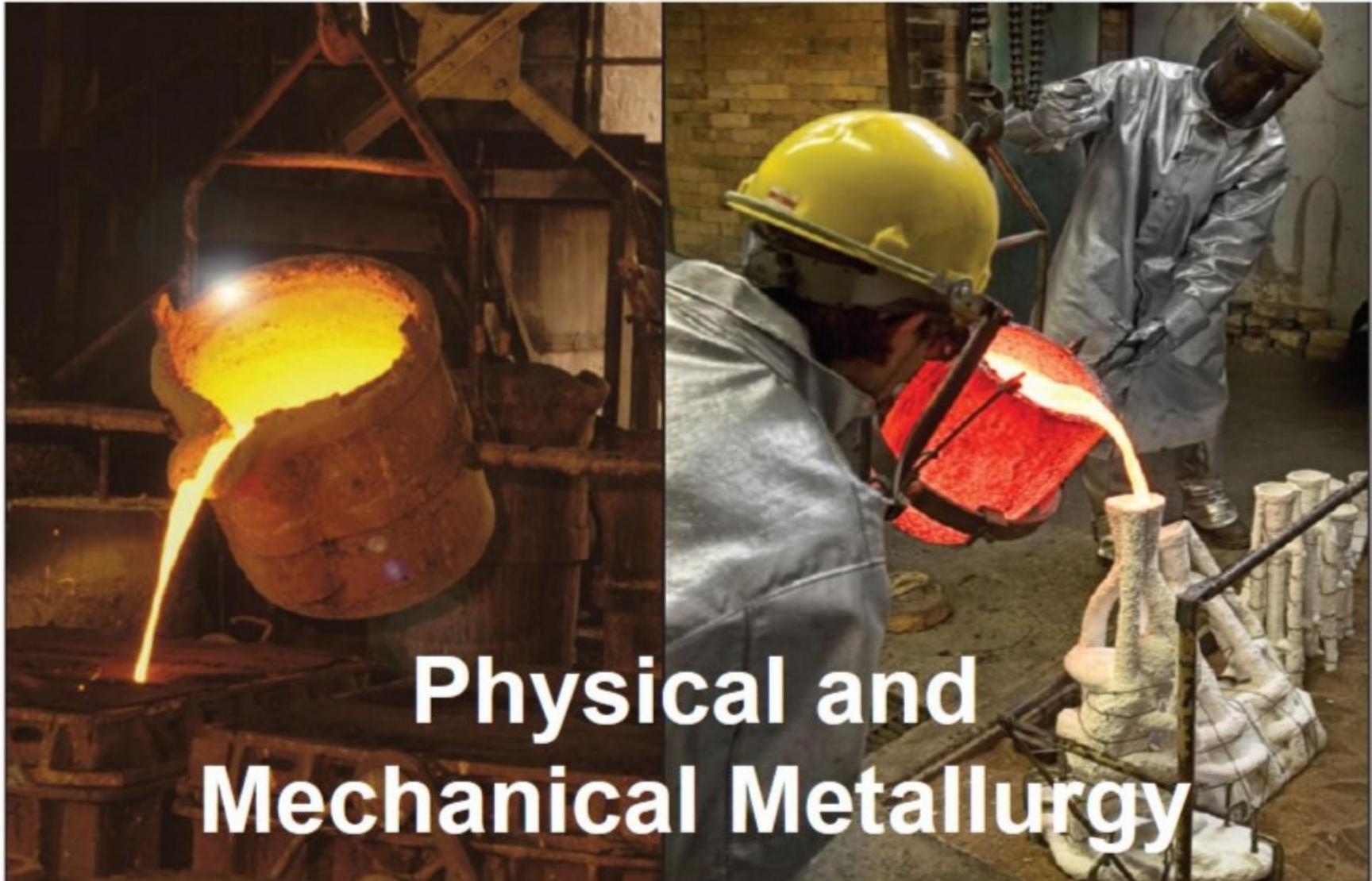


CHY1005 INDUSTRIAL CHEMISTRY FOR ENGINEERS

Module 3 Physical and Mechanical Metallurgy



**Physical and
Mechanical Metallurgy**

Classification, selection and properties of materials

Crystal defects: point, line, surface and volume defects

Alloy formation, solid solution types, solidification of castings

Bragg's law and x-ray diffraction

Structural examination using microscopy

Phase diagrams - isomorphous, eutectic, eutectoid and peritectic systems

Phase transformation in shape-memory alloys

Fe-C phase diagram

Diffusion - Fick's laws, *Interconnects* in IC chips

Brittle fracture - Griffith's theory, fracture toughness

Embrittlement phenomena, Fatigue and creep

Strengthening mechanism: dislocation, solid solution, precipitation, dispersion, grain-boundary.

Metals, metalloids, non-metal

Characteristics, sources, occurrence and physical properties of metals

Manufacture of steel, Bessemer process, modern steel making technology

Furnaces, slags and fluxes

Metal fabrication techniques:

Forming: forging, rolling, extrusion & drawing

Castings: sand, die, investment, lost-foam, continuous

Miscellaneous methods: Welding, Powder metallurgy

3D Printing: direct energy deposition, powder bed fusion.

CHY1005 INDUSTRIAL CHEMISTRY FOR ENGINEERS

MODULE 3 PHYSICAL AND MECHANICAL METALLURGY

INTRODUCTION

Materials Science and Engineering is:

- the study of the properties of solid materials and how those properties are determined by a material's composition and structure.
- an interdisciplinary study that combines metallurgy, physics, chemistry, and engineering to solve real-world problems with real-world materials in an acceptable societal and economical manner.
- the study of Performance, Properties, Structure, Composition, Synthesis and Processing of materials and also interactions of these aspects of such materials.

The principal goals of a materials science and engineering are to (1) make existing materials better and (2) invent or discover new phenomena, materials, devices, and applications.

The heart of materials science is the ability to change the properties and/or behavior of a material to make most materials useful. **Materials science and engineering** (MSE) is an interdisciplinary field of science and engineering that studies and manipulates the composition and structure of materials across length scales to control materials properties through synthesis and processing.

INTRODUCTION

Materials science involves investigating the relationships between the structures and properties of materials.

Materials engineering is based on structure-property correlations, designing or engineering the structure of a material to produce a predetermined set of properties.

One of the most important functions of materials scientists and engineers is to establish the relationships between a material or a device's properties and performance and the microstructure of that material, its composition, and the way the material or the device was synthesized and processed.

The goal of Materials Science and Engineering is to design materials with a certain set of properties, which gives a certain desired performance. Using suitable processing techniques the material can be synthesized and processed. The processing also determines the microstructure of the material.

Materials can be classified based on **Band Structure** (Metals, Semimetals, Semiconductors, Insulators) or **Atomic Structure** (Crystals, Quasicrystals, Amorphous phases).

Composition means the chemical make-up of a material.

Structure means a description of the arrangement of atoms.

Materials scientists and engineers deal with the development of materials and also with the **synthesis** and **processing** of materials and manufacturing processes related to the production of components.

Synthesis refers to how materials are made from naturally occurring or man-made chemicals.

Processing means how materials are shaped into useful components to cause changes in the properties of different materials.

Structure of a material usually relates to the arrangement of its internal components.

Structure levels are subatomic, atomic, microscopic, and macroscopic.

A **property** is a material trait in terms of the kind and magnitude of response to a specific imposed stimulus.

Important properties of solid materials are: mechanical, electrical, thermal, magnetic, optical and deteriorative.

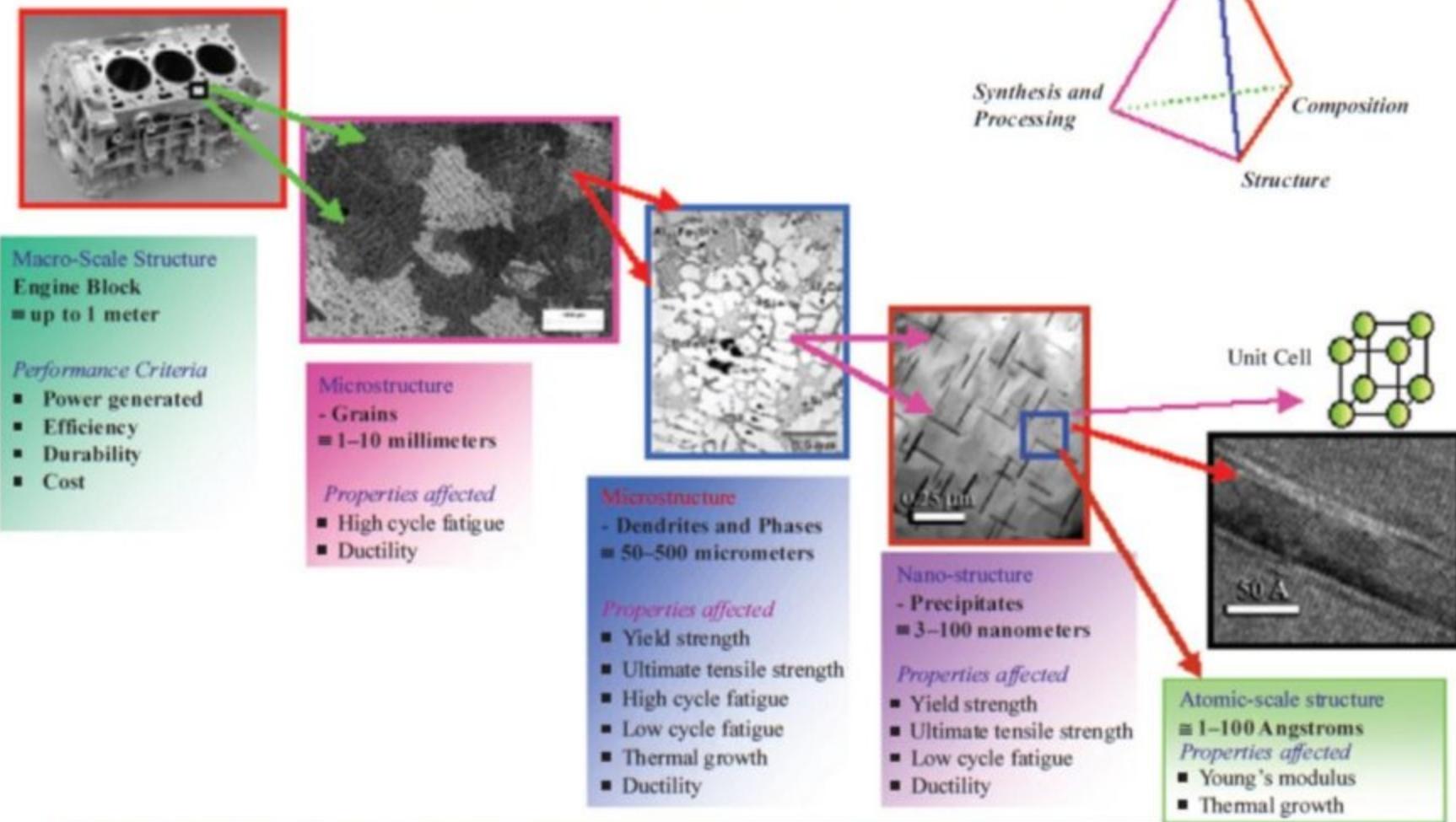
The structure of a material will depend on how it is processed.

A material's performance will be a function of its properties.

The interrelationship between processing, structure, properties, performance, and reuse/recyclability is:



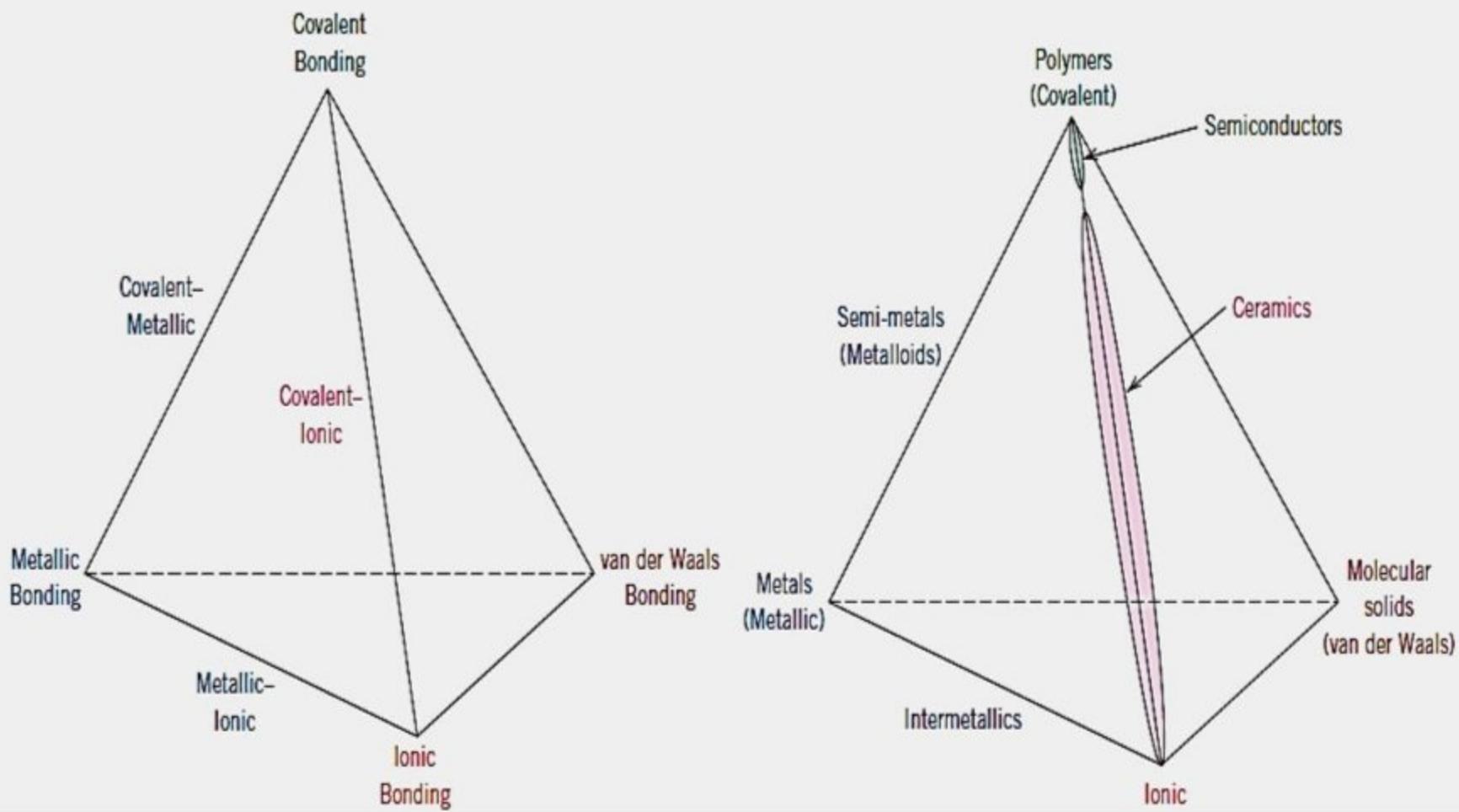
What is Materials Science and Engineering?



A real-world example of important microstructural features at different length-scales resulting from the sophisticated synthesis and processing used, and the properties they influence. The atomic, nano, micro, and macro-scale structures of cast aluminum alloys (for engine blocks) in relation to the properties affected and performance are shown. The materials science and engineering (MSE) tetrahedron that represents this approach is shown in the upper right corner.

(Illustrations Courtesy of John Allison and William Donlon, Ford Motor Company.)

CHEMICAL BONDING & MATERIALS



TRADITIONAL AND MODERN MATERIALS

SOME TRADITIONAL MATERIALS AND USES

| | |
|----------|-------------------------------------|
| Wood | Construction, ships |
| Metal | Tools, weapons |
| Stone | Building |
| Clay | Pottery, record keeping, insulators |
| Glass | Tableware, windows |
| Leather | Clothing, armor, saddles |
| Plastics | Piping, containers, etc. |
| Rubber | Tires |

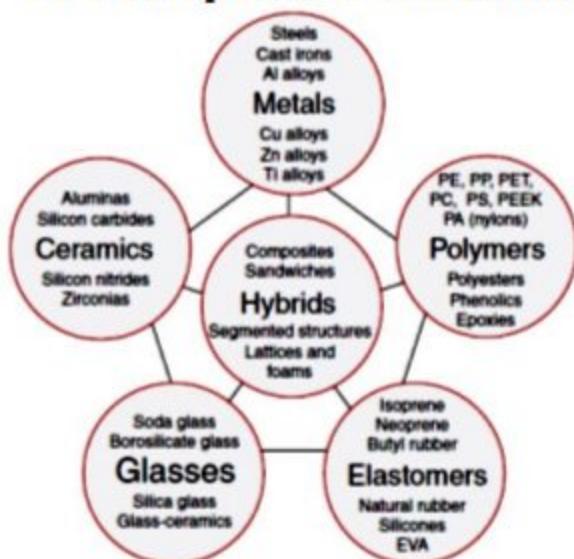
SOME MODERN MATERIALS AND USES

| | |
|--------------------------|---|
| Metal-organic frameworks | Catalyst, gas storage |
| Catalysts | Pollution abatement, wide variety of uses |
| Semiconductors | Electronics |
| Biomaterials | Hard and soft tissue, others |
| Energy materials | Photovoltaics, fuel cells, batteries |
| Nanomaterials | Nano-carbon, fullerenes, etc. |

CLASSIFICATION OF MATERIALS

There are different ways of classifying materials. One way is to describe five groups.

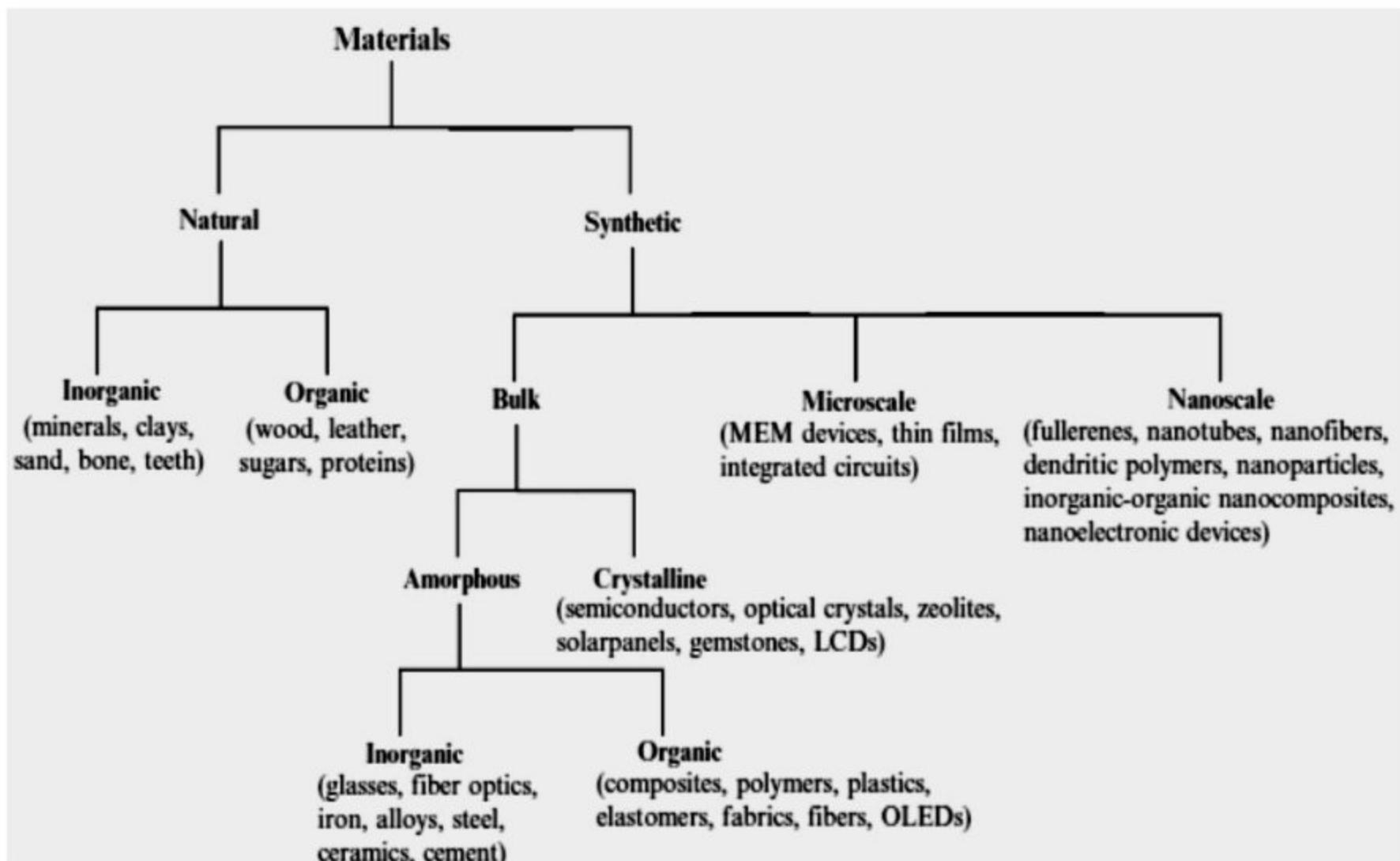
1. **metals and alloys;**
2. **ceramics, glasses, and glass-ceramics;**
3. **polymers (plastics);**
4. **semiconductors;** and
5. **composite materials.**



Metals, ceramics, glasses, polymers, elastomers etc. can be combined in various geometries to create HYBRIDS

TABLE 1-1 ■ Representative examples, applications, and properties for each category of materials

| | Examples of Applications | Properties |
|---|--|---|
| Metals and Alloys | | |
| Copper | Electrical conductor wire | High electrical conductivity, good formability |
| Gray cast iron | Automobile engine blocks | Castable, machinable, vibration-damping |
| Alloy steels | Wrenches, automobile chassis | Significantly strengthened by heat treatment |
| Ceramics and Glasses | | |
| $\text{SiO}_2\text{-Na}_2\text{O-CaO}$ | Window glass | Optically transparent, thermally insulating |
| $\text{Al}_2\text{O}_3, \text{MgO}, \text{SiO}_2$ | Refractories (i.e., heat-resistant lining of furnaces) for containing molten metal | Thermally insulating, withstand high temperatures, relatively inert to molten metal |
| Barium titanate | Capacitors for microelectronics | High ability to store charge |
| Silica | Optical fibers for information technology | Refractive index, low optical losses |
| Polymers | | |
| Polyethylene | Food packaging | Easily formed into thin, flexible, airtight film |
| Epoxy | Encapsulation of integrated circuits | Electrically insulating and moisture-resistant |
| Phenolics | Adhesives for joining plies in plywood | Strong, moisture resistant |
| Semiconductors | | |
| Silicon | Transistors and integrated circuits | Unique electrical behavior |
| GaAs | Optoelectronic systems | Converts electrical signals to light, lasers, laser diodes, etc. |
| Composites | | |
| Graphite-epoxy | Aircraft components | High strength-to-weight ratio |
| Tungsten carbide-cobalt (WC-Co) | Carbide cutting tools for machining | High hardness, yet good shock resistance |
| Titanium-clad steel | Reactor vessels | Low cost and high strength of steel with the corrosion resistance of titanium |



Functional Classification of Materials



Table 1.3 Important grouping of materials

| Material group (1) | Important characteristics (2) | Typical examples of engineering use (3) |
|---|---|--|
| 1. Metals and Alloys | Lusture, hardness, thermal and electrical conductivity, resistance to corrosion, malleability, stiffness and the property of magnetism | Iron and steels, aluminium, copper, silver, gold, zinc, magnesium, brasses, bronzes, manganin, invar, super alloy, boron, rare-earth alloys, conductors, etc. |
| 2. Ceramics and Glasses | Thermal resistance, hardness, brittleness, opaqueness to light, electrical insulation abrasiveness, high temperature strength and resistance to corrosion | Silica, soda-lime-glass, concrete, cement, refractories, Ferrites and garnets, ceramic superconductors, MgO , CdS , Al_2O_3 , SiC , $BaTiO_3$, etc. |
| 3. Organic Polymers | Soft, light in weight, poor conductors of electricity and heat, dimensionally unstable, ductile, combustible, low thermal resistance | <i>Plastics:</i> PVC, PTFE, polyethylene, polycarbonate <i>Fibres:</i> terylene, nylon, cotton, natural and synthetic rubbers, leather <i>Other uses:</i> refrigerants, explosives, insulators, lubricants, detergents, fuels, vitamins, medicines for surface treatment, adhesives, fibre-reinforced plastics, etc. |
| 4. Composites | They are better than any of the individual components as regards to their properties like strength, stiffness, heat resistance, etc. | <ul style="list-style-type: none">• Steel-reinforced concrete, dispersion hardened alloys.• Vinyl coated steel, whisker-reinforced plastics.• Fibre-reinforced plastics, carbon-reinforced rubber. |
| (i) Metals and alloys and ceramics | | |
| (ii) Metals and alloys and organic polymers | | |
| (iii) Ceramics and organic polymers | | |

SELECTION OF MATERIALS

- ❑ Different materials possess different properties to meet the various requirement for engineering purposes.
- ❑ A good grasp of the fundamental principles which control the properties of various materials help to make the optimum selection of material.
- ❑ For selecting a material or designing a component, it is essential to understand:
 - (1) the requirements of the process thoroughly, (2) operating limitations like hazardous or non-hazardous conditions, (3) continuous or non-continuous operation, (4) availability of raw materials as well as spares, (5) availability of alternate materials, (6) life span of the instrument/equipment, (7) reuse/recycle nature, (8) technology level, (9) environmental impact, (10) sustainability, (11) cost etc.
- ❑ The selection of a specific material for a particular engineering use as per specifications is based on
 - (i) operating parameters,
 - (ii) manufacturing processes,
 - (iii) functional requirements and
 - (iv) cost considerations.

Table 1.1 Factors affecting selection of materials

| (i) <i>Manufacturing processes</i> | (ii) <i>Functional requirements</i> | (iii) <i>Cost considerations</i> | (iv) <i>Operating parameters</i> |
|--|---|--|--|
| <ul style="list-style-type: none">• Plasticity• Malleability• Ductility• Machinability• Casting properties• Weldability• Heat• Tooling• Surface finish | <ul style="list-style-type: none">• Strength• Hardness• Rigidity• Toughness• Thermal conductivity• Fatigue• Electrical treatment• Creep• Aesthetic look | <ul style="list-style-type: none">• Raw material• Processing• Storage• Manpower• Special treatment• Inspection• Packaging properties• Inventory• Taxes and custom duty | <ul style="list-style-type: none">• Pressure• Temperature• Flow• Type of material• Corrosion requirements• Environment• Protection from fire• Weathering• Biological effects |

One can list the major factors affecting the selection of materials as

- | | |
|-------------------------------|--------------------------------------|
| (i) Component shape | (ii) Dimensional tolerance |
| (iii) Mechanical properties | (iv) Fabrication requirements |
| (v) Service requirements | (vi) Cost of the material |
| (vii) Cost of processing, and | (viii) Availability of the material. |

PROPERTIES OF MATERIALS

General properties of materials.

| Class | Property |
|---------------------|---|
| General | Density, porosity, moisture content, macrostructure, microstructure |
| Chemical | Composition, pH, resistance to corrosion, weatherability |
| Physiochemical | Liquid absorption/repulsion, shrinkage and swell due to hygroscopic action |
| Mechanical | Strength in: tension, compression, shear, torsion and flexure; static, impact, and endurance, stiffness, toughness, elasticity, plasticity, ductility, hardness |
| Thermal | Specific heat, thermal expansion, thermal conductivity |
| Electrical/magnetic | Conductivity, permeability, galvanic action |
| Acoustic | Transmission, reflection, absorption |
| Optical | Color, transmission, reflection, collimation |

Mechanical Properties

The important mechanical properties affecting the selection of a material are:

- (i) **Tensile Strength:** This enables the material to resist the application of a tensile force. To withstand the tensile force, the internal structure of the material provides the internal resistance.
- (ii) **Hardness:** It is the degree of resistance to indentation or scratching, abrasion and wear. Alloying techniques and heat treatment help to achieve the same.
- (iii) **Ductility:** This is the property of a metal by virtue of which it can be drawn into wires or elongated before rupture takes place. It depends upon the grain size of the metal crystals.
- (iv) **Impact Strength:** It is the energy required per unit cross-sectional area to fracture a specimen, i.e., it is a measure of the response of a material to shock loading.
- (v) **Wear Resistance:** The ability of a material to resist friction wear under particular conditions, i.e. to maintain its physical dimensions when in sliding or rolling contact with a second member.
- (vi) **Corrosion Resistance:** Those metals and alloys which can withstand the corrosive action of a medium, i.e. corrosion processes proceed in them at a relatively low rate are termed corrosion-resistant.
- (vii) **Density:** This is an important factor of a material where weight and thus the mass is critical, i.e. aircraft components.

