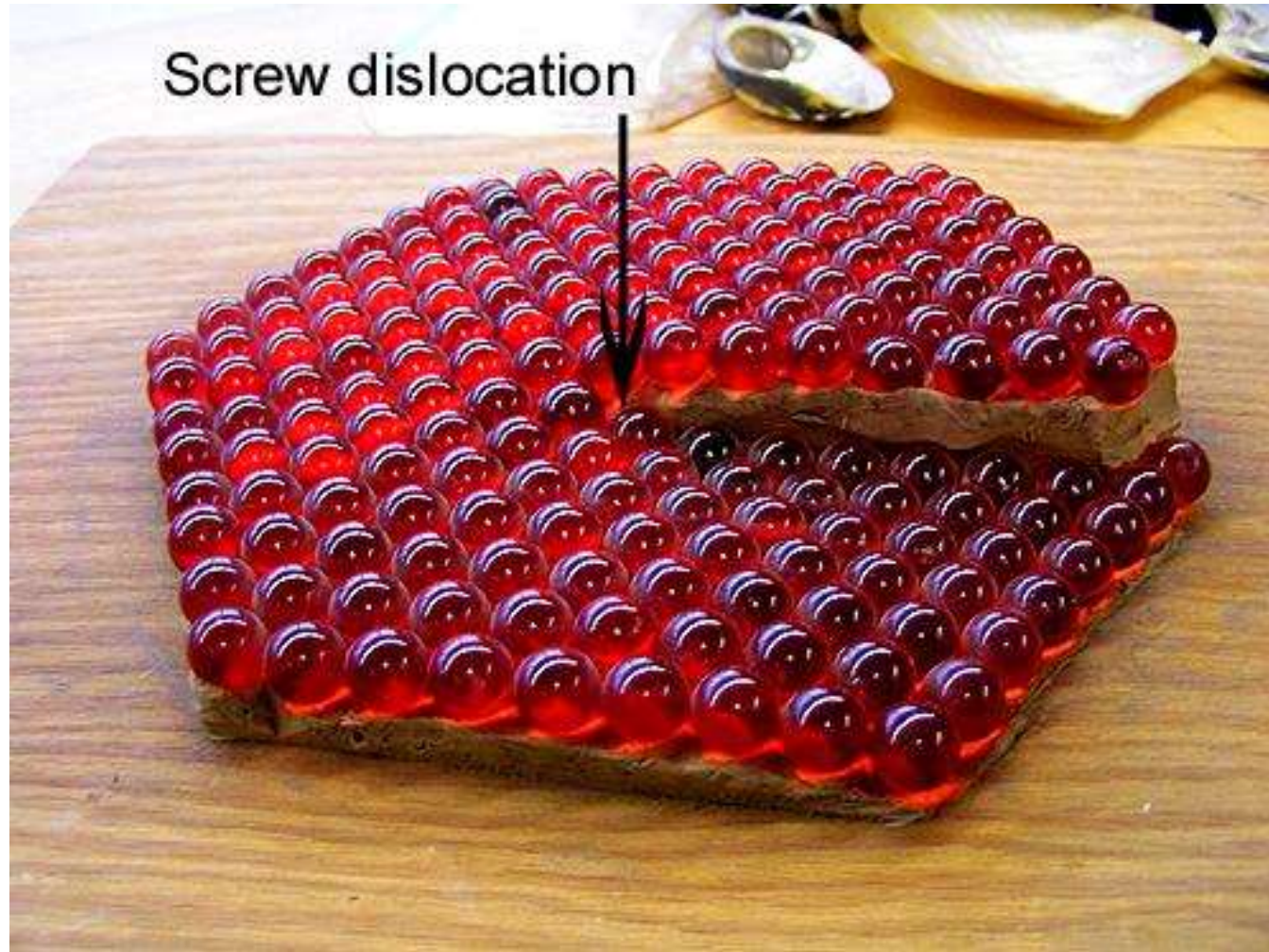
- Presence of plane ABCD causes edge dislocation
- The line AB represents dislocation (dislocation line)
- The magnitude and direction of the lattice distortion associated with a dislocation is expressed in terms of a **Burgers vector**, denoted by a **b**
- An atom by atom circuit is drawn around the dislocation line – Burgers circuit
- In a perfect crystal where there is no dislocation, such a circuit will close without any mismatch
- In the presence of dislocation, the Burgers circuit will not close perfectly
- vector needed to close the circuit – Burgers vector

$$\vec{b}$$

- In an edge dislocation the Burgers vector is seen to be perpendicular to the dislocation line

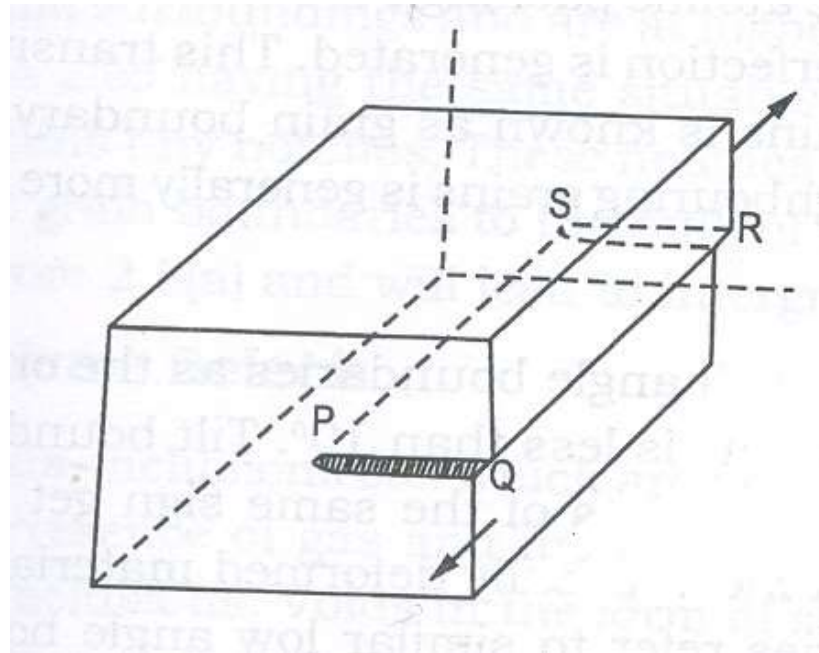


Screw Dislocation



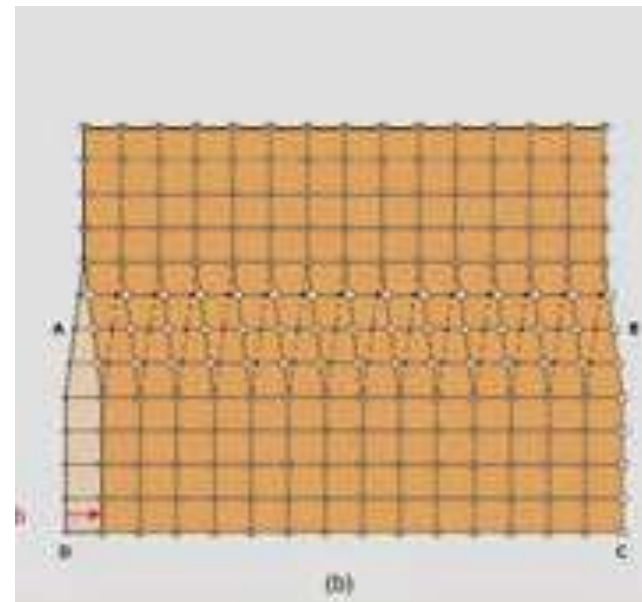
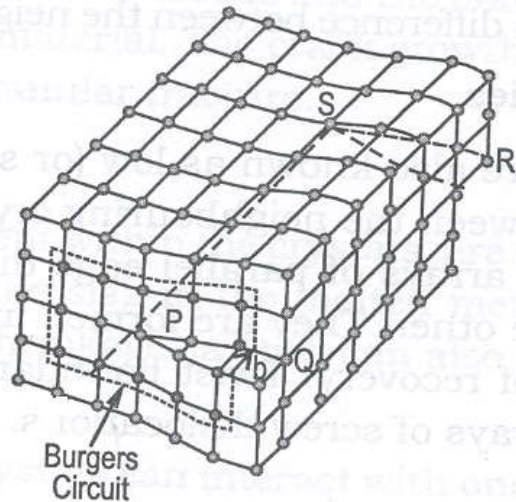
Screw Dislocation

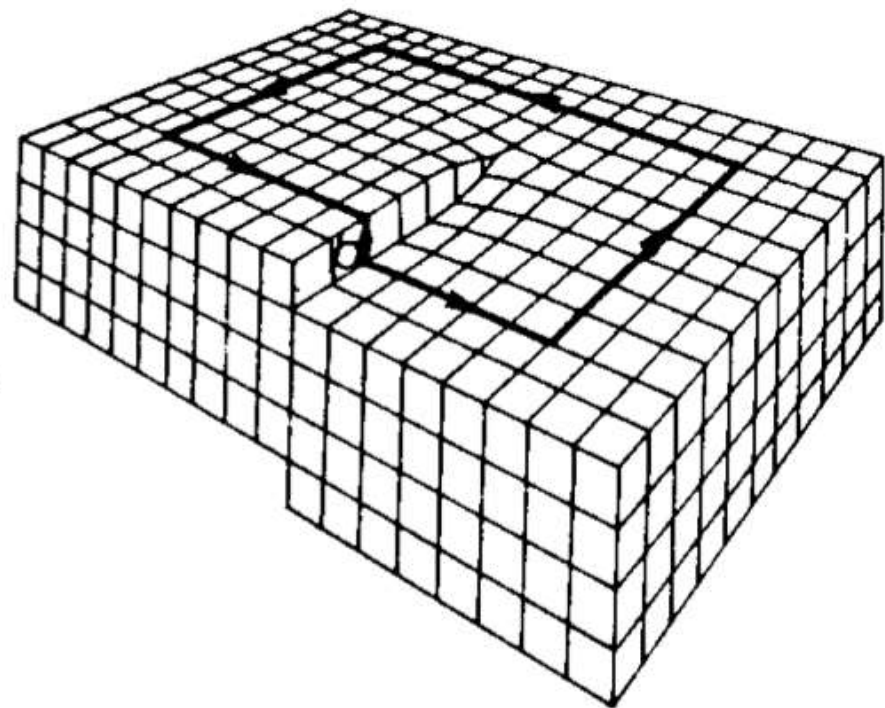
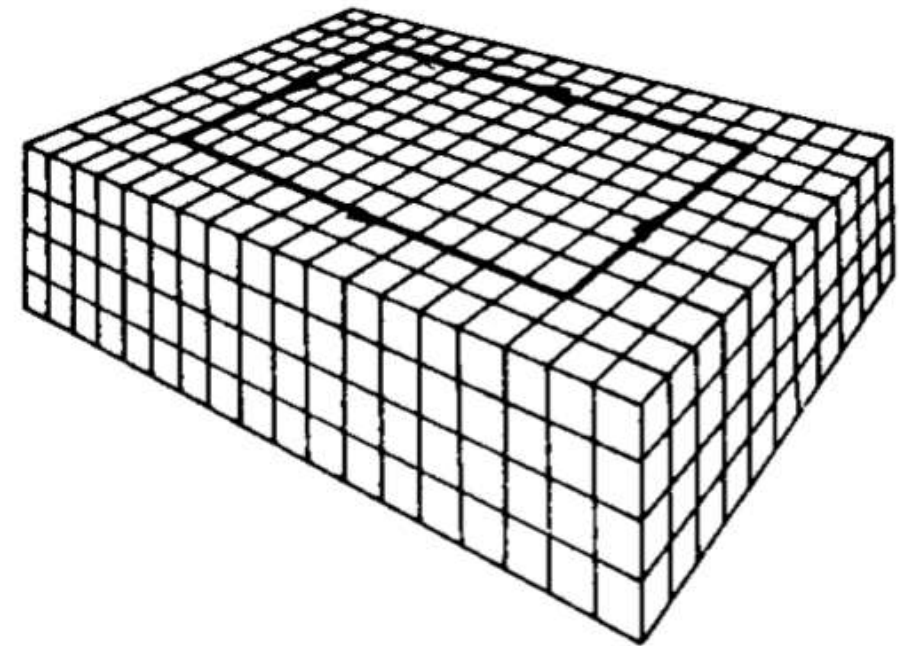
- Disturbed region formed by a shear stress such that a portion of the crystal is shifted relative to the other



- When shear stress applied the upper part of crystal to the right of PS has skipped relative to the lower part

- Line PS is the dislocation line and is parallel to the Burgers vector
- Screw dislocation derives its name from the spiral or helical path that can be traced along the dislocation line by the atomic planes
- Right handed spirals - +ve screw dislocation
- Left handed spirals -ve screw dislocation
- symbols





screw dislocation

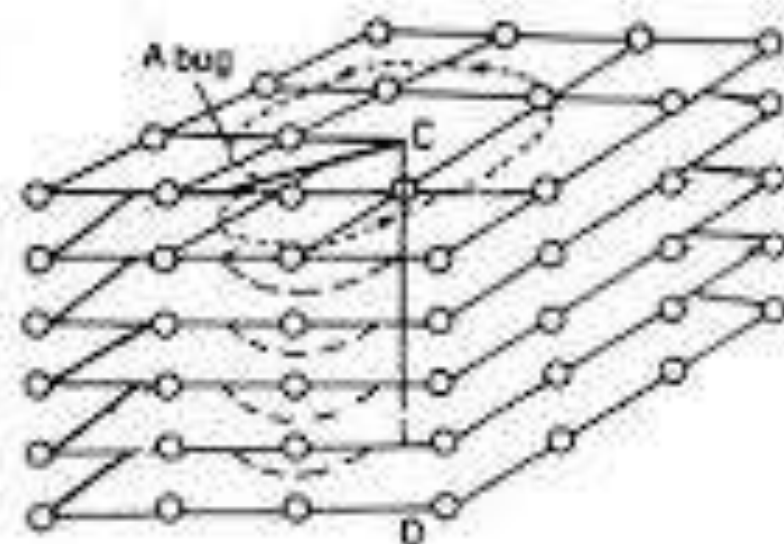
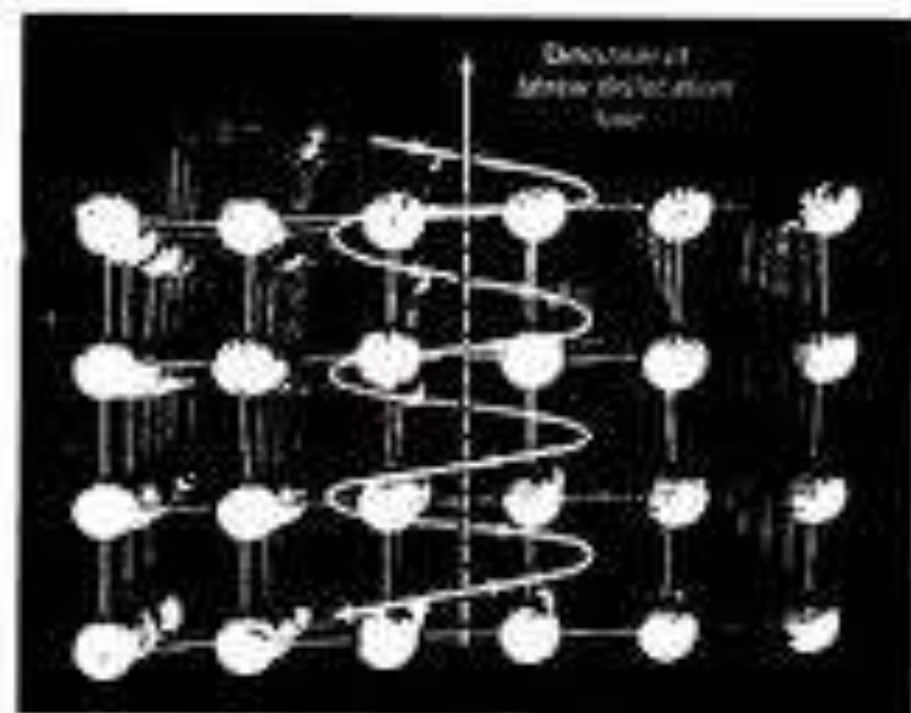
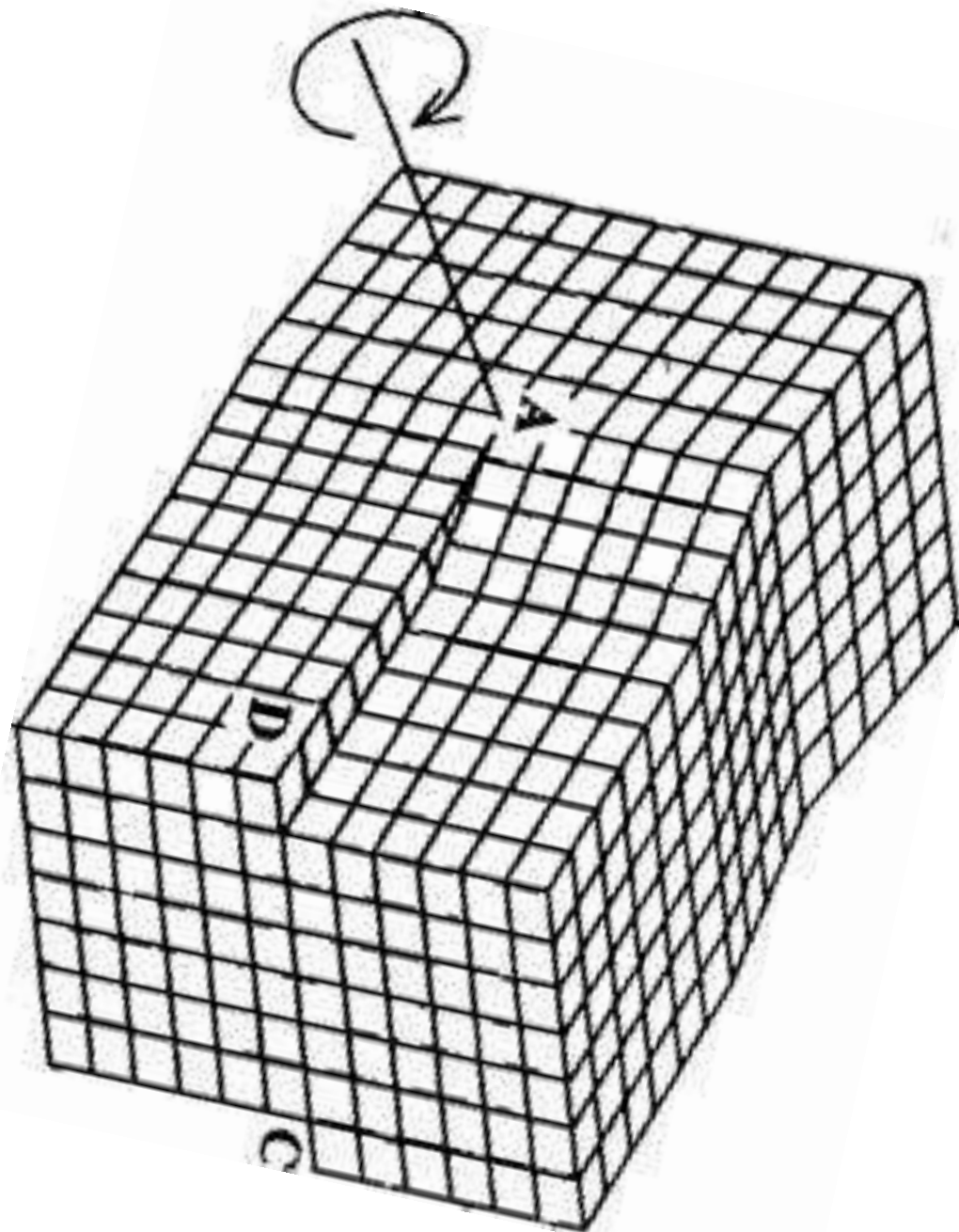
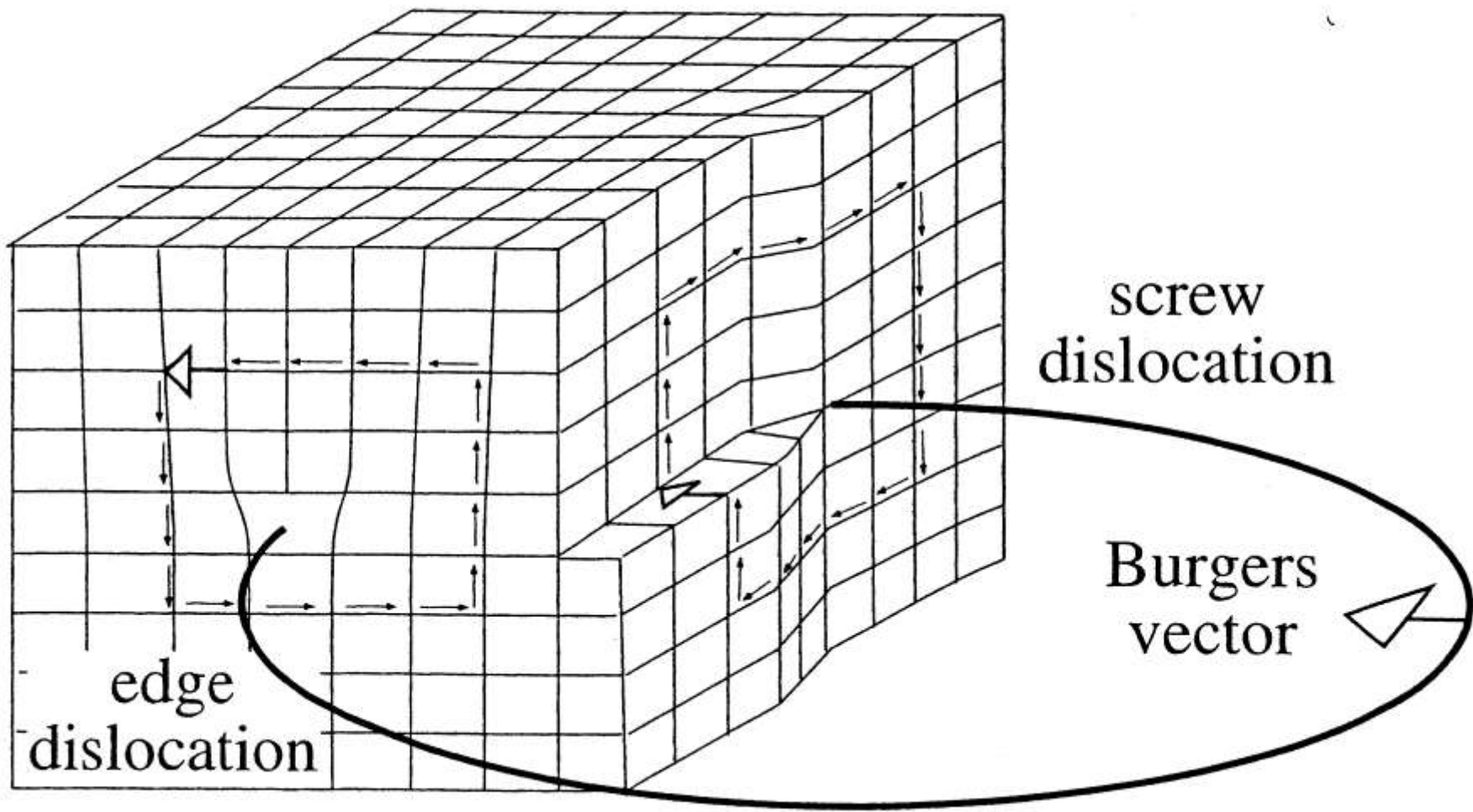


Fig. 4.22. A Screw dislocation (CD)

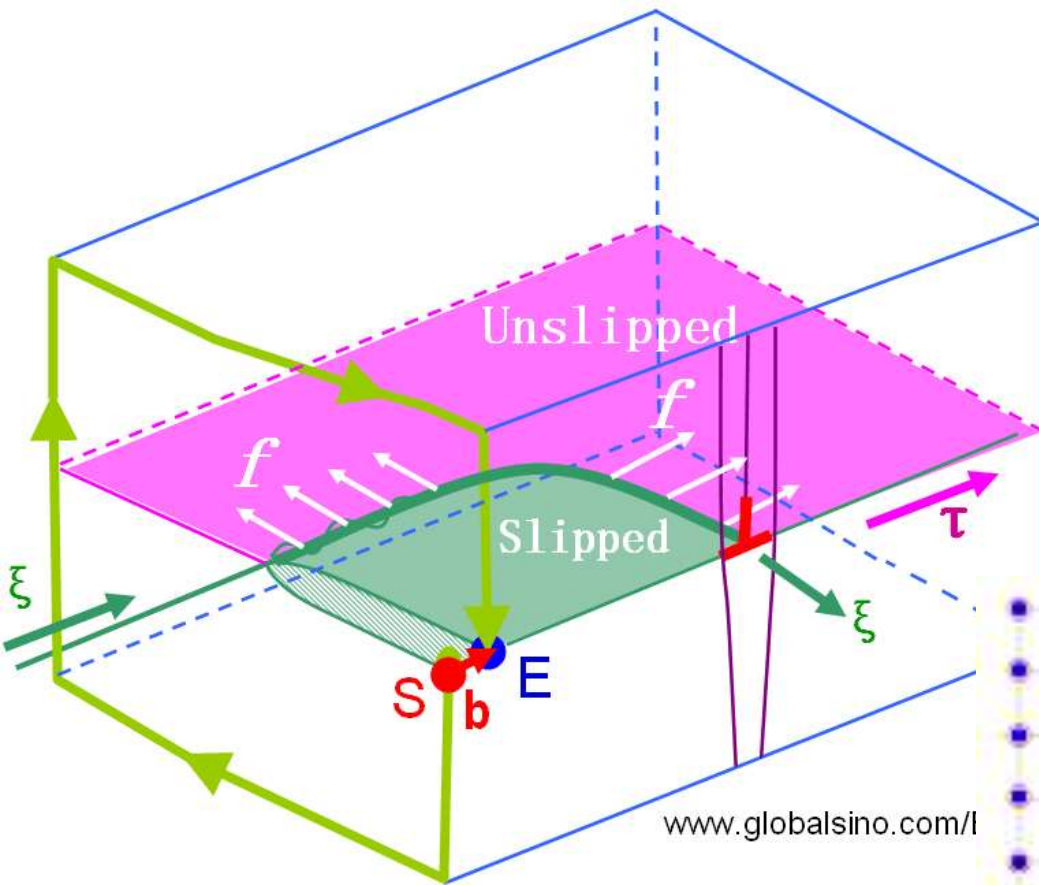




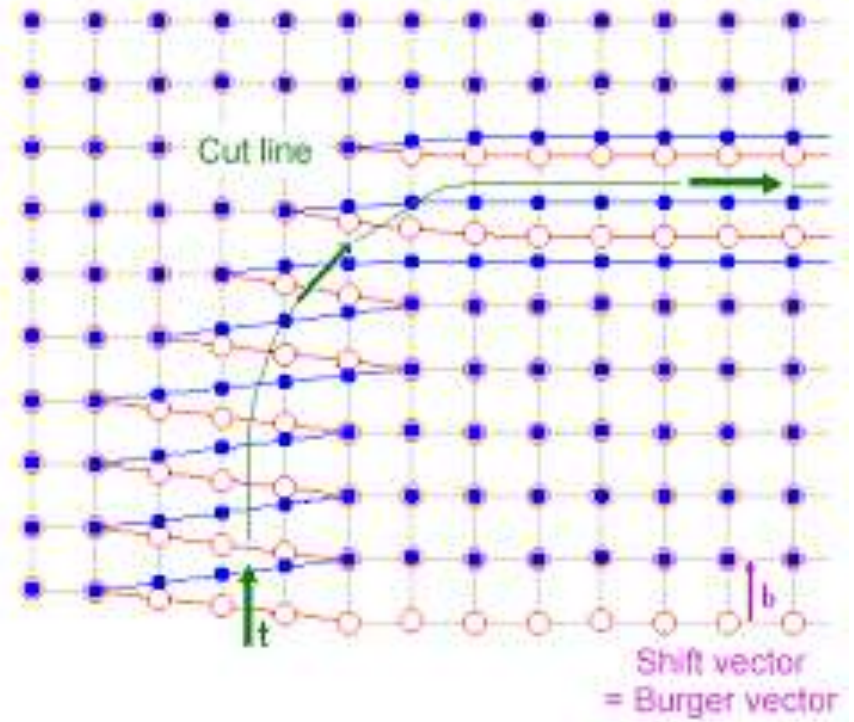
edge
dislocation

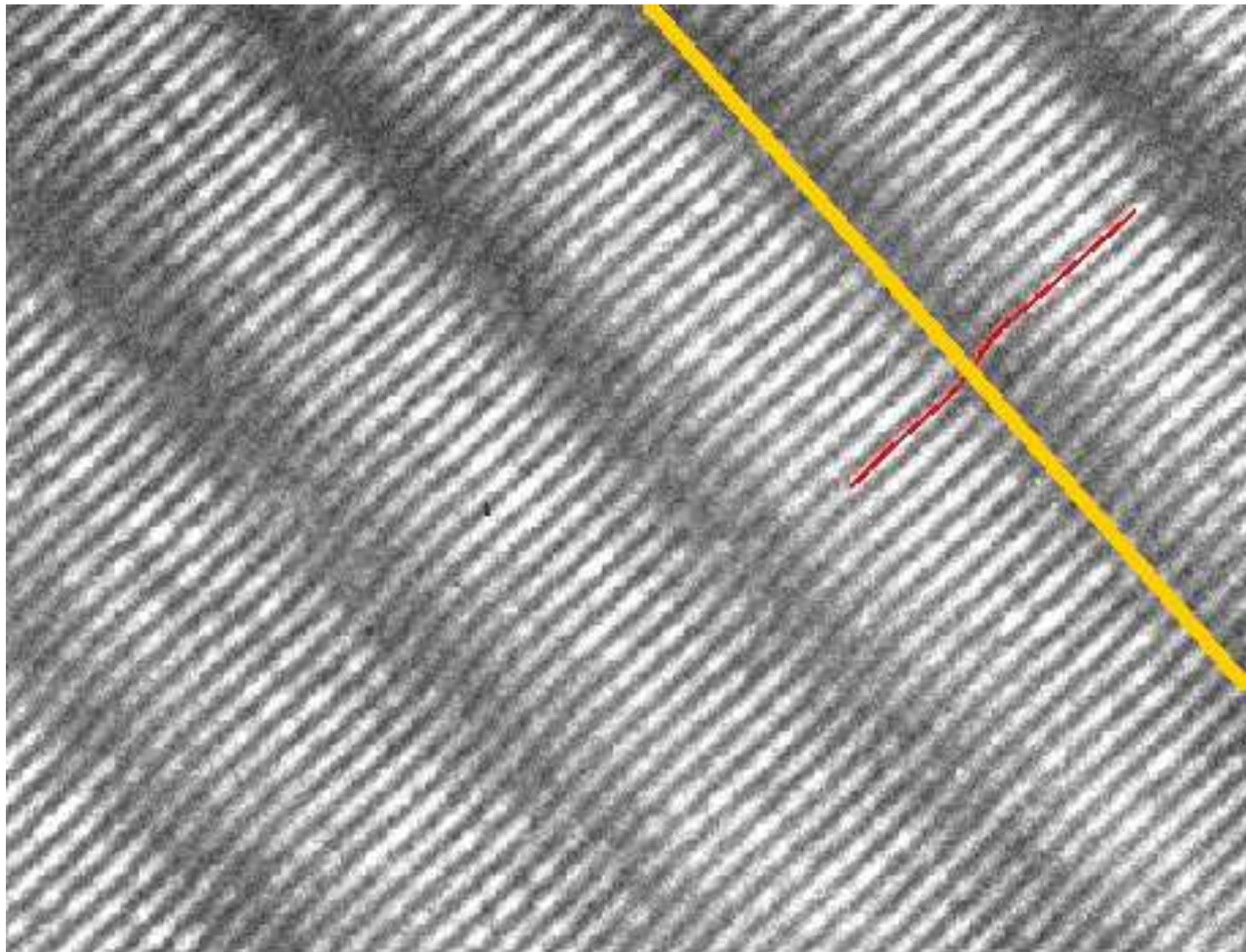
screw
dislocation

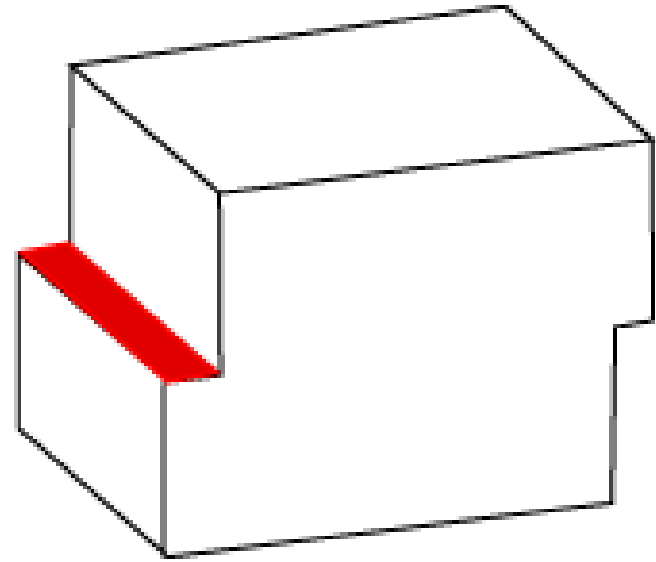
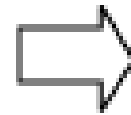
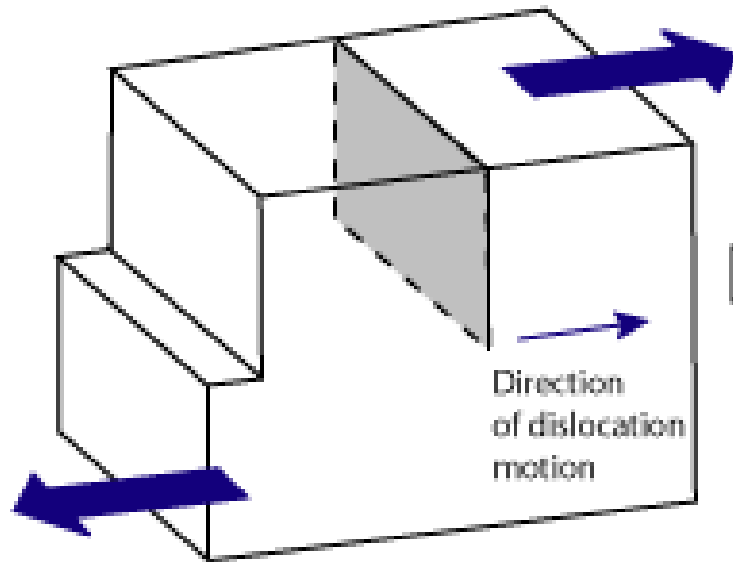
Burgers
vector



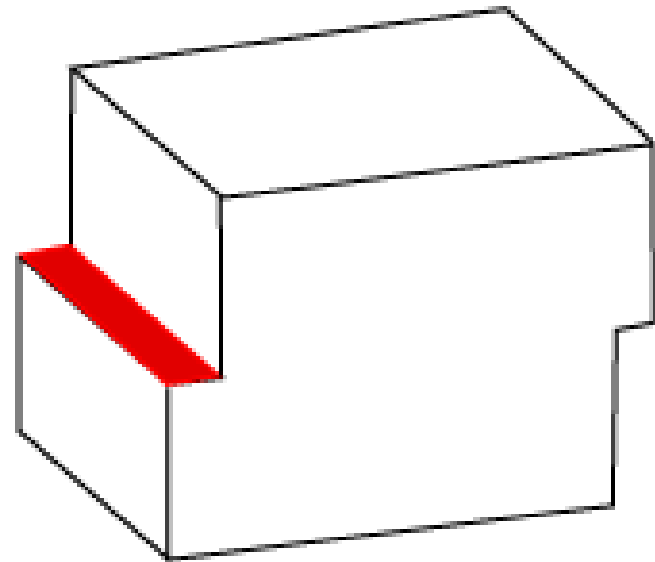
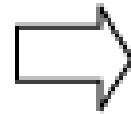
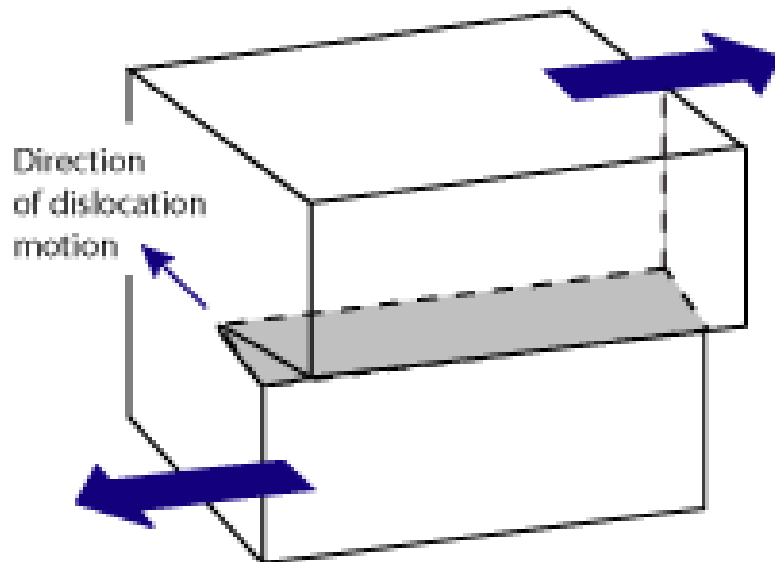
www.globalsino.com/







Edge Dislocation



Screw Dislocation

- When stress applied to a material having dislocations, they move causing plastic deformation
- Edge dislocations move in the direction of Burgers vector
- Screw dislocations move perpendicular to Burgers vector
- Dislocations in real crystals are combinations of edge and screw types – mixed dislocations
- Dislocation line in this case will be curved
- Edge and screw dislocations of opposite signs lying in the same plane attract and can destroy each other

Surface Defects

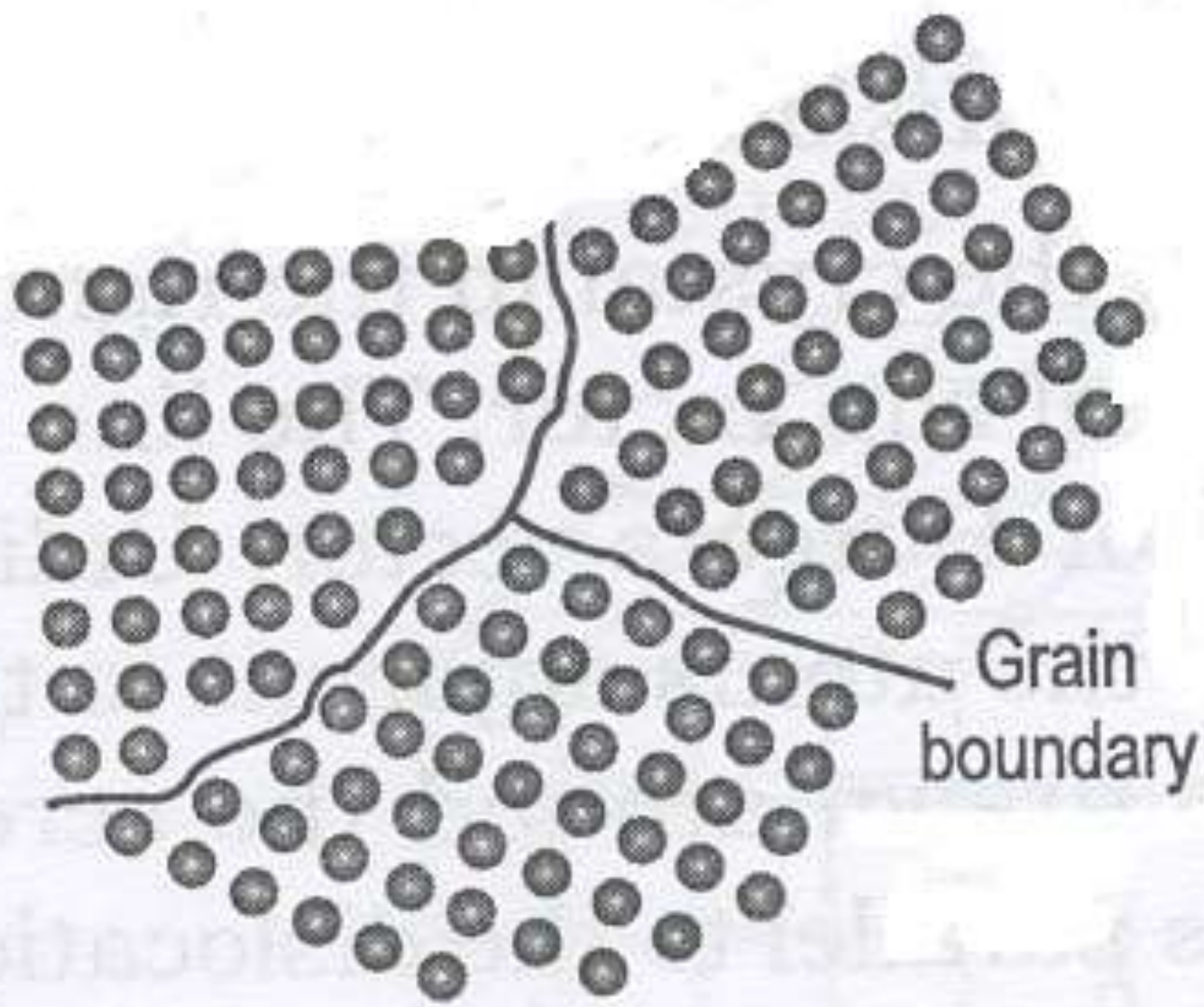
- are two Dimensional in nature – plane defects
- Change in the stacking of atomic planes
- Refer to regions of distortions that lie about a surface having a thickness of a few atomic diameters

Grain boundary



Grain Boundaries

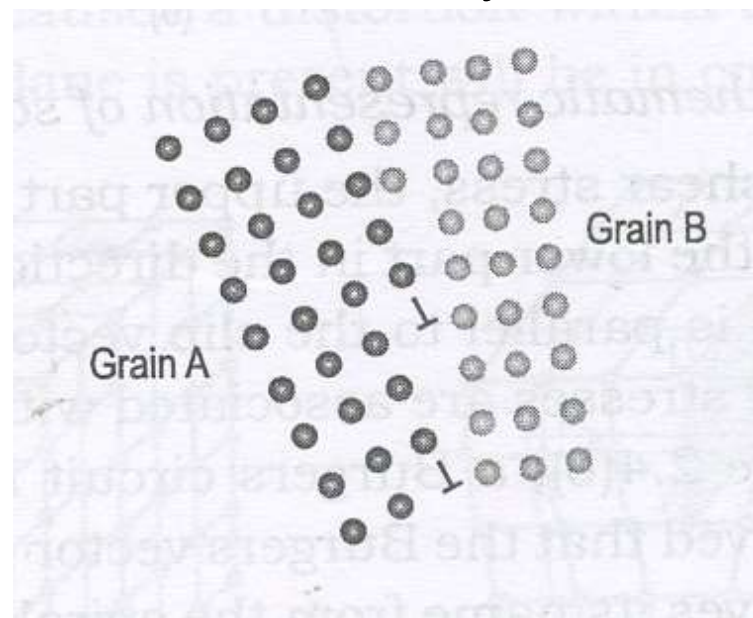
- Surface imperfections which separate crystals of different orientations in a polycrystalline material
- During solidification of liquid metals new crystals are randomly oriented with respect to one another
- They grow by addition of atoms from adjacent regions and finally impinge each other
- Atoms caught in b/w the crystals are pulled by each of the two crystals – but they take up a compromise position – boundary
- Crystal boundary or grain boundary
- The atomic packing b/w the two adjacent grains gets distorted and a region of imperfection is generated
- Low angle and high angle grain boundaries...



Grain
boundary

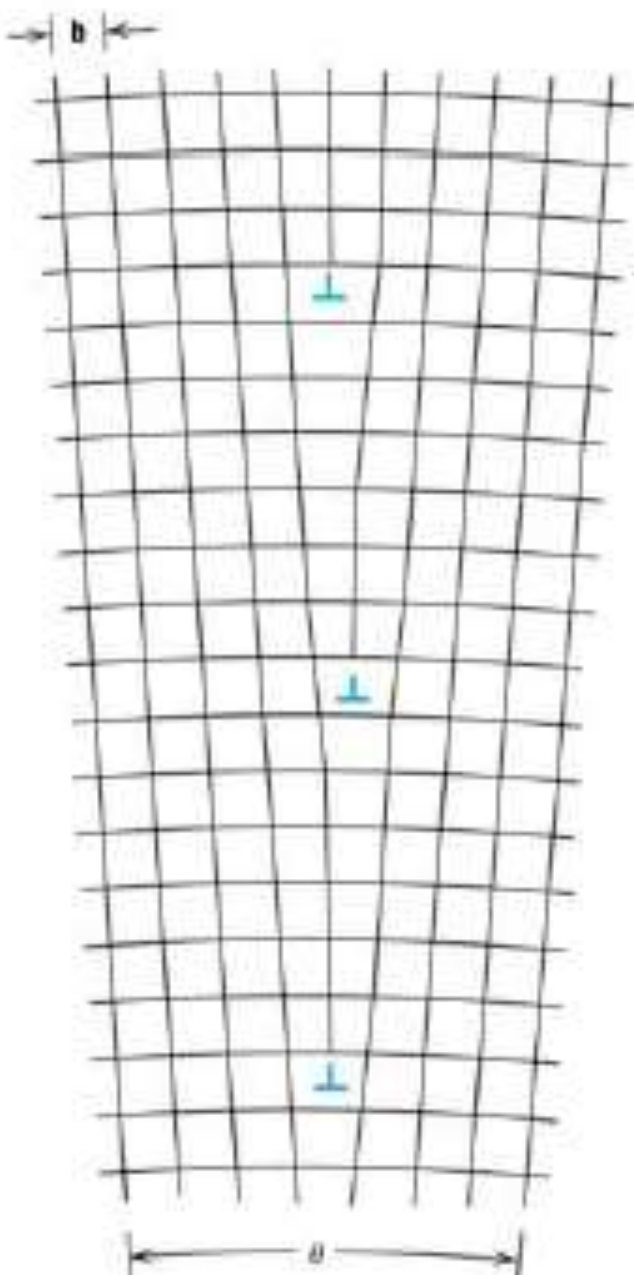
Tilt boundaries

- A type of low angle boundary (less than 10 degrees)
- Are formed when arrays of parallel edge dislocations of the same sign get arranged one above the other
- They are formed in mechanically deformed materials during the process of recovery



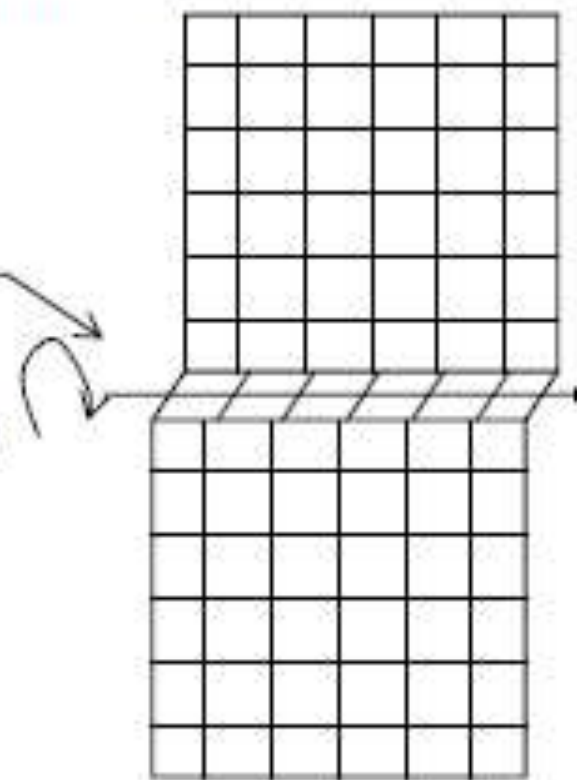
- Twist boundaries – similar low angle boundaries formed by arrays of screw dislocations
- All crystalline materials don't exhibit Tilt boundaries

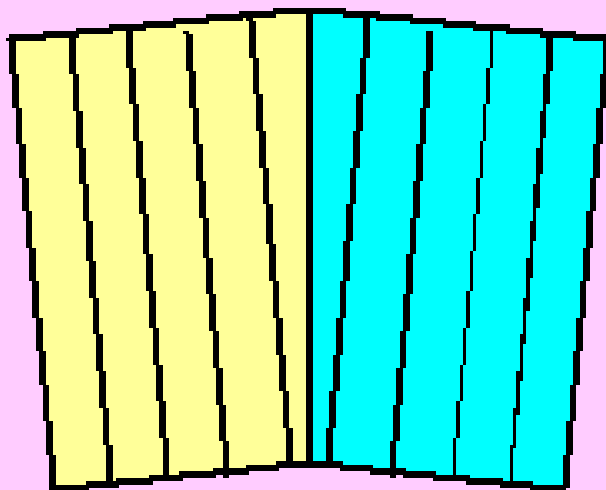
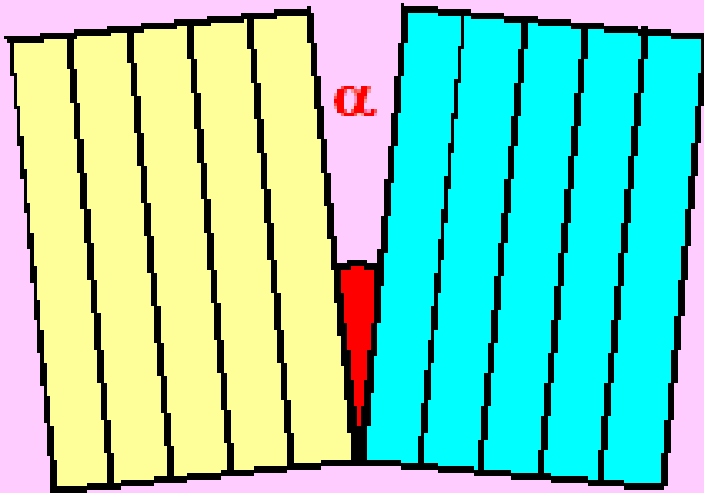
FIGURE 4.8 Demonstration of how a tilt boundary having an angle of misorientation θ results from an alignment of edge dislocations.



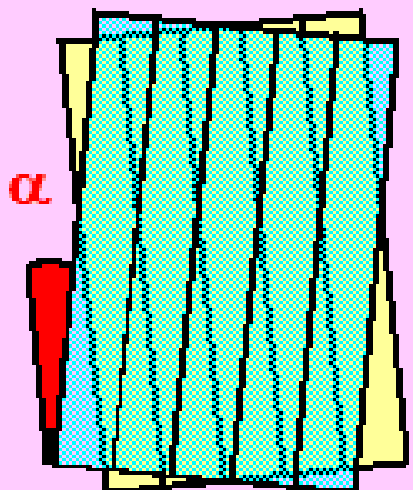
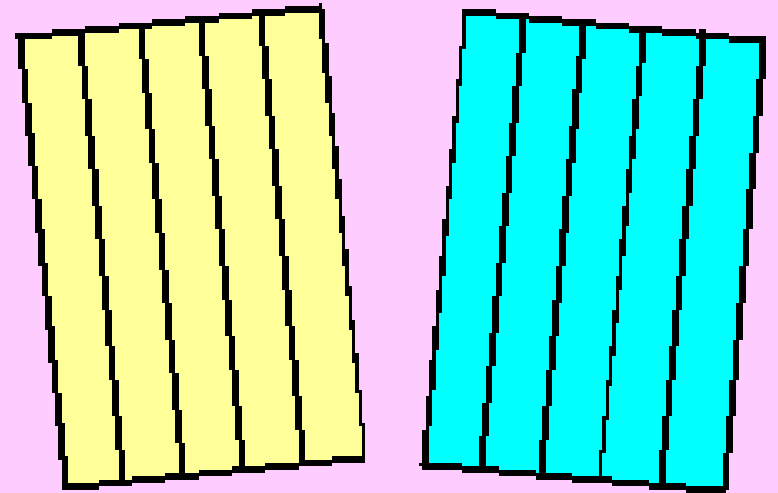
← Small angle grain boundary such as this **tilt boundary** can be considered as an array of edge dislocations.