Thermal Properties

The characteristics of a material, which are functions of the temperature, are termed its thermal properties.

Thermal properties play a vital role in selection of material for engineering applications when materials are considered for high temperature service.

(i) Specific Heat (c): It is the heat capacity of a unit mass of a homogeneous substance. For a homogeneous body, $c = C/M$, where C is the heat capacity and M is the mass of the body. One can also define it as the quantity of heat required to raise the temperature of a unit mass of the substance through 1°C . Its units are $\text{cal/g}^\circ\text{C}$.

(ii) Thermal Conductivity (K): This represents the amount of heat conducted per unit time through a unit area perpendicular to the direction of heat conduction when the temperature gradient across the heat.

(iii) Thermal Expansion: All solids expand on heating and contract on cooling. Thermal expansion may take place either as linear, circumferential or cubical. A solid which expands equally in three mutually orthogonal directions is termed as *thermally isotropic*. The increase in any linear dimension of a solid, e.g. length, width, height on heating is termed as *linear expansion*.

Thermal Properties

- iv. **The coefficient of linear expansion** is the increase in length per unit length per degree rise in temperature. The increase in volume of a solid on heating is called *cubical expansion*. The thermal expansion of solids has its origin in the lattice vibration and lattice vibrations increases with the rise in temperature.
- v. **Thermal Resistance (R_T)**: It is the resistance offered by the conductor when heat flow due to temperature difference between two points of a conductor.
- vi. **Thermal Diffusivity (h)**: is defined as (thermal conductivity/[heat capacity x density]). For a material with low thermal diffusivity more heat must be added to or removed from the material for effecting a temperature change.
- vii. **Thermal Fatigue**: This is the mechanical effect of repeated thermal stresses caused by repeated heating and cooling.

Electrical Properties

Conductivity, resistivity and dielectric strength are few important electrical properties of a material.

A material which offers little resistance to the passage of an electric current is said to be a good conductor of electricity.

On the basis of electrical resistivity materials are divided as:

- (i) Conductors**
- (ii) Semiconductors and**
- (iii) Insulators.**

In general metals are good conductors.

Insulators have very high resistivity.

Ceramic insulators are most common examples and are used on automobile spark plugs, Bakelite handles for electric iron, plastic coverings on cables in domestic wiring.

When a large number of metals and alloys are sufficiently cooled below transition temperature, T_c , enter the state of superconductivity in which the DC resistivity goes to zero.

Magnetic Properties

Materials in which a state of magnetism can be induced are termed magnetic materials. There are five classes into which magnetic materials may be grouped:

- (i) diamagnetic
- (ii) paramagnetic
- (iii) ferromagnetic
- (iv) antiferromagnetic and
- (v) ferrimagnetic.

Iron, Cobalt, Nickel and some of their alloys and compounds possess spontaneous magnetization. Magnetic oxides like ferrites and garnets could be used at high frequencies. Because of their excellent magnetic properties along with their high electrical resistivity these materials today find use in a variety of applications like magnetic recording tapes, inductors and transformers, memory elements, microwave devices, bubble domain devices, recording hard cores, etc.

Hysteresis, permeability and coercive forces are some of the magnetic properties of magnetic substances. These properties are to be considered for the manufacture of transformers and other electronic components.

Chemical Properties

These properties include atomic weight, molecular weight, atomic number, valency, chemical composition, acidity, alkalinity, etc. These properties govern the selection of materials particularly in Chemical plants.

Optical Properties

The optical properties of materials, e.g. refractive index, reflectivity and absorption coefficient etc. affect the light reflection and transmission.

X-RAY CRYSTALLOGRAPHY

Historically, much of our understanding regarding the atomic and molecular arrangements in solids has resulted from x-ray diffraction investigations. X-rays are still very important in developing new materials. The x-ray diffraction phenomenon is used to deduce atomic interplanar distances and crystal structures.

X-rays are a form of electromagnetic radiation that have high energies and short wavelengths—wavelengths on the order of the atomic spacings for solids. When a beam of x-rays impinges on a solid material, a portion of this beam will be scattered in all directions by the electrons associated with each atom or ion that lies within the beam's path. Let us now examine the necessary conditions for diffraction of x-rays by a periodic arrangement of atoms.

Material Structure

Material structure can be classified as: macrostructure, microstructure, substructure, crystal structure, electronic structure and nuclear structure.

(a) Macrostructure

- Macrostructure of a material is examined by low-power magnification or naked eye.
- It deals with the shape, size and atomic arrangement in a crystalline material.
- In case of some crystals, e.g., quartz, external form of the crystal may reflect the internal symmetry of atoms.
- Macrostructure may be observed directly on a fracture surface or on a forging specimen.
- The individual crystals of a crystalline material can be visible, e.g. in a brass doorknob by the constant polishing and etching action of a human hand and sweat.
- Macrostructure can reveal flaws, segregations, cracks etc. by using proper techniques and one can save much expenses by rejecting defective materials at an early stage.

(b) Micro Structure

- This generally refers to the structure of the material observed under optical microscope.
- Optical microscopes can magnify a structure about 1500 to 3000 times linear, without loss of resolution of details of the material structure.
- Cracks, porosity, non-metallic inclusions within materials can be revealed by examining them under powerful optical microscope.

(c) Sub Structure

- When crystal imperfections such as dislocation in a structure are to be examined, a special microscope having higher magnification and resolution than the optical microscope is used.
- *Electron microscope* with magnifications 10^5 are used for this purpose.
- *Field ion microscope* can produce images of individual atoms as well as defects in atomic arrangements.

(d) Crystal Structure

- This reveals the atomic arrangement within a crystal.
- X-ray diffraction techniques and electron diffraction method are commonly used for studying crystal structure.
- It is usually sufficient to study the arrangement of atoms within a *unit cell*.
- The crystal is formed by a very large number of unit cells forming regularly repeating patterns in space.

(e) Electronic Structure

- This refers to the electrons in the outermost shells of individual atoms in the solid.
- Spectroscopic techniques are commonly used for determining the electronic structure.

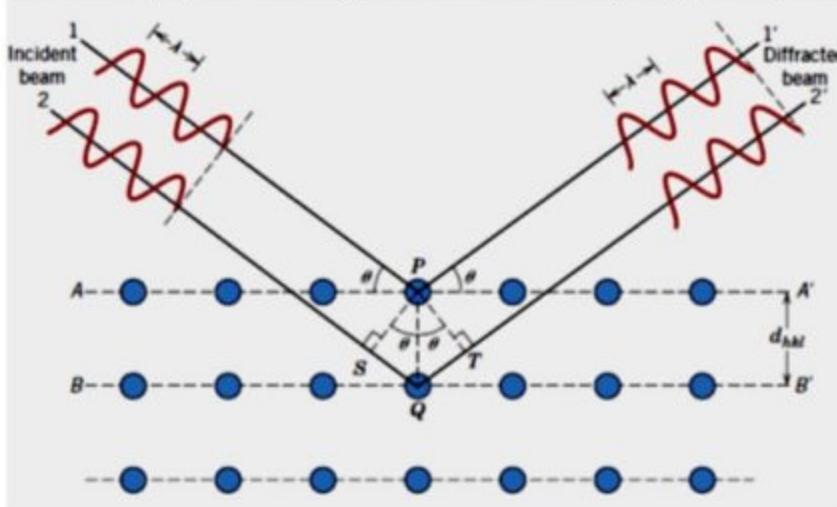
(f) Nuclear Structure

- This is studied by nuclear spectroscopic techniques, e.g., nuclear magnetic resonance (NMR) and Mössbauer spectroscopy.

BRAGG'S LAW

X-rays are a form of electromagnetic radiation that have high energies and short wavelengths — wavelengths on the order of the atomic spacings for solids. When a beam of x-rays impinges on a solid material, a portion of this beam will be scattered in all directions by the electrons associated with each atom or ion that lies within the beam's path.

Consider the two parallel planes of atoms $A-A'$ and $B-B'$ in Figure, which have the same h, k and l Miller indices and are separated by the interplanar spacing d_{hkl} . Now assume that a parallel, monochromatic and coherent (in-phase) beam of x-rays of wavelength λ is incident on these two planes at an angle of θ . Two rays in this beam, labeled 1 and 2, are scattered by atoms P and Q . Constructive interference of the scattered rays $1'$ and $2'$ occurs also at an angle θ to the planes, if the path length difference between $1-P-1'$ and $2-Q-2'$ is equal to a whole number, n , of wavelengths. The condition (Bragg's law) for diffraction is



$$n\lambda = \overline{SQ} + \overline{QT}$$

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

The Bragg's law relates the x-ray wave length and diffraction angle to interplanar spacing. The magnitude of the distance between two adjacent and parallel planes of atoms (i.e., the interplanar spacing d_{hkl}) is a function of the Miller indices (h, k , and l) as well as the lattice parameter(s). For example, for crystal structures that have cubic symmetry,

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

in which a is the lattice parameter (unit cell edge length). Relationships similar to the above equation, but more complex, exist for the other six crystal systems.

X-RAY DIFFRACTION

One common diffraction technique employs a powdered or polycrystalline specimen consisting of many fine and randomly oriented particles that are exposed to monochromatic x-radiation. Each powder particle (or grain) is a crystal, and having a large number of them with random orientations ensures that some particles are properly oriented such that every possible set of crystallographic planes will be available for diffraction. The *diffractometer* is an apparatus used to determine the angles at which diffraction occurs for powdered specimens.

One of the primary uses of x-ray diffractometry is for the determination of crystal structure. The unit cell size and geometry may be resolved from the angular positions of the diffraction peaks, whereas arrangement of atoms within the unit cell is associated with the relative intensities of these peaks. The intensity of the diffraction lines in the diffraction pattern allows identification of the crystal structure. Absences of peaks in certain positions indicate the presence of various planes within the structure as do the relative intensities of the peaks. This procedure is called **crystal structure analysis**.

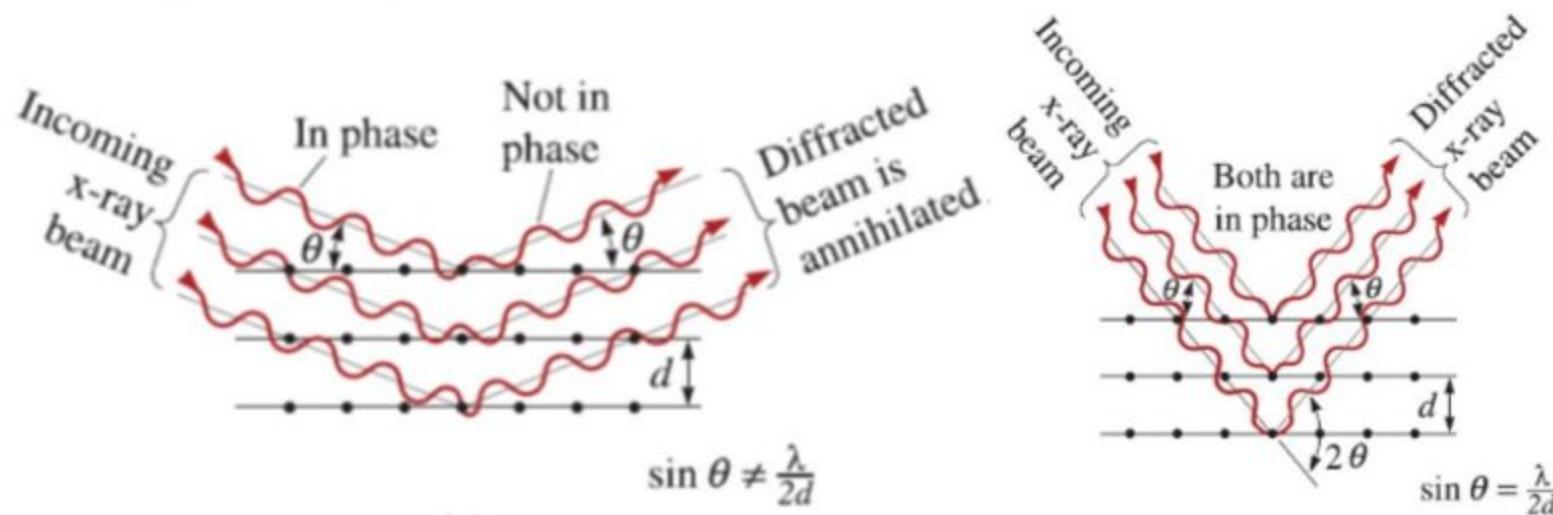
X-rays, as well as electron and neutron beams, are also used in other types of material investigations. For example, crystallographic orientations of single crystals are possible using x-ray diffraction (or Laue) photographs.

Other uses of x-rays include qualitative and quantitative chemical identifications and the determination of residual stresses and crystal size.

DIFFRACTION TECHNIQUES FOR CRYSTAL STRUCTURE ANALYSIS

A crystal structure of a crystalline material can be analyzed using **x-ray diffraction (XRD)** or electron diffraction. When a beam of x-rays having a single wavelength on the same order of magnitude as the atomic spacing in the material strikes that material, x-rays are scattered in all directions. Most of the radiation scattered from one atom cancels out radiation scattered from other atoms; however, x-rays that strike certain crystallographic planes at specific angles are reinforced rather than annihilated. This phenomenon is called **diffraction**. The x-rays are diffracted, or the beam is reinforced, when conditions satisfy **Bragg's law**.

When the material is prepared in the form of a fine powder, there are always at least some powder particles (crystals or aggregates of crystals) with planes oriented at the proper θ angle to satisfy Bragg's law. Therefore, a diffracted beam, making an angle of 2θ with the incident beam, is produced. In a *diffractometer*, a moving x-ray detector records the 2θ angles at which the beam is diffracted, giving a characteristic diffraction pattern. If we know the wavelength of the x-rays, we can determine the interplanar spacings and, eventually, the identity of the planes that cause the diffraction.



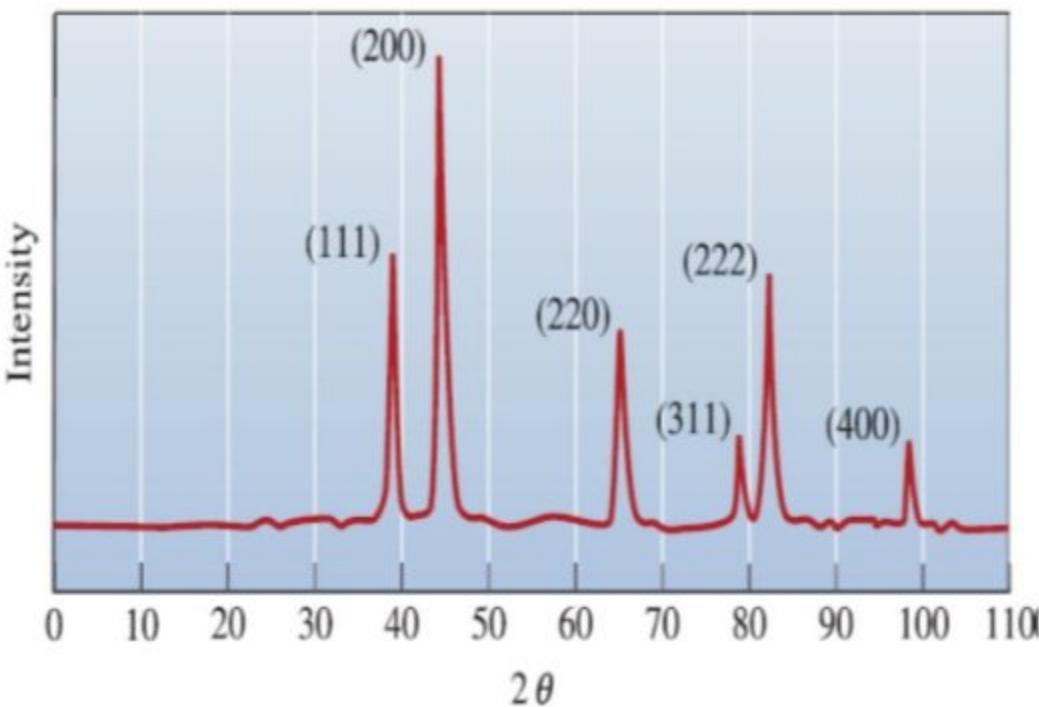
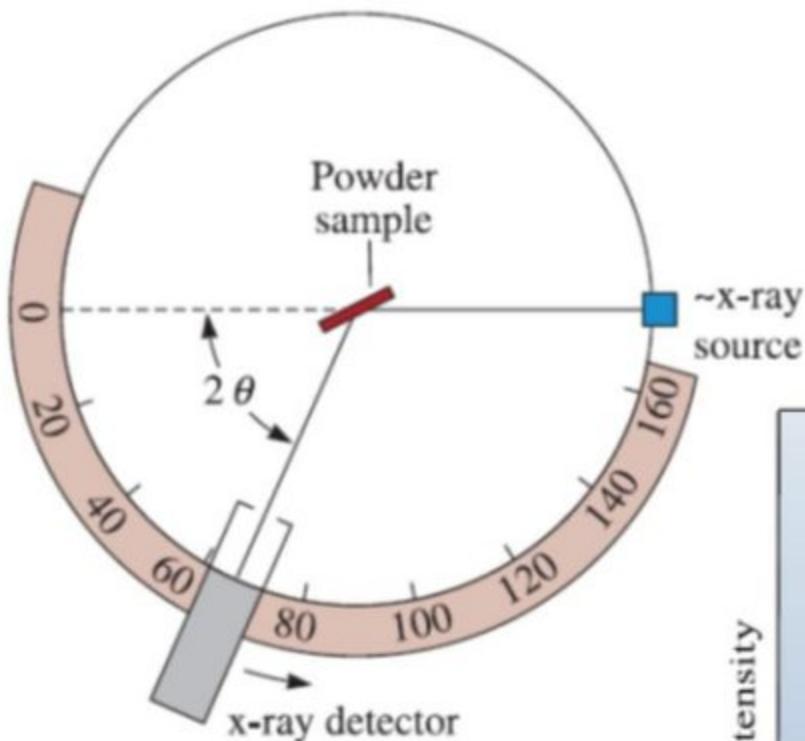


Figure 3-37 (a) Diagram of a diffractometer, showing powder sample, incident and diffracted beams. (b) The diffraction pattern obtained from a sample of gold powder.

CRYSTAL DEFECTS

According to the **law of nature** nothing is perfect and so crystals need not be perfect. They always found to have some defects in the arrangement of their constituent particles.

These defects affect the physical and chemical properties of the solid and play an important role in various processes.

For example, a process called doping leads to a crystal imperfection and it increases the electrical conductivity of a semiconductor material such as silicon.

The ability of ferromagnetic material such as iron, nickel etc., to be magnetized and demagnetized depends on the presence of imperfections.

Natural crystals always contain defects, often in abundance, due to the uncontrolled conditions under which they formed.

The importance of defects depends upon the material, type of defect, and properties, which are being considered.

Some properties, such as density and elastic constants, are proportional to the concentration of defects, and so a small defect concentration will have a very small effect on these.

Other properties, e.g. the colour of an insulating crystal or the conductivity of a semiconductor crystal, may be much more sensitive to the presence of small number of defects.

All solids, even the most ‘perfect’ crystals contain defects. Defects are of great importance as they can affect properties such as mechanical strength, electrical conductivity, chemical reactivity and corrosion. There are several terms used to describe defects which we must consider:

Intrinsic defects – present for thermodynamic reasons.

Extrinsic defects – not required by thermodynamics and can be controlled by purification or synthetic conditions.

Point defects – Occur at single sites. Random errors in a periodic lattice eg absence of atom from usual place (vacancy) or atom in a site not normally occupied (interstitial).

Extended defects – ordered in one, two and three dimensions. Eg. errors in the stacking of planes.

Every solid has a thermodynamic tendency to acquire point defects, as they introduce disorder and therefore increase entropy.

CRYSTAL DEFECTS

Material properties such as stiffness, density and electrical conductivity which are termed structure - insensitive, are not affected by the presence of defects in crystals.

Properties of greatest technical importance such as mechanical strength, ductility, crystal growth, magnetic hysteresis, dielectric strength, condition in semiconductors, which are termed structure sensitive.

Structure sensitive properties are greatly affected by the relatively minor changes in crystal structure caused by defects or imperfections.

Defects in materials have a significant influence on their electrical, optical, and magnetic properties.

Crystalline defects can be classified on the basis of their geometry as:

(i) Point imperfections, (ii) Line imperfections, (iii) Surface and grain boundary imperfections and (iv) Bulk / Volume imperfections.

The dimensions of a point defect are close to those of an interatomic space. With linear defects, their length is several orders of magnitude greater than the width.

Surface defects have a small depth, while their width and length may be several orders larger.

Volume defects (pores and cracks) may have substantial dimensions in all measurements.

Point Defects

Point defects distort the crystal lattice and have a certain influence on the physical properties. In commercially pure metals, point defects, increase the electric resistance and have almost no effect on the mechanical properties. Only at high concentrations of defects in irradiated metals, the ductility and other properties are reduced noticeably.

Point defect types are:

(i) Vacancies A vacancy is produced when an atom/ ion is missing from its normal site in the crystal structure. Arise either from imperfect packing during original crystallisation or from thermal vibrations of the atoms at higher temperatures.

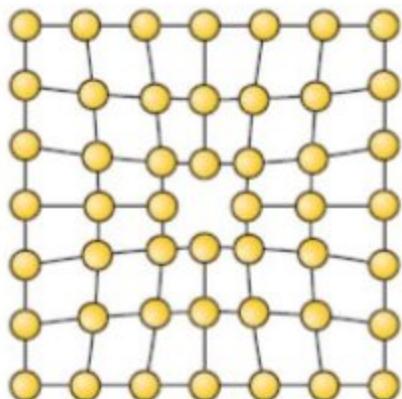
(ii) Interstitial Imperfections An extra atom or ion is inserted in the interstitial space or void between the regularly positioned atoms or ions within the crystal structure.

(iii) Frenkel Defect When an ion jumps from normal lattice point to an interstitial site. It is a combination of vacancy and interstitial defects. More common in ionic crystals.

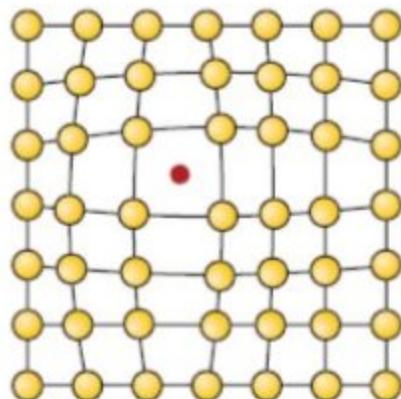
(iv) Schottky Defect a pair of positive and negative ions is missing from a crystal structure. This defect maintains ionic charge neutrality.

(v) Substitutional Defect A foreign atom or ion replaces the parent atom of the lattice and thus occupies the position of parent atom or ion.

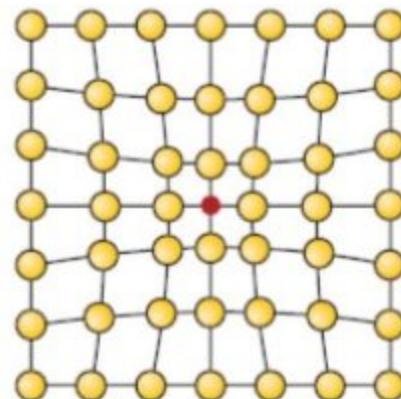
(vi) Phonon When the temperature is raised, thermal vibrations takes place. This results in the defect of a symmetry and deviation in shape of atoms or ions. This defect has much effect on the magnetic and electric properties.



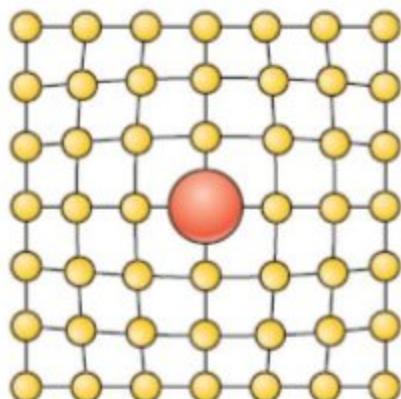
(a)



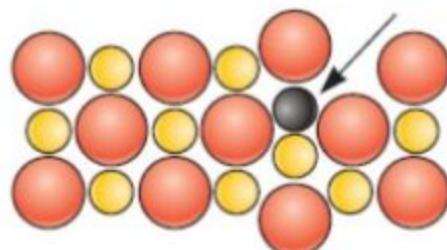
(b)



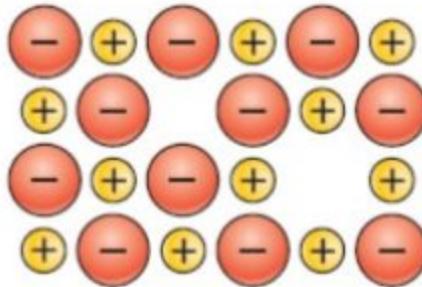
(c)



(d)



(e)



(f)

Figure 4-1 Point defects: (a) vacancy, (b) interstitial atom, (c) small substitutional atom, (d) large substitutional atom, (e) Frenkel defect, and (f) Schottky defect. All of these defects disrupt the perfect arrangement of the surrounding atoms.

Line Defects

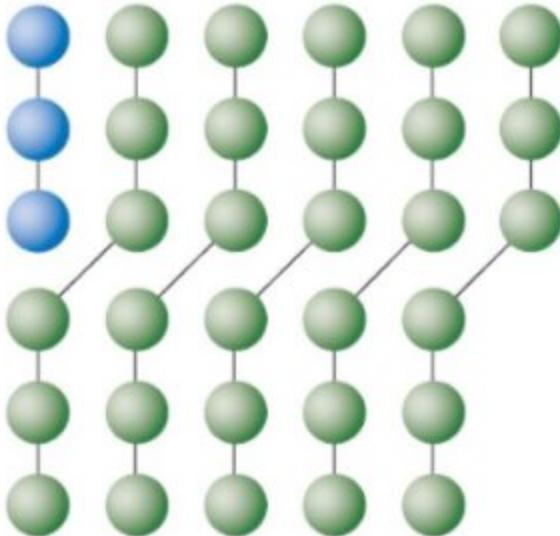
Dislocations are line imperfections in an otherwise perfect crystal. They formed during solidification of the material or when the material is deformed permanently. Dislocations influence electronic and optical properties of materials.

Three types of dislocations are: the screw dislocation, the edge dislocation, and the mixed dislocation.

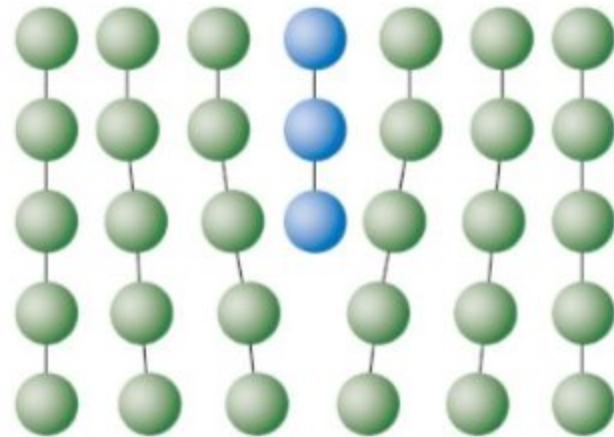
(i) Screw dislocation can be illustrated by cutting partway through a perfect crystal and then skewing the crystal by one atom spacing.

(ii) Edge dislocation can be illustrated by slicing partway through a perfect crystal, spreading the crystal apart, and partly filling the cut with an extra half plane of atoms. The bottom edge of this inserted plane represents the edge dislocation.