Array of screw dislocations lead to a **twist boundary**. 0° misalignment or parallel misalignment.

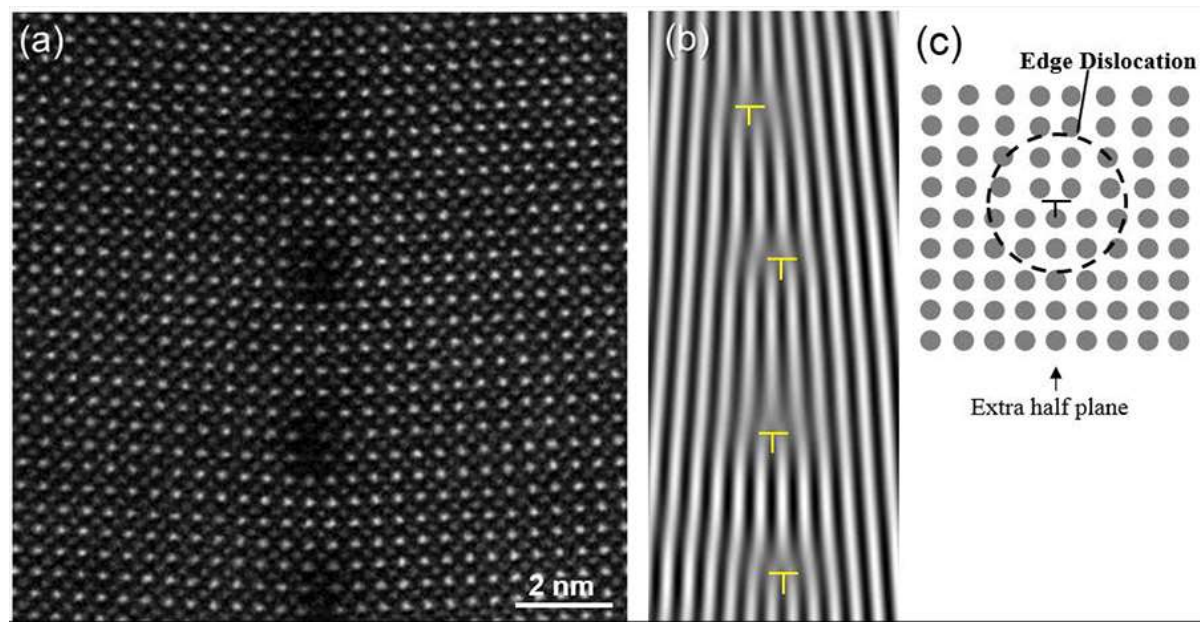
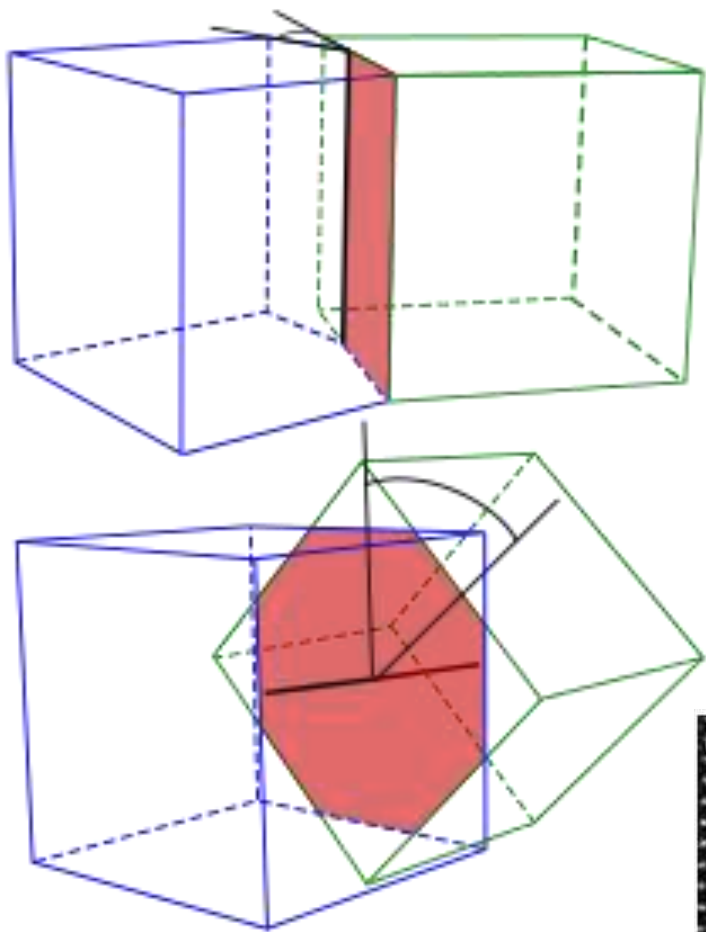




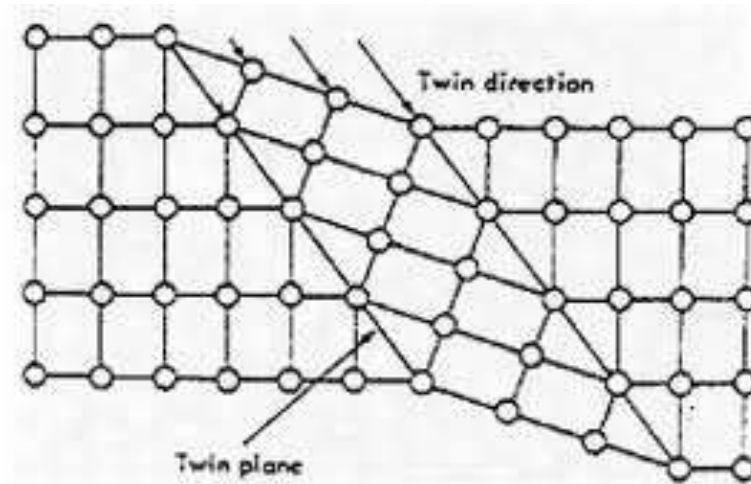
Pure tilt



Pure twist



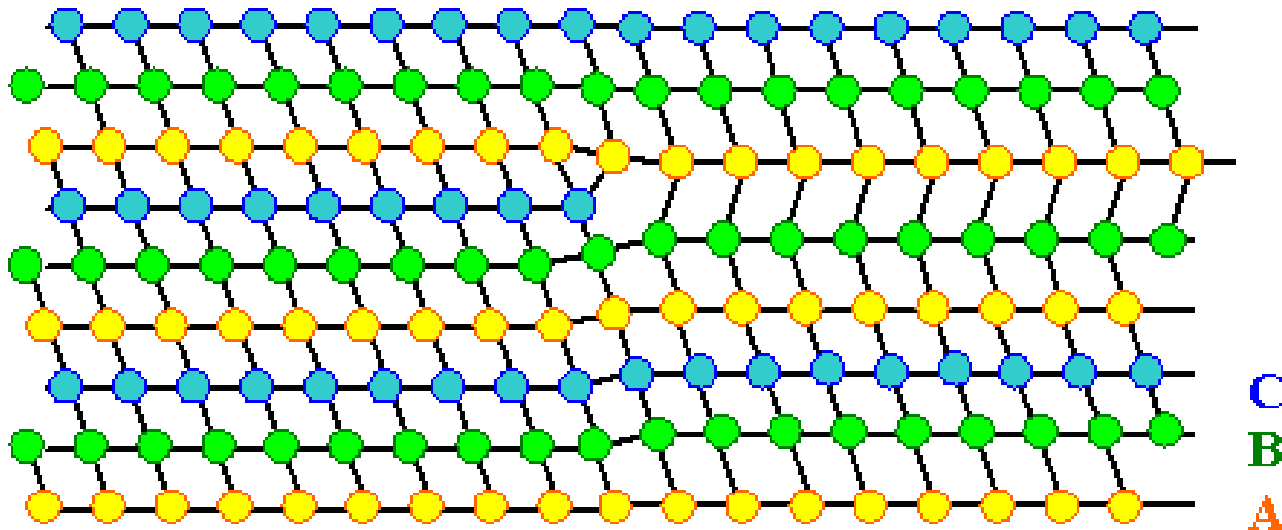
Twin Boundaries



- The arrangement of atoms on one side of the boundary is a mirror image of that on the other side – Twin Boundary
- Occur in pairs, resulting in region of different orientation in a crystal
- Formed during crystal growth, mechanical deformation or annealing...

Stacking fault

- Arise whenever the stacking of atomic planes within a crystal is not in the proper sequence
- Observed in close packed crystal structures (FCC or HCP)
- Ideal FCC crystal – ABC ABC ABC.....
- HCP – AB AB AB.....



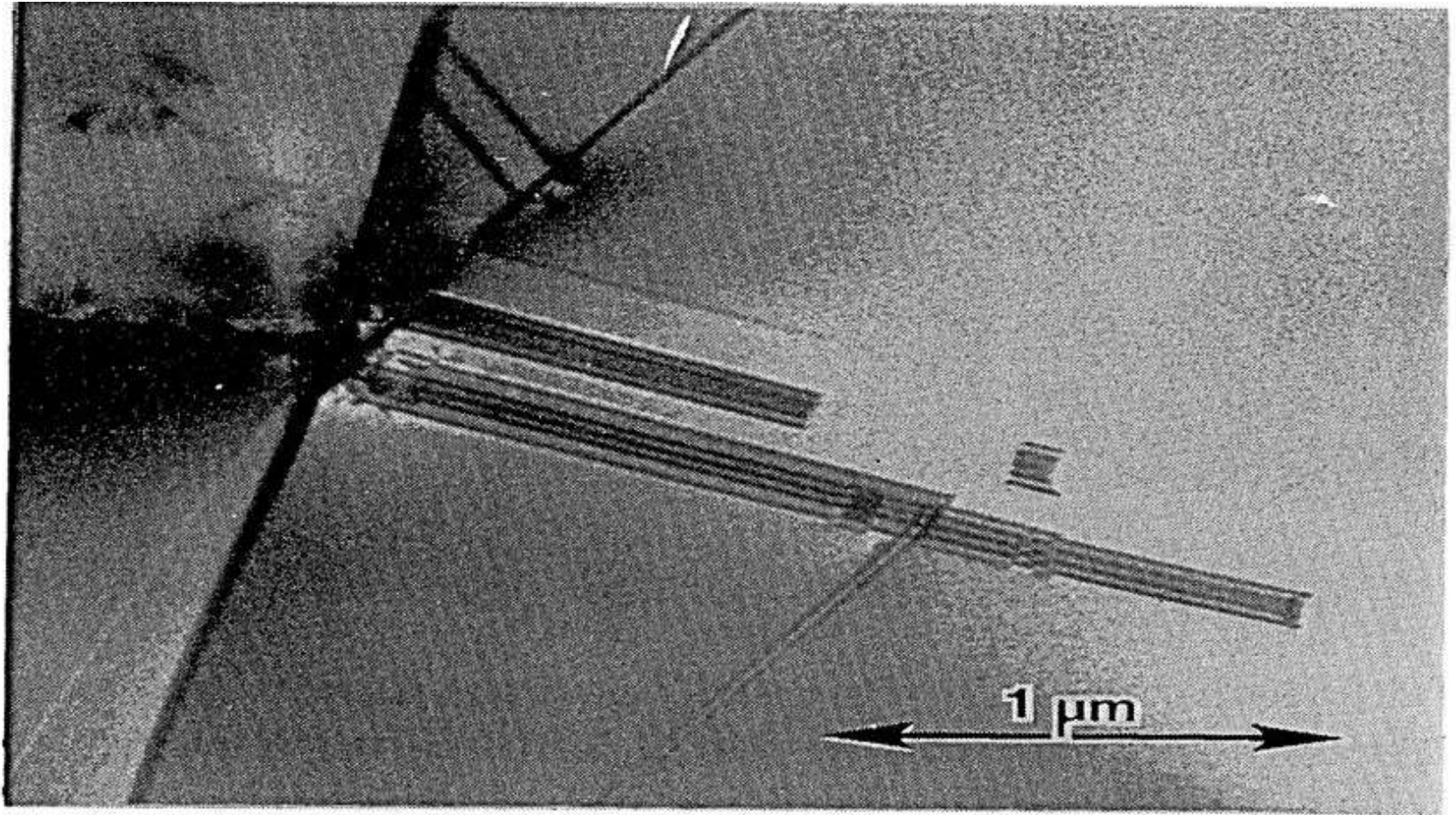
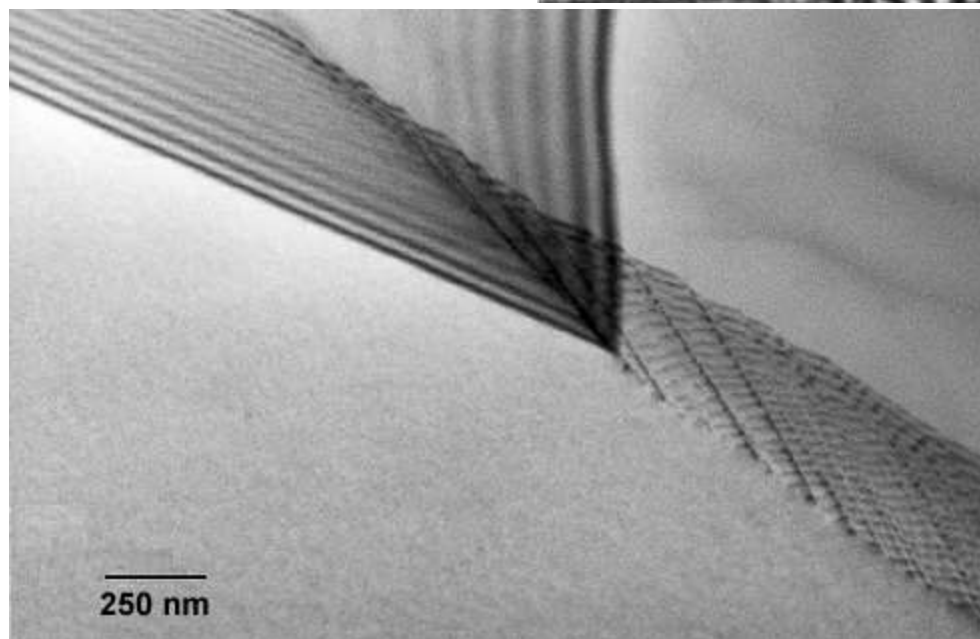
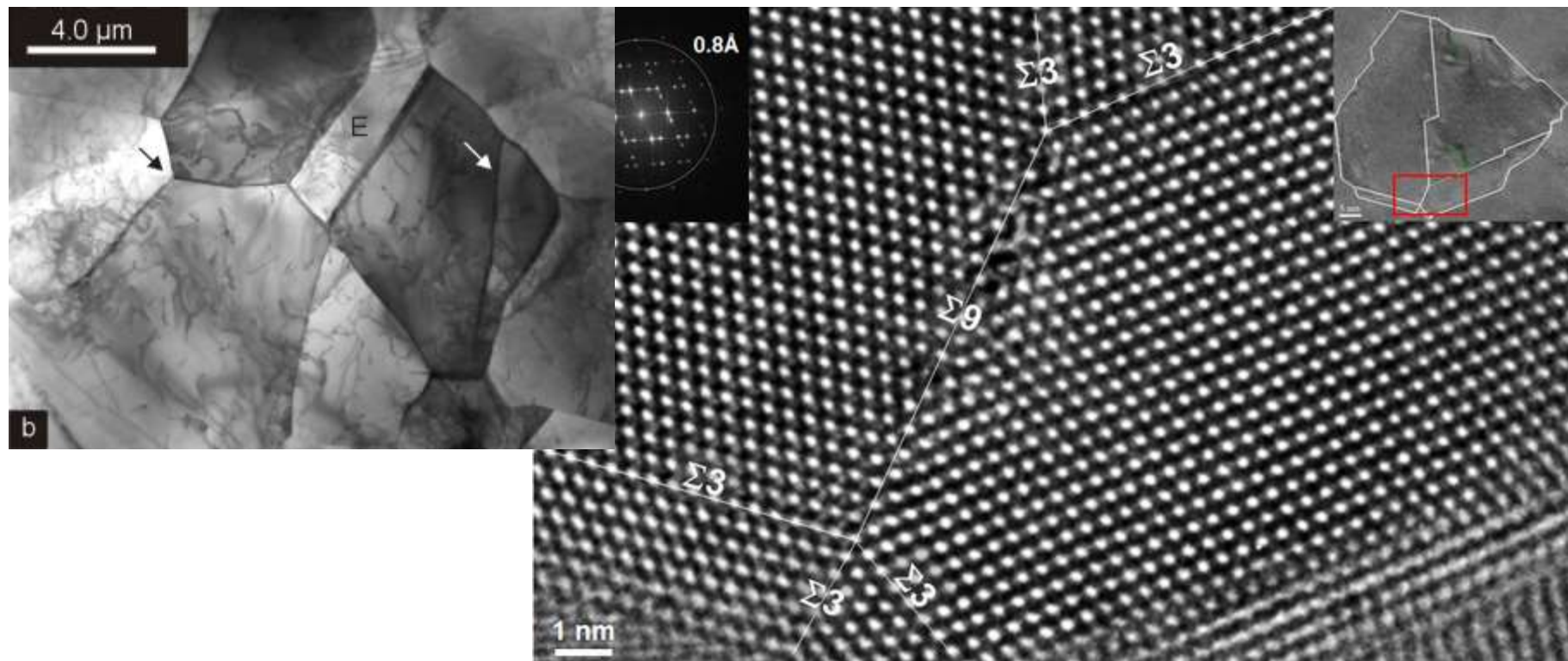


Figure 13-5. Group of stacking faults in 302 stainless steel stopped at boundary on left-hand side.



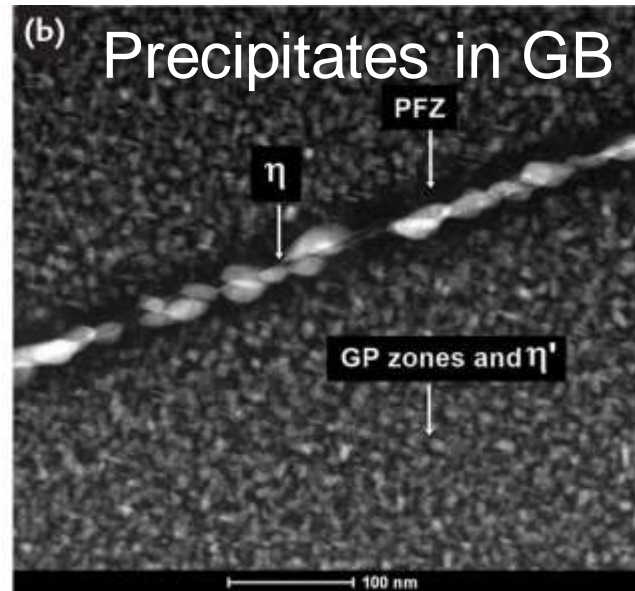
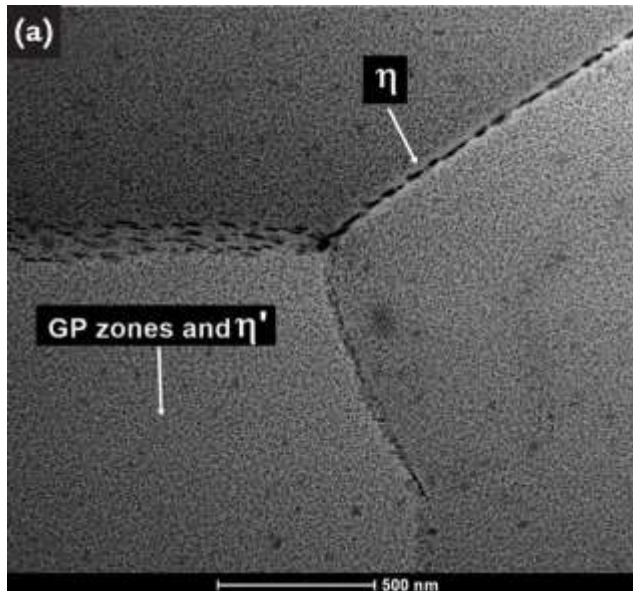
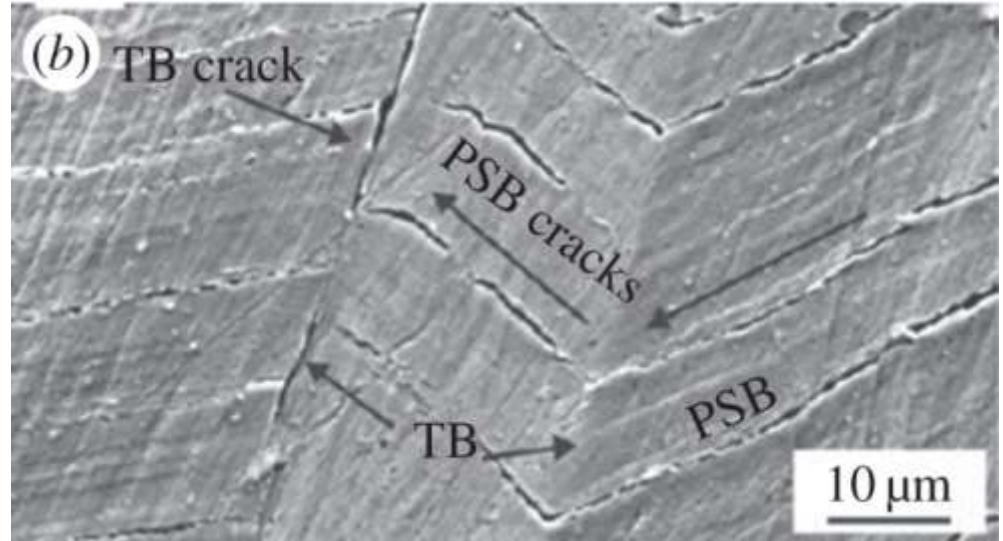
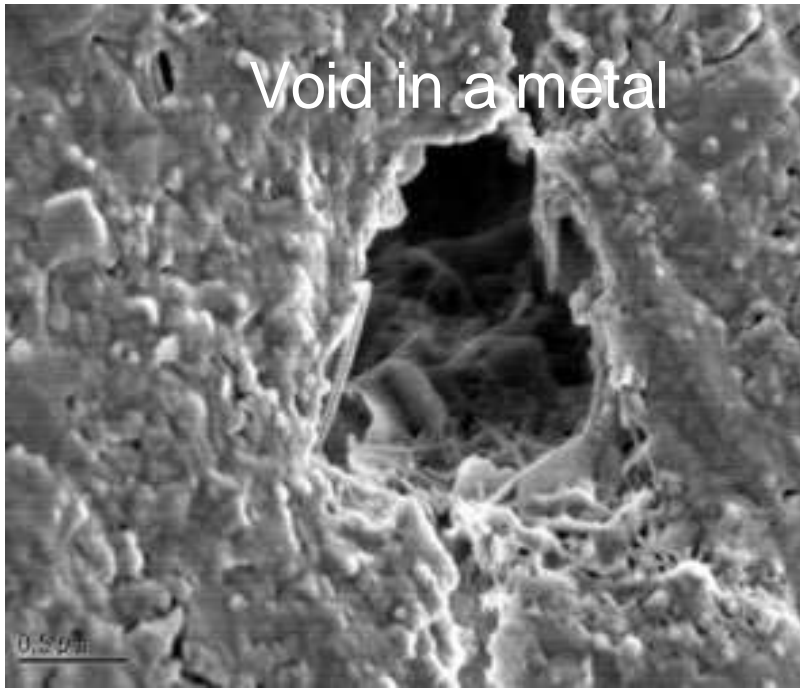
Role of surface defects on crack initiation

- Atoms located near grain boundaries have distorted surroundings – higher energy levels
- Every atom at an exterior surface is also having same situation
- Exterior surface is usually rough and contains tiny notches
- These notches initiates cracks that propagate along grain boundaries – intergranular failure...

Volume defects

- Voids, inclusions etc. present within the crystals are called volume defects
- Voids are regions where there are a large number of atoms missing from the lattice
- Voids due to air bubbles trapped during solidification – porosity
- Voids due to shrinkage of material as it solidifies - cavitation
- Inclusions – due to clustering of impurity atoms in the *molten metal...*

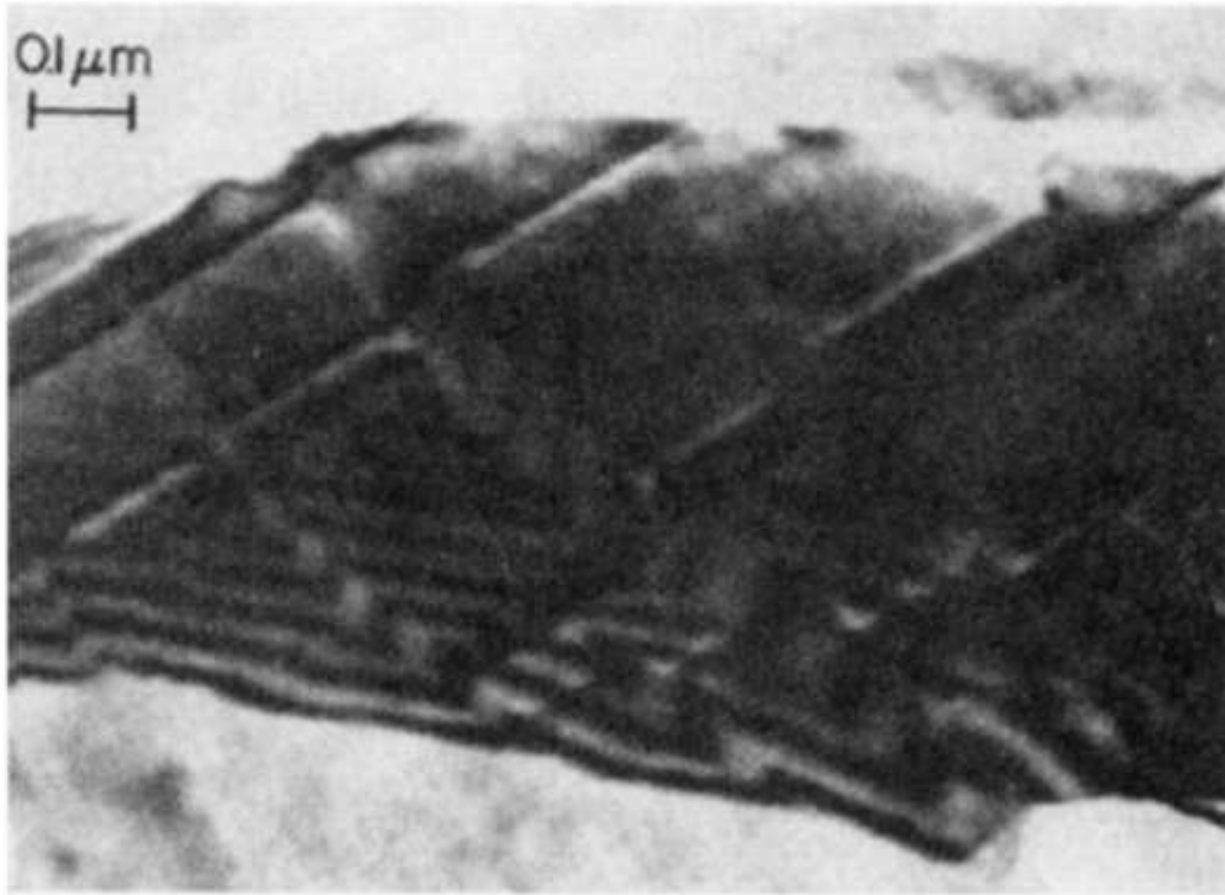
Void in a metal



Effects of grain boundaries on Dislocation and Creep

- A high angle grain boundary - a group of immobile dislocations which forms a step or grain boundary ledge
- As the angle of mismatch increases density of the ledge increases – good sources of dislocation
- Low angle boundary can be considered as a regular array of dislocations

Grain Boundary Ledges



Grain boundary ledges as observed by TEM. (Courtesy of L. E. Murr.)

- Moving dislocations tend to pile up at grain boundaries
- Pile ups introduce stress concentration ahead of the slip planes and generate new dislocations in adjacent grains
- More slip planes are in operation near grain boundaries – high hardness

- When grain size is reduced behavior near the boundary will also be felt at the centre and grains will have uniform hardness
- Fine grained material imposes more barriers to dislocation (greater grain boundary area) – higher strength and Hardness
- Effect of strain hardening will also be greater in a fine grained material

- **creep** is the tendency of a solid material to deform permanently under the influence of stresses
- occurs as a result of long term exposure to high levels of stress that are below the yield strength of the material
- Creep is more severe in materials that are subjected to heat for long periods, and near melting point
- Increases with temperature
- Creep is a "time-dependent" deformation

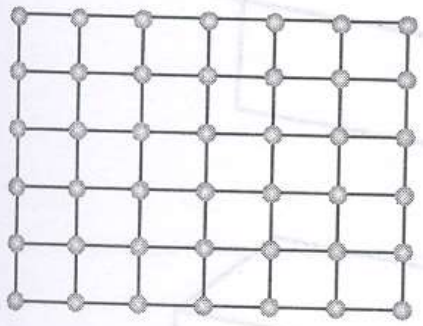
- Grain boundary sliding above $0.5 T_m$
- Becomes prominent with increasing temperature and deformation occurs by sliding along grain boundaries – leads to creep

Movement of dislocations

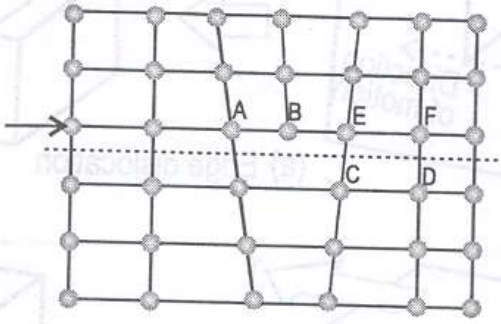
- Motion of a large no: of dislocations within a crystal – plastic deformation
- Dislocation movement types:
 - a) slip or glide
 - b) cross slip
 - c) dislocation climb

Dislocation slip or Glide

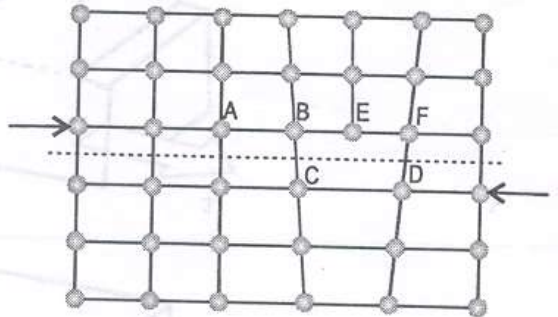
- Edge dislocation slip or glide in a direction perpendicular to its length
- Atomic bonds at one point are broken and re-established at another point
- The presence of dislocation makes the process easier than in a perfect crystal
- In crystals where dislocations are present atomic movement is relatively easy...



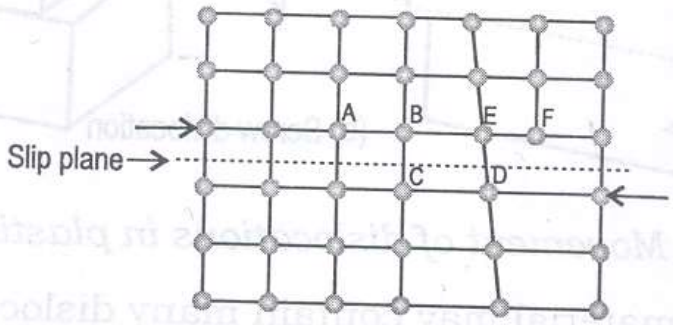
(a) Perfect Crystal



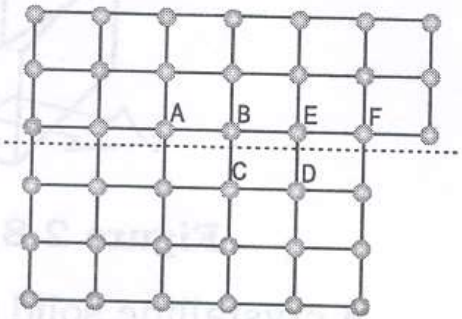
(b)



(c)



(d)



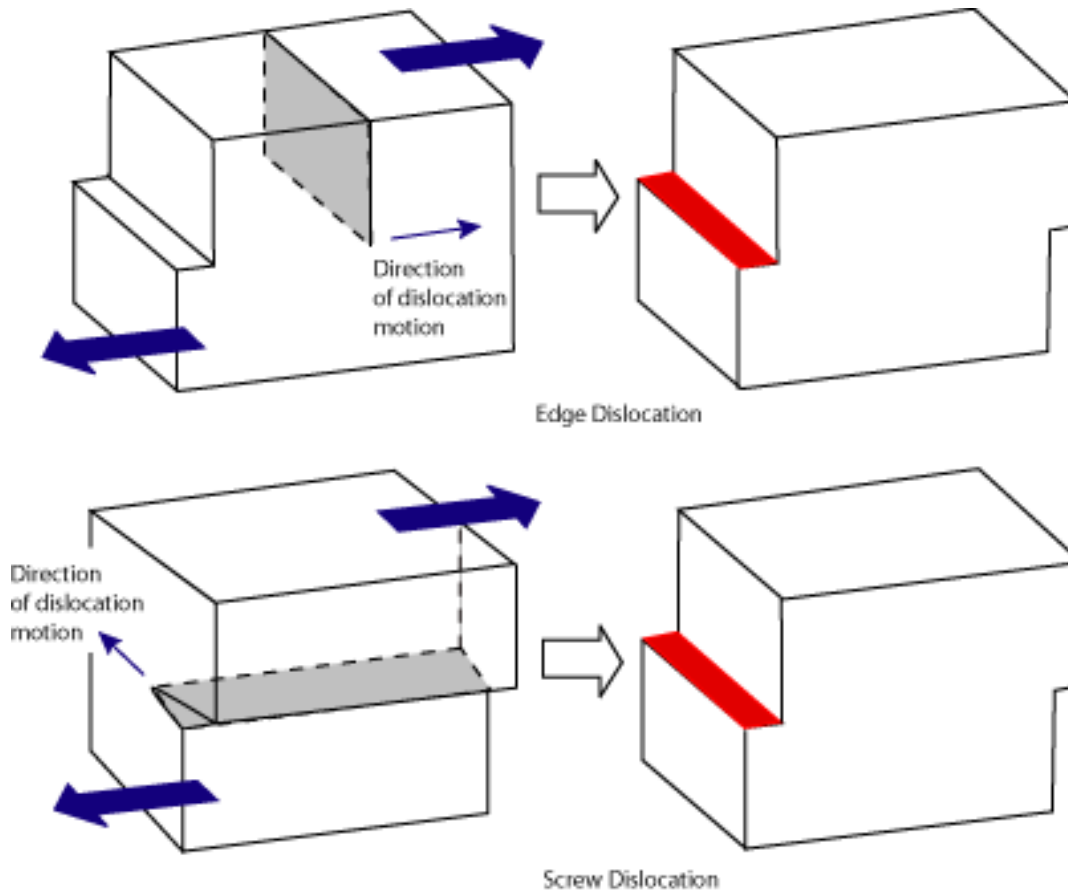
(e)

Crystal in which an edge dislocation is present and moving

Figure 2.7 Movement of dislocation

- Thus in real crystals plastic deformation is effected by the movement of dislocations
- Edge dislocations move perpendicular to the dislocation line but parallel to Burger's vector
- Screw dislocations move perpendicular to both dislocation line and Burger's vector

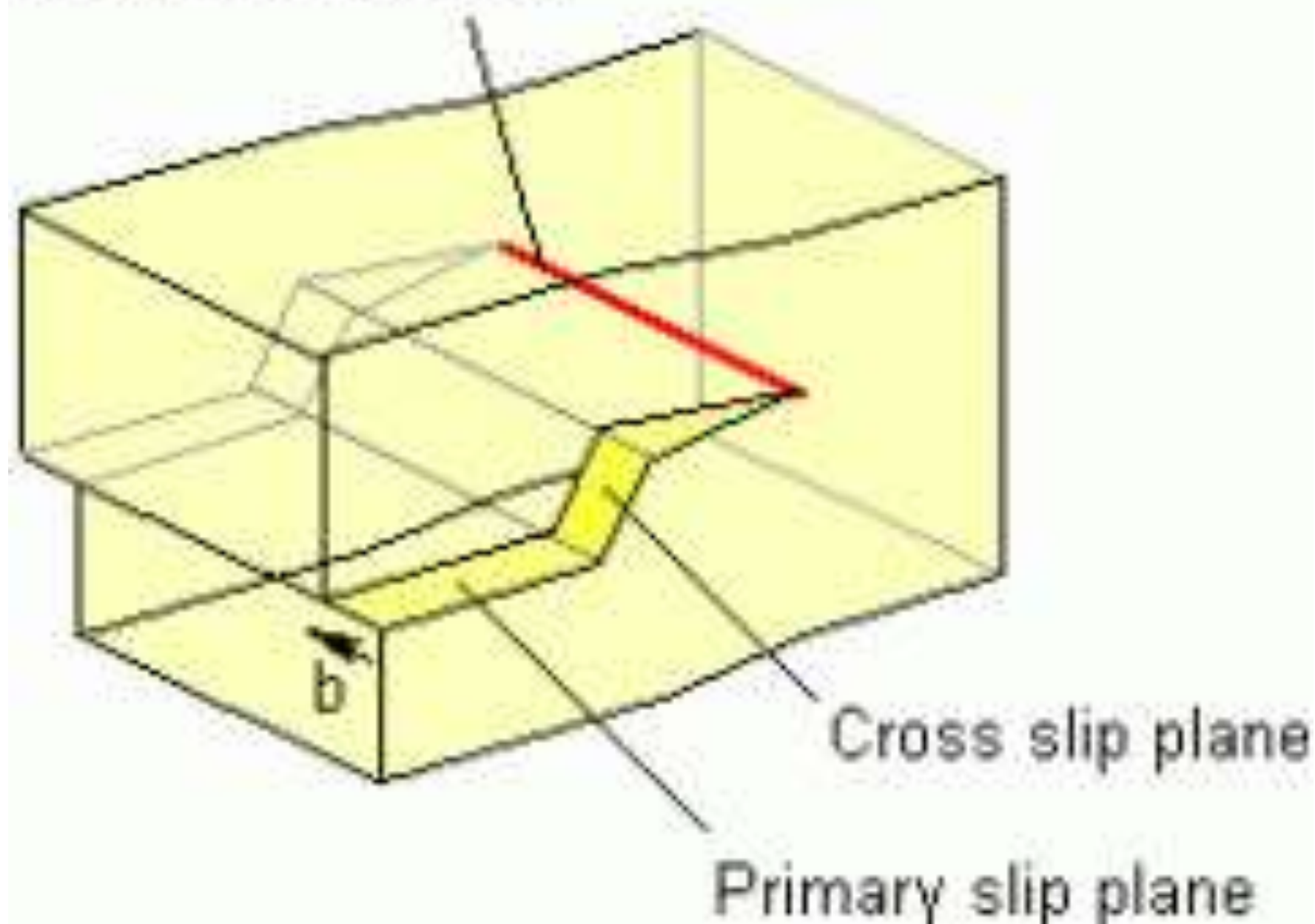
- Movement of dislocations may come across barriers
- Dislocation pile ups and stress concentration

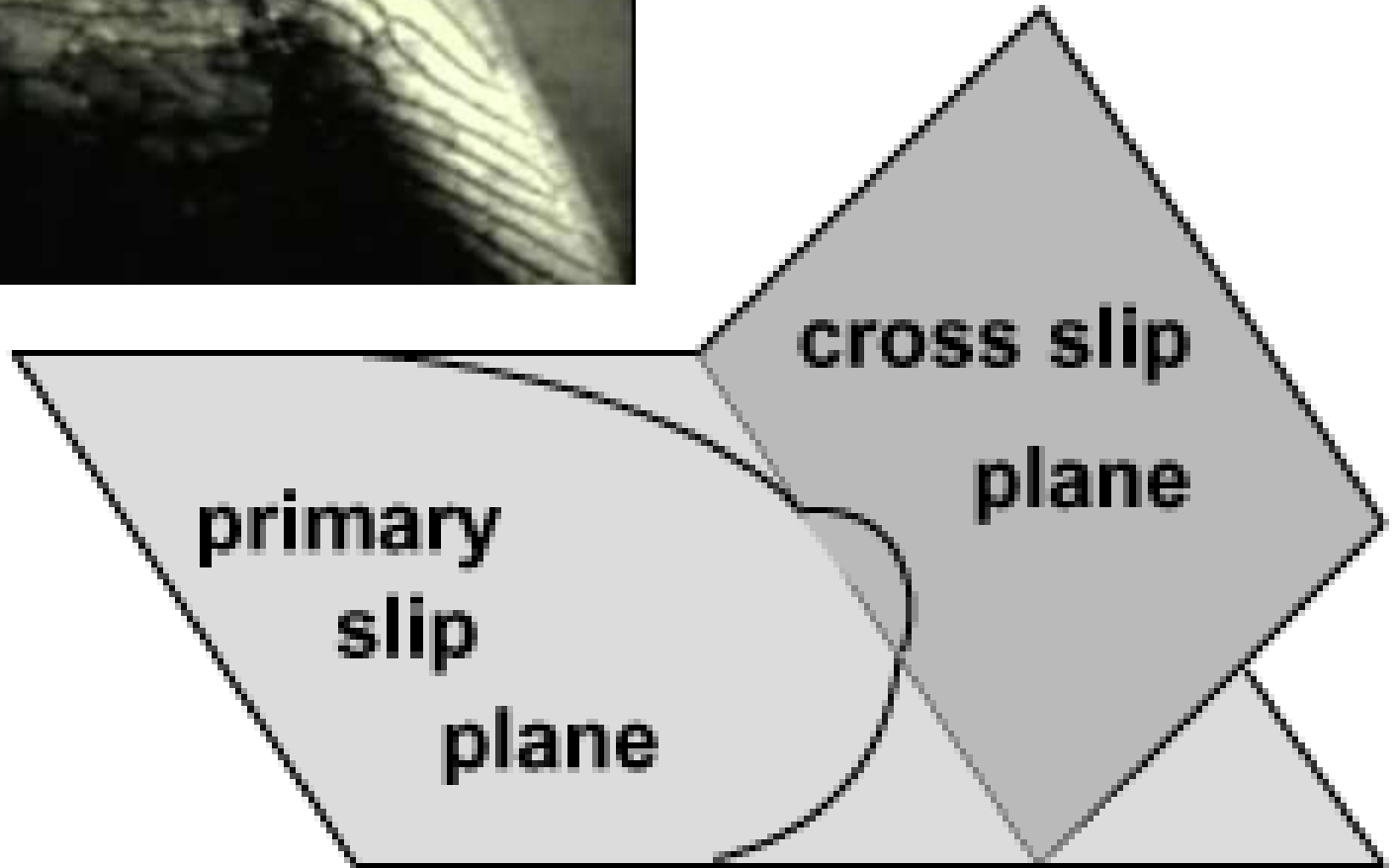
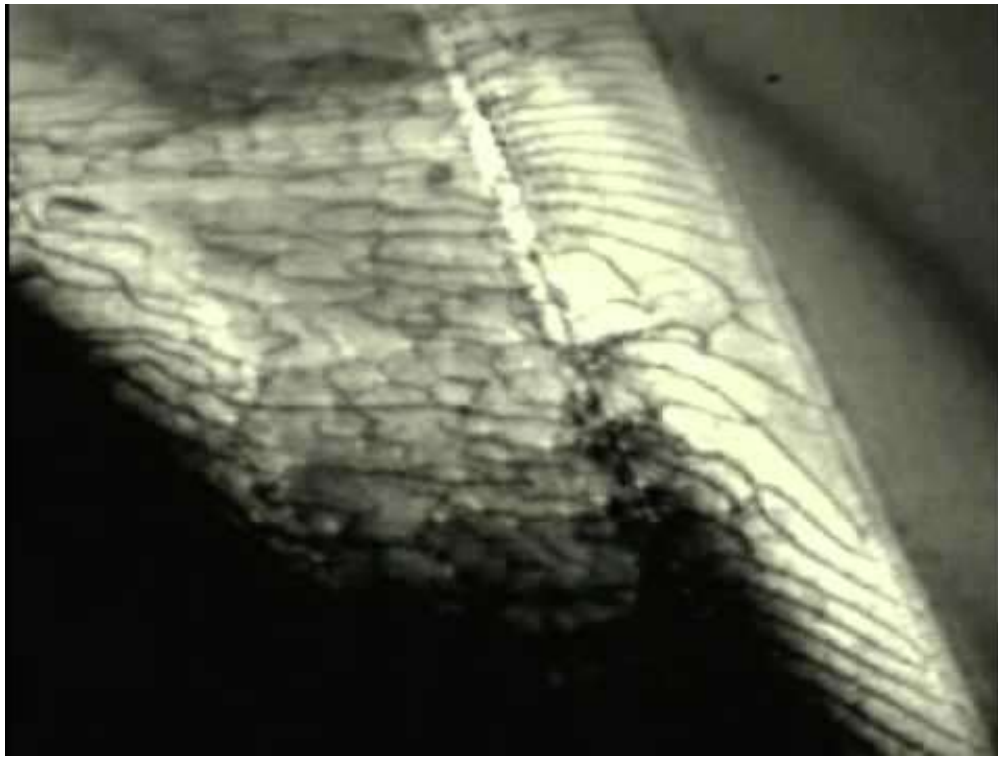


Cross Slip

- When a screw dislocation moving along a slip plane meets an obstacle, it is blocked from further movement along the plane
- The dislocation now shifts to another intersecting slip plane and continue to move along this plane – cross slip
- Occur only with screw dislocation
- Intersecting slip planes are present in FCC and BCC – hence more ductile than HCP
- In HCP slip planes are parallel, hence no cross slip

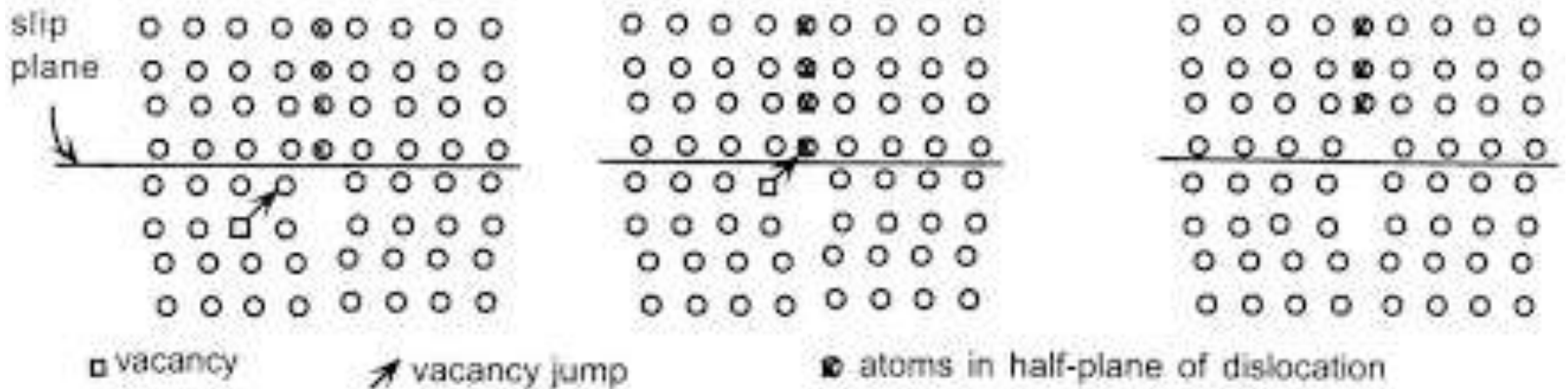
Screw dislocation





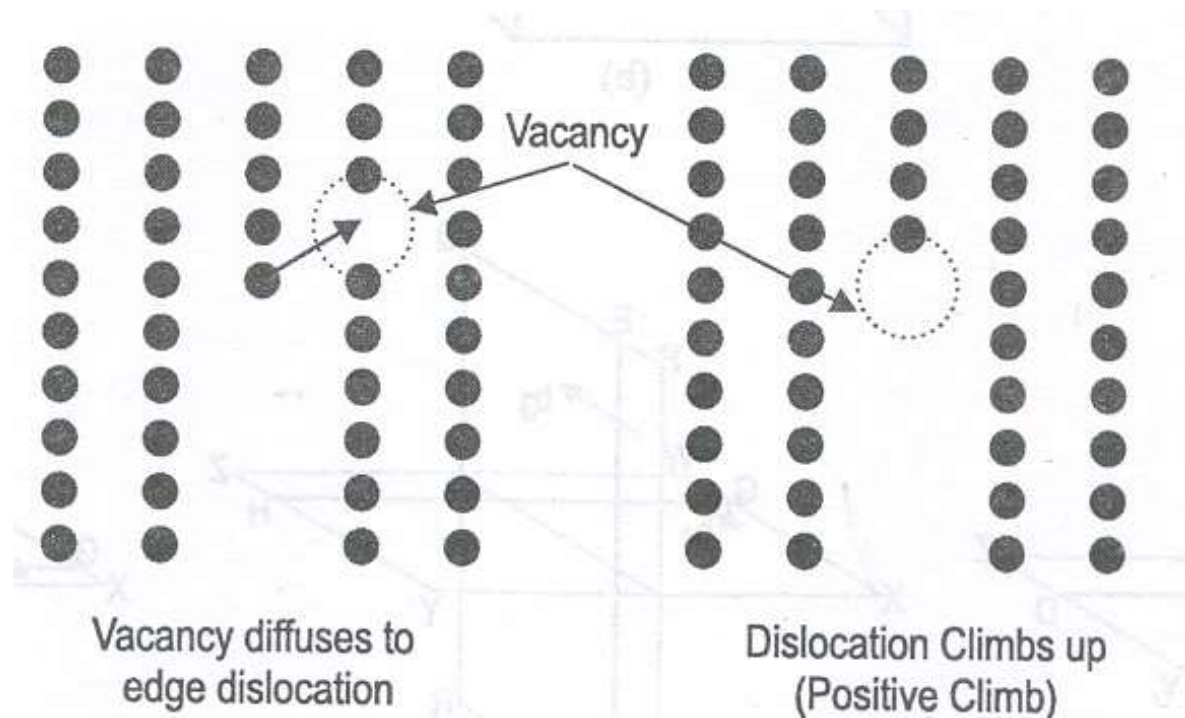
Dislocation climb

- Edge dislocation can move out of its slip plane to another parallel slip plane above or below it – dislocation climb
- Climb up (+ve climb) and climb down (-ve climb)