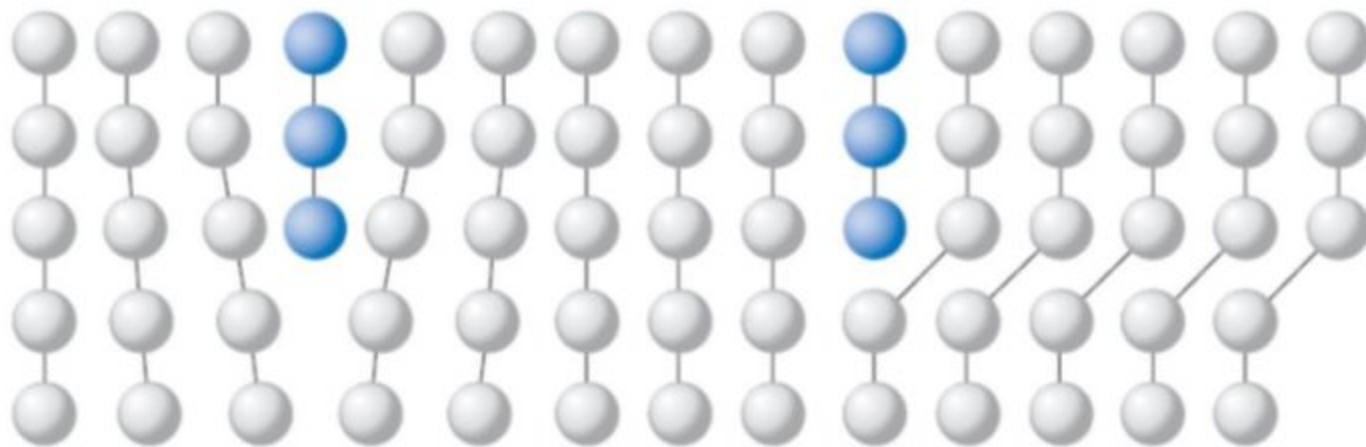
(iii) Mixed dislocations have both edge and screw components, with a transition region between them.



Screw dislocation



Edge dislocation



Mixed dislocation

Surface Defects

Surface defects are the boundaries, or planes, that separate a material into regions. For example, each region may have the same crystal structure but different orientations.

Material Surface: The exterior dimensions of the material represent surfaces at which the crystal abruptly ends. Each atom at the surface no longer has the proper coordination number, and atomic bonding is disrupted. The exterior surface may also be very rough, may contain tiny notches, and may be much more reactive than the bulk of the material. In petroleum refining and many other areas of technology, we make use of high surface area catalysts for enhancing the kinetics of chemical reactions.

Grain Boundaries: The microstructure of many engineered ceramic and metallic materials consists of many grains. A **grain** is a portion of the material within which the arrangement of the atoms is nearly identical; however, the orientation of the atom arrangement, or crystal structure, is different for each adjoining grain. A **grain boundary**, the surface that separates the individual grains, is a narrow zone in which the atoms are not properly spaced.

Surface Defects (... continued)

Small Angle & High Angle Grain Boundaries are arrays of dislocations that produce mis-orientations between the adjoining crystals. Because the energy of the surface is less than that of a regular grain boundary, the small angle grain boundaries are not as effective in blocking slip. Small angle boundaries formed by edge dislocations are called **tilt boundaries**, and those caused by screw dislocations are called **twist boundaries**.

Phase Boundaries exist in multiphase materials, wherein a different phase exists on each side of the boundary. Each of the constituent phases has its own distinctive physical and/or chemical characteristics. Phase boundaries play an important role in determining the mechanical characteristics of some multiphase metal alloys.

Surface Defects (... continued)

A twin boundary is a special type of grain boundary across which there is a specific mirror lattice symmetry. Atoms on one side of the boundary are located in mirror-image positions of the atoms on the other side.

It is a plane across which there is a special mirror image misorientation of the crystal structure. Twinning occurs on a definite crystallographic plane and in a specific direction, both of which depend on the crystal structure.

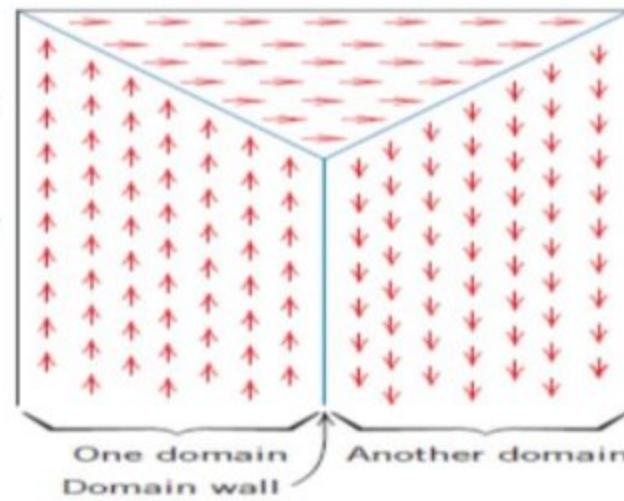
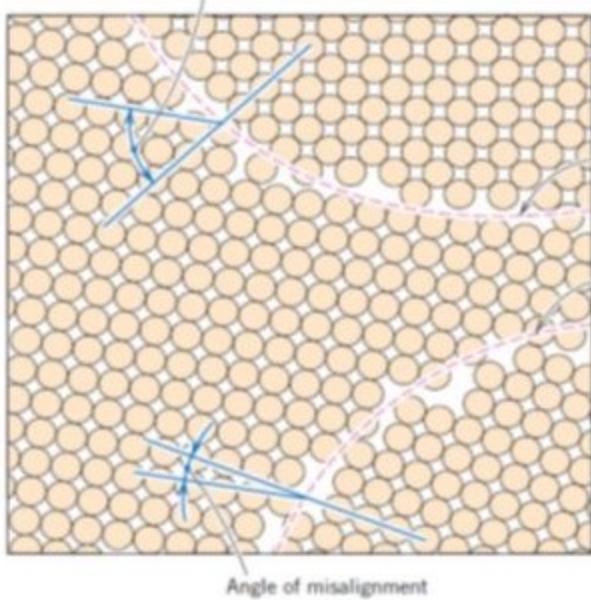
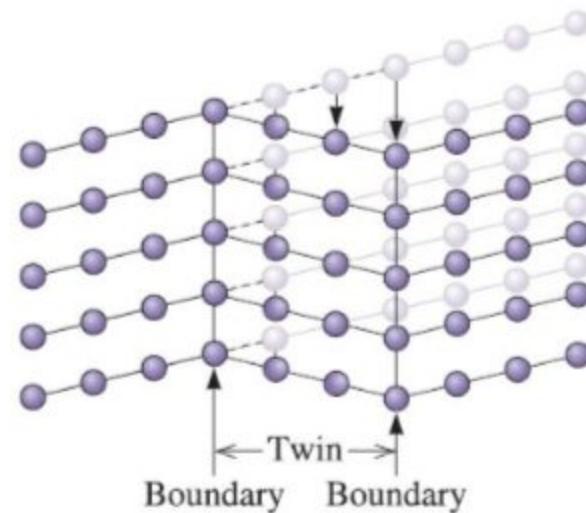
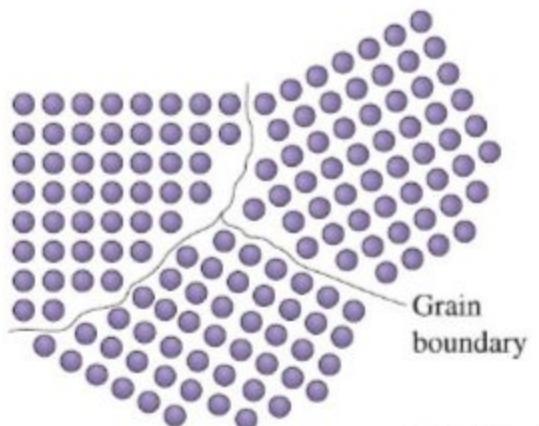
Twins can be produced when a shear force, acting along the twin boundary, causes the atoms to shift out of position. Twinning occurs during deformation or heat treatment of certain metals.

Twins result from atomic displacements that are produced from applied mechanical shear forces (mechanical twins), and also during annealing heat treatments following deformation (annealing twins).

The twin boundaries interfere with the slip process and increase the strength of the metal.

Stacking faults occur in FCC metals, an error in the stacking sequence of close-packed planes. Stacking faults interfere with the slip process.

Magnetic domain wall is the boundary that separates regions having different directions of magnetization.



Bulk / Volume Defects

Bulk / volume defects include pores, cracks, foreign inclusions, and other phases. They are normally introduced during processing and fabrication steps.

IMPORTANCE OF CRYSTAL DEFECTS

Dislocations are most significant in metals and alloys since they provide a mechanism for plastic deformation.

Plastic deformation refers to irreversible deformation or change in shape that occurs when the force or stress that caused it is removed. The applied stress causes dislocation motion, and the cumulative effect of slip of numerous dislocations is plastic deformation.

The slip process is particularly important in understanding the mechanical behavior of metals.

First, slip explains why the strength of metals is much lower than the value predicted from the metallic bond. If slip occurs, only a tiny fraction of all of the metallic bonds across the interface need to be broken at any one time, and the force required to deform the metal is small.

Second, slip provides ductility in metals. If no dislocations were present, an iron bar would be brittle and could not be shaped by metalworking processes, such as forging, into useful shapes.

Third, we control the mechanical properties of a metal or alloy by interfering with the movement of dislocations. An obstacle introduced into the crystal prevents a dislocation from slipping unless we apply higher forces. Thus, the presence of dislocations helps strengthen metallic materials.

IMPORTANCE OF CRYSTAL DEFECTS (... continued)

Enormous numbers of dislocations are found in materials.

The **dislocation density**, or total length of dislocations per unit volume, is usually used to represent the amount of dislocations present.

Dislocation densities of 10^6 cm/cm^3 are typical of the softest metals, while densities up to 10^{12} cm/cm^3 can be achieved by deforming the material.

Dislocations also influence **electronic** and **optical properties** of materials. For example, the resistance of pure copper increases with increasing dislocation density.

The resistivity of pure copper also depends strongly on small levels of impurities.

Dislocation-free silicon crystals are used since this allows the charge carriers such as electrons to move more freely through the solid.

IMPORTANCE OF CRYSTAL DEFECTS (... continued)

Dislocations has a deleterious effect on the performance of photo detectors, light emitting diodes, lasers, and solar cells.

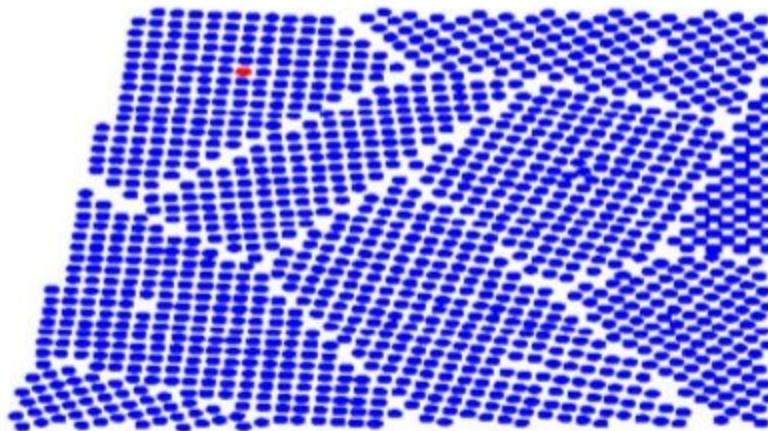
These devices are often made from compound semiconductors such as gallium arsenide-aluminum arsenide (**GaAs-AlAs**).

Dislocations in these materials can originate from concentration inequalities in the melt from which crystals are grown or stresses induced because of thermal gradients that the crystals are exposed to during cooling from the growth temperature.

Extended and point defects play a major role in influencing mechanical, electrical, optical, and magnetic properties of engineered materials.

Crystal defects

- Defects are imperfections (lattice irregularity) in materials

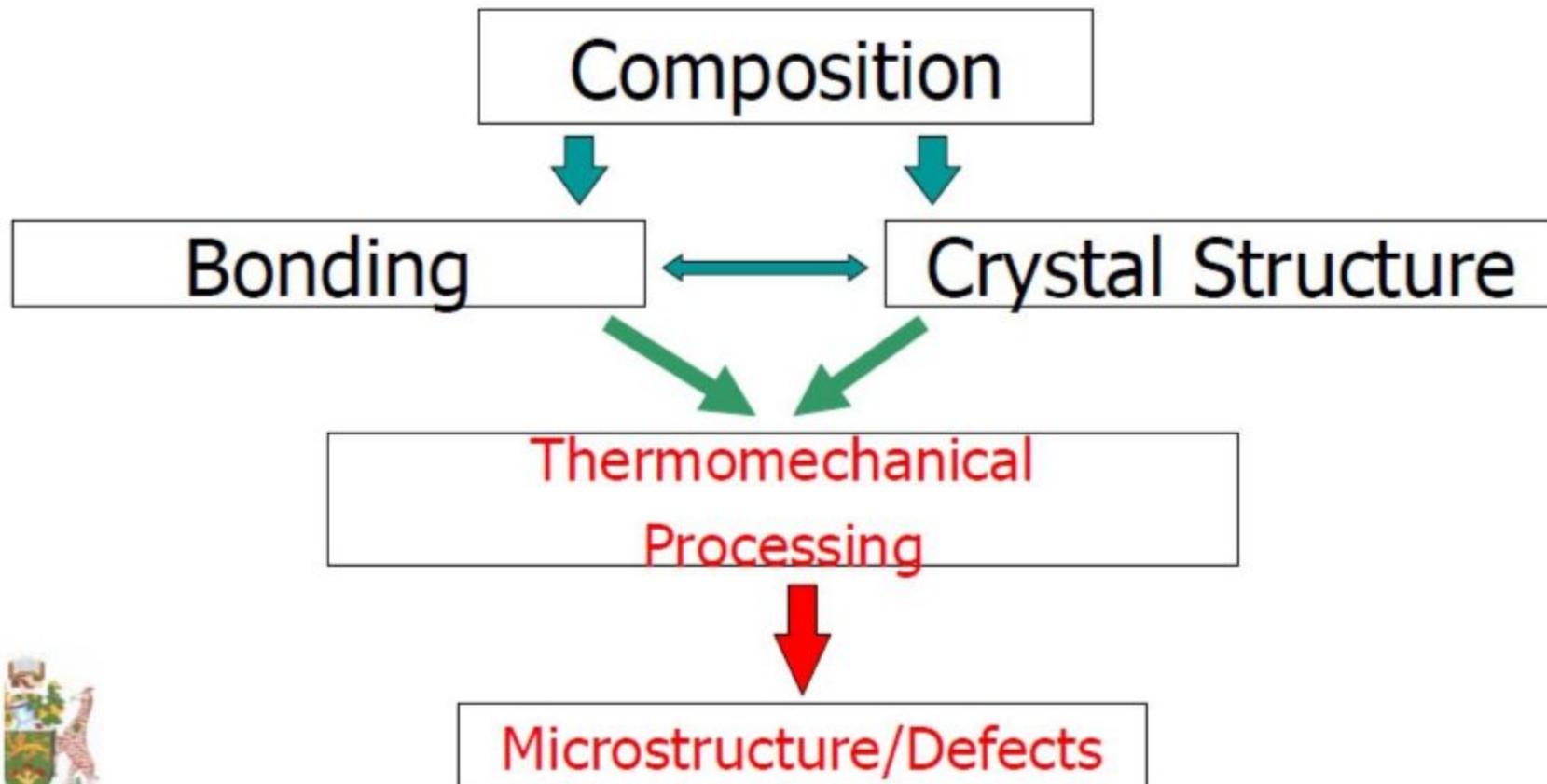


poly-crystal with many defects

“Crystals are like people, it is the defects that make them interesting!” - Colin Humphreys.



- Defects arise during processing due to
 - Accidents in crystal growth from the melt
 - Mechanical deformation of the crystal etc
- Processing determines nature and type of defects



Imperfections in Crystalline Solids

There is no such thing as a perfect crystal.

- Real crystals contain various types of imperfections. We have briefly touched on the fact that many engineering materials are polycrystals
- Many of the important properties of materials are due to the presence of these imperfections

"Solids are like people - imperfect!"

"It's the defects that make them unique & interesting"

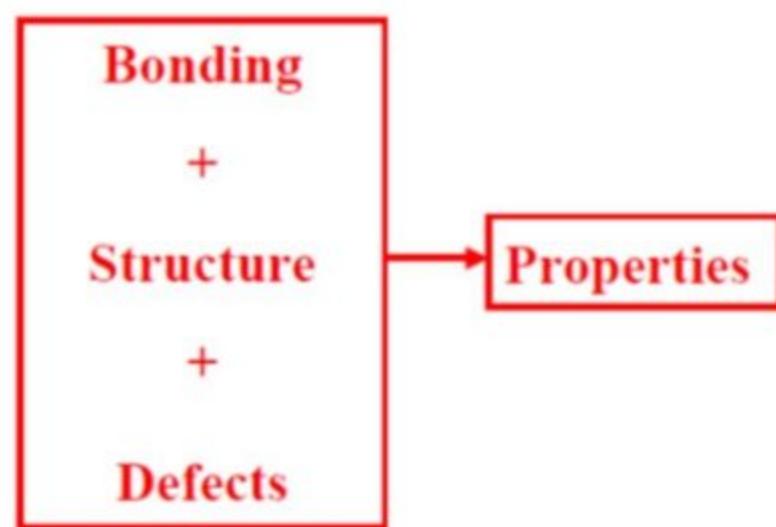
- Point, line and area defects arise in solids.
- The number and type of defects depend on several factors (e.g. the concentration of vacancies can be controlled by temperature).
- The properties of the materials are affected by defects (e.g. defects control mechanical, electrical, optical properties...)
- Defects can be wanted or unwanted depending of the specific application.
- Defects have a profound impact on the macroscopic properties of materials.

Type of defects

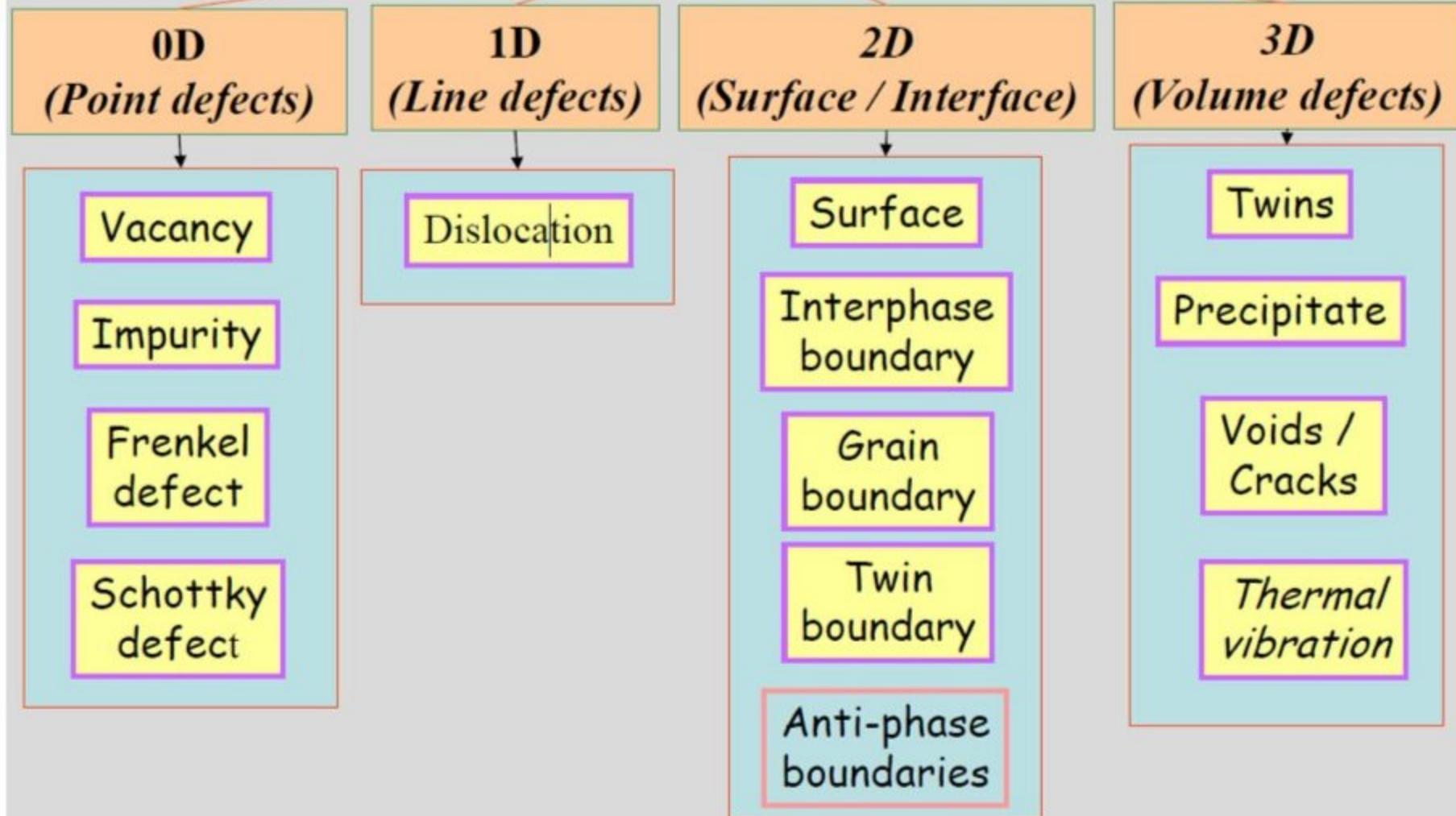
Defects are classification according to geometry (point, line or plane).

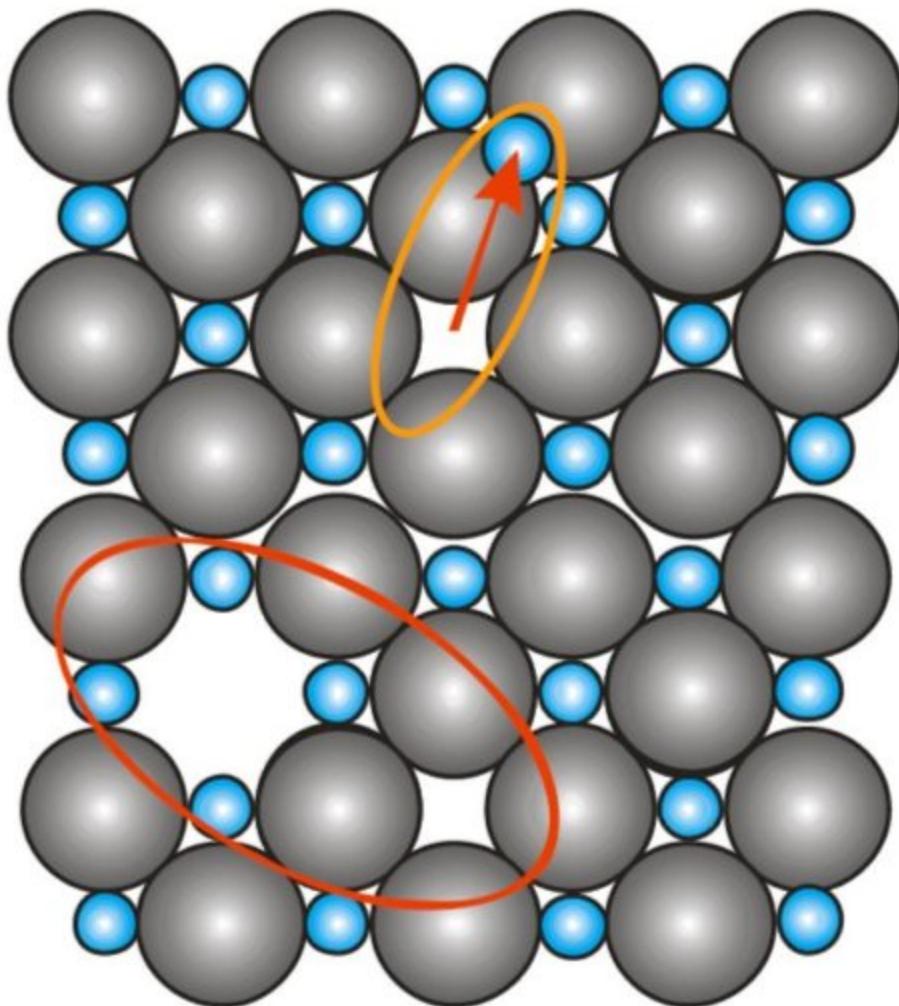
☰ 4 categories

- Point defects - 0D
- Line defects - 1D
- Area defects - 2D
- Volume defects - 3D



CLASSIFICATION OF DEFECTS BASED ON DIMENSIONALITY





Frenkel defect

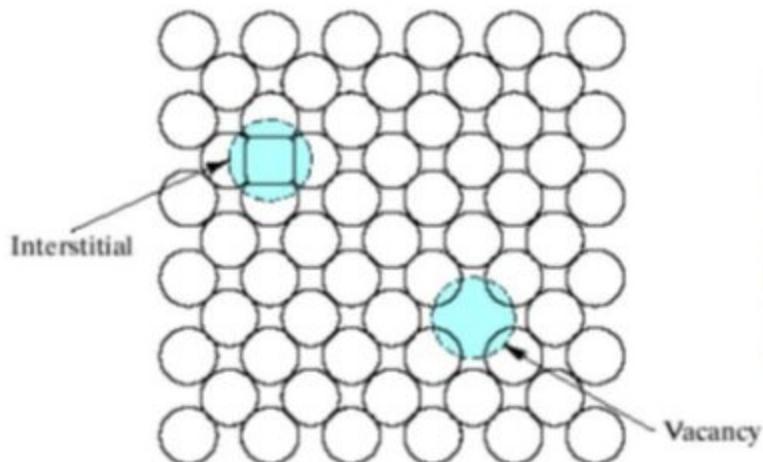
Cation vacancy
+
cation interstitial

A vacancy-interstitially combination

Schottky defect

Cation vacancy
+
anion vacancy

A pair of oppositely charged ion vacancies



Vacancy - Unoccupied atom site

Interstitial - An atomic occupying an interstitial site

Substitutions in Ionic Solids

- Rules for substitution
 - Similarity of sites
 - Charge balance
- Effect on ionic conductivity

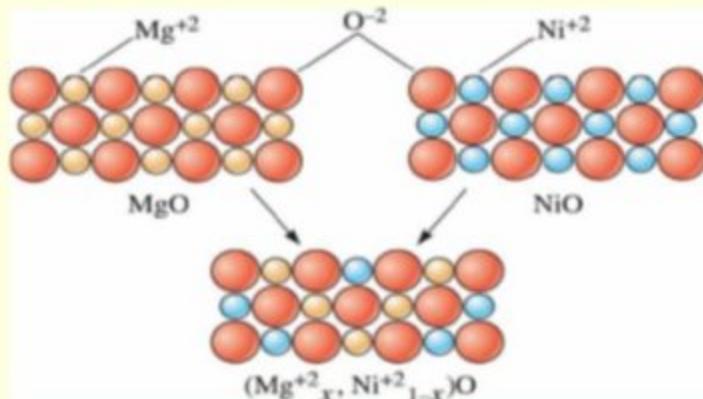
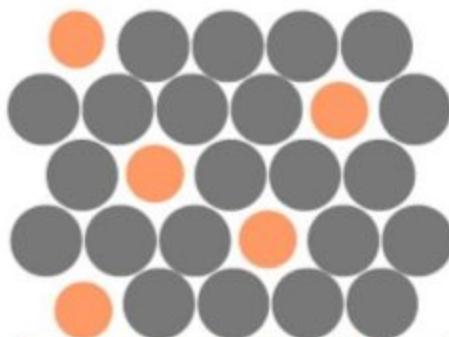


Figure 10-6
MgO and NiO have similar crystal structures, ionic radii, and valences; thus the two ceramic materials can form solid solutions.

Point Defects in Alloys

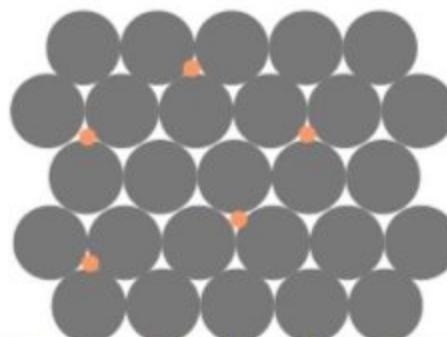
Two outcomes if impurity (B) added to host (A):

- Solid solution of B in A (i.e., random dist. of point defects)



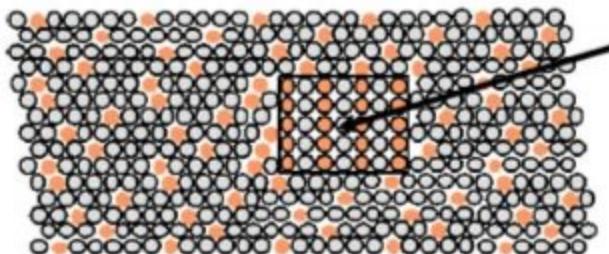
Substitutional solid soln.
(e.g., Cu in Ni)

OR



Interstitial solid soln.
(e.g., C in Fe)

- Solid solution of B in A plus particles of a **new phase** (usually for a larger amount of B)



Second phase particle
--different composition
--often different structure.

Linear Defects or Dislocations

Linear defects or dislocations : Associated with mechanical deformation

Edge Dislocation

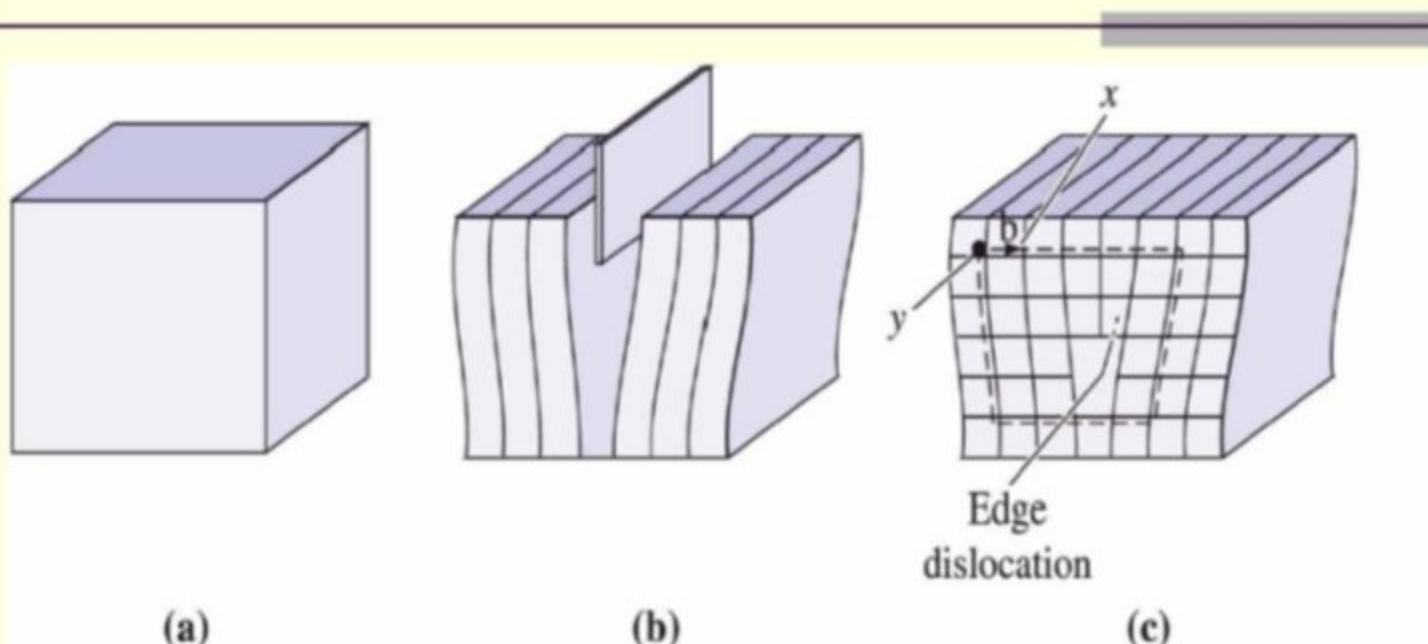
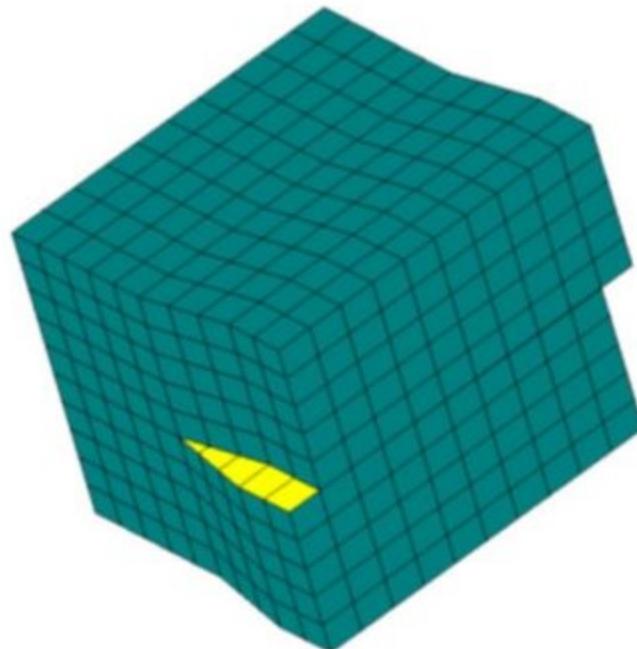
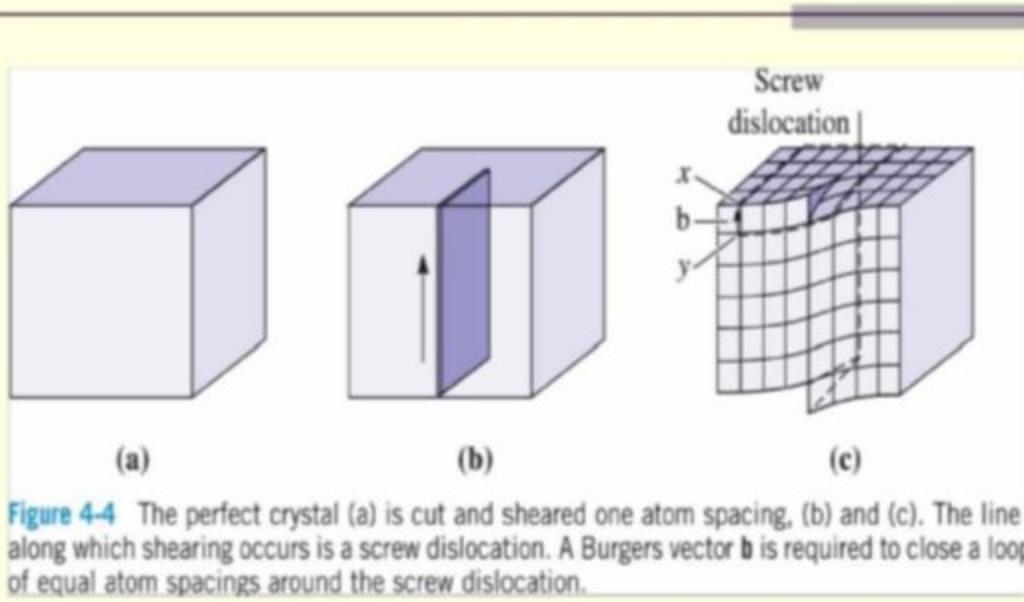


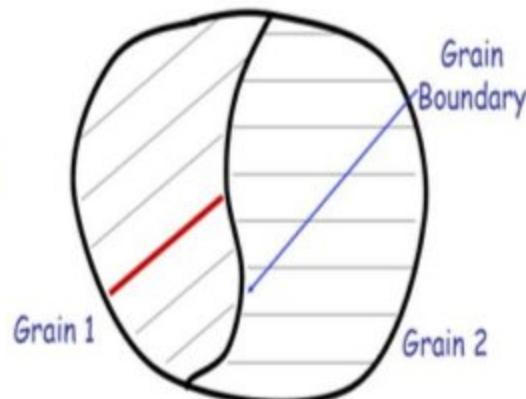
Figure 4-5 The perfect crystal in (a) is cut and an extra plane of atoms is inserted (b). The bottom edge of the extra plane is an edge dislocation (c). A Burgers vector \mathbf{b} is required to close

Screw Dislocation



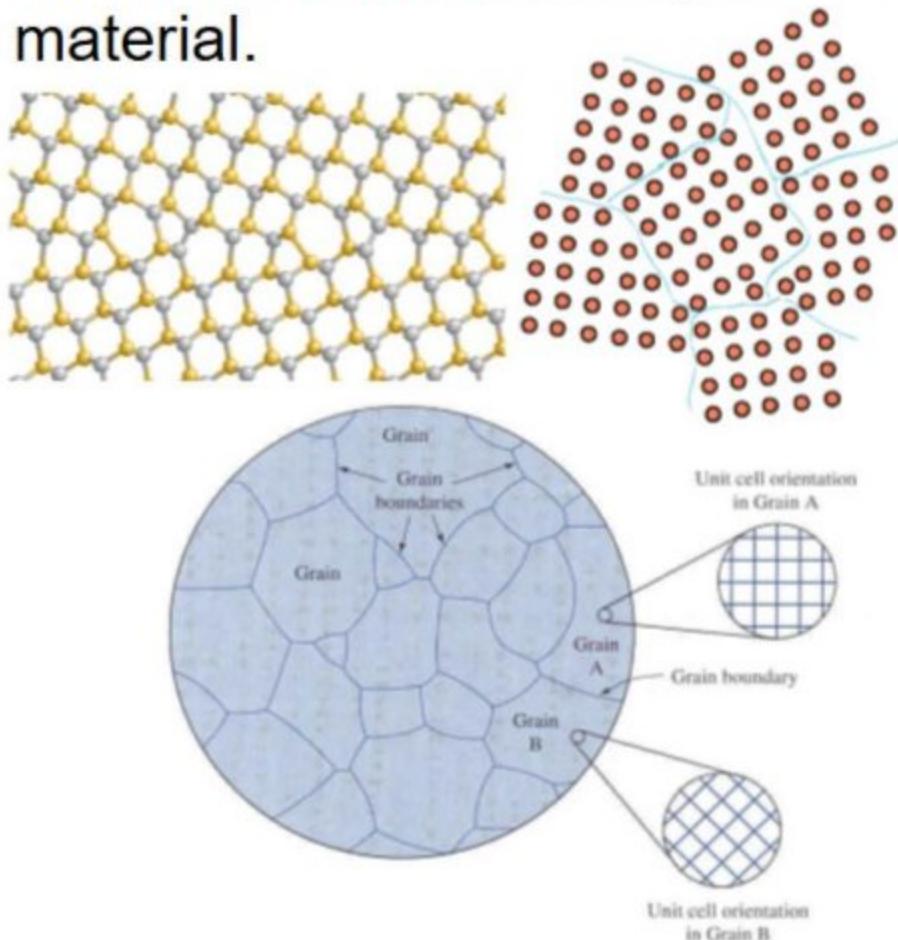
A dislocation line cannot end abruptly inside a crystal.

It can end on (1) Free surfaces, (2) Grain boundaries, (3) On other dislocations at a point called a node, and (4) On itself forming a loop



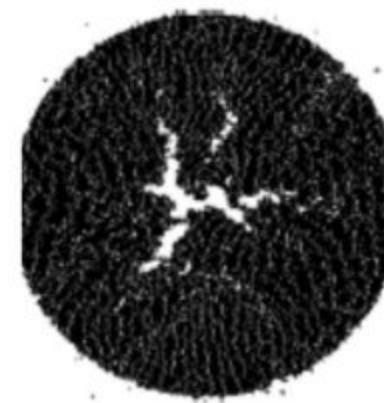
Surface Defects

A grain boundary is the interface between two grains in a polycrystalline material and it tends to decrease the electrical and thermal conductivity of the material.



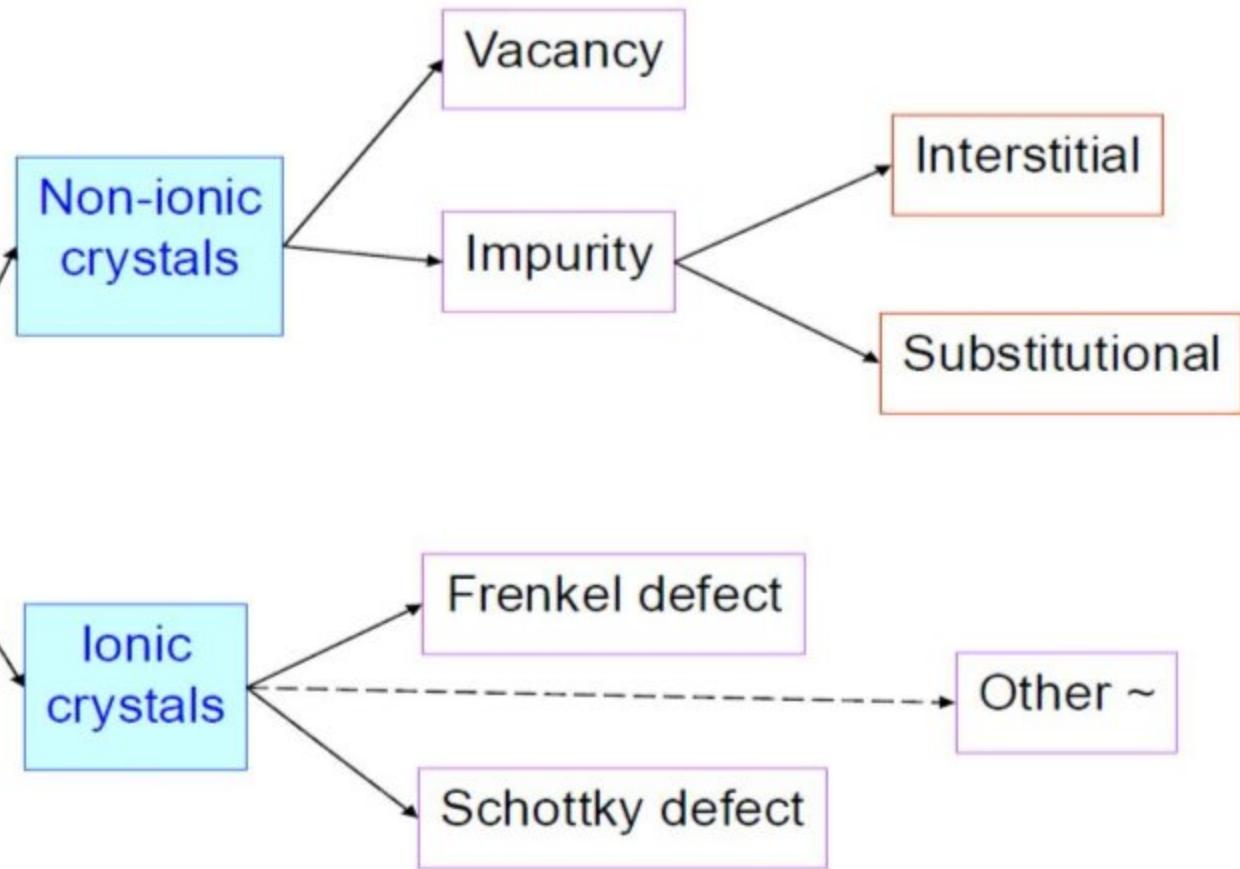
Bulk or Volume Defects

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



A cluster of microcracks in a melanin granule irradiated by a short laser pulse. Computer simulation by L. V. Zhigilei and B. J. Garrison.

0D *(Point defects)*



Addition of impurities may be intentional or unintentional, e.g.,

- Small amounts of carbon in iron makes steel stronger.
- Boron in silicon change its electrical properties.

Alloys - deliberate mixtures of metals to make a stronger material

(b) Linear defects - (dislocations)

- Are 1-D defects around which atoms are misaligned. Are also called Dislocations
- Are responsible for causing plastic deformation
- \exists two types

(i) Edge dislocation (symbol \perp)

- Associated with extra half-plane of atoms inserted in a crystal

(ii) Screw dislocation (symbol)

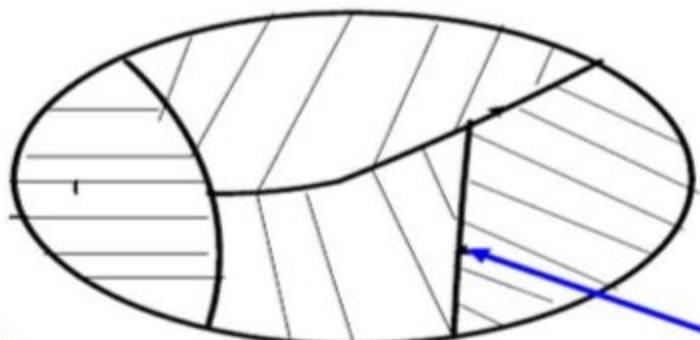
- Associated with spiral planar ramp resulting from shear deformation

Role of dislocations

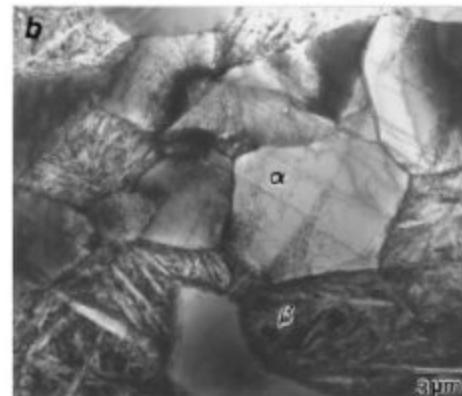
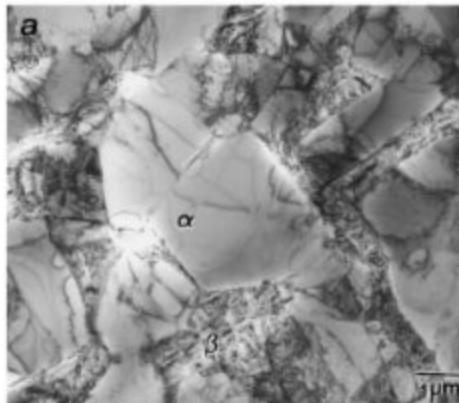
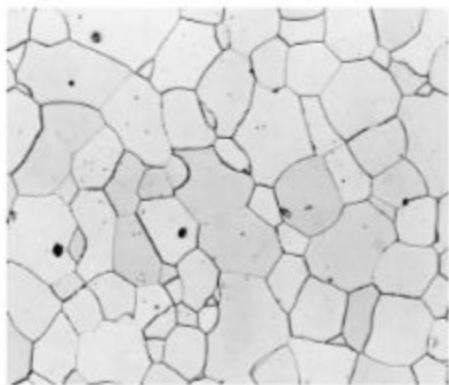
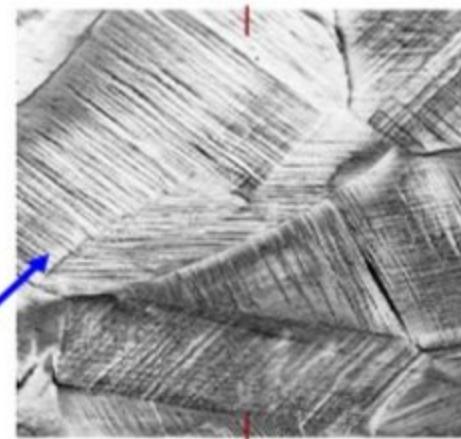
- Play an important role in deformation processes like creep, Fatigue and fracture.
- Play a 'constructive role' in crystal growth.
- Provide short circuit paths for diffusion (pipe diffusion).

(c) Area defects (Grain boundaries)

- Are interfaces between crystals that have different crystallographic orientation, composition or dimensions of the crystals lattice.
- They impede dislocation motion (crystal slip).



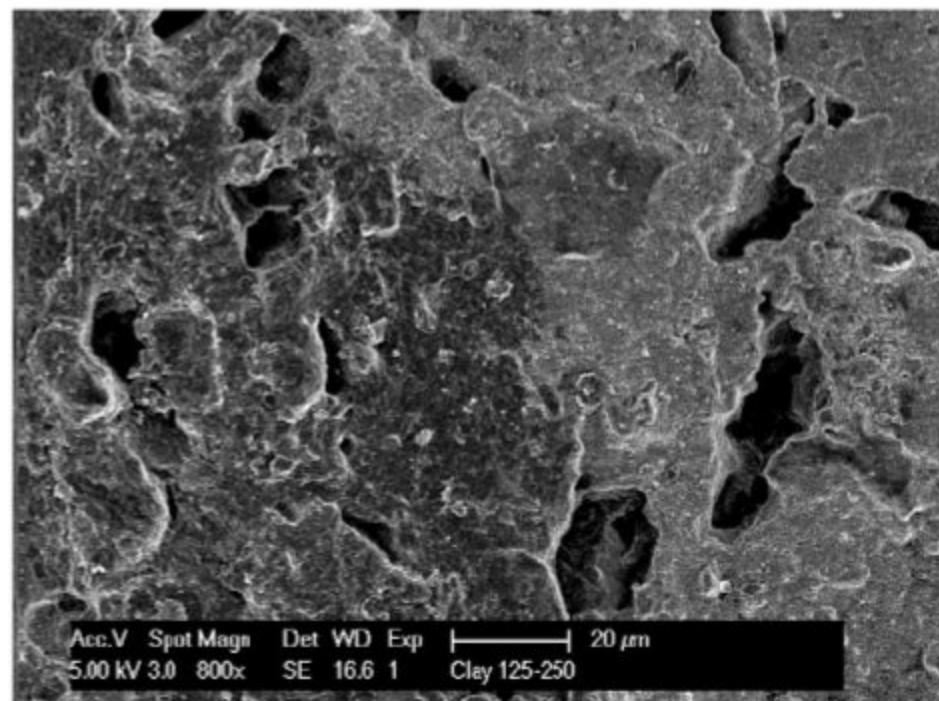
Grain boundaries



(d) Volume defects

- **Are distributed** within volume of material (pores, cracks, foreign inclusions etc)
- They affect optical, thermal, mechanical & electrical properties

PORES IN CERAMICS



The ability of a metal to deform depends on the ability of dislocations to move

Restricting dislocation motion makes the material stronger

Mechanisms of strengthening in single-phase metals:

- grain-size reduction
- solid-solution alloying
- strain hardening

Ductile metals become stronger when they are deformed plastically at temperatures well below the melting point.

The reason for strain hardening is the increase of dislocation density with plastic deformation. The average distance between dislocations decreases and dislocations start blocking the motion of each other.

- *Macrostructure* can be observed with naked eye
 - coarse grains can be revealed this way (e.g. Al streetlight posts e.g. zinc galvanized garbage cans)
- *microstructure* is when the grains can only be observed with a microscope → *microscopy*
 - imaged using a camera for archiving
 - *photomicrograph*

ALLOY FORMATION

A homogeneous mixture of two or more metals or a metal and a non-metal when fused together at a certain temperature forms a new metal after solidification, termed as an alloy.

Alloys are normally harder than their components, less ductile and may have a much lower conductivity, whereas the highly purified single crystal of a metal is very soft and malleable, with high electrical conductivity.

Pure metals are used only for specific applications. The alloy is usually more corrosion resistant and less affected by atmospheric conditions.

In the solid state an alloy may be present in one or more of the following forms:

- (i) As a solid solution
- (ii) As an intermediate phase or intermediate chemical compound
- (iii) As a finely divided mechanical mixture of solid solution
- (iv) As a finely divided mechanical mixture of the metals
- (v) As a finely divided mechanical mixture of chemical compounds of metals, the individual metals and solid solutions.

ALLOY FORMATION (... continued)

When an alloy is in a liquid state the atoms of the constituent are distributed randomly through the liquid. When solidification takes place, there appears number of possibilities. A number of different types of solutions may form, as follows:

(a) Simple Eutectic Type In this case the two components of an alloy system (binary) are soluble in liquid state but separate out in the solid state, each maintaining its own separate identity. In the solid state, the two components are said to be insoluble in each other. The solution is called *eutectic type*.

(b) Solid Solution Type When the two components of a binary alloy remain completely mixed in each other both in liquid and solid state, the two components are said to be soluble in each other and a different type of solution may be formed. It is called a solid solution.

(c) Combination Type On solidifying, the two components of a binary alloy may show limited solubility in each other. This type of solution combines characteristics of both components.

(d) Inter-metallic Compounds It is observed that the elements may combine to form inter-metallic compounds on solidification, when their affinity is great. These types of compounds may find place in between the solid solution and chemical compound.

SOLID SOLUTION TYPES

A solid solution forms when the solute atoms are added to the host material, the crystal structure is maintained, and no new structures are formed.

In other words, when elements completely dissolve in each other in liquid and or solid state the resulting phase is called *solid solution*.

Solid solution may be defined as solution in the solid state which consists of two (or more) kinds of atoms combined in one type of crystal structure (space lattice).

Solid solutions form readily when solvent and solute atoms have similar sizes and electron structures, so that it is compositionally or chemically homogeneous and the component atoms of the elements cannot be distinguished physically or separated mechanically.

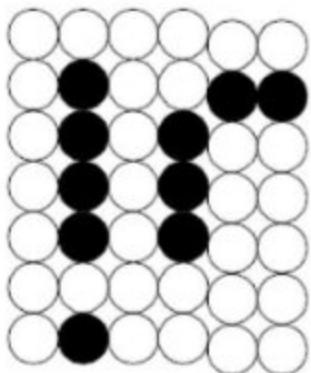
There is a homogeneous distribution of the constituents in the solid state so as to form a single phase or solid solution.

Basically, solid solutions are of two types:

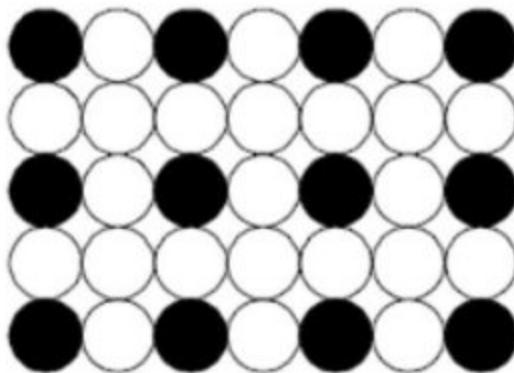
(i) Substitutional Solid Solution

- (a)** Random substitutional solid solutions
- (b)** Ordered substitutional solid solutions.

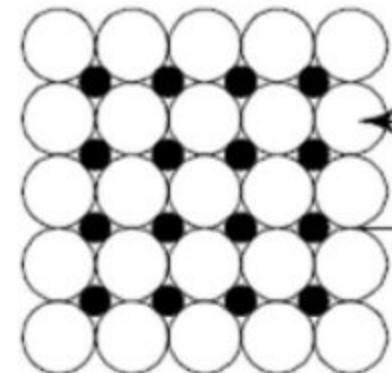
(ii) Interstitial Solid Solution.



Random
solid solution



Ordered
solid solution



Interstitial
solid solution

Substitutional Solid Solution: The solute or impurity atoms replace or substitute for the host atoms (as shown in Figure). Several features of the solute and solvent atoms determine the degree to which the solute dissolves in the solvent, are: (1) *Atomic size factor*, (2) *Crystal structure*, (3) *Electronegativity* and (4) *Valences*.

Interstitial Solid Solution: In crystal lattice of many metals, small sized atoms of H, N, C and B can occupy the empty spaces (interstices) to form interstitial solid solutions.

In some alloys both interstitial and substitutional solid solutions are formed to some good extent. For example, in Cr-Ni Steel the carbon occupies interstitially whereas Cr and Ni occupy substitutionally in the iron lattice.

SOLID SOLUTION TYPES

Lattice distortions appearing on the formation of interstitial solid solutions are stronger than those in the substitutional solid solutions.

Increase in solute concentration in a solid solution increases the electric resistance, coercive force, hardness and strength, but decreases the ductility and toughness.

Solid solutions are crystals whose properties are close to those of the solvent, since they retain the same crystal lattice and type of bond.

Solid solutions of metals have high plasticity and they are readily deformable in the hot state and many of them, in the cold state.

Solid solutions are the bases of most commercial structural and special alloys.

Summary: Solid solutions in metallic or ceramic materials exist when elements or compounds with similar crystal structures form a single phase that is chemically homogeneous.

- Solid-solution strengthening is accomplished in metallic materials by the formation of solid solutions. The point defects created restrict dislocation motion and cause strengthening.
- The degree of solid-solution strengthening increases when (1) the amount of the alloying element increases and (2) the atomic size difference between the host material and the alloying element increases.
- The amount of alloying element (or compound) added to produce solid solution strengthening is limited by the solubility of the alloying element or compound in the host material.
- Solid-solution strengthening increases strength and hardness; decreases ductility and electrical conductivity of metallic materials. It also provides good high-temperature properties to the alloy.
- In solid-solution formation, solidification begins at the liquidus temperature and is completed at the solidus temperature.