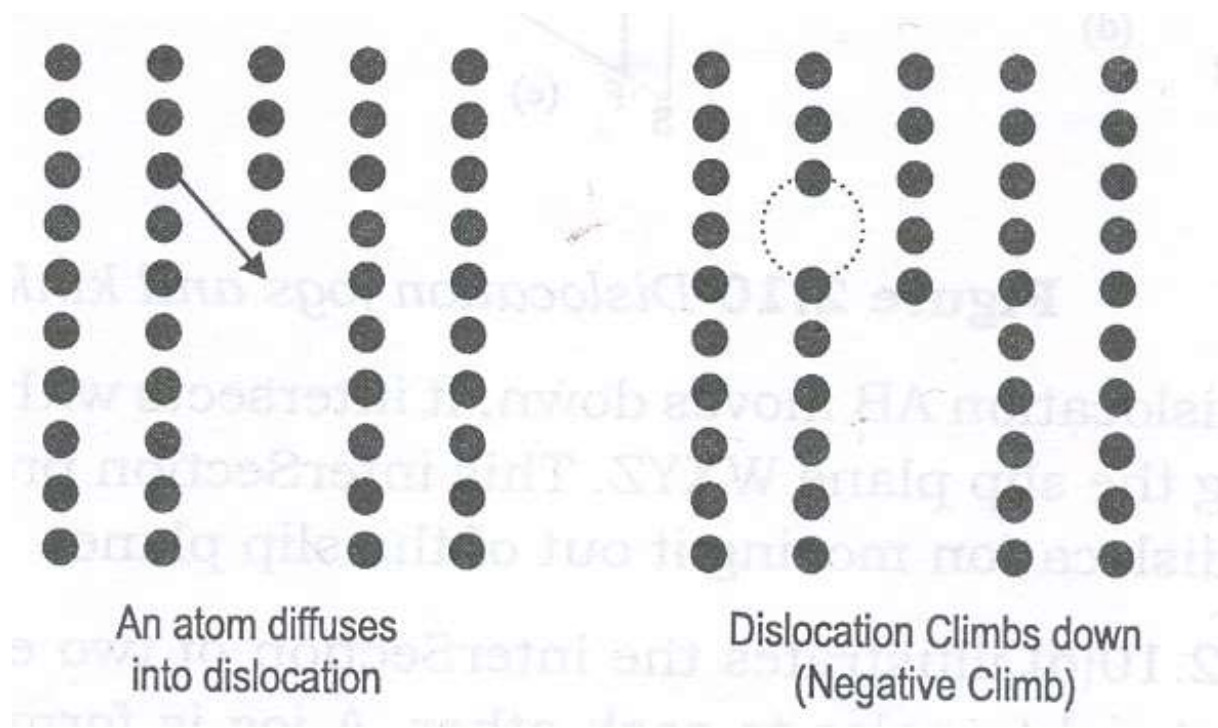


Dislocation Climb

- Climb up (+ve climb)
- Occurs by diffusion of vacancies to the site of dislocation

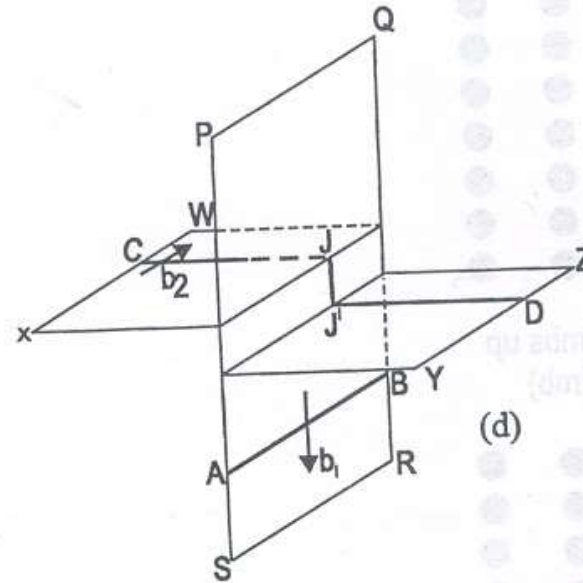
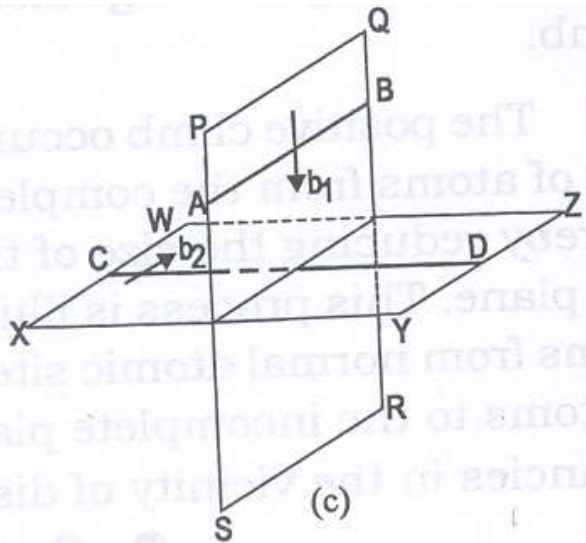
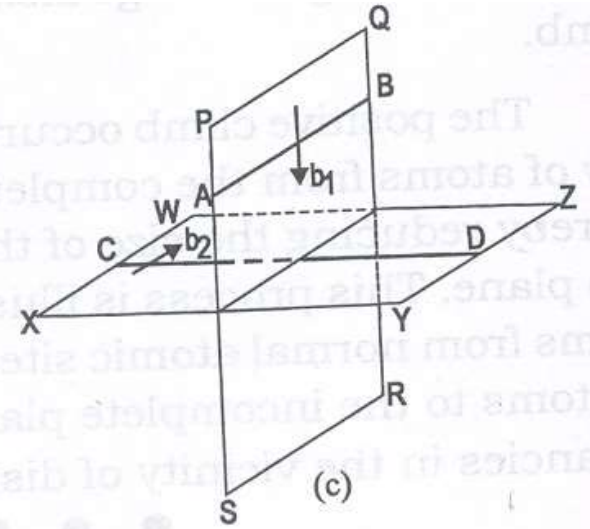
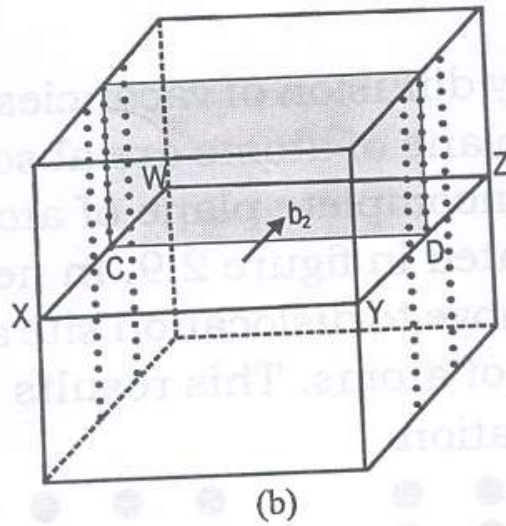
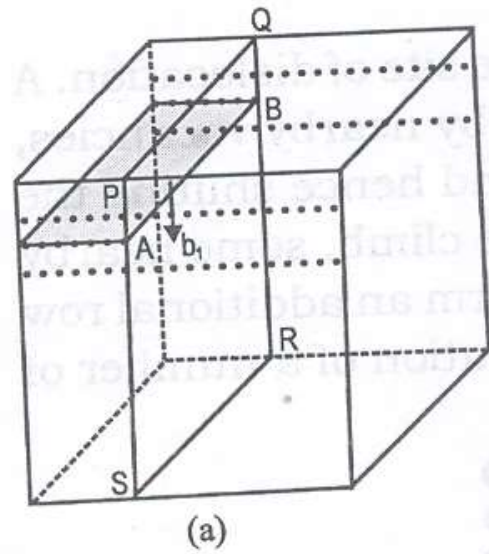


- Climb down (-ve climb)
- Some nearby atoms from normal atomic sites move to dislocation site

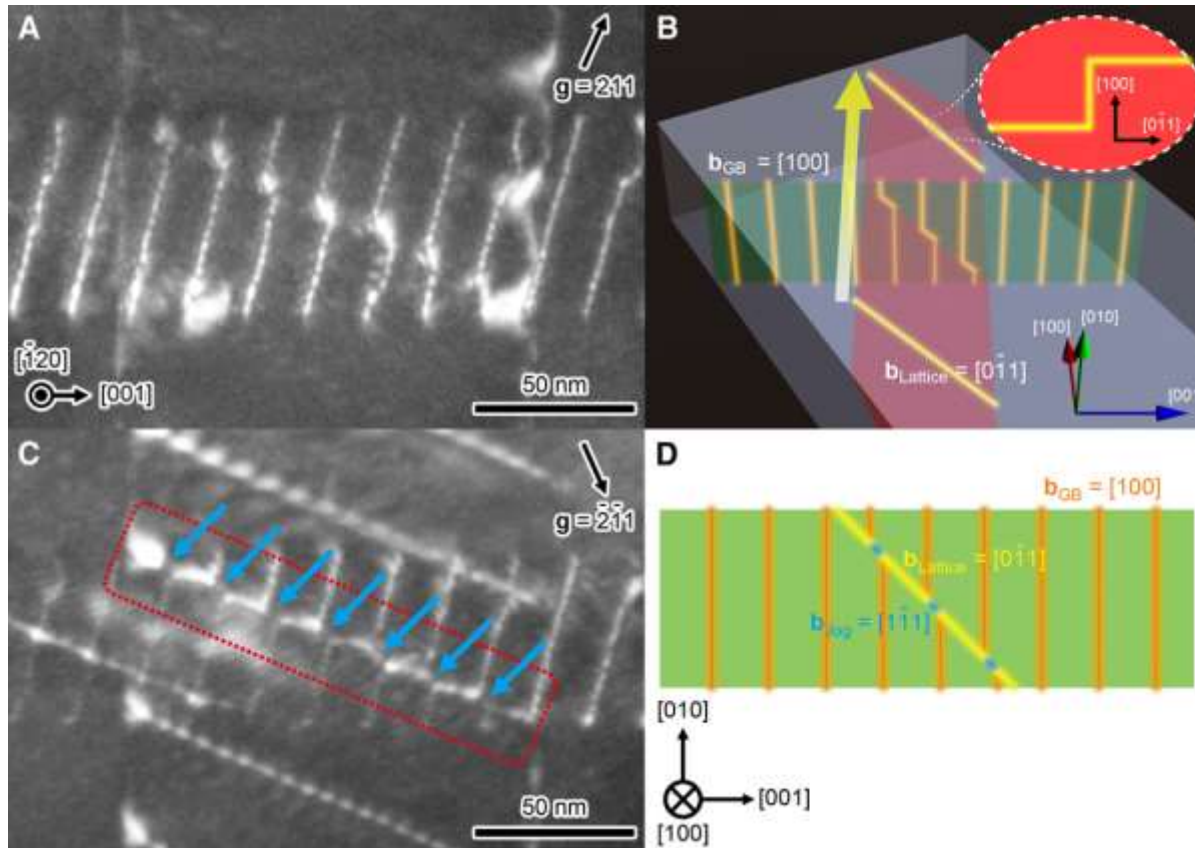


- Dislocation climb is due to self diffusion and is controlled by the temperature level
- At higher temp diffusion, hence climb faster
- Not possible with screw dislocation, since no incomplete plane of atoms involved

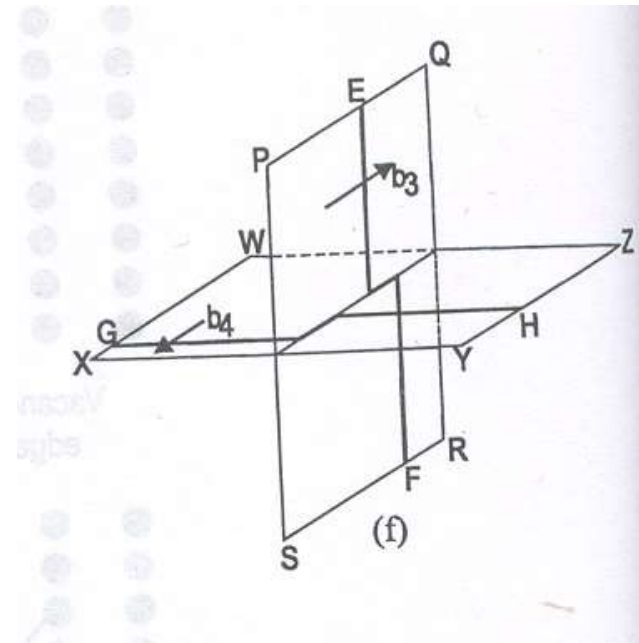
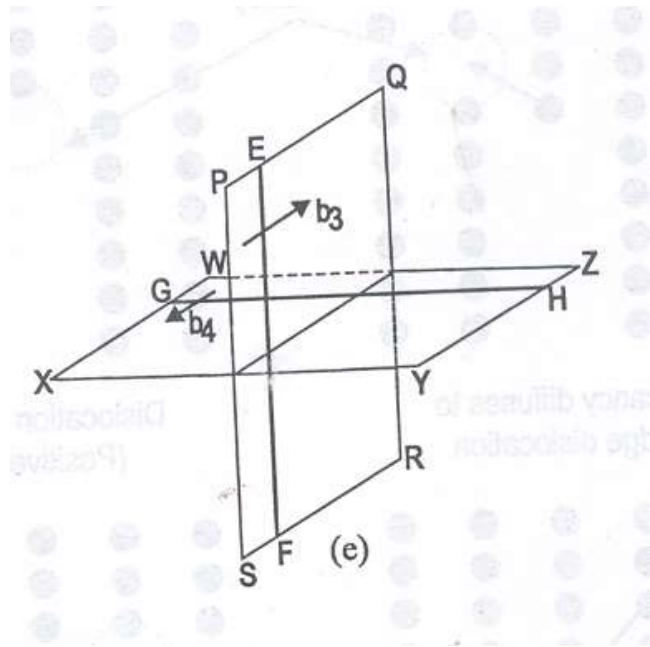
Jogs and Kinks



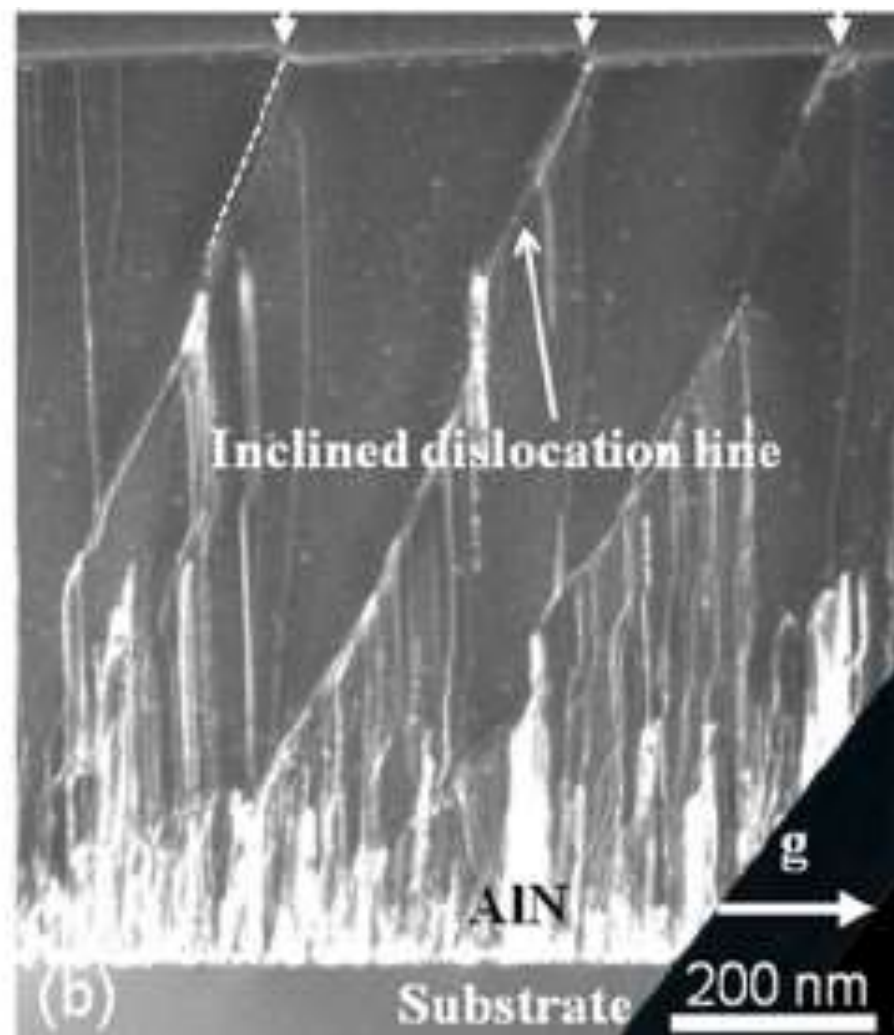
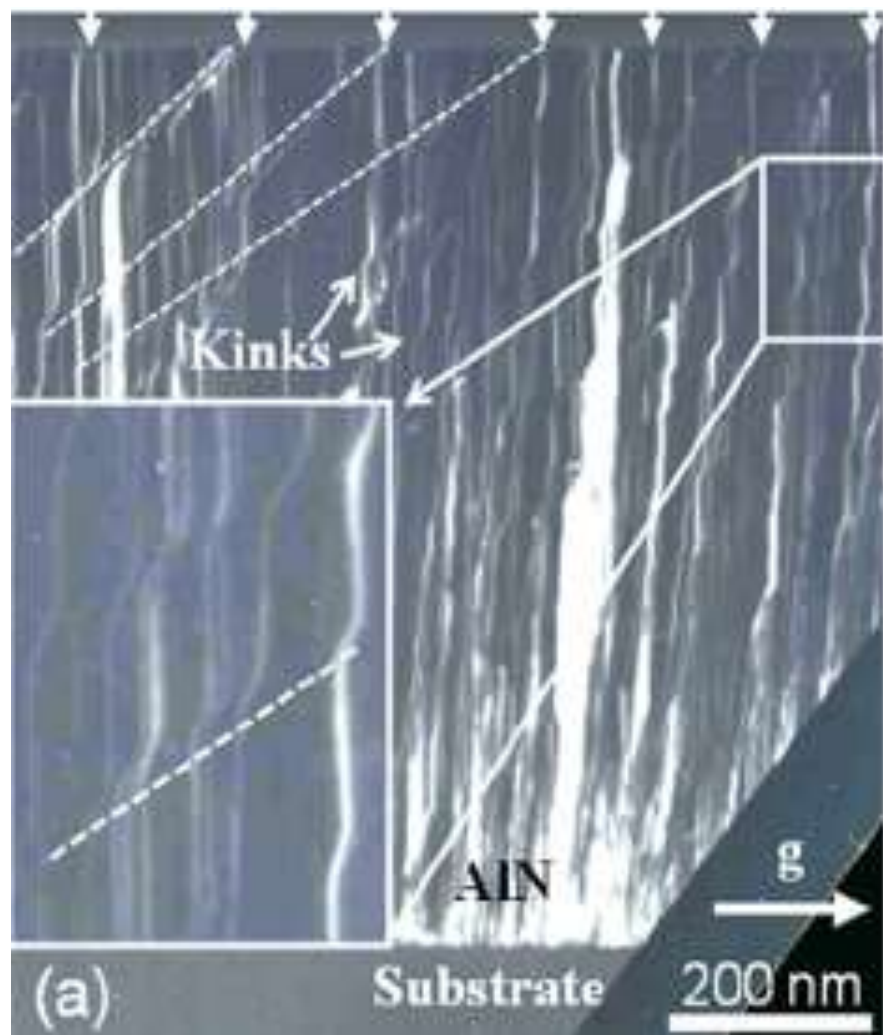
- A jog is formed when the Burger's vector of the intersecting dislocation is normal to the other dislocation line
- b_1 is normal to CD, hence jog is formed in CD
- b_2 is parallel to AB, no jog is formed in AB
- Resulting jog is parallel to b_1 and its length equal to b_1 ...



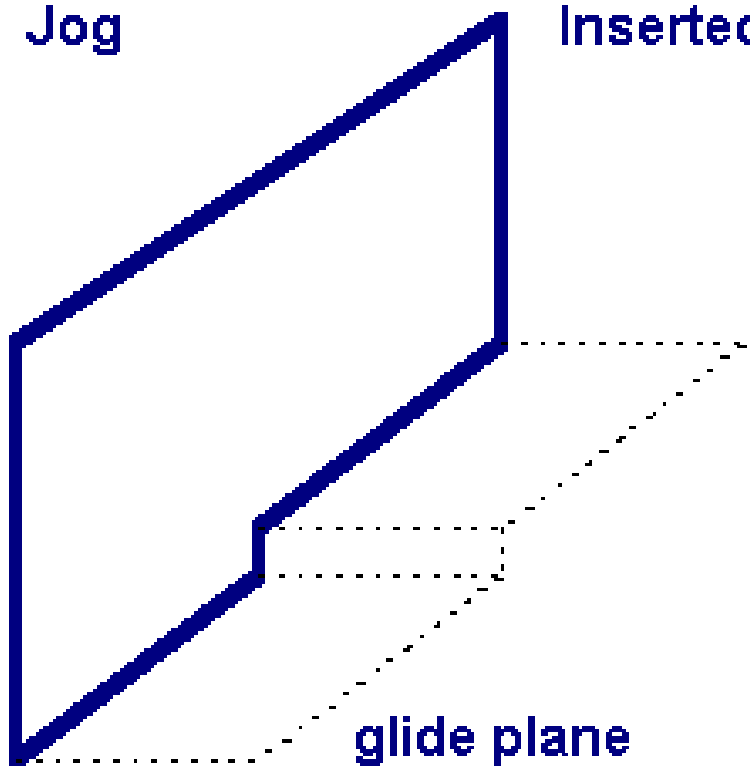
Kinks



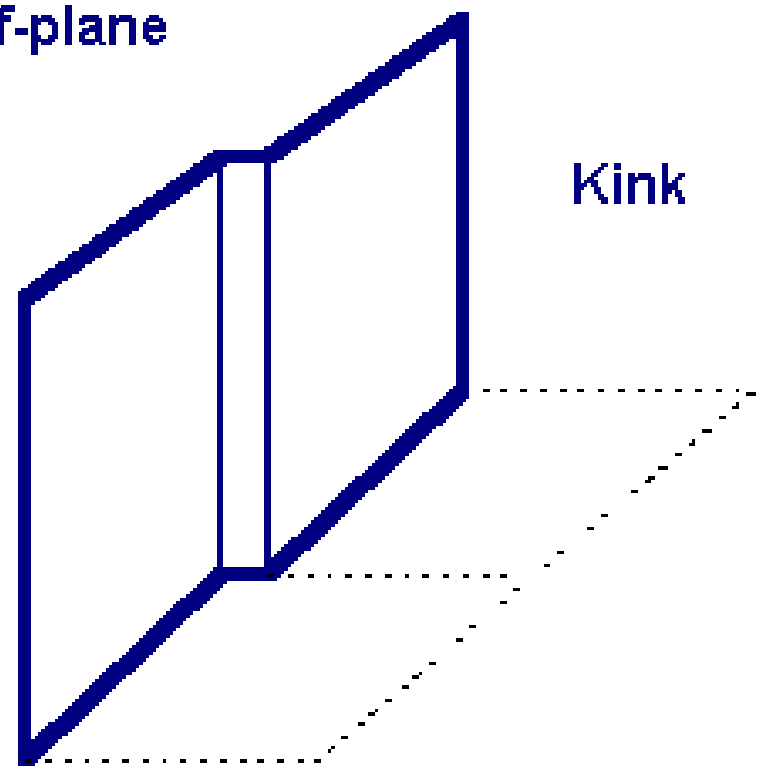
- When Burger's vectors of intersecting dislocations are parallel to each other, jogs will be formed on both the dislocations
- These jogs will lie on the original slip planes - Kinks



Jog



Inserted half-plane

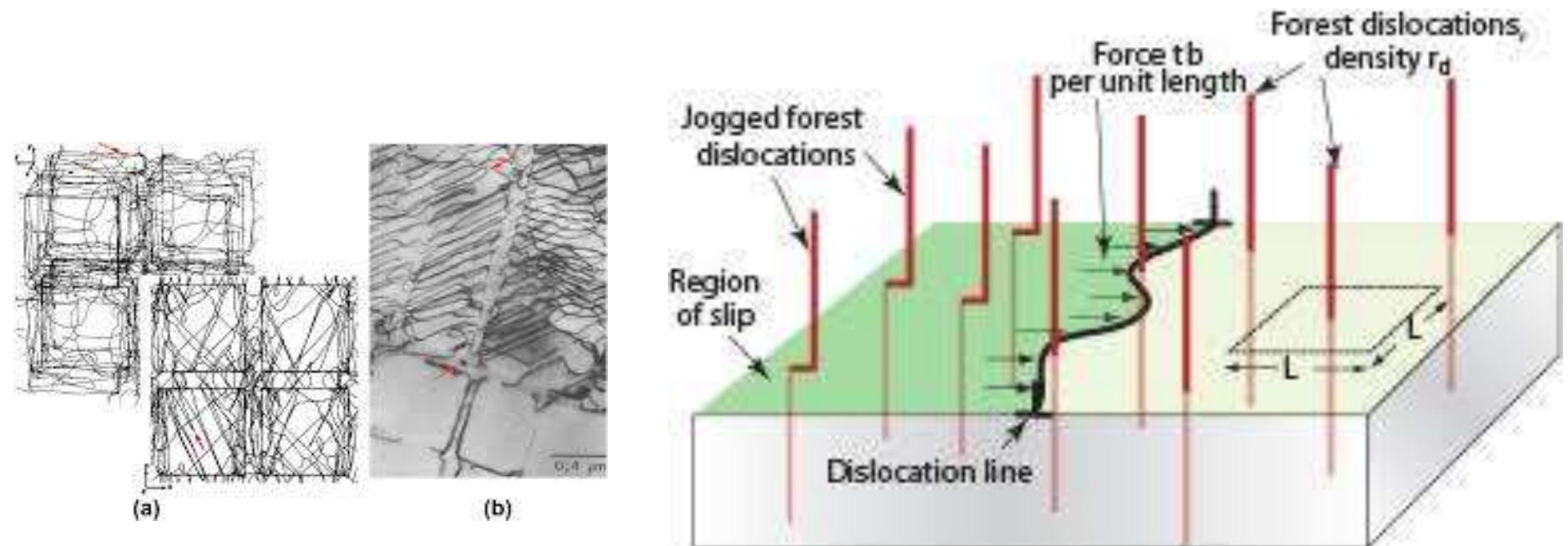


Kink

glide plane

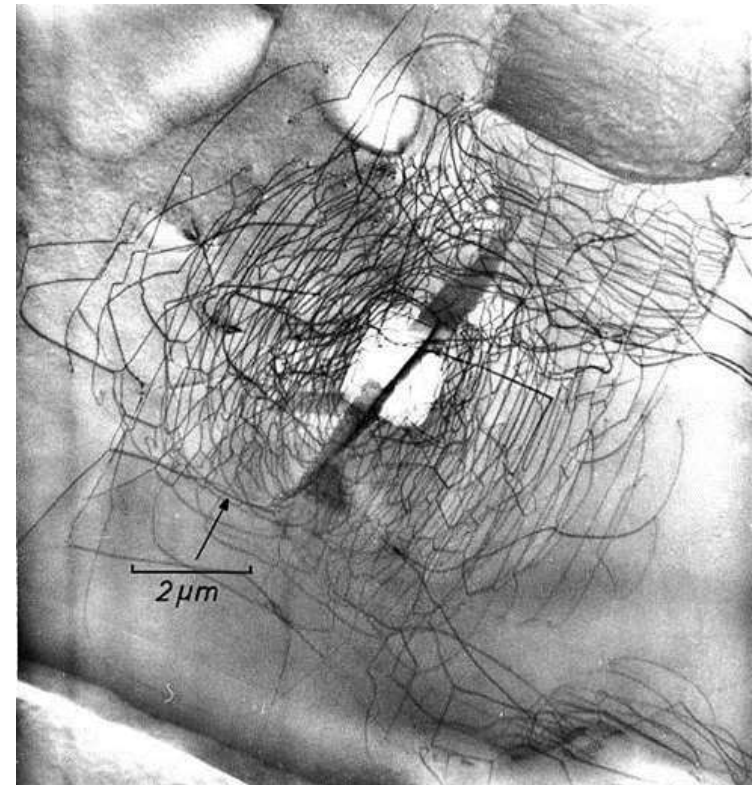
Forest of Dislocations

- Dislocations moving one behind the other along an active slip plane
- When a dislocation forest intersect with other dislocations, jogs are formed – restrict further movement of dislocations



Dislocation density

- Total length of dislocation lines per unit volume
- OR the no: of dislocation lines that move across a unit c/s area
- Usual unit is mm^{-2}
- Cold working and annealing affect dislocation density
- Completely annealed material... 10^4 to 10^6 mm^{-2}
- Cold worked material... 10^8 to 10^{10} mm^{-2}



Correlation of Dislocation density with strength

- Usually all crystalline materials have a certain level of dislocation density
- Cold working increases dislocation density, but leads to strain hardening
- Strength of a material is inversely related to mobility of dislocations
- Even though cold working increases dislocation density, strain hardening imposes barriers on mobility – increase in strength...

Nano concept

- In nano crystalline metals the grain size is very small
- This leads to a considerable degree of strengthening in nanocrystalline materials.
- The additional increment of strengthening generated through strain hardening is relatively small
- The yield strength of nanomaterials is also determined by the ease with which dislocations can be moved through the material...

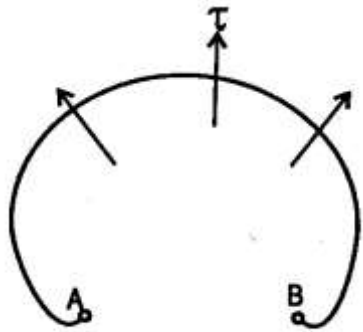
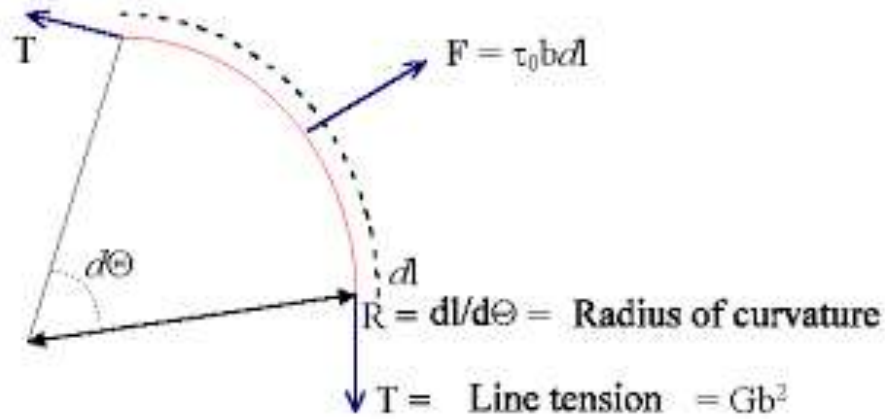
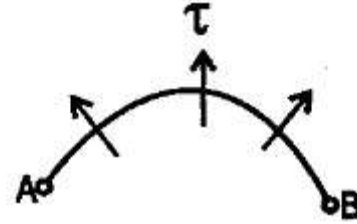
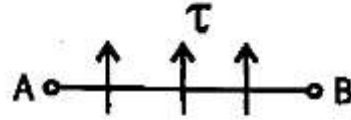
Sources of Dislocation

- Large no: of dislocations are necessary in a crystal to facilitate plastic deformation
- Originated through different sources
- Natural consequence of crystal formation and growth
- Even in nearly perfect crystals there is an equilibrium concentration of imperfections including dislocations

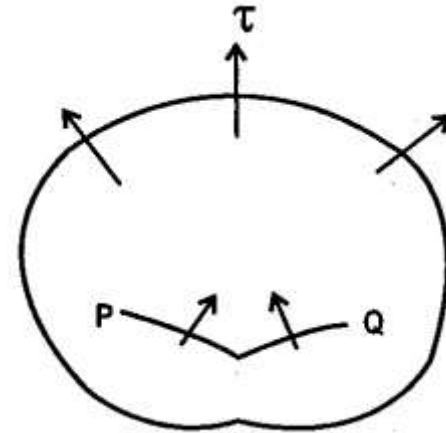
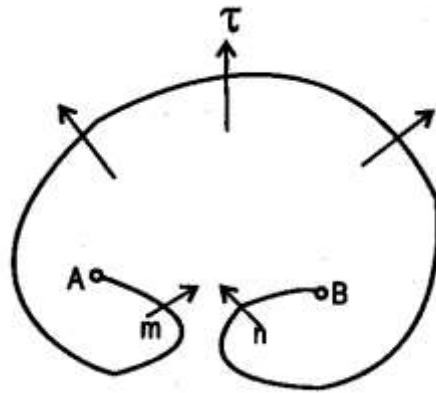
Frank-Read Source

- During plastic deformation dislocation density in the crystals are increased by 2 to 6 orders of magnitude depending upon the amount of deformation undergone by the crystal
- ie, there must be a mechanism of generating dislocations in a cold worked metal
- A source from which dislocations can be generated from existing dislocations is known as **Frank-Read source**

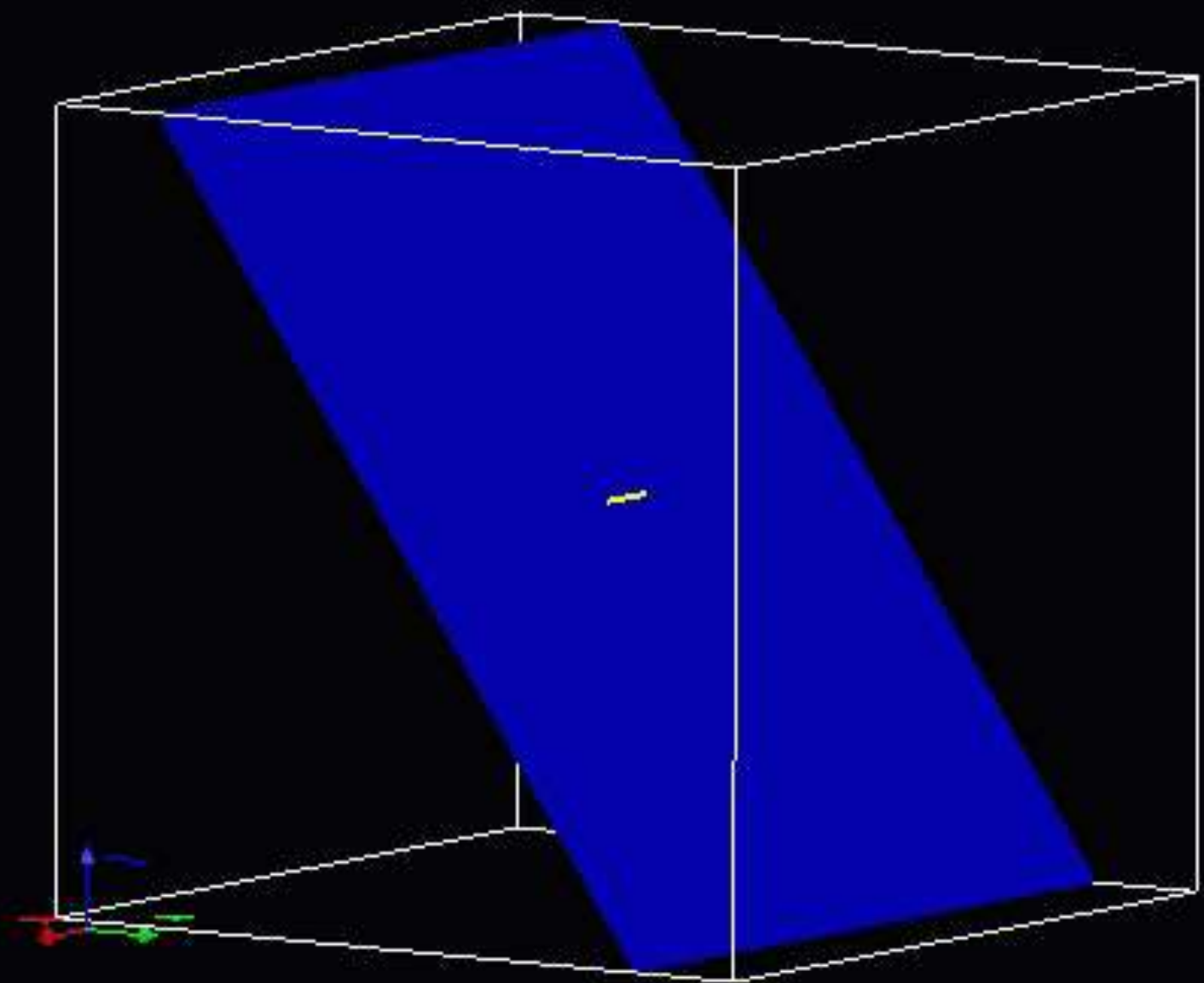
Frank-Read source

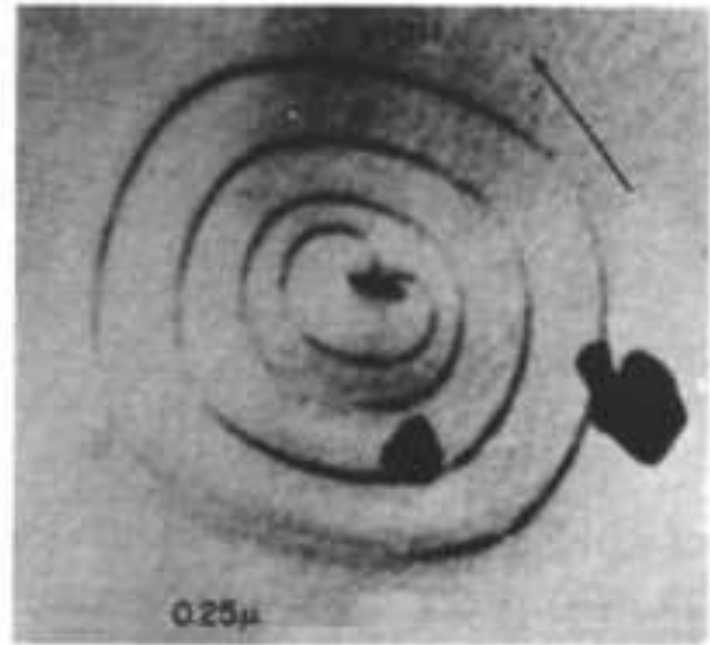
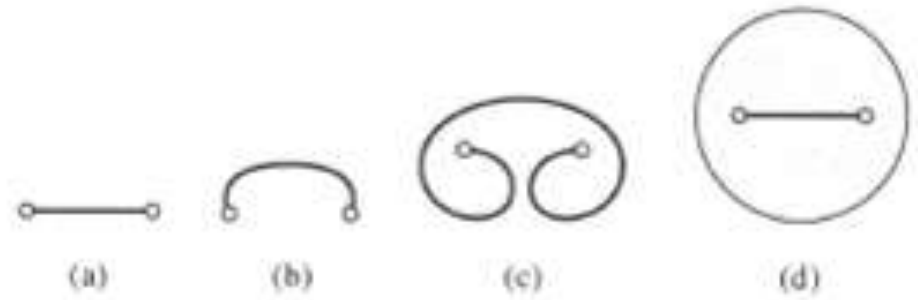
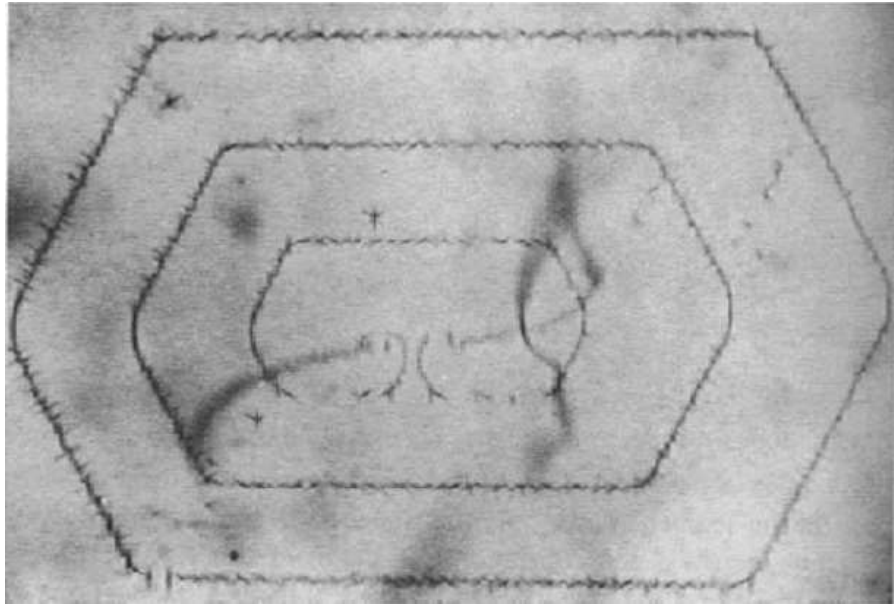


(c)



0 [step] 0.100E-08 [sec/step] 0.0000000E+00 [sec]





(e)

- A Frank-Read source can operate continuously and produce an infinite no: of loops, provided the loops move out and disappear at the surface of the crystal
- But if the loops piled up against an obstacle, back stresses build up at the source and it gradually dies out

Microstructure Determination

- Study of microstructure of metals by microscopic examination using optical or electron microscopes – Metallography
- Reveal the structure of grains, grain boundaries, line and surface defects etc.
- Their effect on material behaviour can also be studied
- Specimen preparation

Specimen preparation stages

1. Selecting and cutting the specimen
2. Rough grinding
3. Intermediate and fine grinding
4. Polishing
5. Etching

1. Selecting and cutting the specimen

- True representative of the material being studied
- Specimen should be from the regions of specific interest
- Cut the specimen using a saw or abrasive wheel
- Ensure, specimen is not heated up
- Size 10-15mm diameter or square
- Mounting – thermoplastic resins

Specimen moulding machine



Moulded specimens



2. Rough Grinding

- To make the surface absolutely flat
- File, grinding wheel, motor driven emery belt
- Ensure, specimen is not heated up
- Wash – to remove coarse filings and abrasive particles

3. Intermediate and fine grinding

- Sequence of emery papers of progressively finer abrasive grit size
- 220, 320 ,400 and 600 (coarse to fine)
- Rubbing against emery paper
- One direction
- Grinding process
- OR m/c grinding
- Wash
- Fine grinding – fine grit emery papers (1/0, 2/0, 3/0, 4/0)
- Shining and scratch free surface

Specimen grinding machine



4. Polishing

- To remove very small parallel grooves
- Polished by means of a rotating cloth pad pasted with a suitable polishing medium
- Wheel rotation – specimen movement
- Harder specimens (iron, steel) – 2 stage polishing
- 1st stage using polishing agents with particle size 6 microns
- 2nd with 0.5 to 1 microns
- Diamond powder, alumina powder, magnesia powder
- Mirror like surface
- Washed and examined under microscope

5. Etching

- To remove 'flowed' or amorphous layer of metal on the polished surface
- An etchant is used to dissolve this layer and remove it
- Cleaning before etching and drying
- Dip and agitate the specimen in etching reagent for a few to several seconds
- Quickly washed in running water

- Examined with naked eye
- Bright appearance is lost and become uniformly dull
- If not successful, repolishing and etching again
- Examined immediately before oxidation

Optical microscope

- 3 optical systems
 1. illuminating system
 2. objective lens
 3. eye piece
- Max: magnification 1000x

Optical microscope



Scanning Electron microscope

- When higher magnification is needed electron microscopy is used
- Instead of light rays electron beams used
- Specimens need not be polished or etched but should be electrically conductive
- Non conductive materials thin metallic coating will be sufficient
- In SEM the surface of specimen is scanned with an electron beam and the reflected beam is displayed using a CRT
- 50,000x and 10^{-4} to 10^{-8} resolution



Transmission electron microscope

- Electron beam is passed through the specimen
- Specimen should be in the form of a thin foil
- The differences in scattering beams due to different elements of the microstructure results in contrasts in images obtained
- 1,000,000x and 10^{-4} to 10^{-10} resolution
- Short wavelength (3.7×10^{-3} nm) of electron beams makes it possible to have higher magnifications and resolving powers

Specimen preparation

- Using a low speed diamond cutter, wafers of 250 micro meter are sectioned from a bulk material
- Ground to thickness of 125-150 micro meter
- Final thinning by chemical and electrochemical method or by ion milling

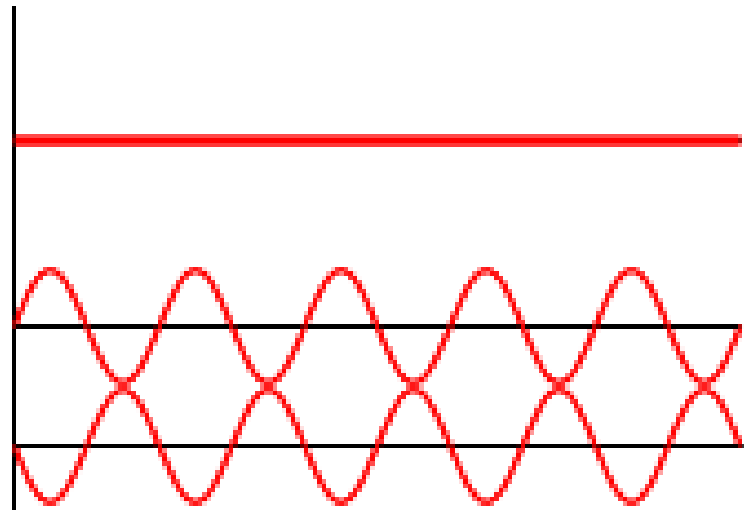
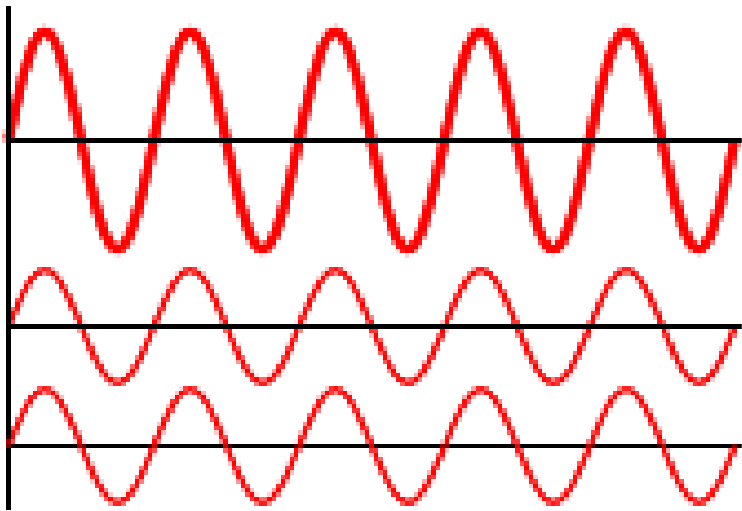
Dislocations, stacking fault, etc can be studied



Crystal structure determination

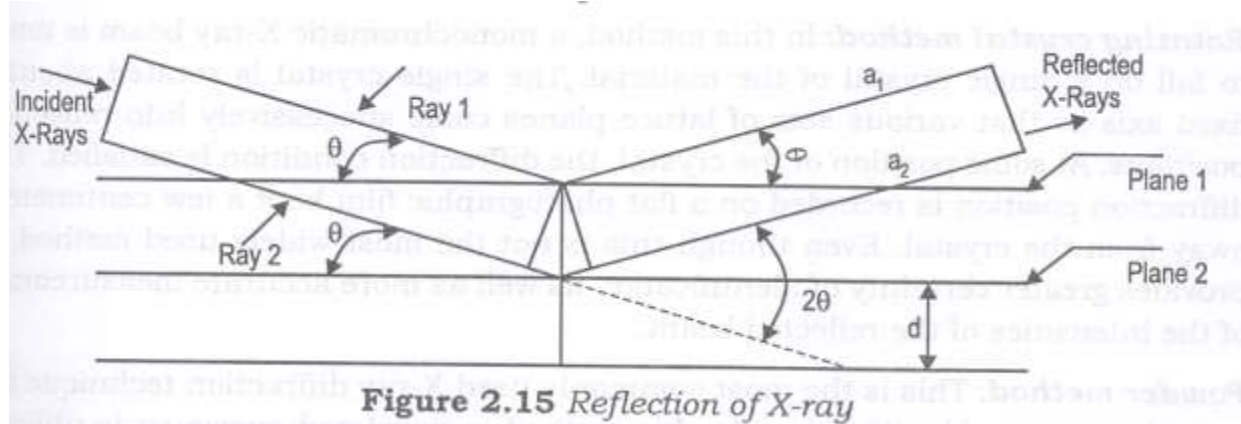
X – Ray diffraction

- X-ray diffraction



Crystal structure determination

- X-ray diffraction



- $n\lambda = 2d\sin\theta$Bragg's law
- Constructive interference – reflected beam leaves the crystal as a narrow pencil of rays capable of producing sharp images on a photographic film
- By analyzing the data crystal structure of the metal can be found

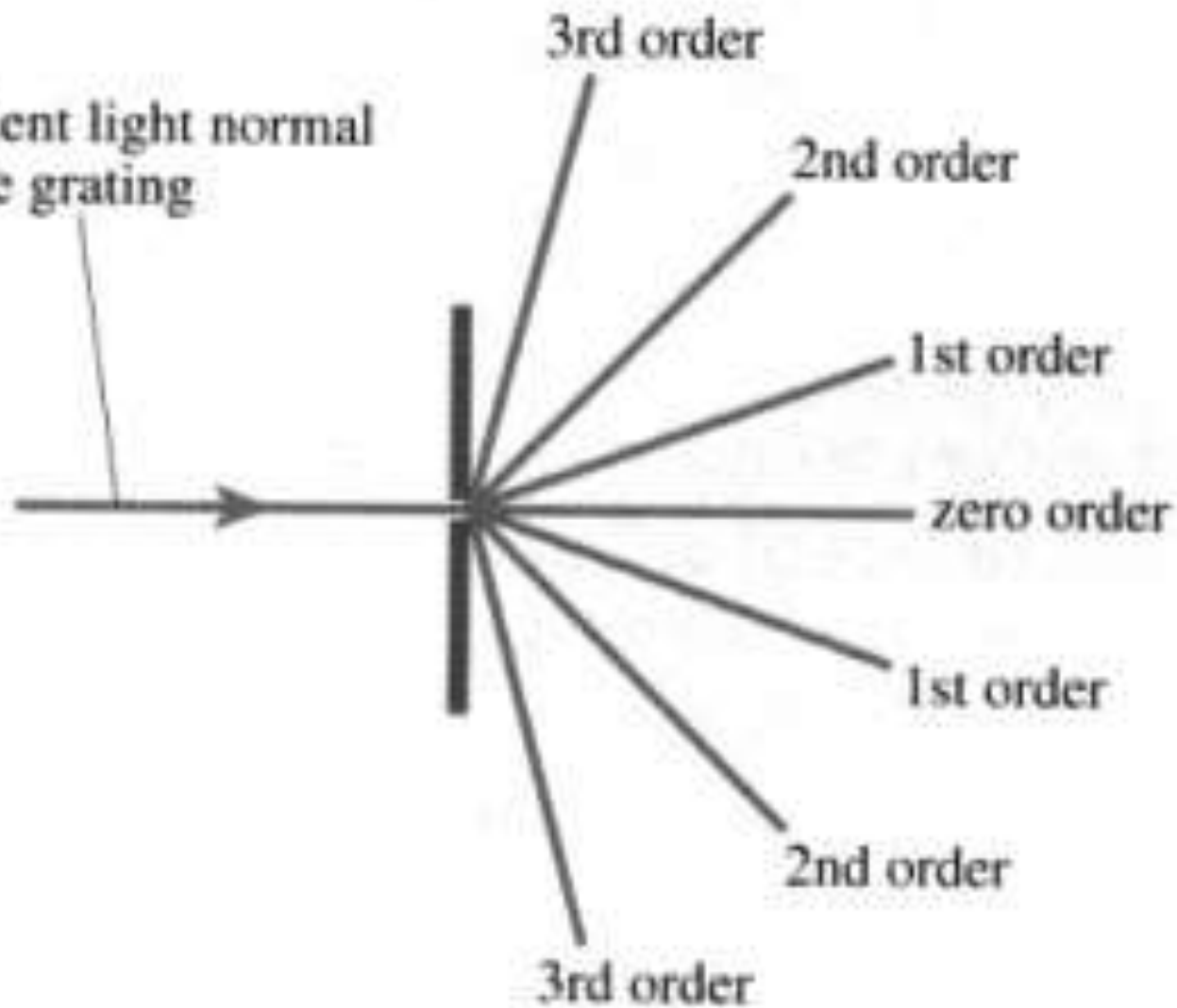
$n\lambda=2d\sin\theta$Bragg's law

In most cases first order diffraction is used
where $n=1$

Hence Bragg's law takes the form

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

incident light normal
to the grating



X-Ray Diffraction techniques

- Laue method
- Rotating crystal method
- Powder method

Laue method

- A single crystal is held stationary on the path of the beam
- A white radiation is directed at the crystal
- The right combination will lead to the proper diffraction pattern