STRUCTURAL EXAMINATION USING MICROSCOPY

Optical, electron, and scanning probe microscopes are commonly used to investigate the microstructural features of materials. Microscopy is an extremely useful tool in the study and characterization of materials.

- 1. Optical microscopy:** Conventional light microscopy, Fluorescence microscopy, confocal/multiphoton microscopy and Stimulated emission depletion microscopy.
- 2. Scanning probe microscopy:** Scanning tunnelling microscopy (STM), Atomic force microscopy (AFM), Near-field scanning optical microscopy and others.
- 3. Electron microscopy:** Scanning electron microscopy (SEM), Transmission electron microscopy (TEM), Scanning transmission electron microscopy (STEM), Focus ion beam microscopy (FIB)

Important applications of microstructural examinations are as follows:

1. to ensure that the associations between the properties and structure (and defects) are properly understood,
2. to predict the properties of materials once these relationships have been established,

STRUCTURAL EXAMINATION USING MICROSCOPY (... continued)

3. to design alloys with new property combinations,
4. to determine whether a material has been correctly heat-treated, and
5. to ascertain the mode of mechanical fracture.
6. to analyse the material structural aspects in
 - (i) *Crystal morphology and symmetry* - Crystal fragments, classify isotropic and anisotropic substances, check possible symmetry.
 - (ii) *Phase identification, purity and homogeneity* - standard optical data (refractive indices and optical axes) for comparison, phase analysis (impurities with separated crystalline / amorphous phase), single vs. twinned crystal;
 - (iii) *Crystal defects* – grain boundaries and dislocations and;
 - (iv) *Refractive index* determination.

Optical Microscopy

With optical microscopy, the light microscope is used to study the microstructure; optical and illumination systems are its basic elements.

For materials that are opaque to visible light (all metals and many ceramics and polymers), only the surface is subject to observation, and the light microscope must be used in a reflecting mode.

Contrasts in the image produced result from differences in reflectivity of the various regions of the microstructure. Investigations of this type are often termed *metallographic*, because metals were first examined using this technique.

Normally, careful and meticulous surface preparations are necessary to reveal the important details of the microstructure. The specimen surface must first be ground and polished to a smooth and mirror-like finish.

The microstructure is revealed by a surface treatment using an appropriate chemical reagent in a procedure termed *etching*. The chemical reactivity of the grains of some single-phase materials depends on crystallographic orientation.

Optical Microscopy

Consequently, in a polycrystalline specimen, etching characteristics vary from grain to grain.

The lustre or texture of each grain depends on its reflectance properties.

Also, small grooves form along grain boundaries as a consequence of etching. Because atoms along grain boundary regions are more chemically active, they dissolve at a greater rate than those within the grains.

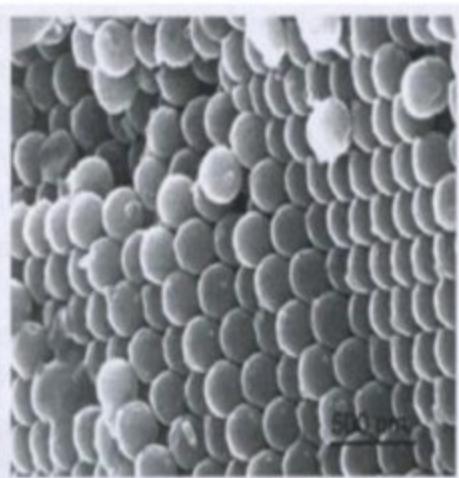
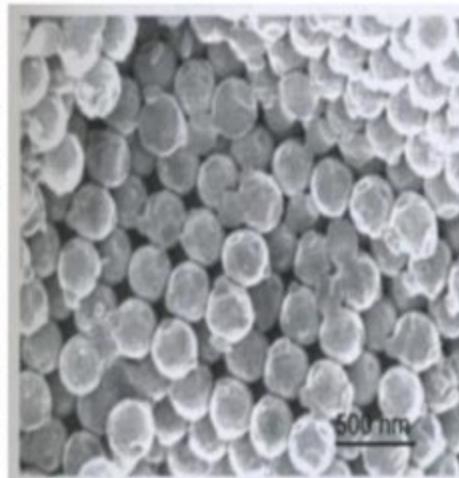
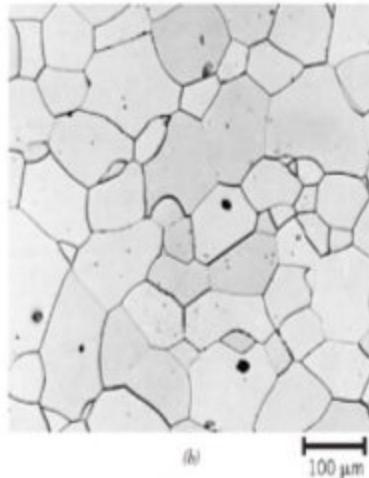
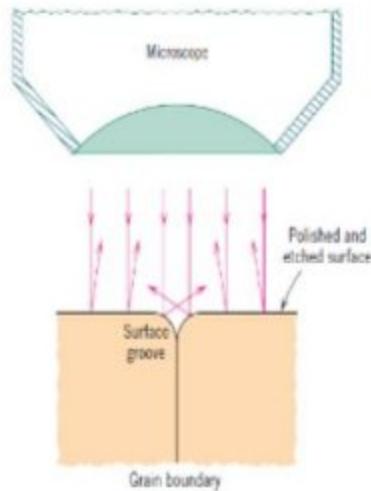
These grooves become discernible when viewed under a microscope because they reflect light at an angle different from that of the grains themselves.

When the microstructure of a two-phase alloy is to be examined, an etchant is often chosen that produces a different texture for each phase so that the different phases may be distinguished from each other.

Electron Microscopy

The upper limit to the magnification possible with an optical microscope is approximately 2000 times. Consequently, some structural elements are too fine or small to permit observation using optical microscopy. Under such circumstances the electron microscope, which is capable of much higher magnifications, may be employed.

An image of the structure under investigation is formed using beams of electrons instead of light radiation. High magnifications and resolving powers of these microscopes are consequences of the short wavelengths of electron beams. The electron beam is focused and the image formed with magnetic lenses; otherwise the geometry of the microscope components is essentially the same as with optical systems. Both transmission and reflection beam modes of operation are possible for electron microscopes.



Transmission Electron Microscopy

The image seen with a **Transmission Electron Microscope (TEM)** is formed by an electron beam that passes through the specimen.

Details of internal micro-structural features are accessible to observation; contrasts in the image are produced by differences in beam scattering or diffraction produced between various elements of the microstructure or defect.

Because solid materials are highly absorptive to electron beams, a specimen to be examined must be prepared in the form of a very thin foil; this ensures transmission through the specimen of an appreciable fraction of the incident beam.

The transmitted beam is projected on to a fluorescent screen or a photographic film so that the image may be viewed.

Magnifications approaching **1,000,000x** are possible with transmission electron microscopy, which is frequently used to study dislocations.

Scanning Electron Microscopy

A more recent and extremely useful investigative tool is the **Scanning Electron Microscope (SEM)**.

The surface of a specimen to be examined is scanned with an electron beam, and the reflected (or back-scattered) beam of electrons is collected, and then displayed at the same scanning rate on a cathode ray tube (similar to a CRT television screen).

The image on the screen, which may be photographed, represents the surface features of the specimen.

The surface may or may not be polished and etched, but it must be electrically conductive; a very thin metallic surface coating must be applied to non-conductive materials.

Magnifications ranging from **10 x** to **50,000 x** are possible, as are also very great depths of field.

Accessory equipment permits qualitative and semi-quantitative analysis of the elemental composition of much localized surface areas.

Scanning Probe Microscopy

The **Scanning Probe Microscope (SPM)** generates a topographical map, on an atomic scale, that is a representation of surface features and characteristics of the specimen being examined.

Some of the features that differentiate the SPM from other microscopic techniques are as follows:

- Examination on the nanometer scale is possible in as much as magnifications as high as **10⁹X** are possible; much better resolutions are attainable than with other microscopic techniques.
- Three-dimensional magnified images are generated that provide topographical information about features of interest.
- Some SPMs may be operated in a variety of environments (e.g., vacuum, air, liquid); thus, a particular specimen may be examined in its most suitable environment.

Scanning probe microscopes employ a tiny probe with a very sharp tip that is brought into very close proximity (i.e., to within on the order of a nanometer) of the specimen surface. This probe is then raster-scanned across the plane of the surface.

During scanning, the probe experiences deflections perpendicular to this plane, in response to electronic or other interactions between the probe and specimen surface.

Scanning Probe Microscopy (... continued)

The in-surface-plane and out-of-plane motions of the probe are controlled by piezoelectric ceramic components that have nanometer resolutions.

Furthermore, these probe movements are monitored electronically and transferred to and stored in a computer, which then generates the three-dimensional surface image.

These new SPMs, which allow examination of the surface of materials at the atomic and molecular level, have provided a wealth of information about a host of materials, from integrated circuit chips to biological molecules.

Indeed, the advent of the SPMs has helped to usher in the era of nanomaterials - materials whose properties are designed by engineering atomic and molecular structures.

Scanning Tunnelling Microscopy

A scanning tunnelling microscope works by moving an exceptionally sharp piezoelectric tip (often only one atom thick at its point) across the surface of a conductive solid, such as a piece of crystalline nickel in an evacuated chamber.

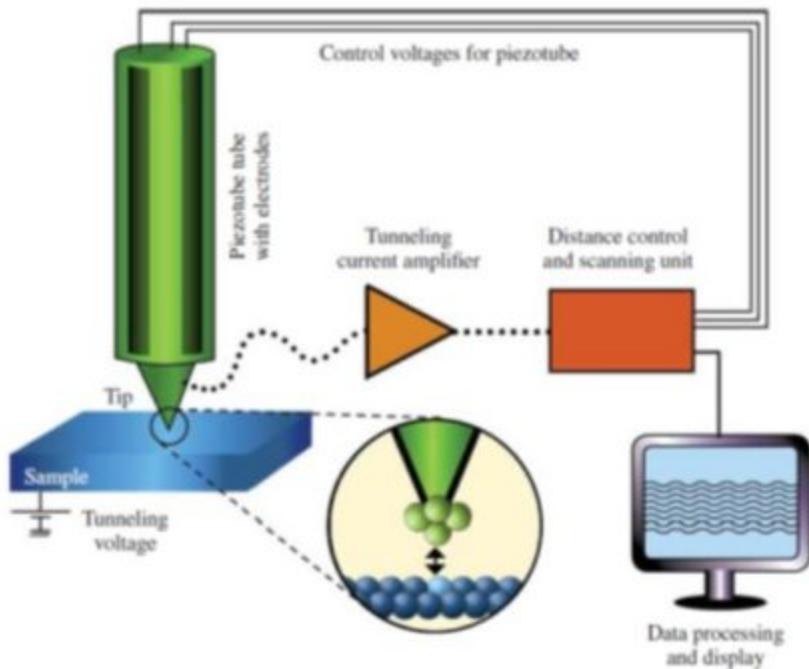
When a small voltage is applied to the tip of the STM, a tunnelling current develops whenever the tip is close to the surface of a Ni atom. This tunnelling current is proportional to the distance between the tip of the probe and the atoms on the surface of the crystal.

By adjusting the STM so that the tunnelling current is a constant, the tip will move up and down as it crosses the surface of the crystal and encounters electron density around the nuclei of the nickel atoms.

A computer is used to map out the three-dimensional contour of the nickel surface and to colour it different shades of blue in this case, depending on the distance that the tip has moved.

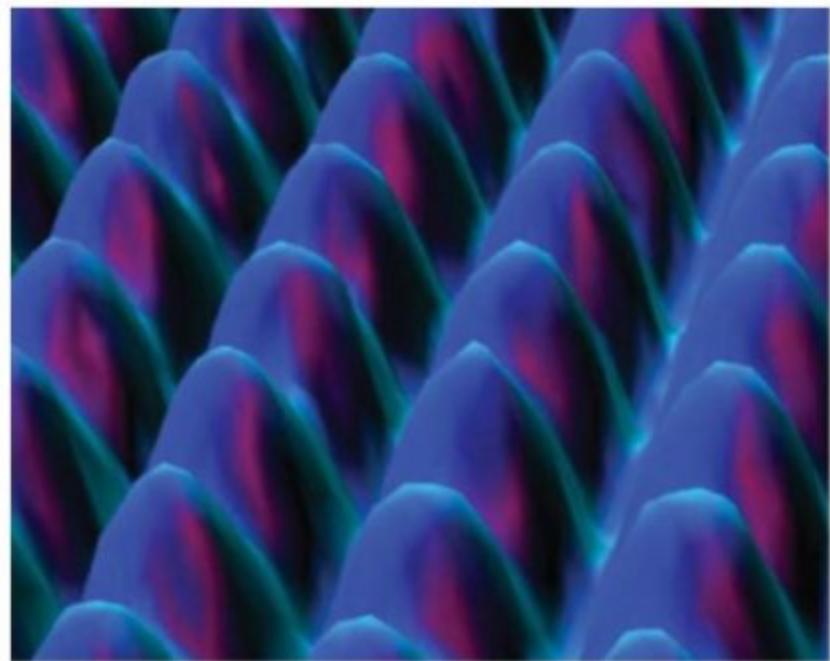
The STM can also be used to pick up atoms and to move them around on a surface.

Scanning Probe Microscopy (... continued)



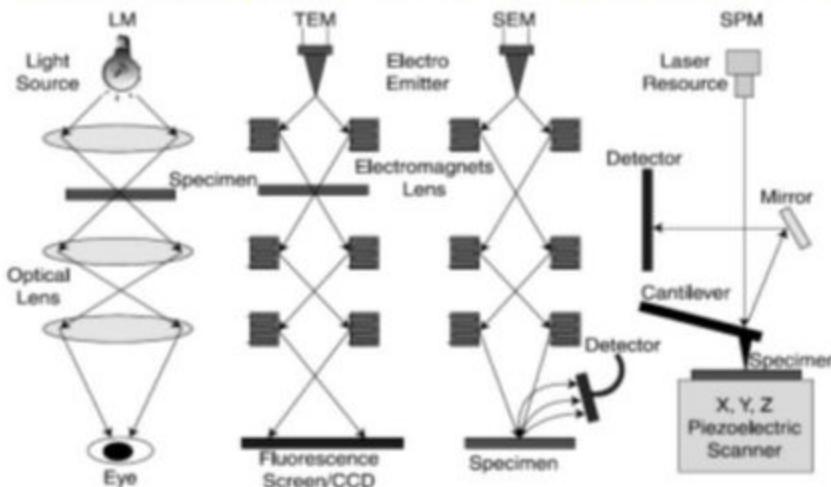
STM Microscope Principle

STM image of (110) face of Ni crystal



Atomic Force Microscopy

Atomic Force Microscope works by monitoring the interaction force between sample surface atoms with the cantilever sensor tip to for **(1) Image** - surface topology in nanometer resolution of polymers, **(2) Force mapping** - surface variation of softness, elasticity and stickiness of composite materials, **(3) Dip-pen nanolithography** and **(4) Nanofabrication**.



Schematic depiction of: Light microscopy (LM), transmission electron microscopy (TEM), Scanning electron microscopy (SEM) and Scanning probe microscopy (SPM).

- Crystallites (grains) and grain boundaries vary considerably in size.
- Can be quite large
 - ex: Large single crystal of quartz or diamond or Si
 - ex: Aluminum garbage can - see the individual grains
- Can be quite small (mm to nm)
necessary to observe with a microscope,
even a TEM. "microstructure", "nanostructure"

Microscopy

Optical resolution ca. 10^{-7} m = 0.1 μ m = 100 nm
For higher resolution need higher frequency

X-Rays? Difficult to focus.

Electrons (SEM, TEM, STEM)

- wavelengths ca. 3 pm (0.003 nm)
(Magnification - 1,000,000X)
- Atomic resolution possible
- Electron beam focused by magnetic lenses.

Scanning Probe Microscopies (STM, AFM, ...)

Summary: Microscopic Techniques

The microstructure of a material consists of defects and structural elements that are of microscopic dimensions. Microscopy is the observation of microstructure using some type of microscope.

Both optical and electron microscopes are employed, usually in conjunction with photographic equipment.

Transmissive and reflective modes are possible for each microscope type; preference is dictated by the nature of the specimen as well as the structural element or defect to be examined.

In order to observe the grain structure of a polycrystalline material using an optical microscope, the specimen surface must be ground and polished in order to produce a very smooth and mirror-like finish.

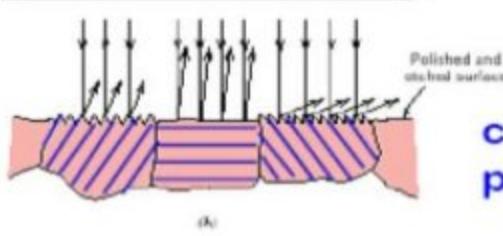
Some type of chemical reagent (or etchant) must then be applied in order to either reveal the grain boundaries or produce a variety of light reflectance characteristics for the constituent grains.

The two types of electron microscopes are transmission (TEM) and scanning (SEM). For TEM an image is formed from an electron beam that, while passing through the specimen, is scattered and/or diffracted.

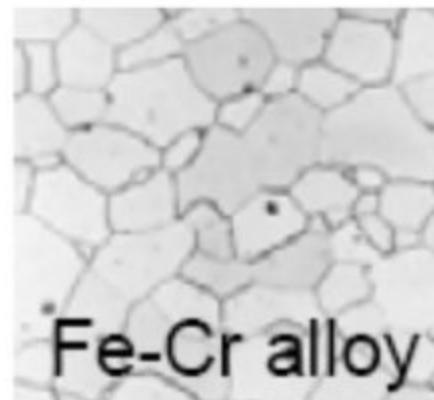
SEM employs an electron beam that raster-scans the specimen surface; an image is produced from back-scattered or reflected electrons.

A scanning probe microscope employs a small and sharp-tipped probe that raster-scans the specimen surface. Out-of-plane deflections of the probe result from interactions with surface atoms. A computer generated and three-dimensional image of the surface results having nanometer resolution.

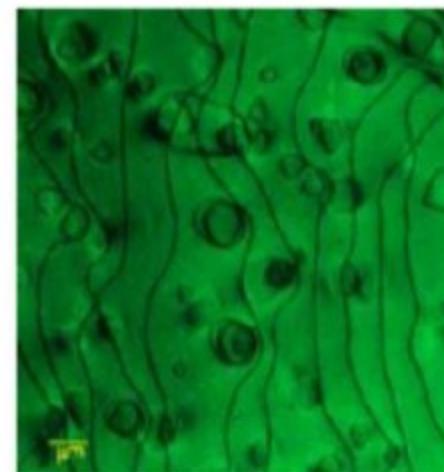
- Useful up to 2000X magnification.
- Polishing removes surface features (e.g., scratches)
- Etching changes reflectance, depending on crystal orientation.



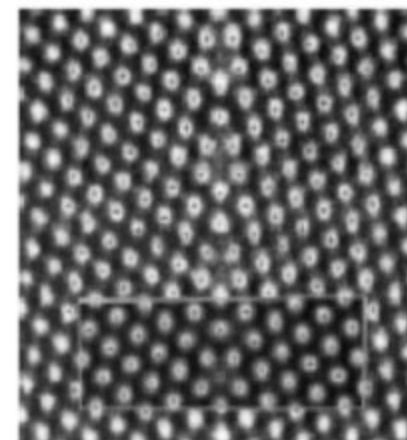
micrograph of
Brass (Cu and
Zn)



Electron Microscopy (*not tested*)



Dislocations in Nickel (the dark lines and loops), transmission electron microscopy image, Manchester Materials Science Center.



High-resolution Transmission Electron Microscope image of a tilt grain boundary in aluminum, Sandia National Lab.

SOLIDIFICATION OF CASTINGS

Solidification is the transformation of materials from the liquid to the solid crystalline state on cooling.

During solidification, the disordered structure of the liquid transforms to the orderly arrangement characteristic of the crystal.

Under industrial casting conditions, the kinetics of solidification is often controlled by heat flow.

When a melt is poured into a container or mould, which is at a much lower temperature than the melt, large supercooling occurs in regions where the melt comes into direct contact with the cold container walls.

This large super cooling results in a rapid rate of nucleation and a *layer of fine crystals* is formed adjacent to the wall.

The latent heat released in the formation of this layer increases the temperature and reduces the nucleation rate in the liquid next to it.

Heat flows out of the mould through the walls.

As a result, the crystals adjacent to the layer of fine crystals at the mould wall grow inwards towards the centre (in a direction opposite to that of the heat flow) producing long columnar crystals.

SOLIDIFICATION OF CASTINGS (... continued)

These crystals ultimately meet near the centre of the mould and a cross-section after solidification shows crystals radially branching out from the centre.

Quite often, crystals grow preferentially along certain crystallographic directions, along which the thermal conductivity (and hence the heat extraction) is a maximum.

This fact is exploited in certain applications to produce a polycrystalline material with crystals aligned in a particular crystal direction.

In magnetic applications, for example, the alignment of crystals enhances the magnetic properties.

In manufacturing components by casting, molten metals are often poured into mould and permitted to solidify.

The mould produces a finished shape, known as a casting. In other cases, the mould produces a simple shape, called an ingot.

An ingot usually requires extensive plastic deformation before a finished product is created.

A macrostructure, sometimes referred to as the ingot structure, consists of as many as three parts.

SOLIDIFICATION OF CASTINGS (... continued)

Chill Zone: The chill zone is a narrow band of randomly oriented grains at the surface of the casting.

The metal at the mold wall is the first to cool to the freezing temperature.

The mold wall also provides many surface sites at which heterogeneous nucleation takes place.

Columnar Zone: The columnar zone contains elongated grains oriented in a particular crystallographic direction.

As heat is removed from the casting by the mold material, the grains in the chill zone grow in the direction opposite the heat flow, or from the coldest toward the hottest areas of the casting.

This tendency usually means that the grains grow perpendicular to the mold wall.

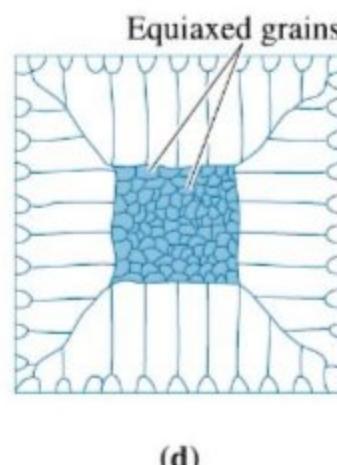
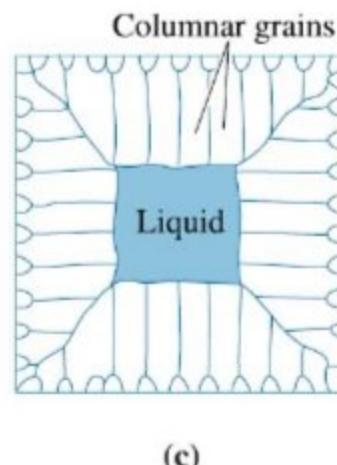
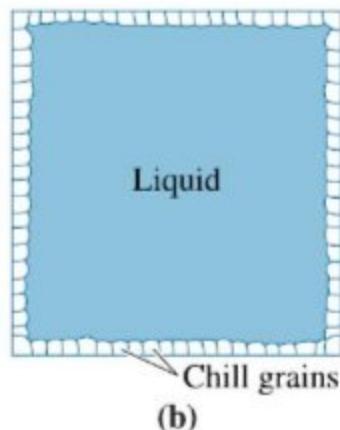
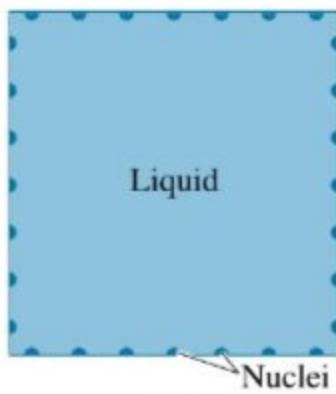
Equiaxed Zone: Although the solid may continue to grow in a columnar manner until all of the liquid has solidified, an equiaxed zone frequently forms in the center of the casting or ingot.

The equiaxed zone contains new, randomly oriented grains, often caused by a low pouring temperature, alloying elements, or grain refining or inoculating agents.

SOLIDIFICATION OF CASTINGS (... continued)

Small grains or dendrites in the chill zone may also be torn off by strong convection currents that are set up as the casting begins to freeze. These also provide the heterogeneous nucleation sites for what ultimately become equiaxed grains. These grains grow as relatively round, or equiaxed, grains with a random orientation, and they stop the growth of the columnar grains.

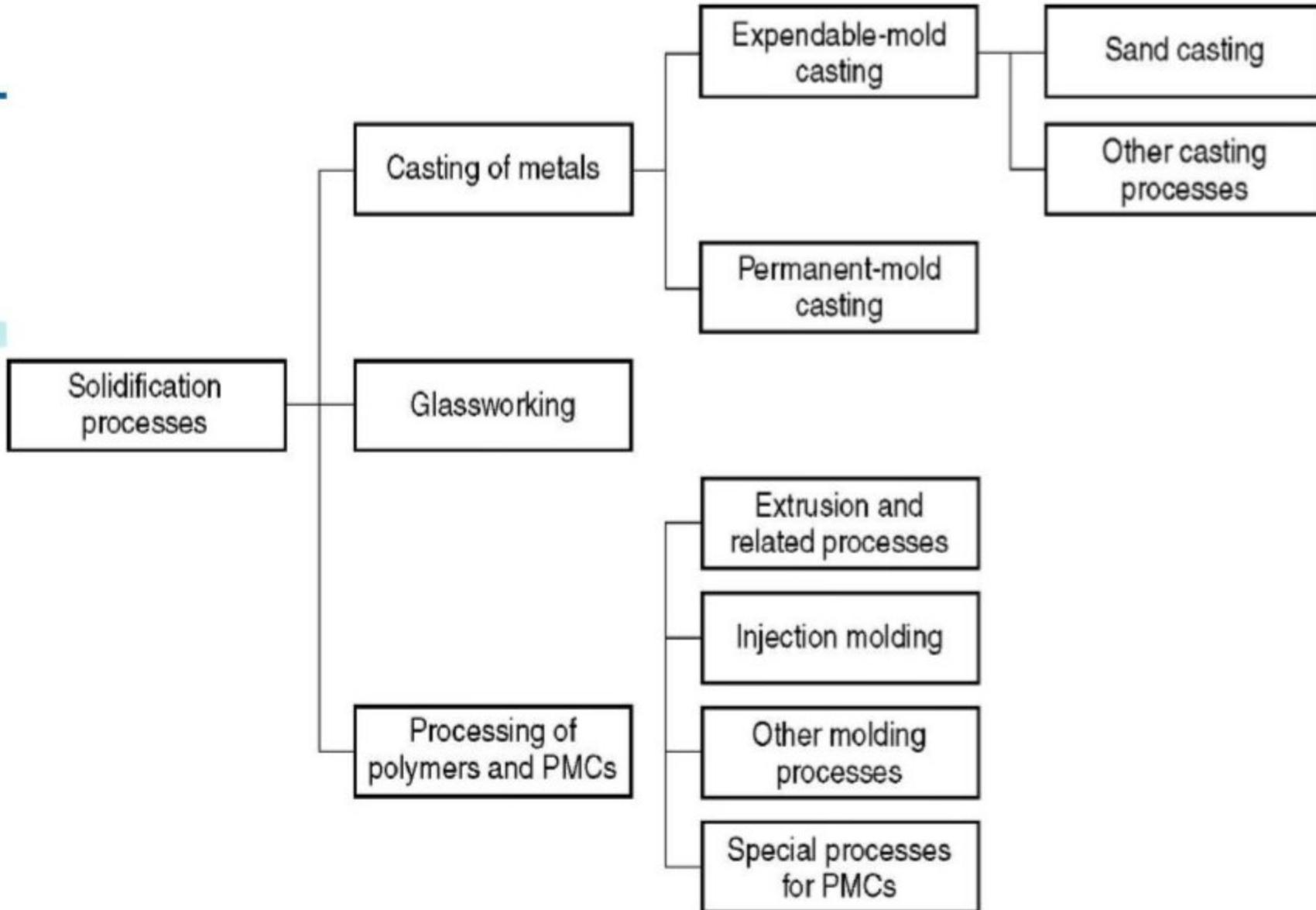
The formation of the equiaxed zone is a nucleation controlled process and causes that portion of the casting to display isotropic behavior.



Development of the ingot structure of a casting during solidification:

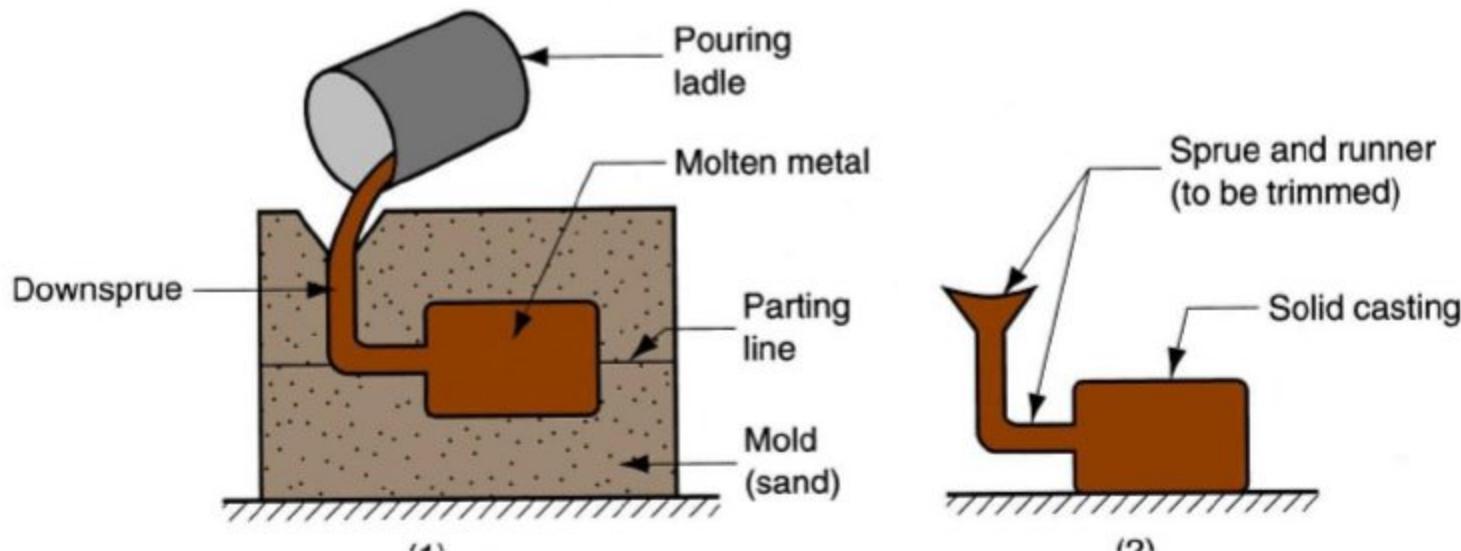
(a) Nucleation begins, (b) the chill zone forms, (c) preferred growth produces the columnar zone, and (d) additional nucleation creates the equiaxed zone.

Solidification Processes



Solidification Processes

- Starting material is heated sufficiently to transform it into a liquid or highly plastic state
- Examples: metal casting, plastic molding



Ancient Cast Structures



4000 -5000 Years

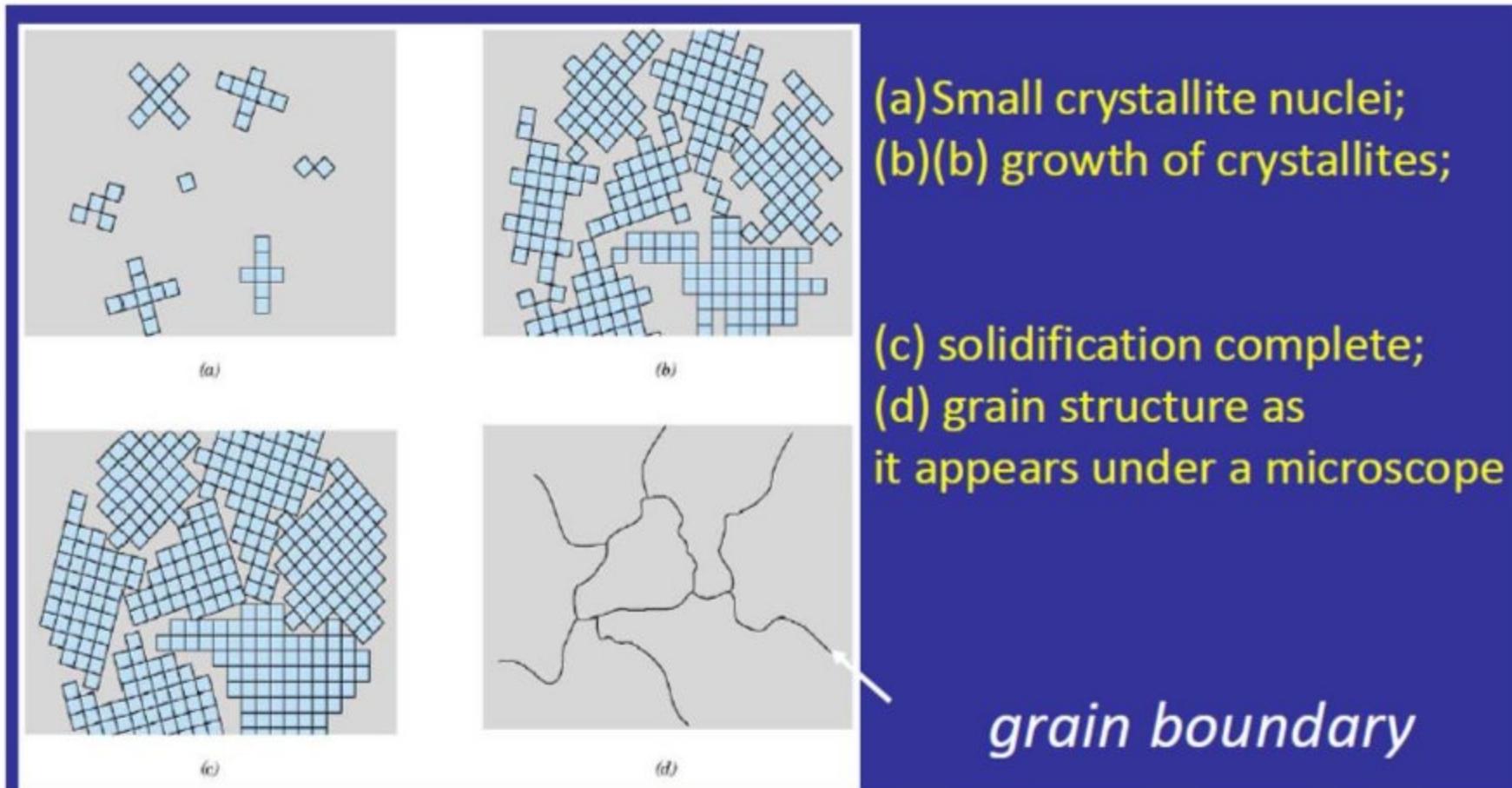
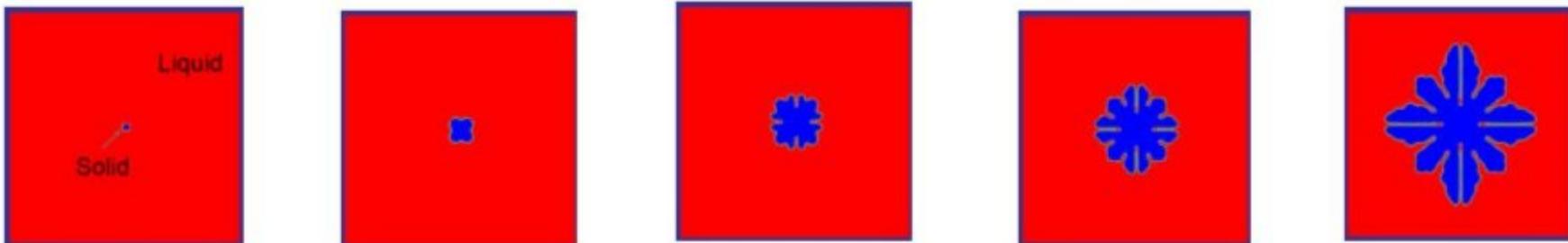


4000 -5000 Years



1000 years

Solidification



Various Stages during Solidification of a Crystalline Material

Solidification

- Nucleation
- Growth

Nucleation

- Formation of tiny stable solid particles from liquid (50-60 atoms of ~ 1-2 nm dia.)
- “Undercooling” provides energy for creation of new S/L interface
- Larger the extent of undercooling, greater will be the number of nuclei formed

Types of nucleation

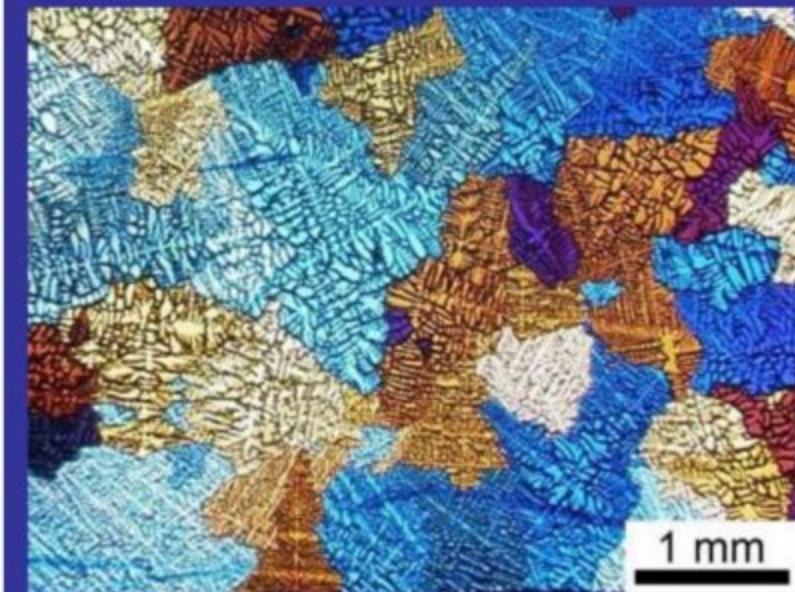
Two types of nucleation

- Heterogeneous
 - Nuclei form on a solid surface (wall of the mold, particulates in the liquid)
- Homogeneous
 - Nuclei form on their own
- Heterogeneous nucleation easier to occur
 - See where ice formation starts in the ice tray

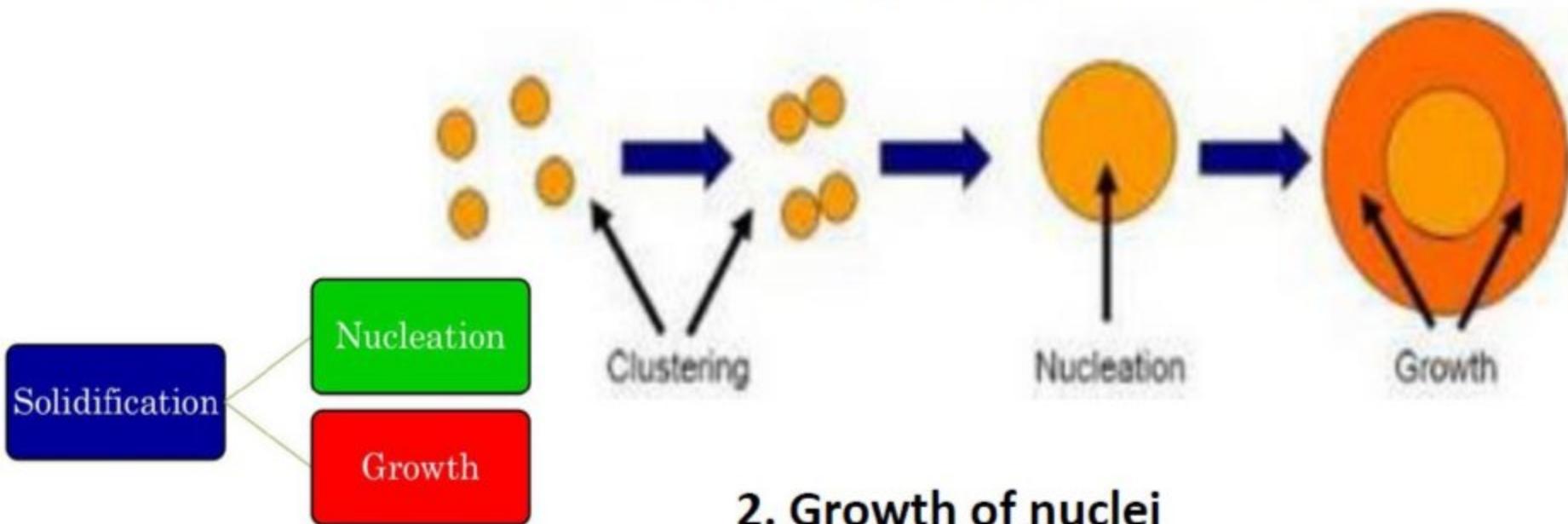
Growth

- Nuclei grow and then aggregate
- Occurs as heat of fusion is extracted from liquid
- Direction and rate of growth can be controlled by the manner of heat removal
 - Directional solidification
 - Movement of S/L interface opposite to the direction of heat flow.
 - Modify heat flow direction

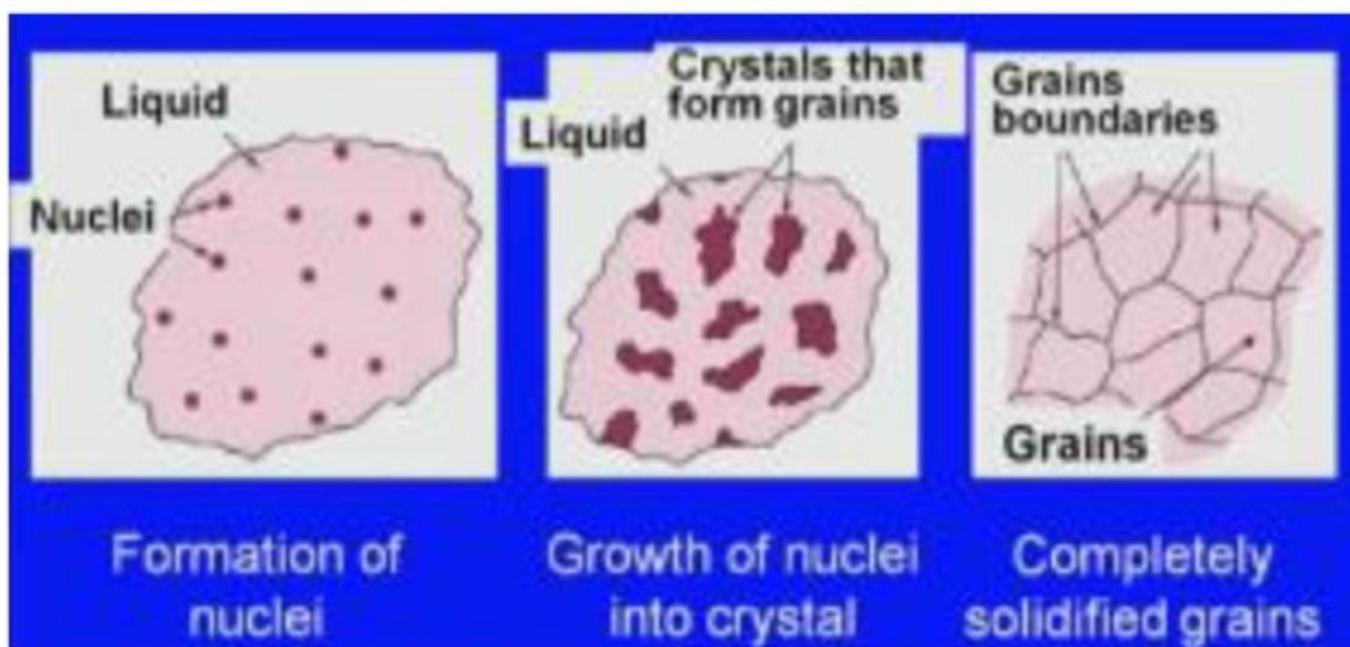
Grain structure during solidification



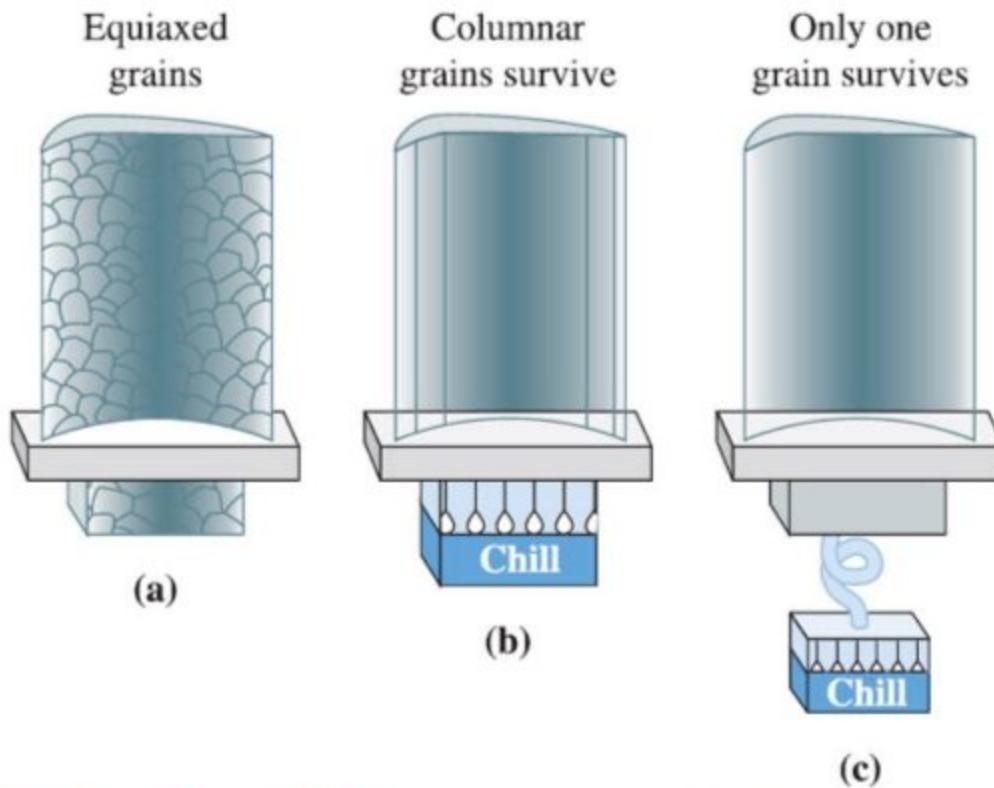
1. Nucleation (formation of nuclei)



2. Growth of nuclei



INDUSTRIAL APPLICATIONS: Directional Solidification, Single Crystal Growth, and Epitaxial Growth



Directional Solidification (DS): The mold is heated from one end and cooled from the other, producing a columnar microstructure with all of the grain boundaries running in the longitudinal direction of the part. No grain boundaries are present in the transverse direction.

INDUSTRIAL APPLICATIONS: Directional Solidification, Single Crystal Growth, and Epitaxial Growth (Continued.)

Single Crystal (SC) Growth: Solidification of columnar grains again begins at a cold surface but, due to a helical cavity in the mold between the heat sink and the main mold cavity, only one columnar grain is able to grow to the main body of the casting. The single-crystal casting has no grain boundaries, so its crystallographic planes and directions can be directed in an optimum orientation.

For semiconductors of doped silicon, high-purity single large crystals up to 12 in. dia. are grown and cut into silicon wafers of a few mm thick. Single crystals of silicon, GaAs, lithium niobate (LiNbO_3), and many other materials produced are used in making novel and useful devices.

Epitaxial Growth: The material is made to grow in an oriented fashion using a substrate that is crystallographically matched with the material being grown. If the lattice matching between the substrate and the film is good, it is possible to grow highly oriented or single crystal thin films.

Shape-Memory Alloys

- ❖ *Shape-memory alloys (SMAs)*, after deformed, return to its predeformed size and shape upon an appropriate heat treatment.
- ❖ The material “remembers” its previous size/shape.
- ❖ Deformation normally is carried out at a relatively low temperature, whereas shape memory occurs upon heating.
- ❖ Examples are nickel–titanium alloys (Nitinol - trade name), and some copper-base alloys (Cu–Zn–Al and Cu–Al–Ni alloys).
- ❖ The two polymorphic crystal structures (or phases), and shape-memory effect involves phase transformations between them.
- ❖ One phase (termed an *austenite phase*) has a body-centered cubic structure that exists at elevated temperatures.
- ❖ Upon cooling, the austenite transforms to a martensite phase.
- ❖ It is diffusionless, involves an orderly shift of large groups of atoms, and occurs very rapidly, and the degree of transformation is dependent on temperature.
- ❖ Under the influence of an applied stress, deformation of martensite occurs by the migration of twin boundaries — some twinned regions grow while others shrink; this deformed martensitic.

Shape-Memory Alloys (... continued)

- ❖ Furthermore, when the stress is removed, the deformed shape is retained at this temperature.
- ❖ And, finally, upon subsequent heating to the initial temperature, the material reverts back to (i.e., “remembers”) its original size and shape.
- ❖ One important SMA application is in weldless, shrink-to-fit pipe couplers used for hydraulic lines on aircraft, for joints on undersea pipelines, and for plumbing on ships and submarines.
 - Each coupler (in the form of a cylindrical sleeve) is fabricated so as to have an inside diameter slightly smaller than the outside diameter of the pipes to be joined.
 - It is then stretched (circumferentially) at some temperature well below the ambient.
 - Next the coupler is fitted over the pipe junction and then heated to room temperature.
 - Heating causes the coupler to shrink back to its original diameter, thus creating a tight seal between the two pipe sections.

Shape-Memory Alloys (... continued)

- ❖ There is a host of other applications for alloys displaying this effect. Examples are:
 - eyeglass frames, tooth-straightening braces, collapsible antennas, greenhouse window openers, Anti-scald control valves on showers, women's foundation garments, fire sprinkler valves, and in biomedical applications (such as blood-clot filters, self extending coronary stents, and bone anchors).
- ❖ Shape-memory alloys also fall into the classification of "smart materials" because they sense and respond to environmental (i.e., temperature) changes.
- ❖ SMA materials have the ability to store a large amount of mechanical strain or elastic energy in a small volume.
- ❖ Used for such diverse applications as circuit breakers, switches, automatic window openers, steam-release valves, hydraulic controls for aircraft, rock cracking, sealing rings, and actuators.
- ❖ They can even be used to unfurl antennas on satellites, where a bulky motor assembly may be replaced by a simple SMA.
- ❖ A limitation on their use, however, is their slow response time, being limited by thermal conduction.

Biomedical Shape-Memory Alloys

Shape memory alloys are used in stents for heart patients.

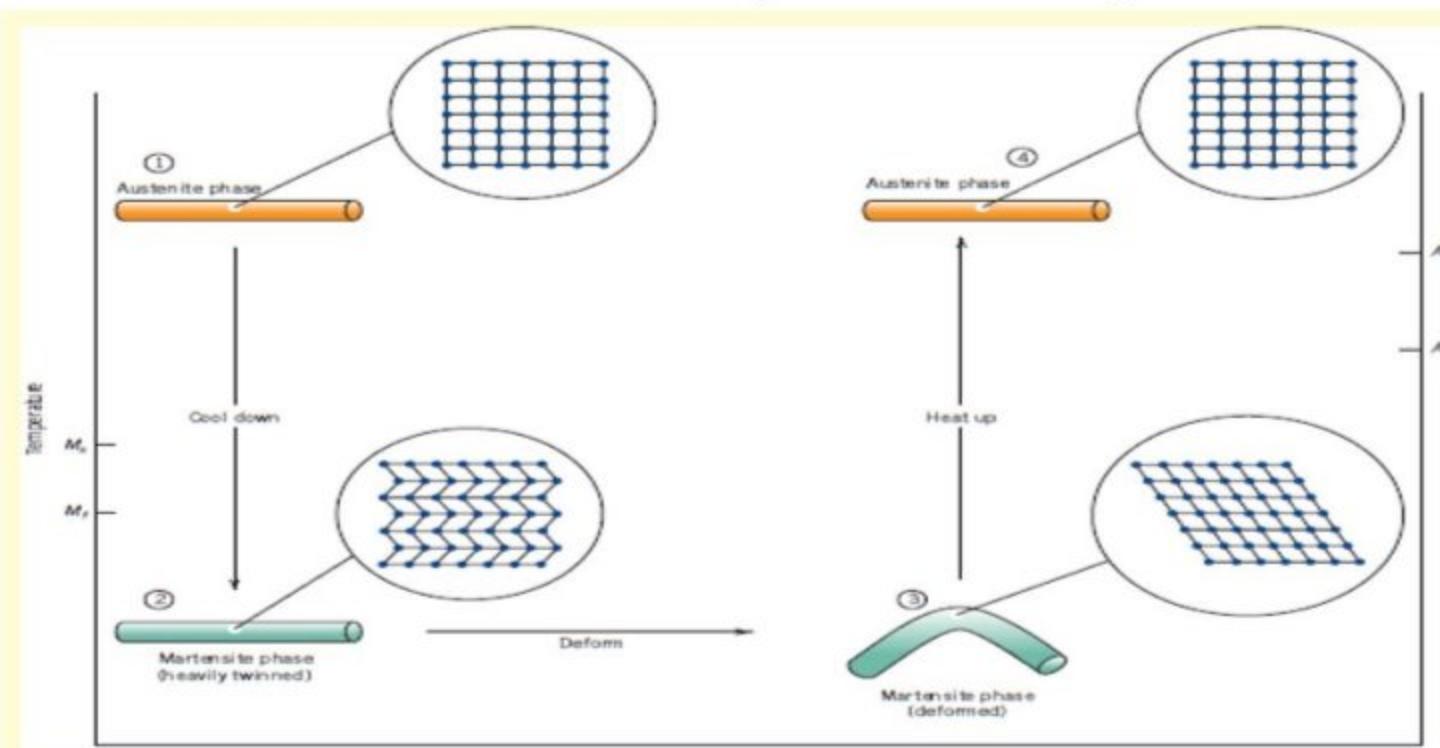
A tube of woven NiTi wire (Nitinol) can be stretched out at low temperatures so that it can be inserted into an artery.

At body temperature the stent assumes its original, larger-diameter shape and keeps the artery open.



Recently, Shape-Memory Plastics

(which have viscoelastic nature) are developed.



Phase Diagrams

A **phase diagram** shows the phases and their compositions at any combination of temperature and alloy composition.

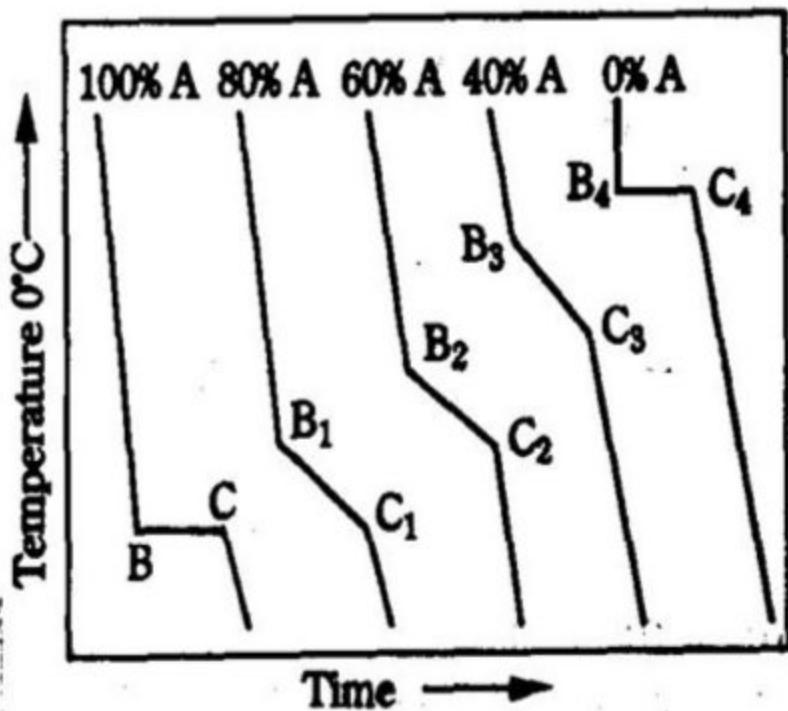
When only two elements or two compounds are present in a material, a **binary phase diagram** can be constructed. The phase diagram can be treated as a road map.