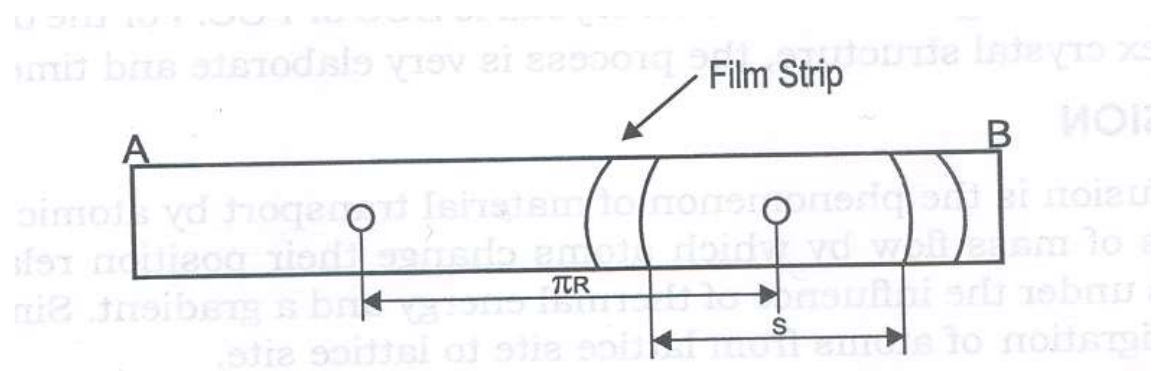
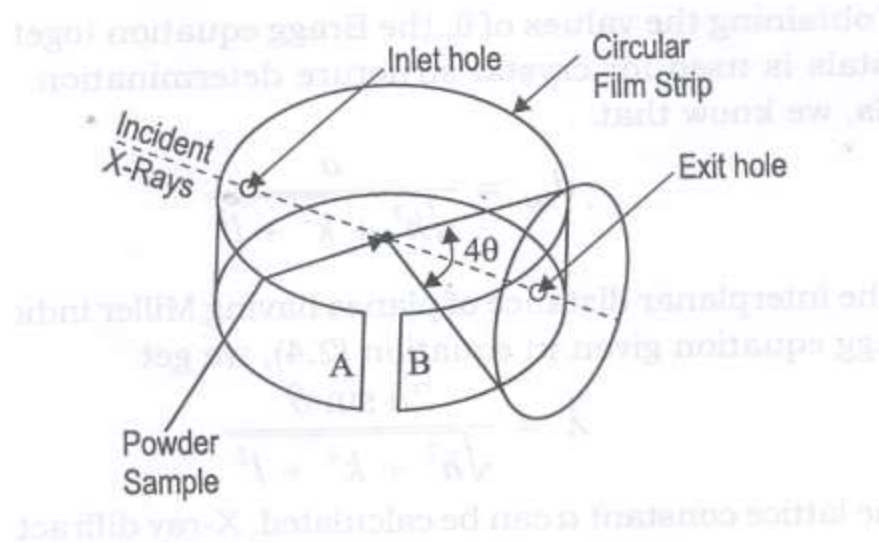
Rotating crystal method

- X-ray beam is made to fall on a single crystal of the material
- It is rotated about a fixed axis
- Various sets of lattice planes come successively in to reflecting positions
- At some position diffraction condition is satisfied
- The diffraction position is recorded on a flat photographic film

Powder method

- Most commonly used X-ray diffraction technique
- A powdered specimen is utilized, so that there will be random orientation of many crystals
- Some of the crystals will be oriented along the X-ray beam to satisfy the diffraction conditions of the Bragg's law

Powder method

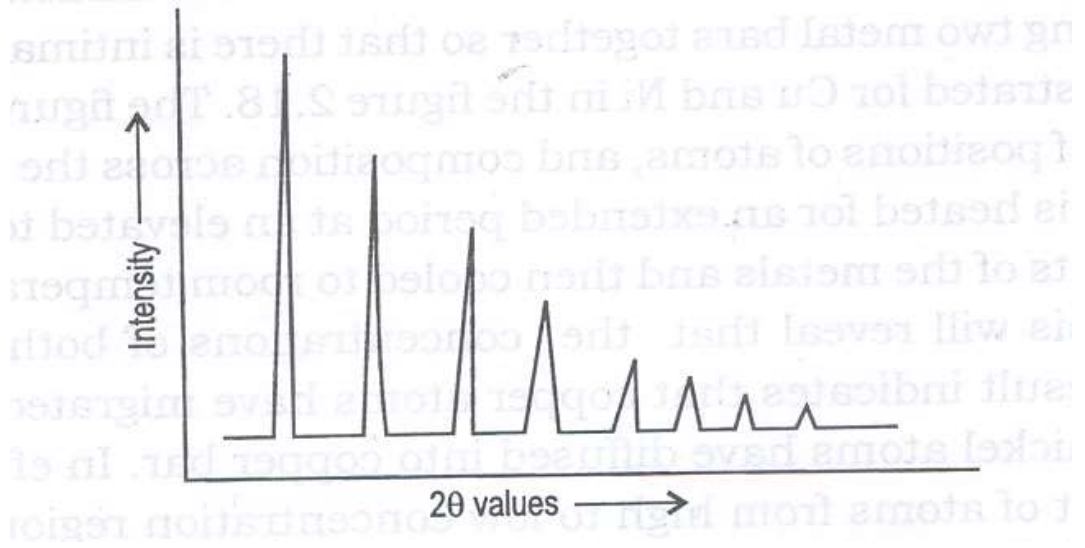


- $S=R \times 4\theta$

R=Radius of powder camera

θ =Bragg angle in radians

- X-ray diffractometer – a radiation counter to detect the angle and intensity of diffracted beam



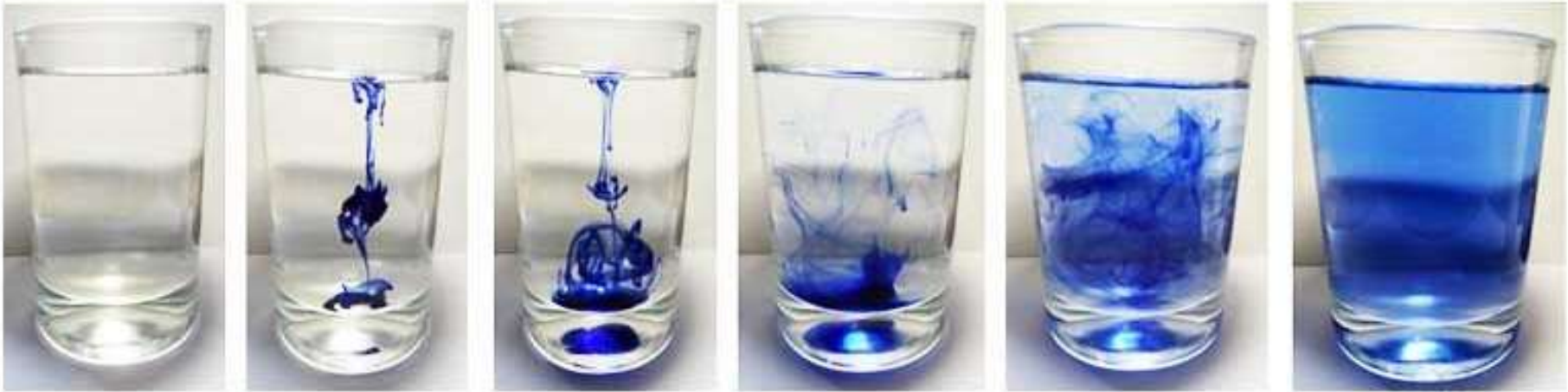
- After obtaining θ , Bragg equation together with known data for crystals is used for crystal structure determination

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}}$$

$$\lambda = \frac{2a \sin\theta}{\sqrt{h^2+k^2+l^2}}$$

- From which lattice constant 'a' can be calculated

Diffusion in nature

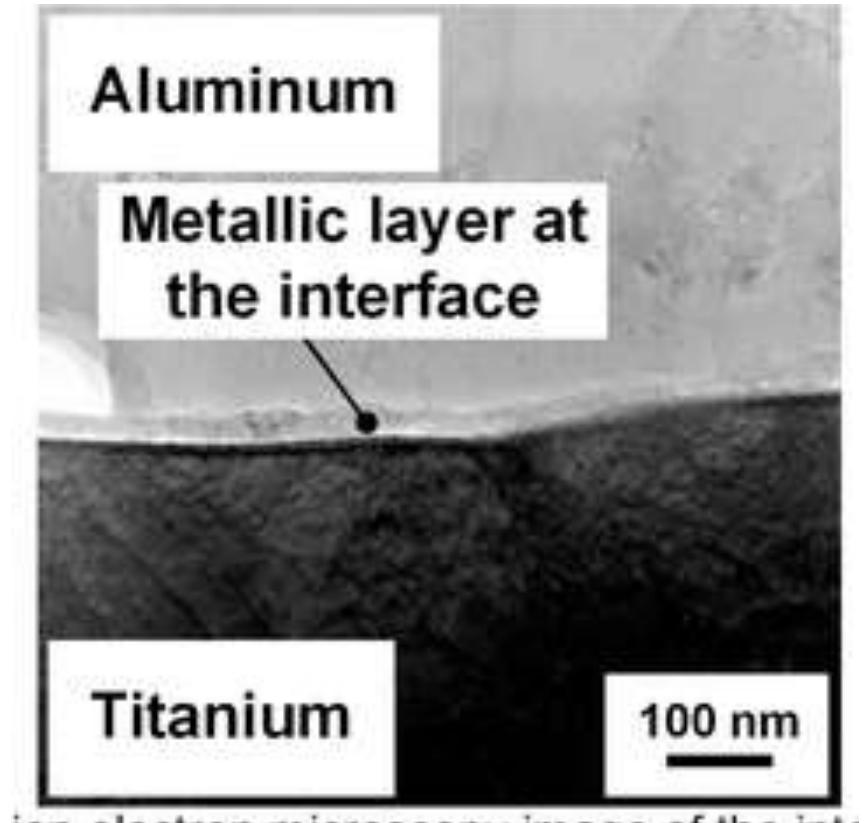
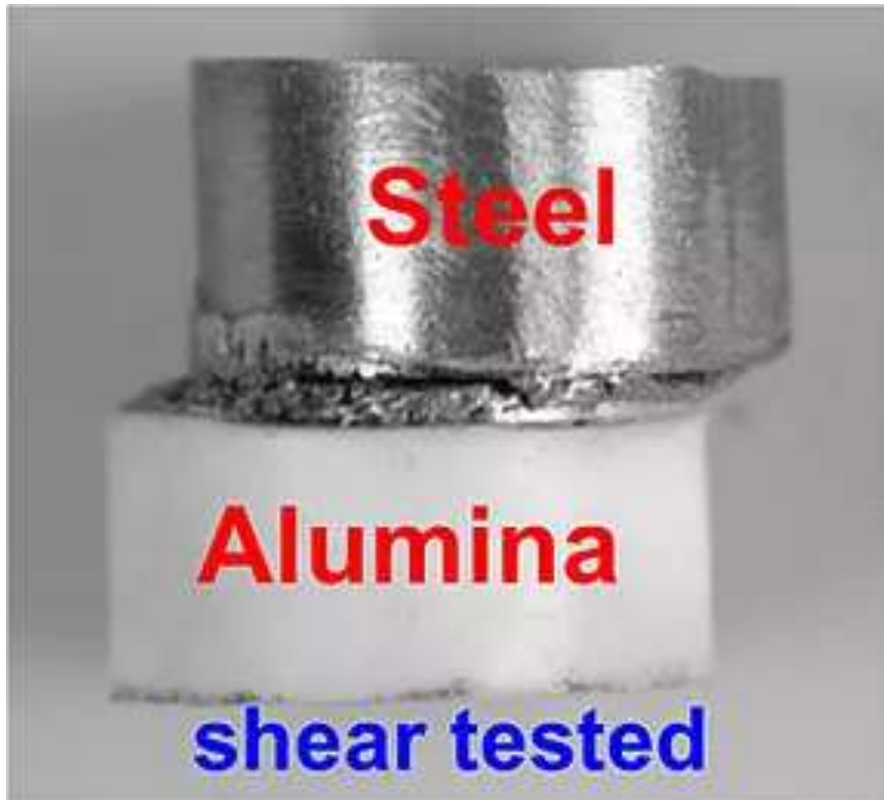


An Ink Drop Gradually Dissolves into a Glass of Water by DIFFUSION

www.easybiologyclass.com



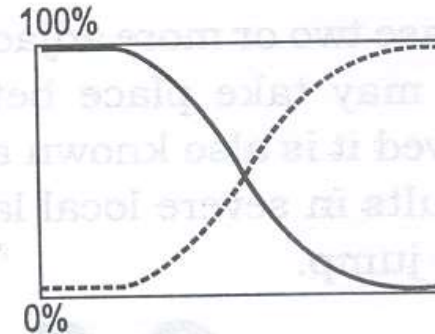
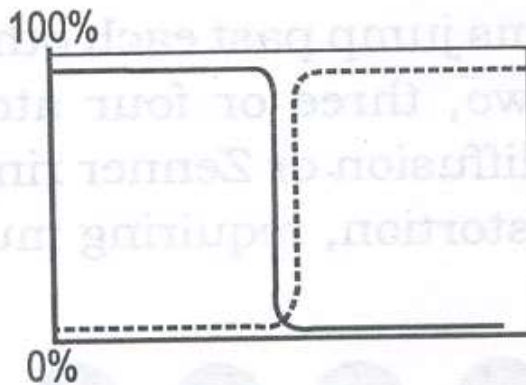
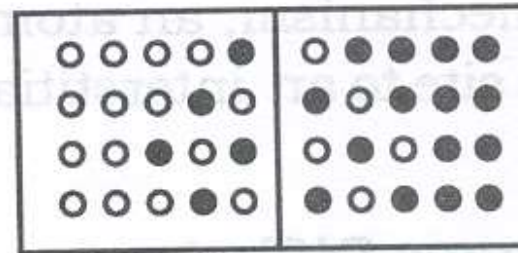
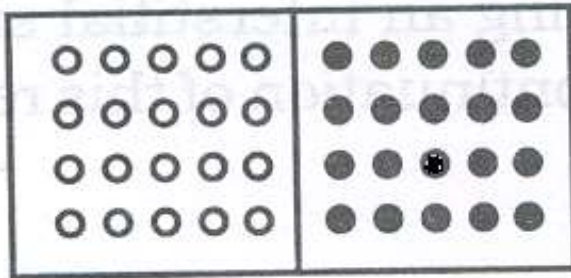
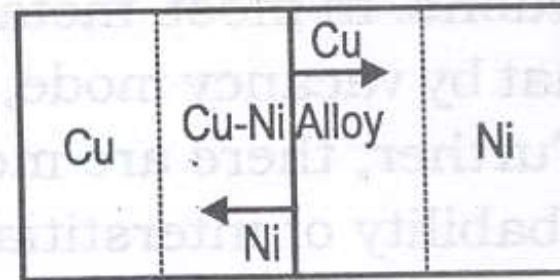
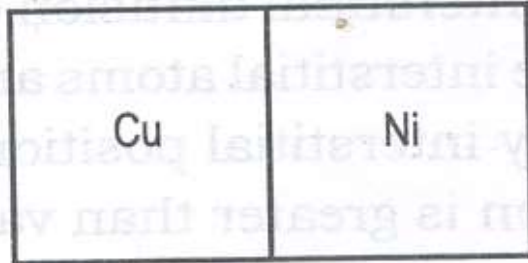
Diffusion in metals



Diffusion

- Phenomenon of material transport by atomic motion
- Process of mass flow by which atoms change their position relative to their neighbors under the influence of thermal energy and a gradient
- Rate of diffusion has a major role in achieving optimum properties in a solid material
- Also assists introducing very small concentration of impurities in semiconductors
- Interdiffusion or impurity diffusion
- Self diffusion.....

Diffusion couple



Conditions of diffusion

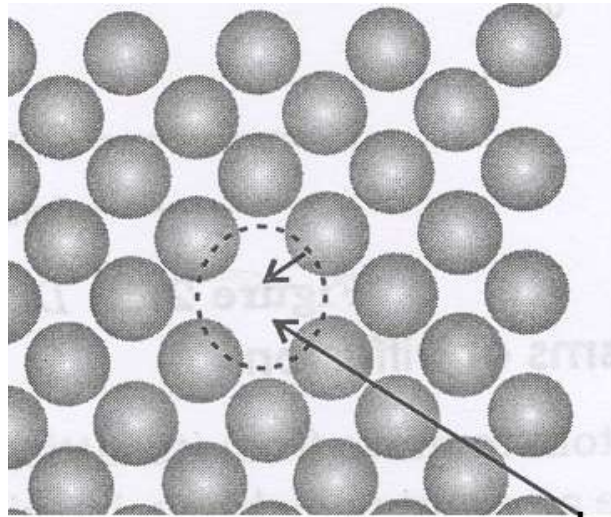
Conditions for an atom to move from its lattice site:

1. There must be an empty neighboring site
2. The atom must have sufficient energy to break bonds with surrounding atoms

Mechanisms of diffusion

Vacancy Diffusion

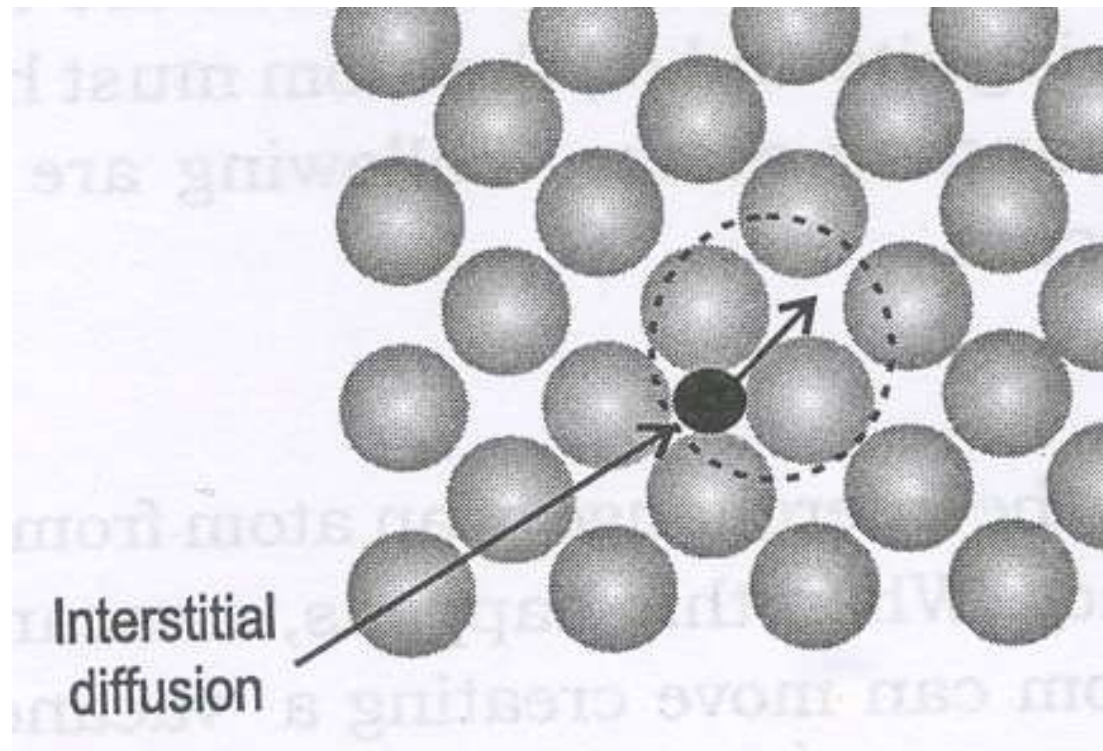
- Interchange of atom from a normal site to an adjacent vacancy



Vacancy Diffusion

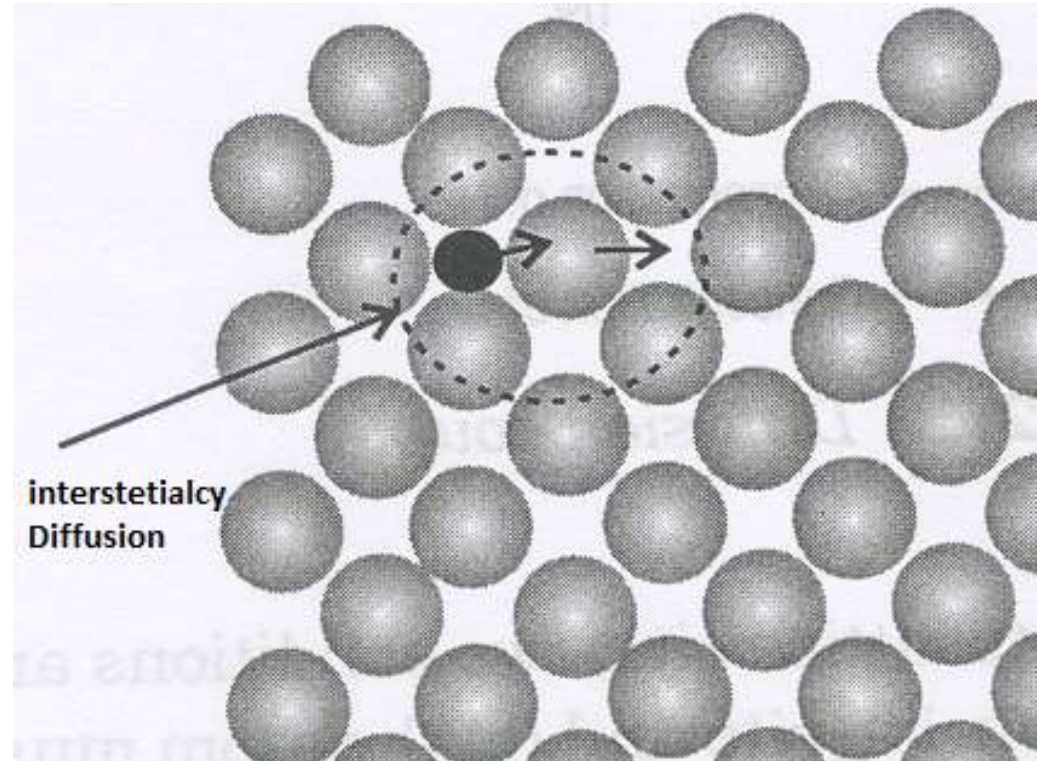
Interstitial diffusion

- Migration of atoms from one interstitial position to a neighboring one that is empty



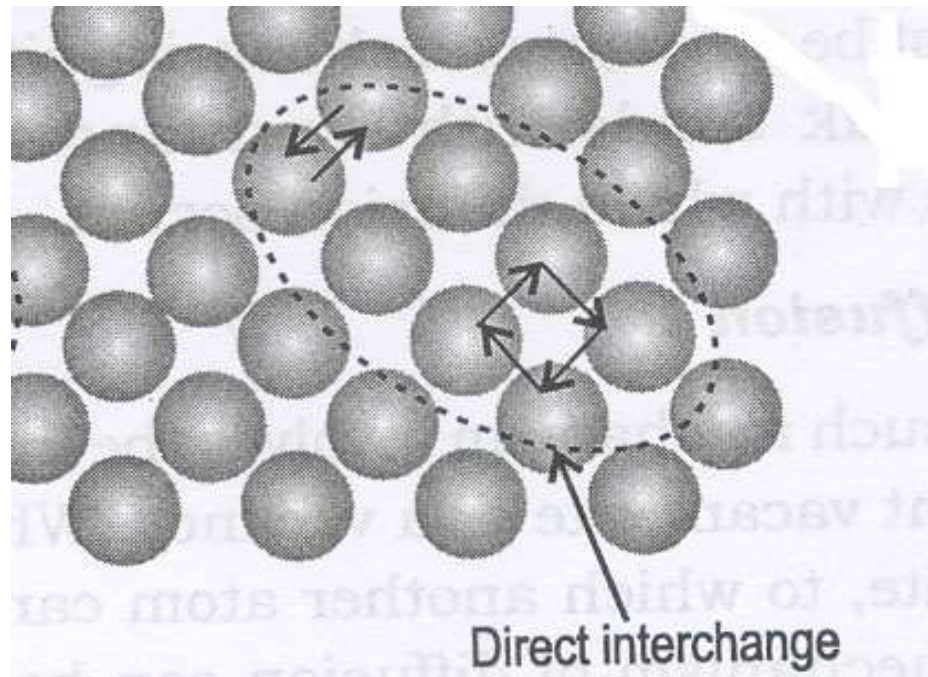
Interstitialcy diffusion

- An atom occupying an interstitial site forces a regular atom to leave its site to an interstitial site