If we know the coordinates - temperature and alloy composition - we can determine the phases present, assuming thermodynamic equilibrium exists.

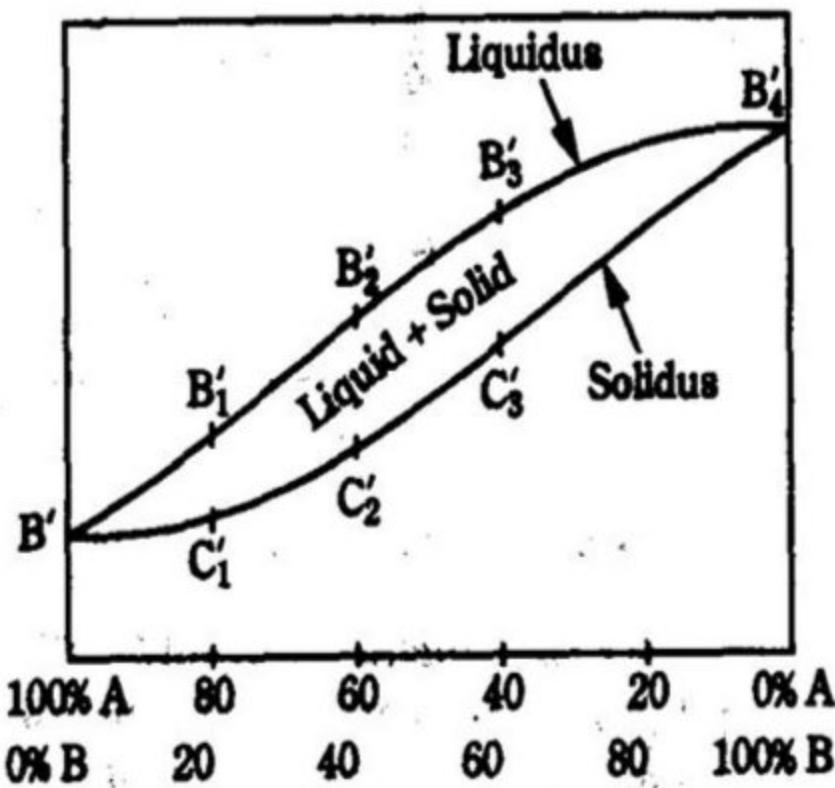
The binary phase diagram can be constructed by using the '**Cooling Curves**' of pure metals and alloys of the two metals with different compositions.

- A knowledge about phase diagrams helps us for materials selection and processing such as casting and heat treatment.
- For casting the metal or alloy must be initially all liquid. Metal casting needs liquid metal which is above its melting point.
- Alloy casting requires melting temperature above the 'liquidus line' which varies with alloy composition.
- To heat treat an alloy component one must be sure that no liquid forms during the process.
- Heat treatment temperatures, and hence, treatment methods are governed by 'solidus line' for an alloy system.
- For example, Cu-Ni alloy - with 40% Cu - melting above 1280°C is needed for casting whereas, heat treating the alloy component should be done below 1240°C only.
- Different solid phases have different properties. For example, BCC iron is ferromagnetic but FCC iron is not.

- Changes of phase in the solid state involve a redistribution of the atoms and the kinetics of the change depend upon the rate of atomic migration.
- The transport of atoms through the crystal is termed as diffusion. This occurs more easily with the aid of vacancies, since the basic act of diffusion is the movement of an atom to an empty adjacent atomic site.
- During a phase change an atom is moved from an α -phase lattice site to a more favourable β -phase lattice site.
- Most phase changes occur by a process of nucleation and growth.
- Chance in thermal fluctuations provide a small number of atoms with sufficient activation energy to break away from the matrix (the old structure).
- And form a small nucleus of the new phase, which then grows at the expense of the matrix until the whole structure is transformed.



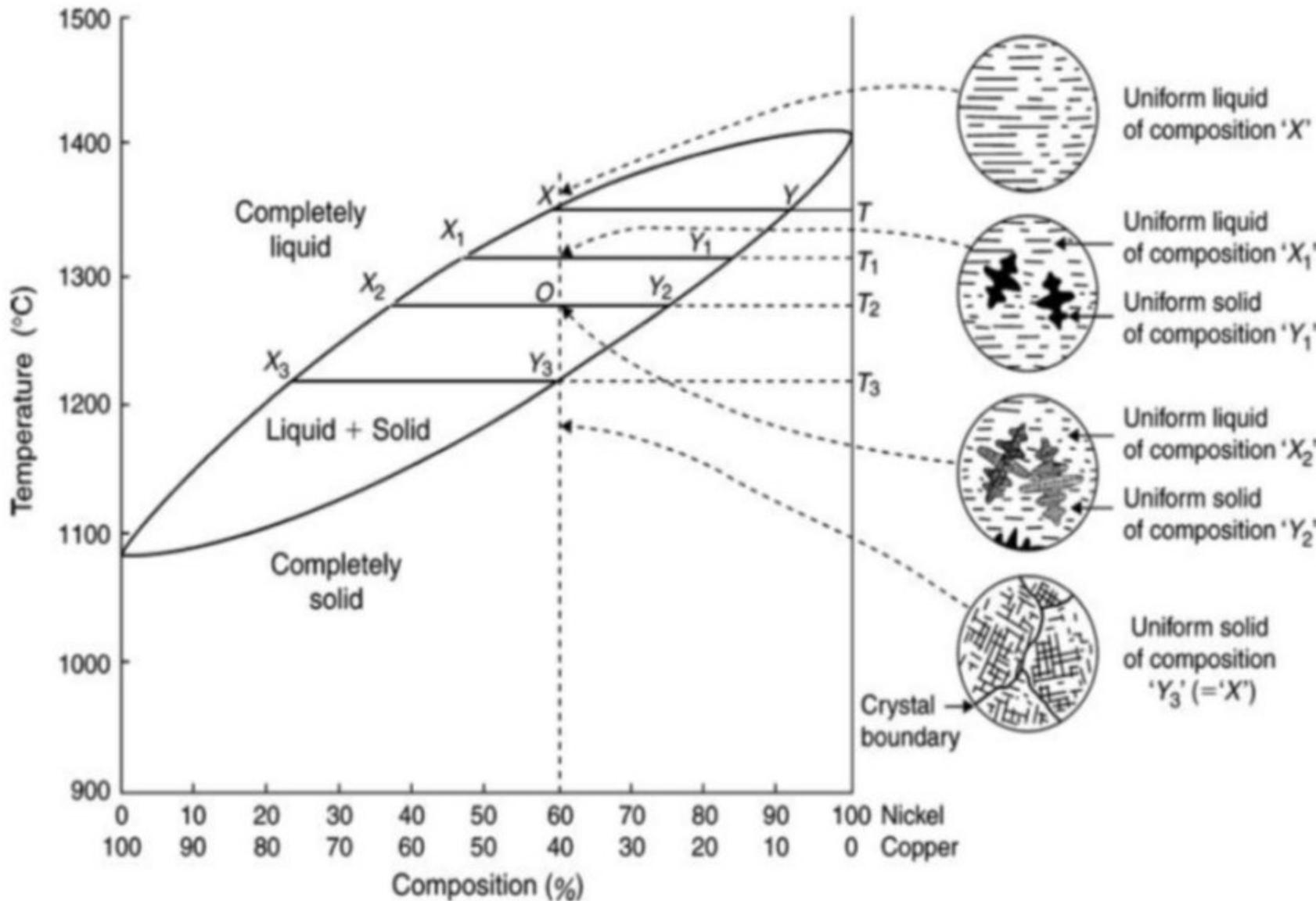
(a) Cooling curves



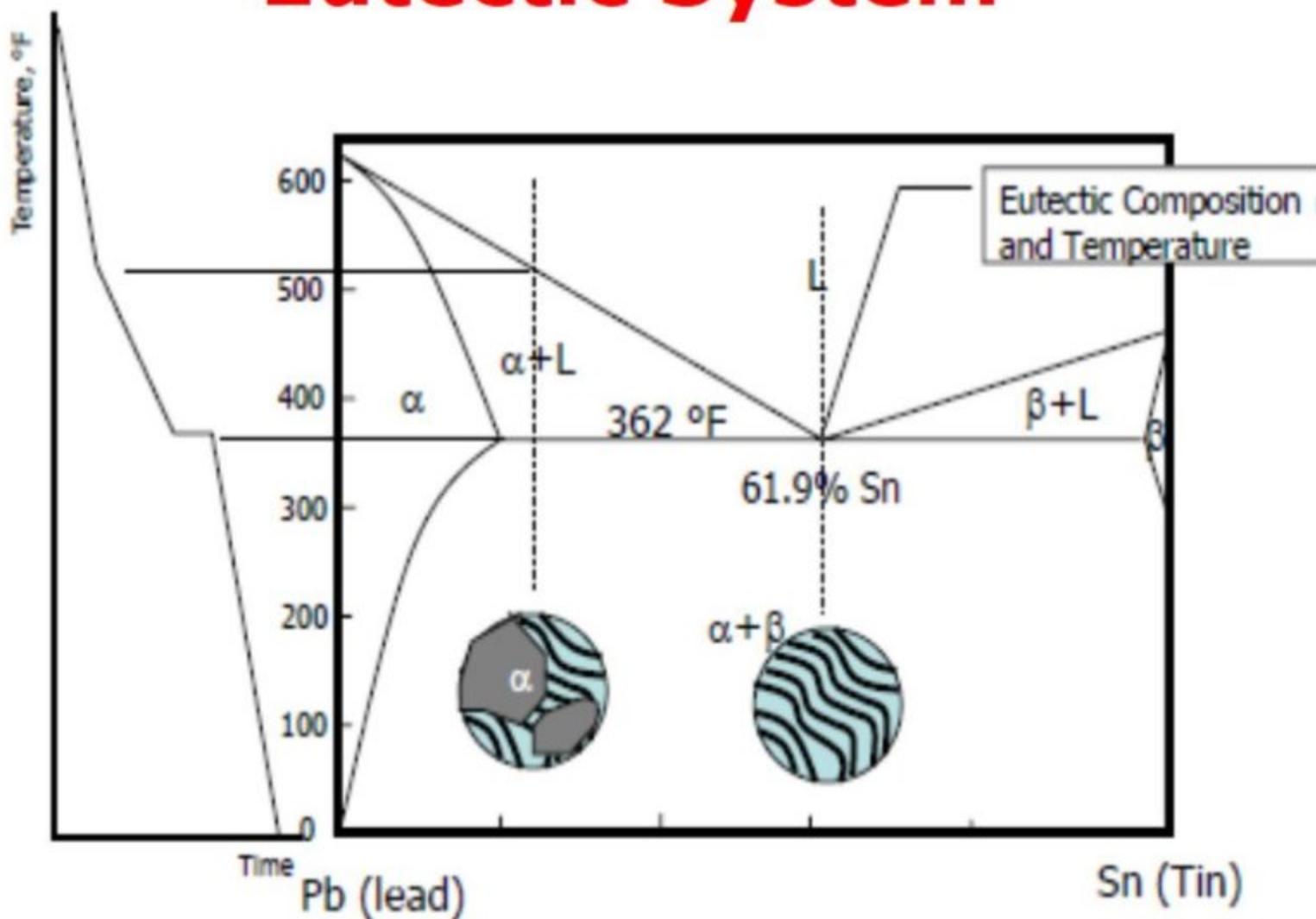
(b) Equilibrium diagram

Construction of Isomorphous phase diagram using 'cooling curves'

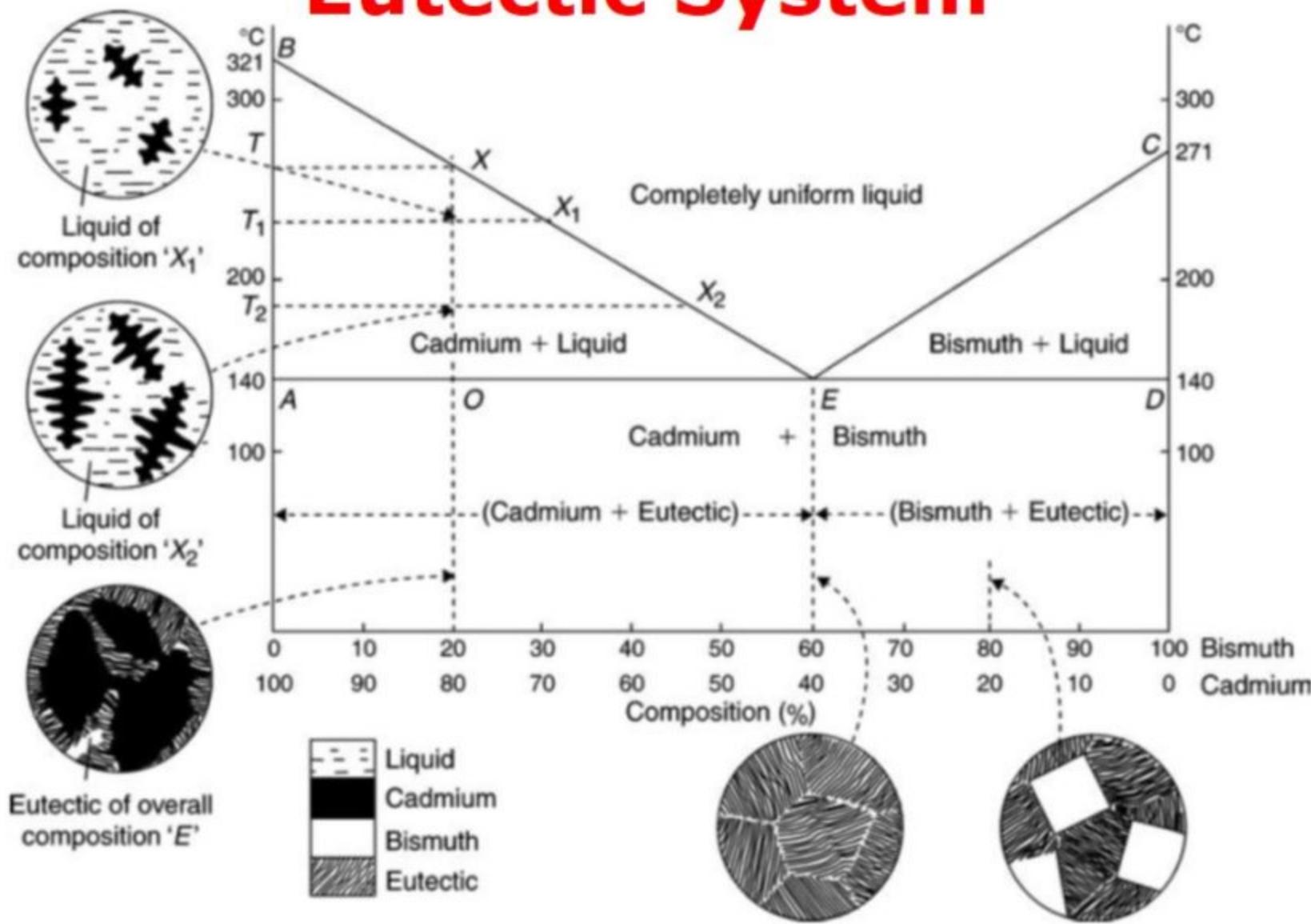
Isomorphous System



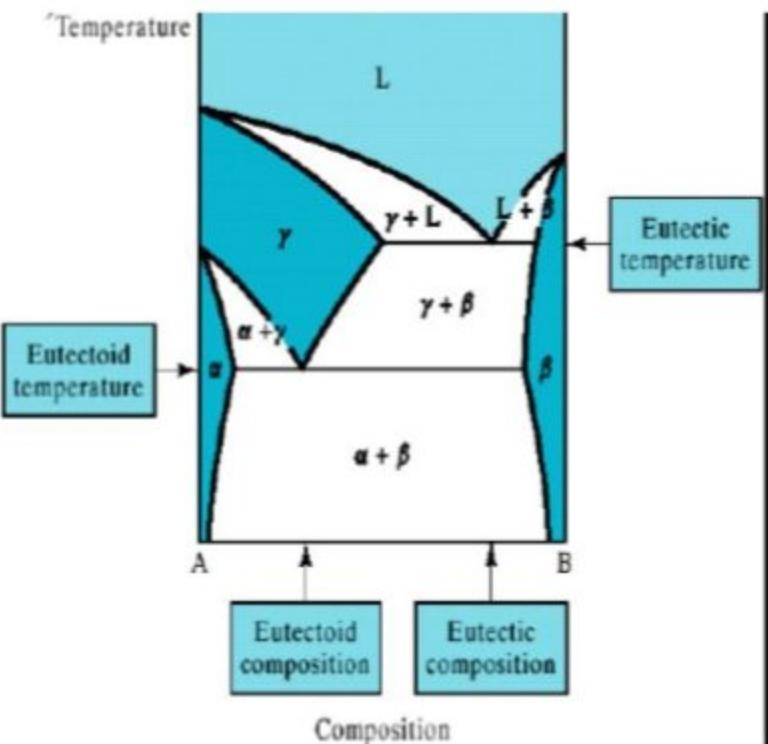
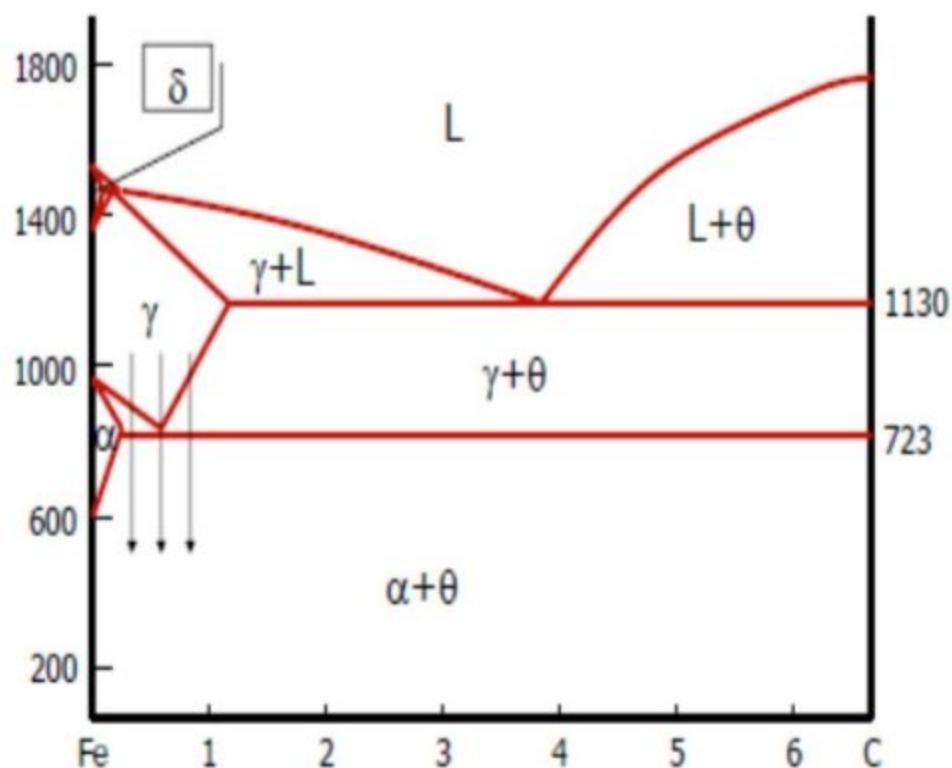
Eutectic System



Eutectic System

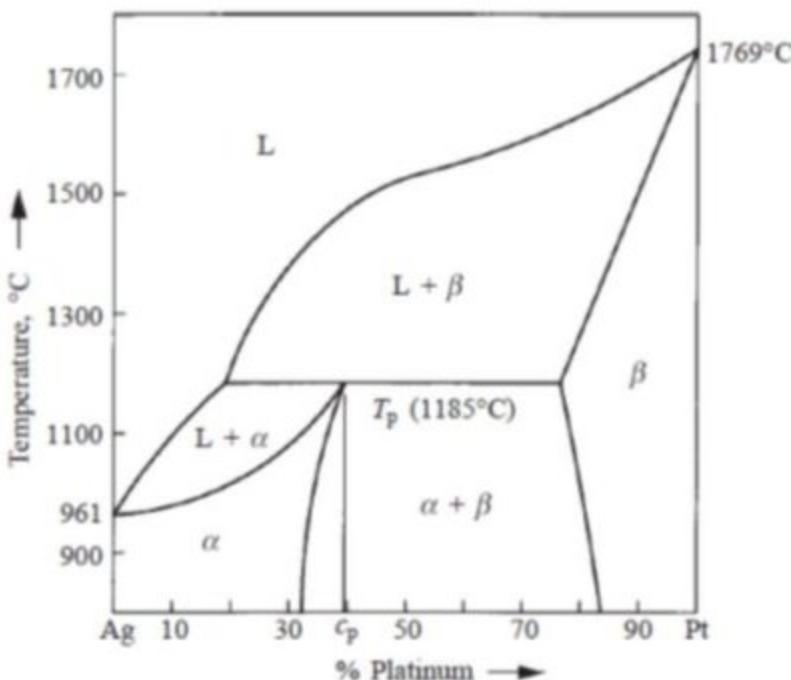


Eutectoid System

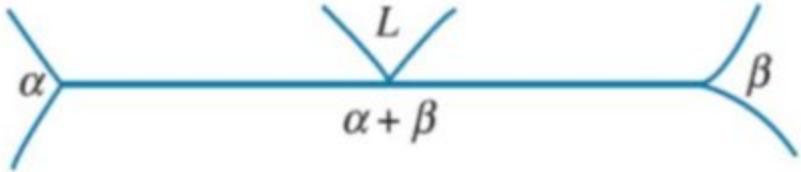
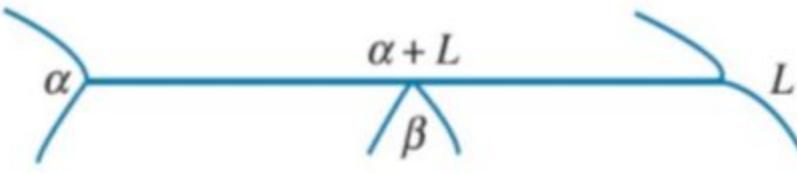
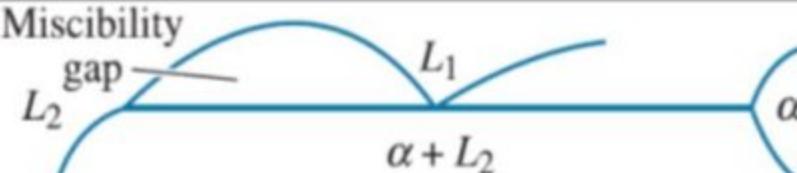
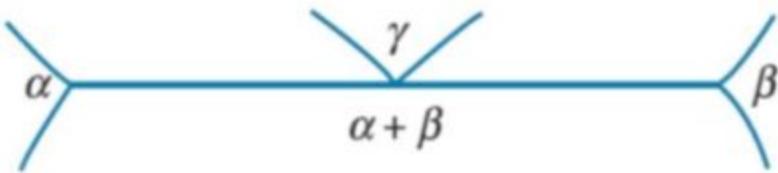
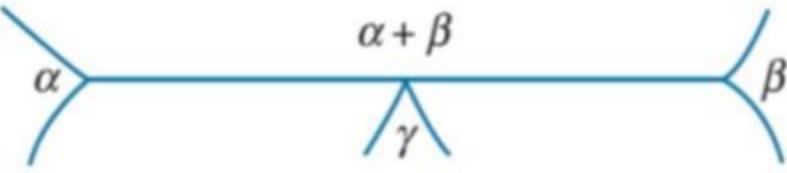


Iron-Carbon Phase Diagram

Peritectic System

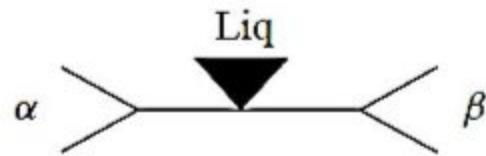
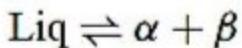


The peritectic reaction is often found when the components have a large difference in melting points. Usually they occur in the more complicated systems.

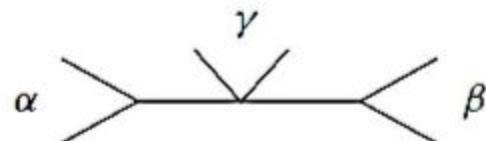
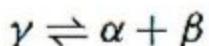
| | | |
|-------------|-------------------------------------|--|
| Eutectic | $L \rightarrow \alpha + \beta$ |  |
| Peritectic | $\alpha + L \rightarrow \beta$ |  |
| Monotectic | $L_1 \rightarrow L_2 + \alpha$ |  |
| Eutectoid | $\gamma \rightarrow \alpha + \beta$ |  |
| Peritectoid | $\alpha + \beta \rightarrow \gamma$ |  |

Eutectic-type
reactions

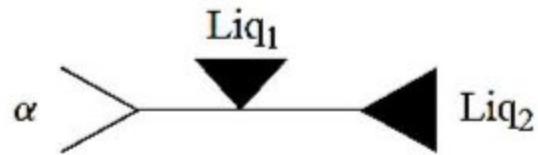
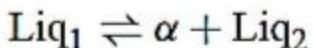
Eutectic



Eutectoid

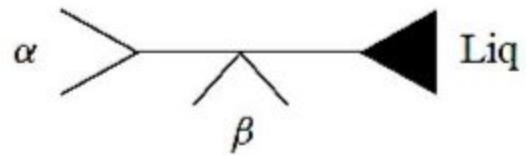
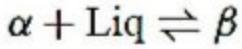


Monotectic

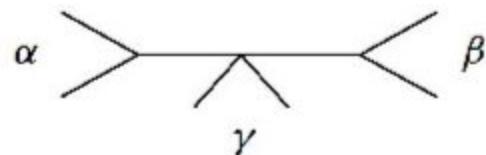
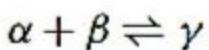


Peritectic-type
reactions

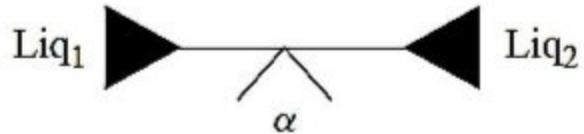
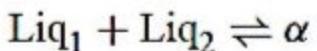
Peritectic