Direct interchange diffusion

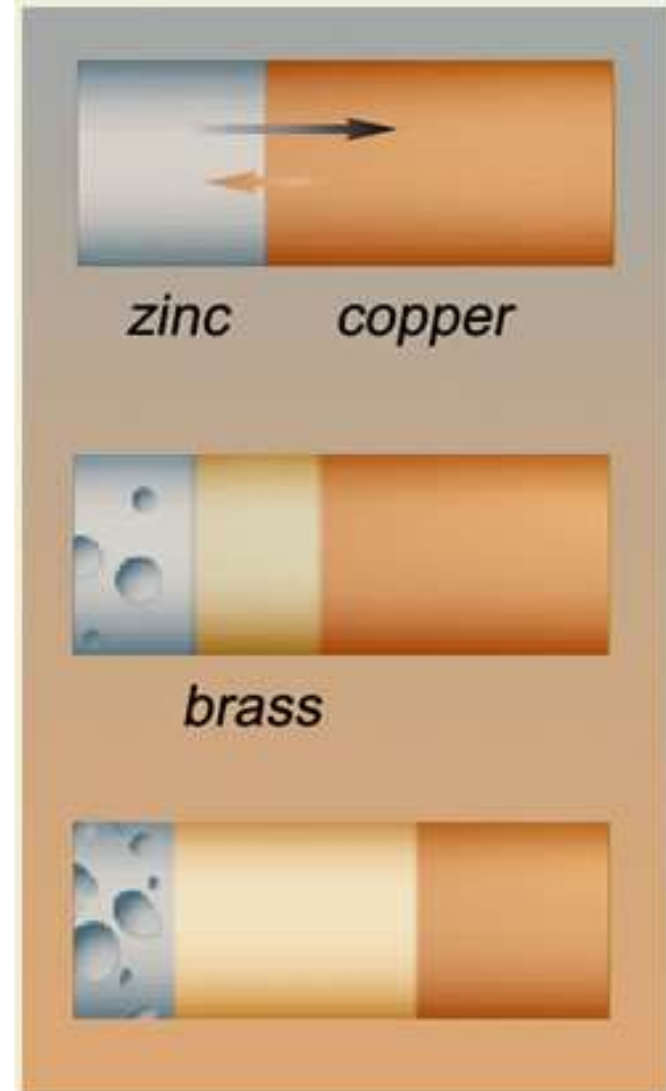
- Two or more adjacent atoms jump past each other and exchange positions



Definitions

Kirkendal effect

- In a binary diffusion couple, of metals A and B, the rate at which A and B diffuse in each other are not necessarily equal
- Lower melting component diffuses more faster in to the other than vice-versa



Diffusion flux (J)

- Mass flow per unit area per unit time
- OR no: of atoms of one plane moving to unit area of another plane per unit time
- $J = M / (A \times t)$

Concentration gradient

- When concentration (C) of the diffusing element is plotted versus position or distance (x) within the solid, the slope of the curve – concentration gradient (dC/dx)

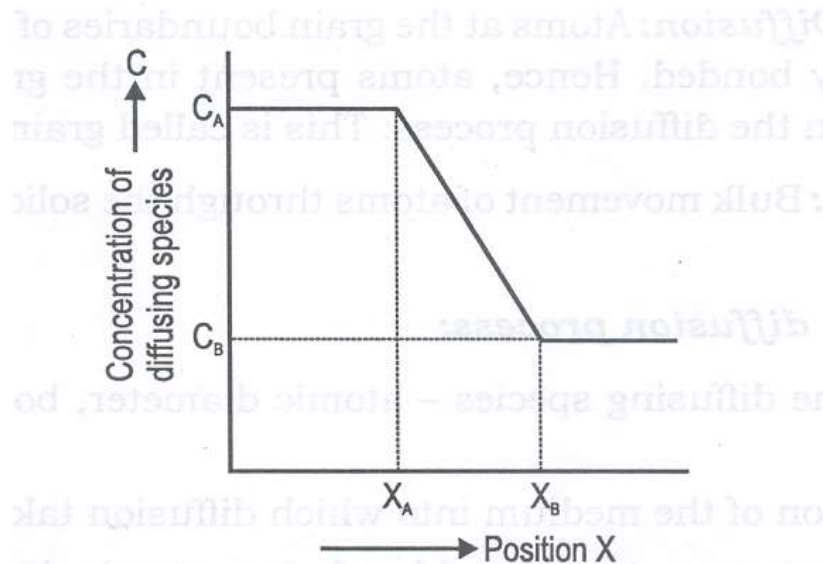


Figure 2.20 Concentration gradient

Steady state and non steady state diffusion

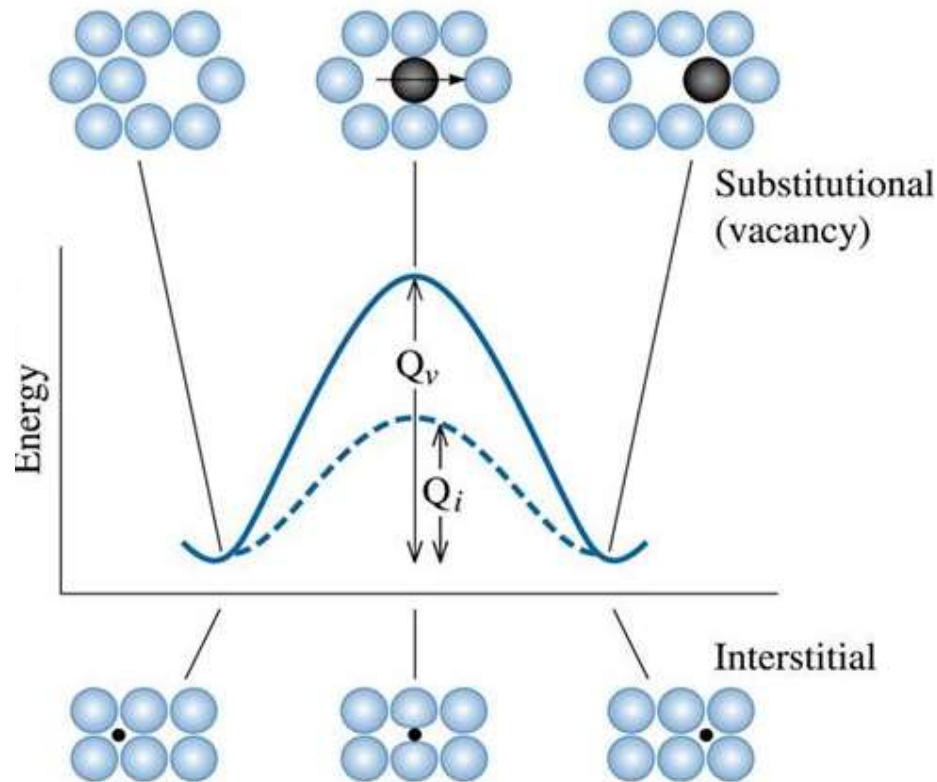
- The diffusion flux does not change with time (flux at any c/s plane along the diffusion distance remains constant) – steady state
- At any given instant the flux at different c/s planes along the diffusion directions are not equal – non steady state

Diffusion coefficient or Diffusivity (D)

- An index of the rate at which atoms diffuse
- It's a material property which depends upon Diffusing species, the composition of the medium into which diffusion occur, and the temperature
- Can also be considered as how fast one species can diffuse in to another

Activation energy

- Atoms will have to overcome the resistance and barriers to their movement
- The energy required for this - Activation Energy (Q)



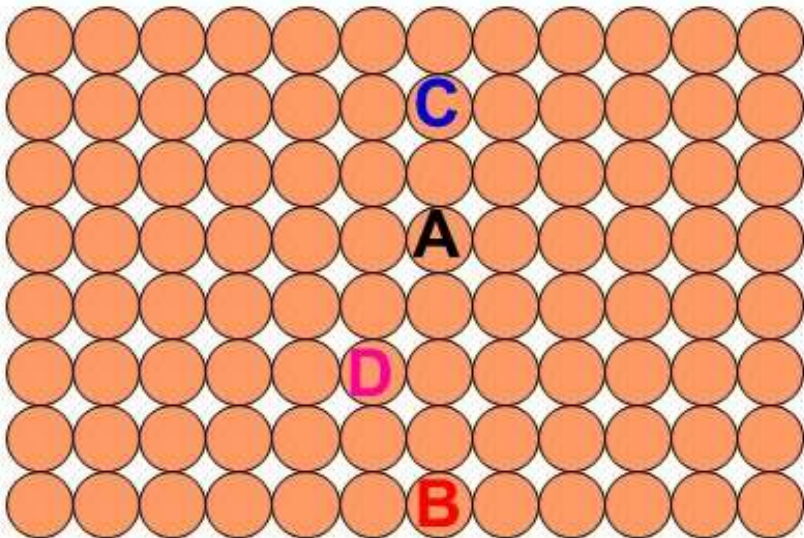
Classification of diffusion processes

- 1) Self diffusion
- 2) Inter diffusion
- 3) Surface diffusion
- 4) Grain boundary diffusion
- 5) Volume diffusion

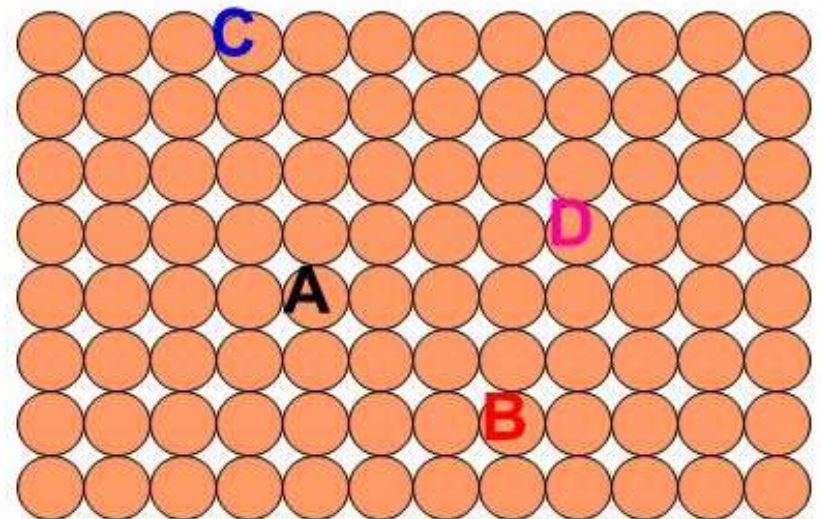
Self-Diffusion

- **Self-diffusion:** In an elemental solid, atoms also migrate.

specific atom movement

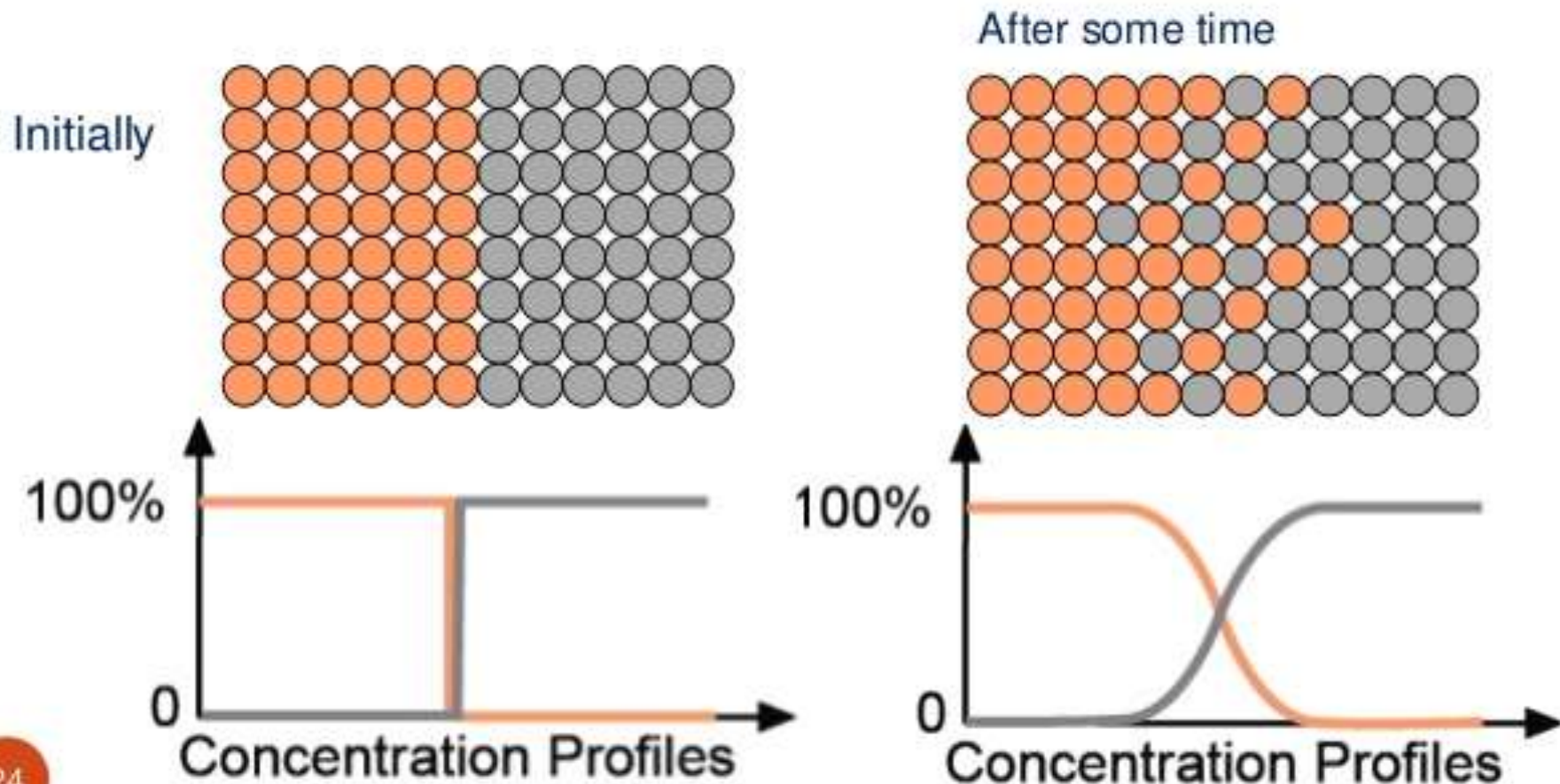


After some time

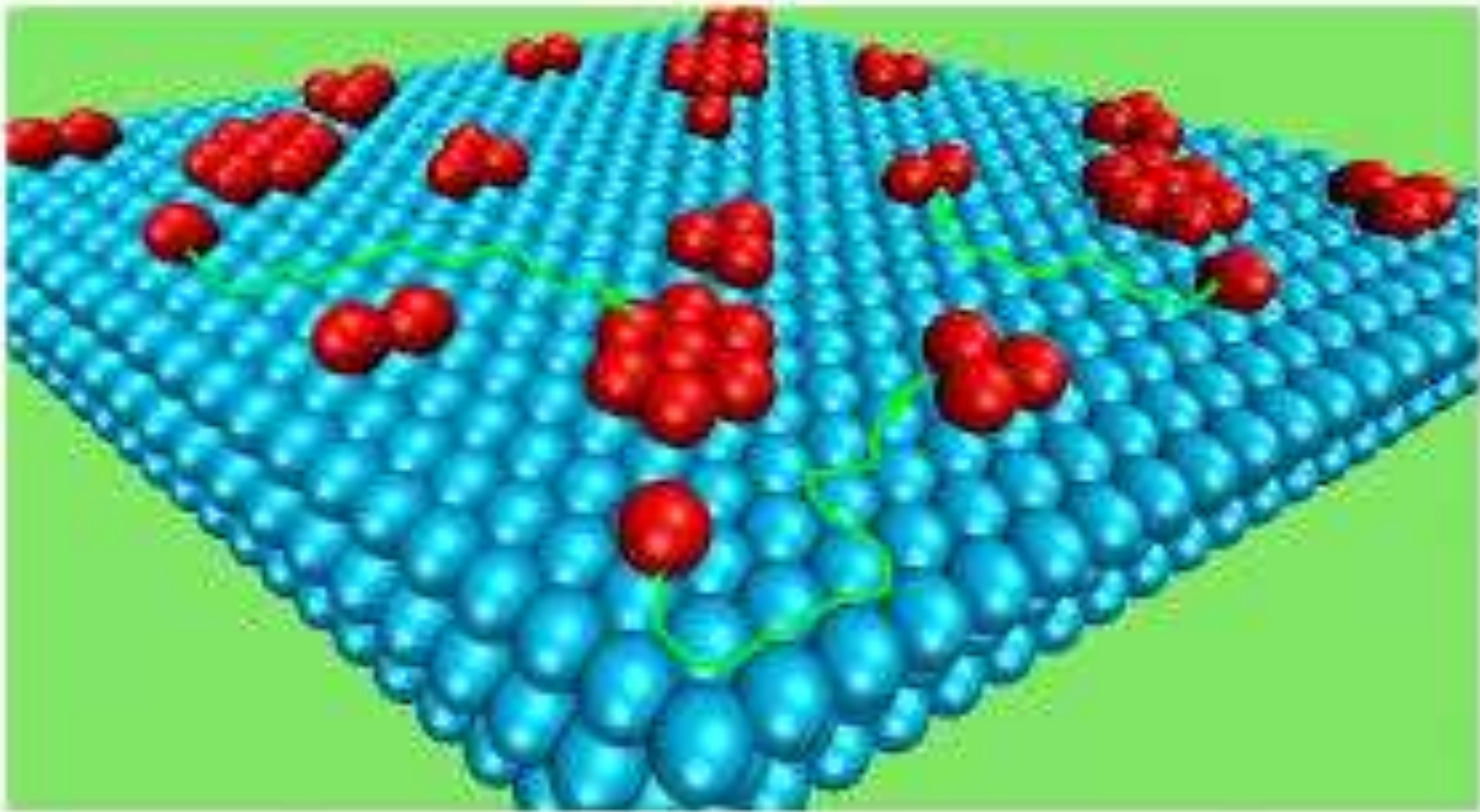


Inter diffusion

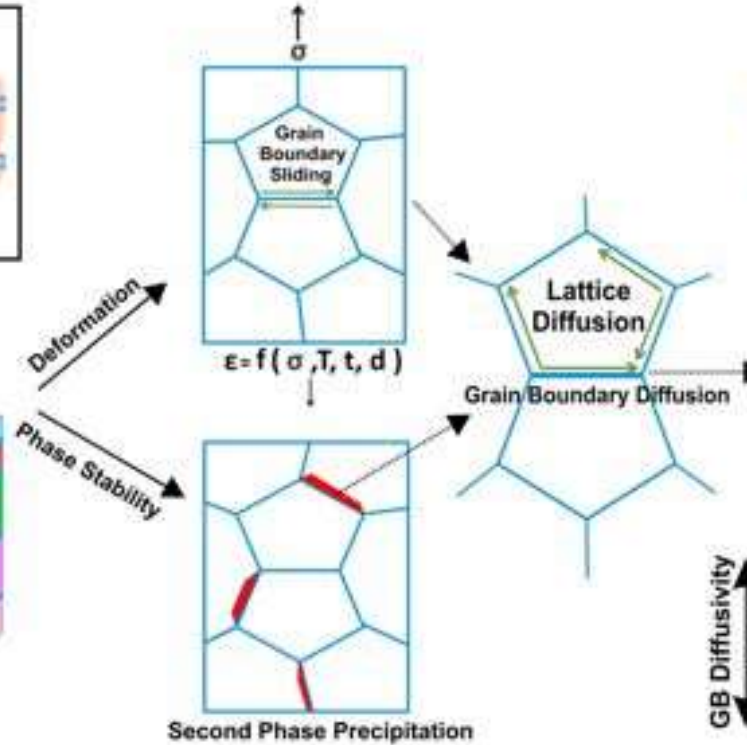
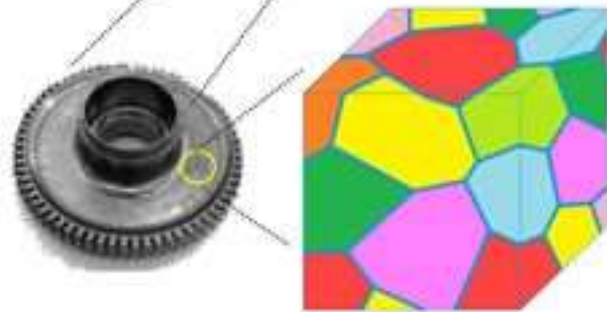
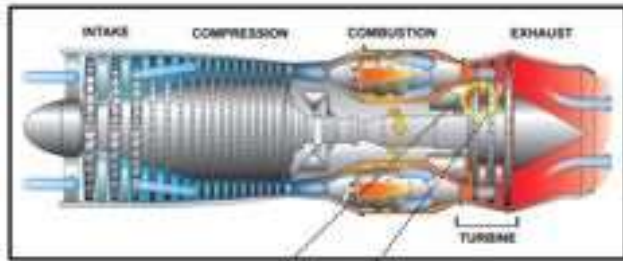
This result indicates that copper atoms have migrated or diffused into the nickel, and that nickel has diffused into copper. This process, whereby atoms of one metal diffuse into another, is termed **inter diffusion, or impurity diffusion**.

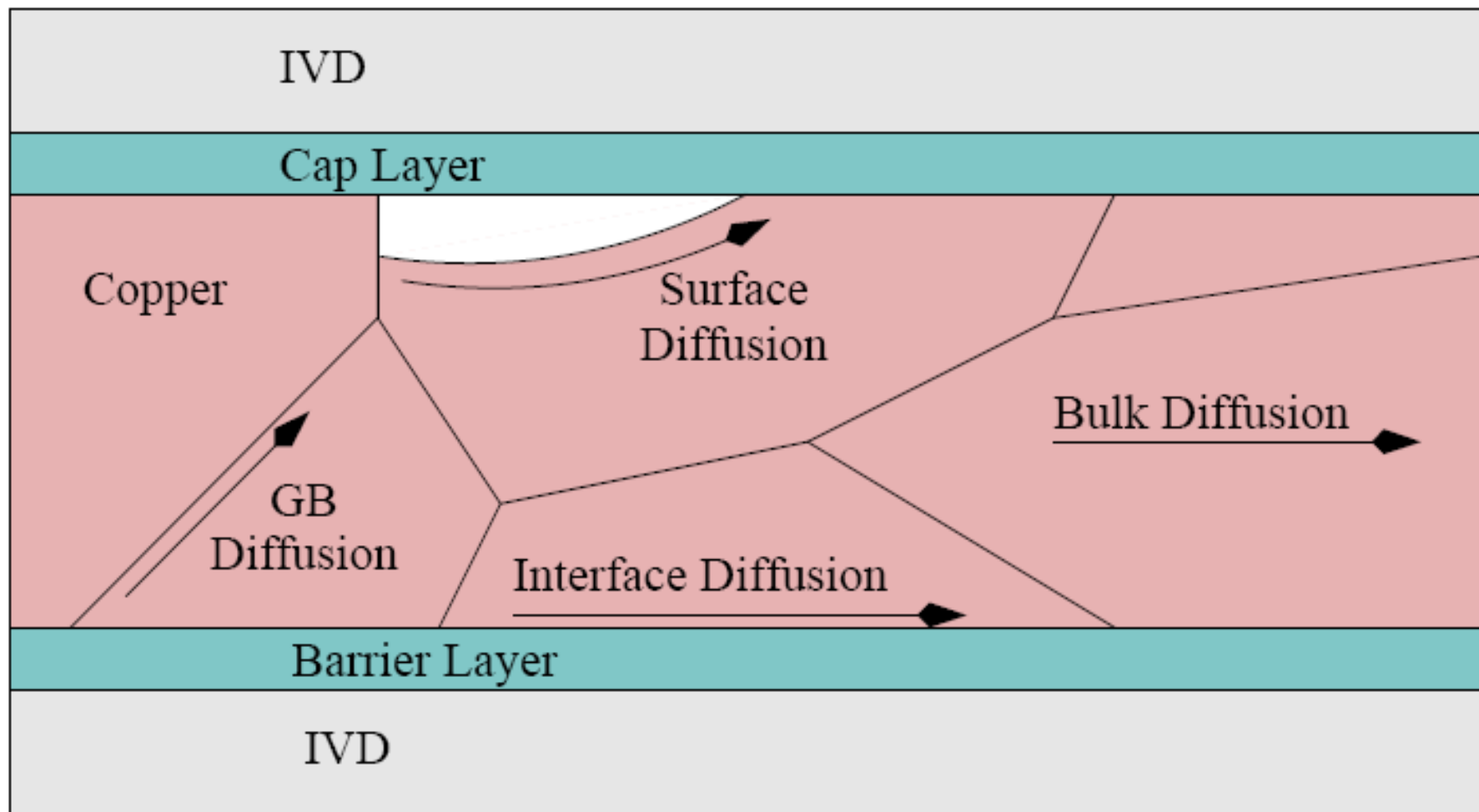


Surface diffusion



Grain boundary diffusion





Fick's laws of Diffusion

1. Fick's First law for steady state diffusion
2. Fick's second law for non-steady state diffusion

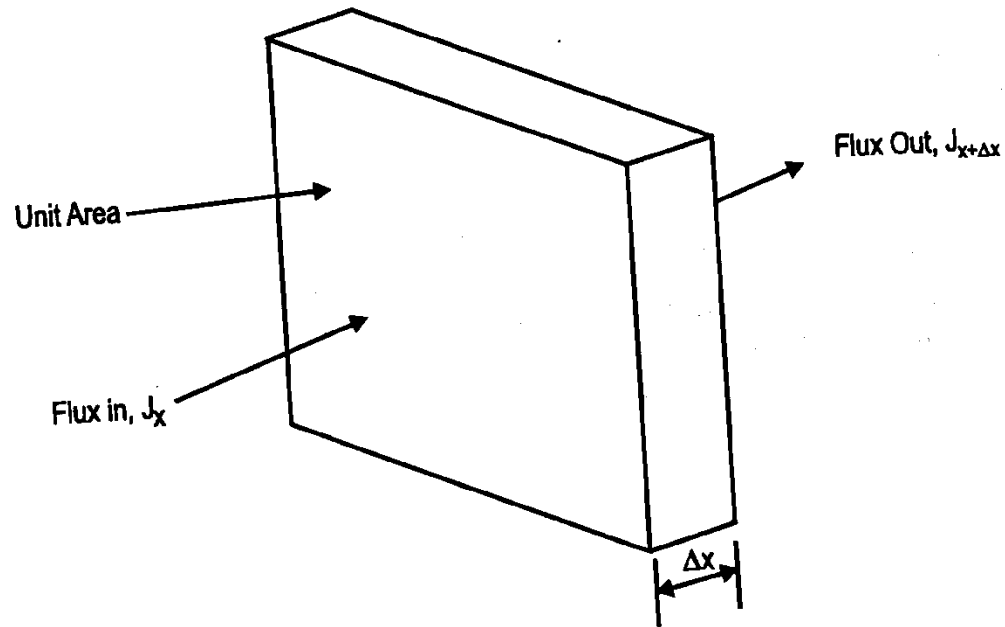


Figure 2.21 Diffusion through a plate