Peritectoid



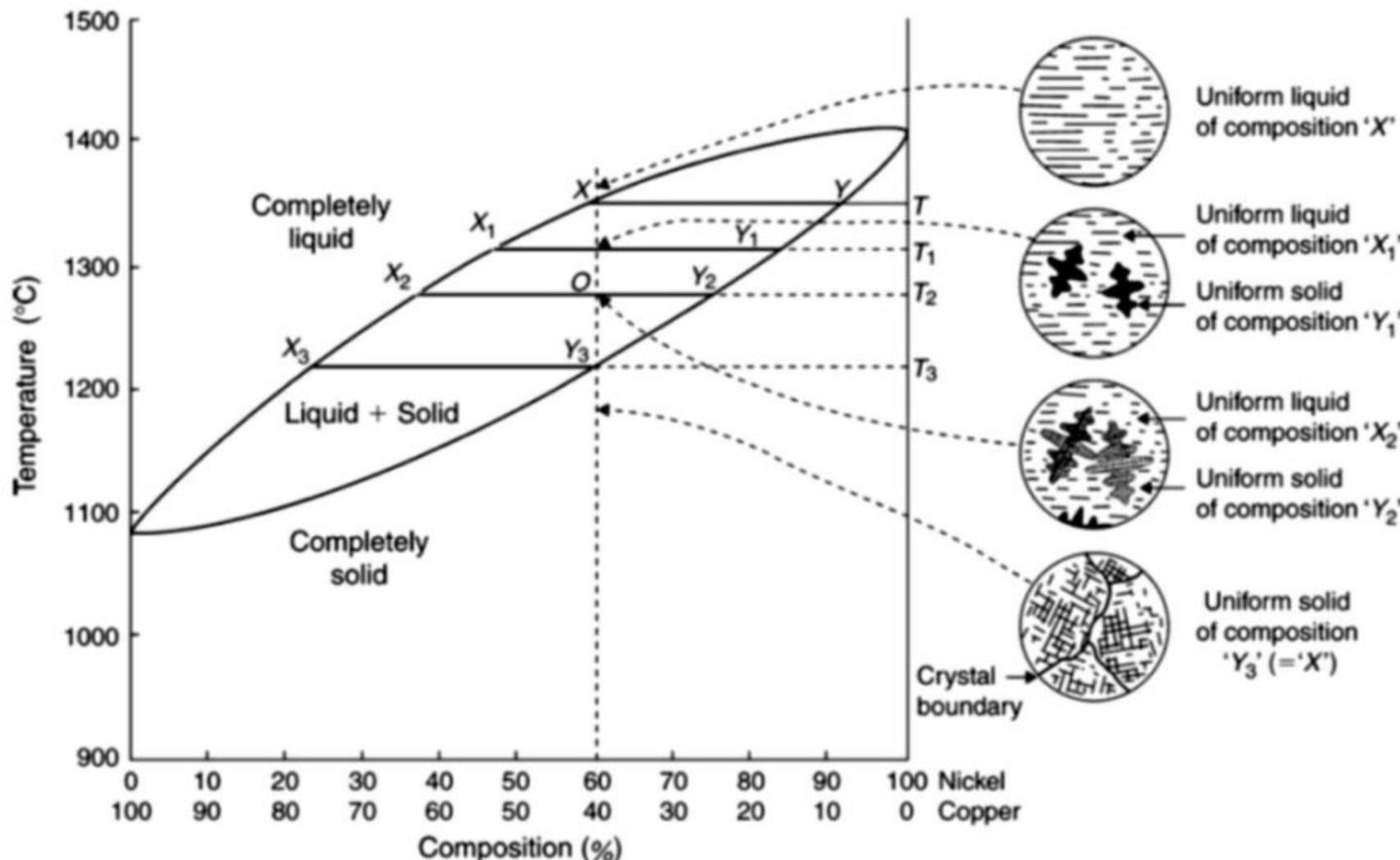
Syntectic



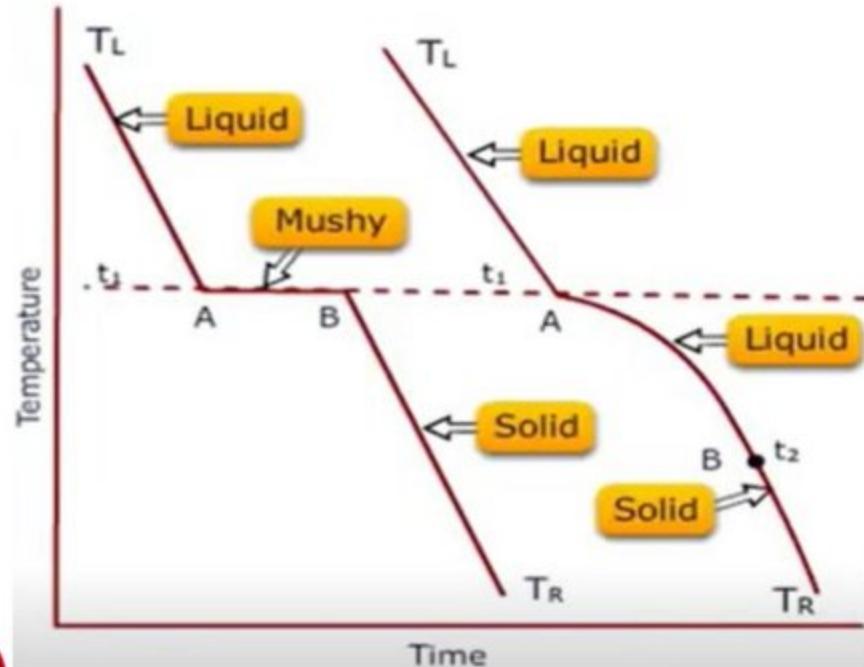
PHASE DIAGRAMS

1. ISOMORPHOUS SYSTEM

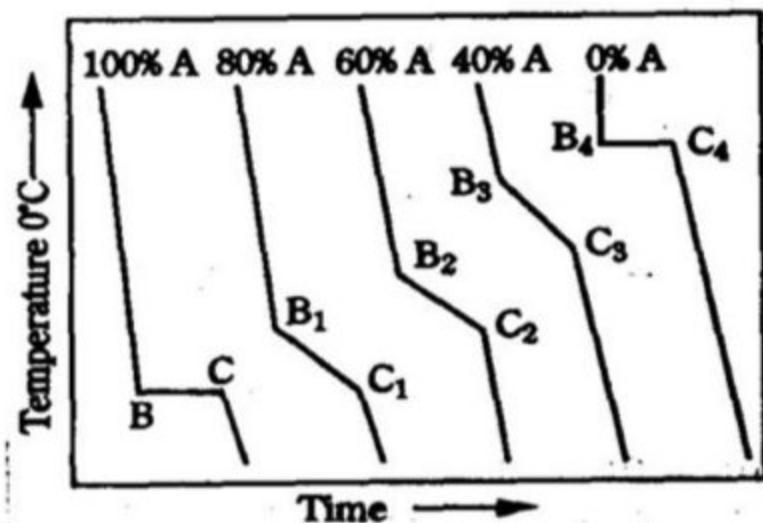
In isomorphous systems the two components have unlimited solubility, which means that they are like water and alcohol when they mix - they always form a solid solution regardless of the ratio of atoms/molecules.



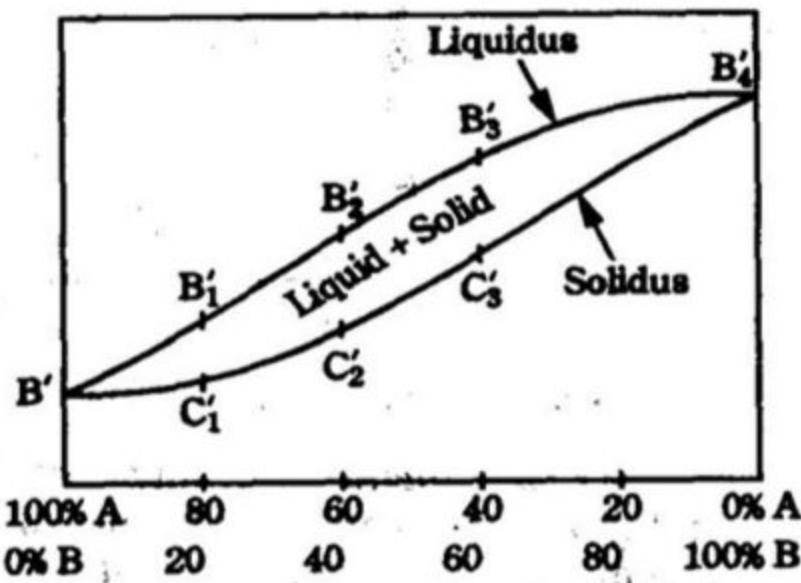
COOLING CURVES: Pure metal & An alloy



**COOLING CURVES
(isomorphous system)**



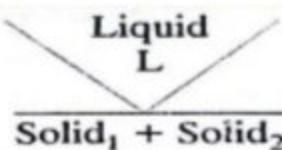
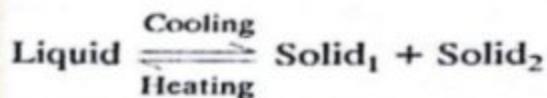
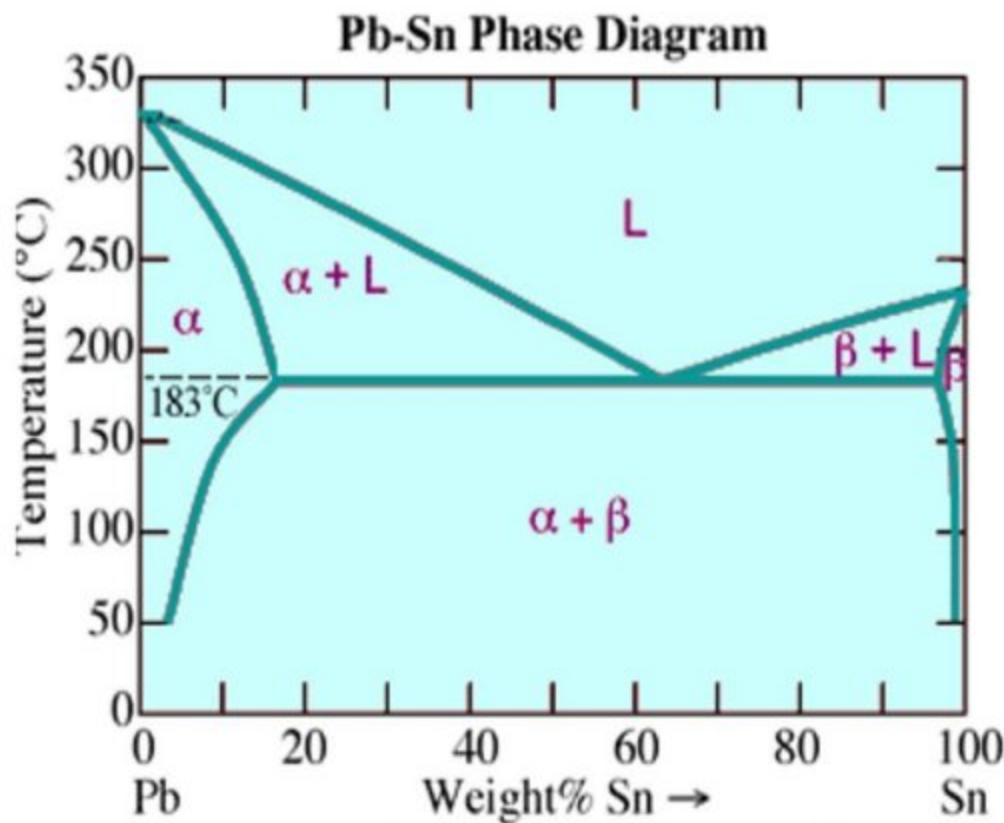
(a) Cooling curves



(b) Equilibrium diagram

2. EUTECTIC SYSTEM

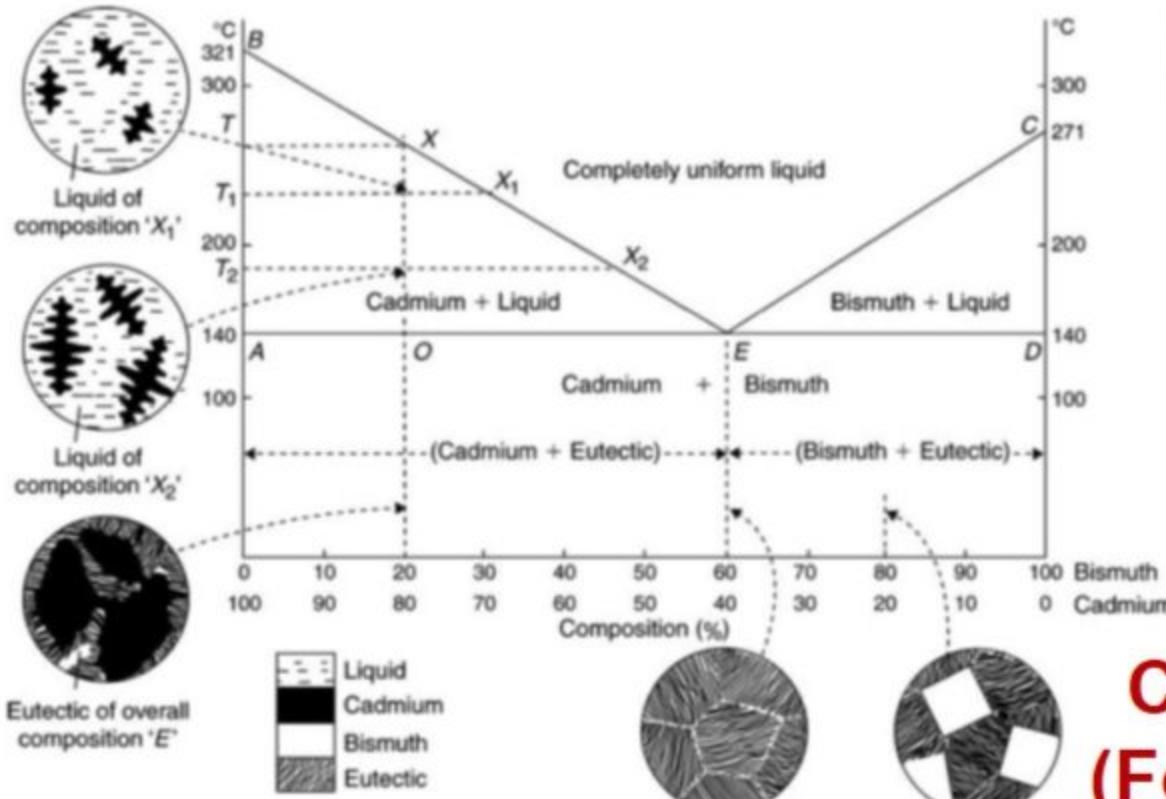
A eutectic system has two components, and they have limited solubility. Upon cooling, the liquid will transform into two mixed solid phases. We will use the Pb-Sn phase diagram as an example. Pb-Sn alloys are used as common solders.



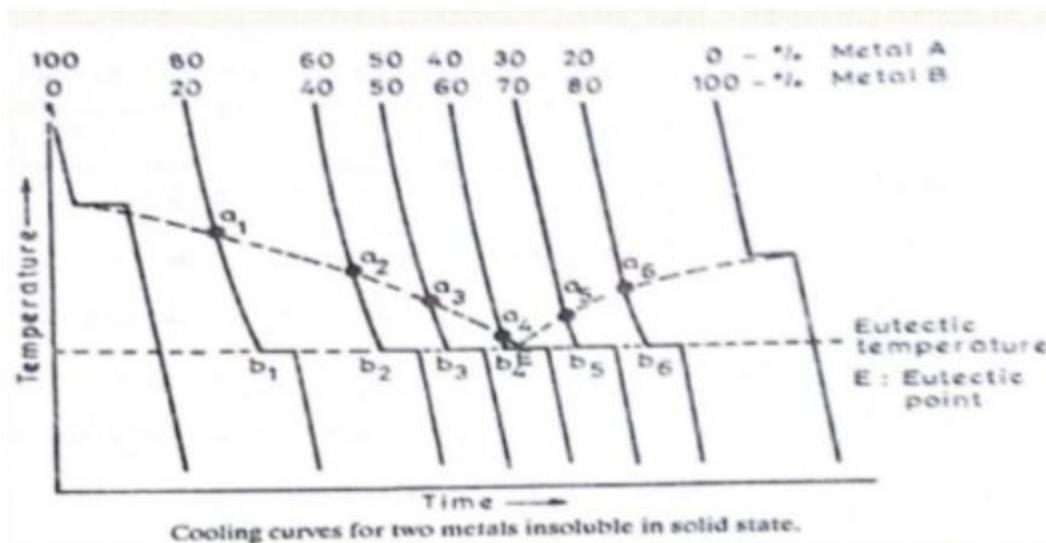
In eutectic reaction the liquid phase solidifies to produce two solid phases. In eutectic systems the two components have limited solubility, which means that they form other solid solutions or compounds above the solubility limit.

In the diagram, the α phase is a solid solution of Pb with a small amount of Sn as the solute.

Eutectic System

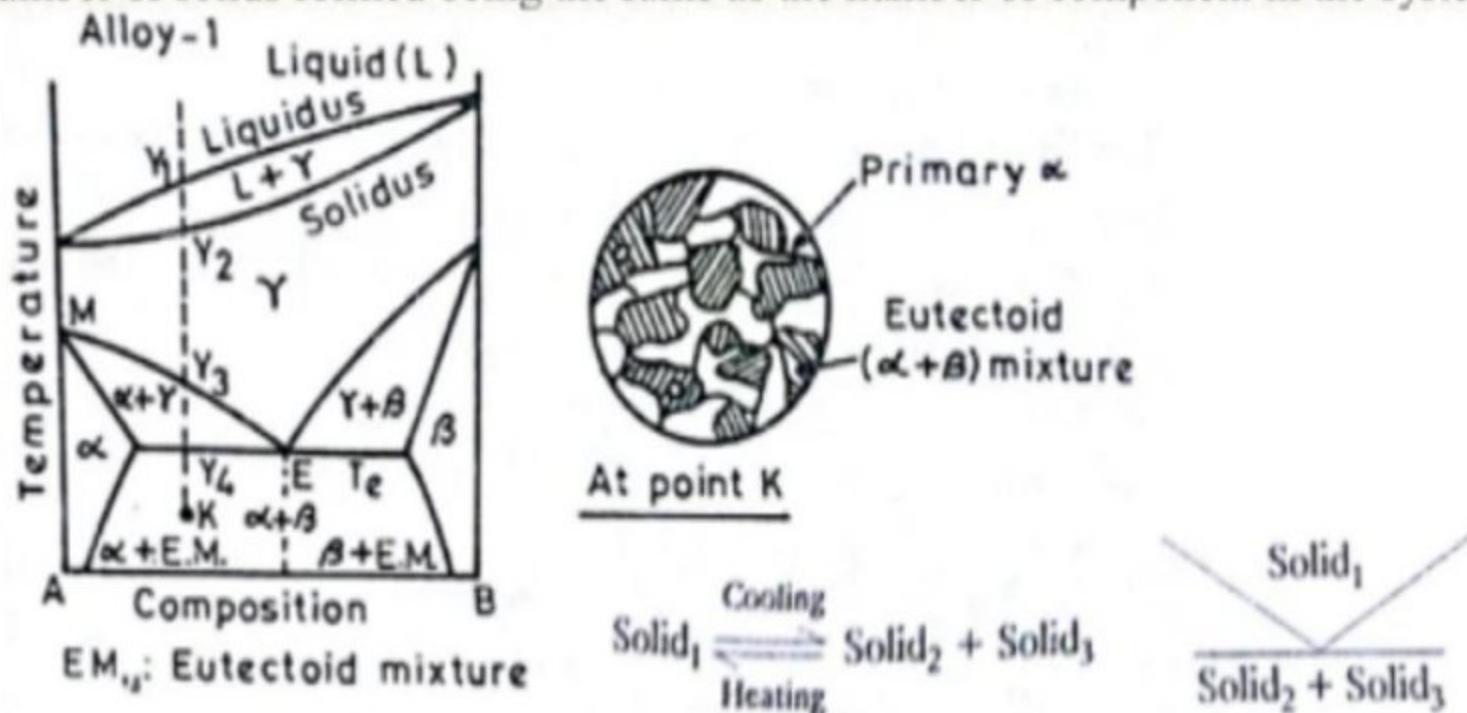


COOLING CURVES (For eutectic system)



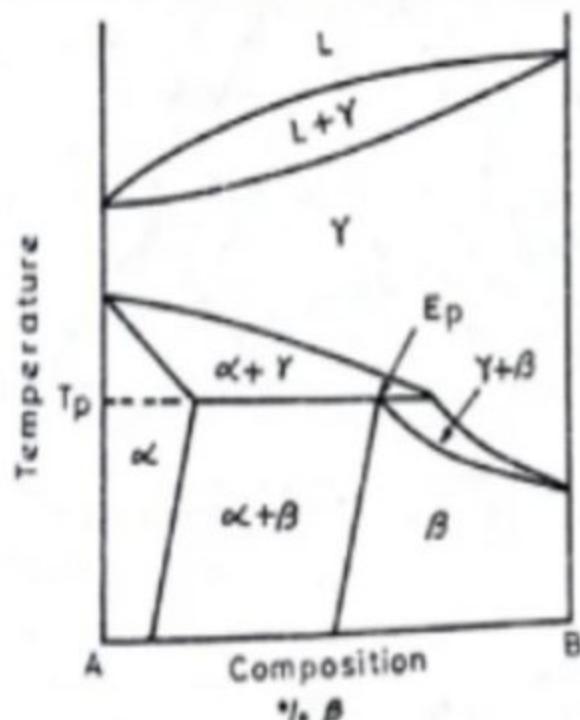
2. Eutectoid Transformation

- Eutectoid reaction is an isothermal reversible reaction in which a solid phase (usually solid solution) is converted into two or more intimately mixed solids on cooling, the number of solids formed being the same as the number of component in the system.

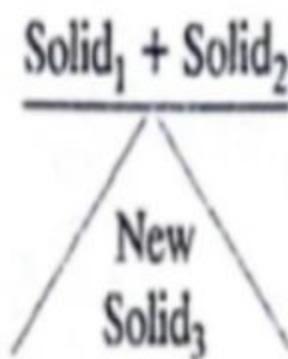
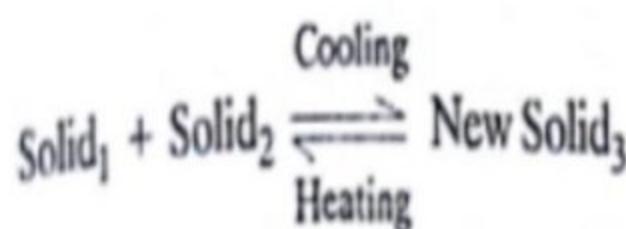


3. Peritectoid Transformation

- The peritectoid reaction is the transformation of two solid into a third solid.



A peritectoid diagram.

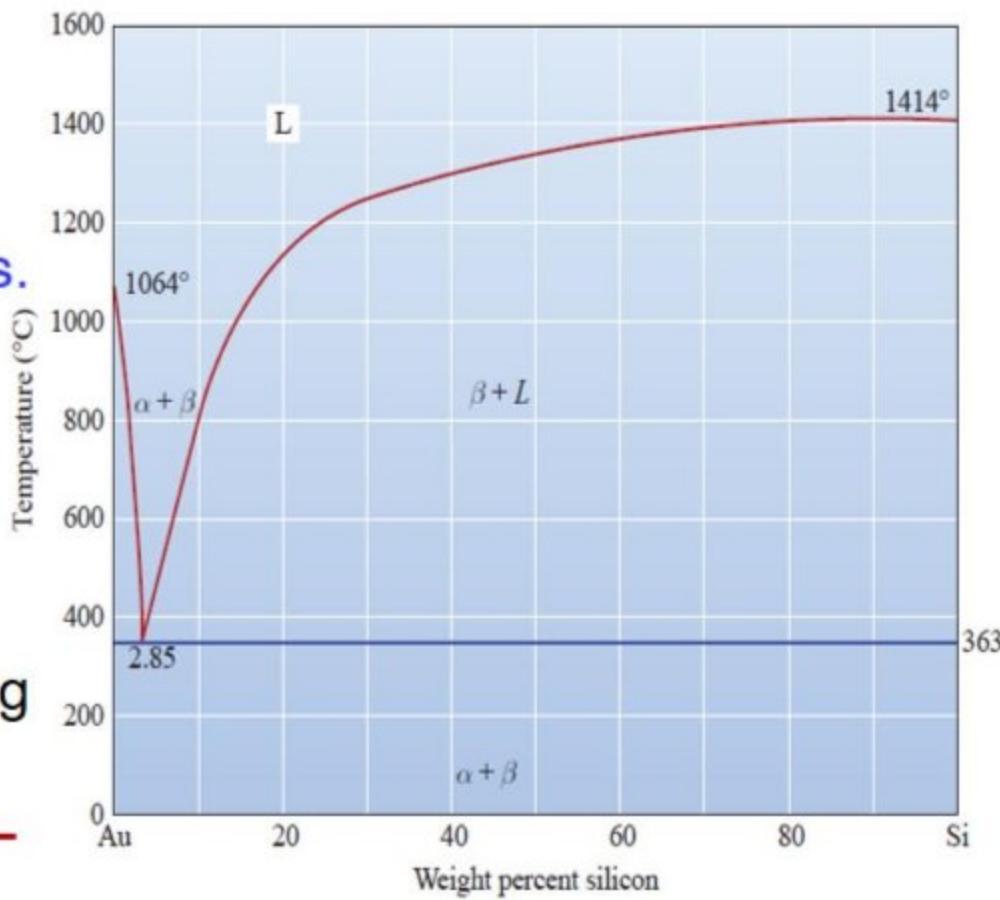


INDUSTRIAL APPLICATIONS: Nanowires (Eutectic Phase Diagram)

Nanowires have great technological interest due to their electrical, mechanical, chemical, and optical properties.

Potential applications include **biological and chemical sensors** and **electrical conductors** in nanoelectronic devices.

A common method of fabricating nanowire materials is through the technique known as **Vapor-Liquid–Solid (VLS) growth**.



The growth of silicon nanowires can be understood by considering the gold–silicon binary phase diagram.

- In VLS silicon nanowire growth, the first step is to deposit a thin layer of pure gold on a substrate. When the substrate is heated, the gold dewets from the substrate forming a series of gold nanoparticles on the substrate surface.
- These gold nanoparticles (nanowire catalysts) act as the template for silicon nanowire growth.
- Silane gas SiH_4 then flows through a chamber holding the substrate with the gold nanoparticles.
- The substrate is heated to a temperature above the gold–silicon eutectic temperature of 363°C but below the melting temperature of the gold (1064°C).
- The silane gas decomposes to Si and H_2 gas. The silicon that is produced adsorbs to the gold nanoparticles and diffuses into them, causing the gold nanoparticles to begin to melt (thus forming the “liquid” of VLS growth).
- The gold nanoparticles are held at a constant temperature, the silicon content increases, and the gold–silicon alloy enters a two-phase region in the gold–silicon phase diagram, as shown in the Gold-Silicon binary eutectic phase diagram.

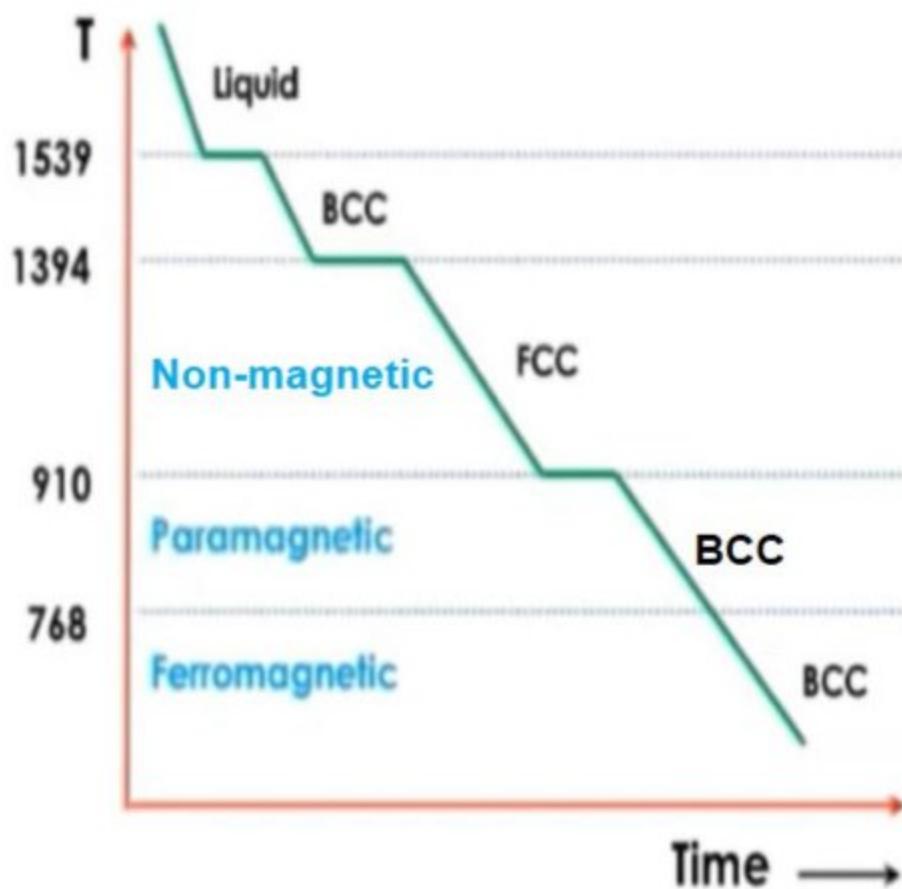
- Silicon continues to diffuse into the gold nanoparticles until the nanoparticles are completely molten.
- The nanoparticles retain their shape as roughly hemispherical balls due to surface-energy considerations between the nanoparticles and the substrate.
- As the silicon content increases due to the diffusion of silicon into the nanoparticles, a solid phase begins to form.
- The solid phase that forms is the silicon nanowire. It is pure silicon. The nanowire has the same diameter as the gold nanoparticle.
- The liquid phase that is present rides atop the silicon nanowire as it grows upward from the substrate.
- The nanowire continues to grow in length while silane gas is supplied to the substrate.
- When the nanowire growth is complete, the temperature is reduced, and the liquid phase atop the silicon nanowire solidifies on cooling through the eutectic temperature.

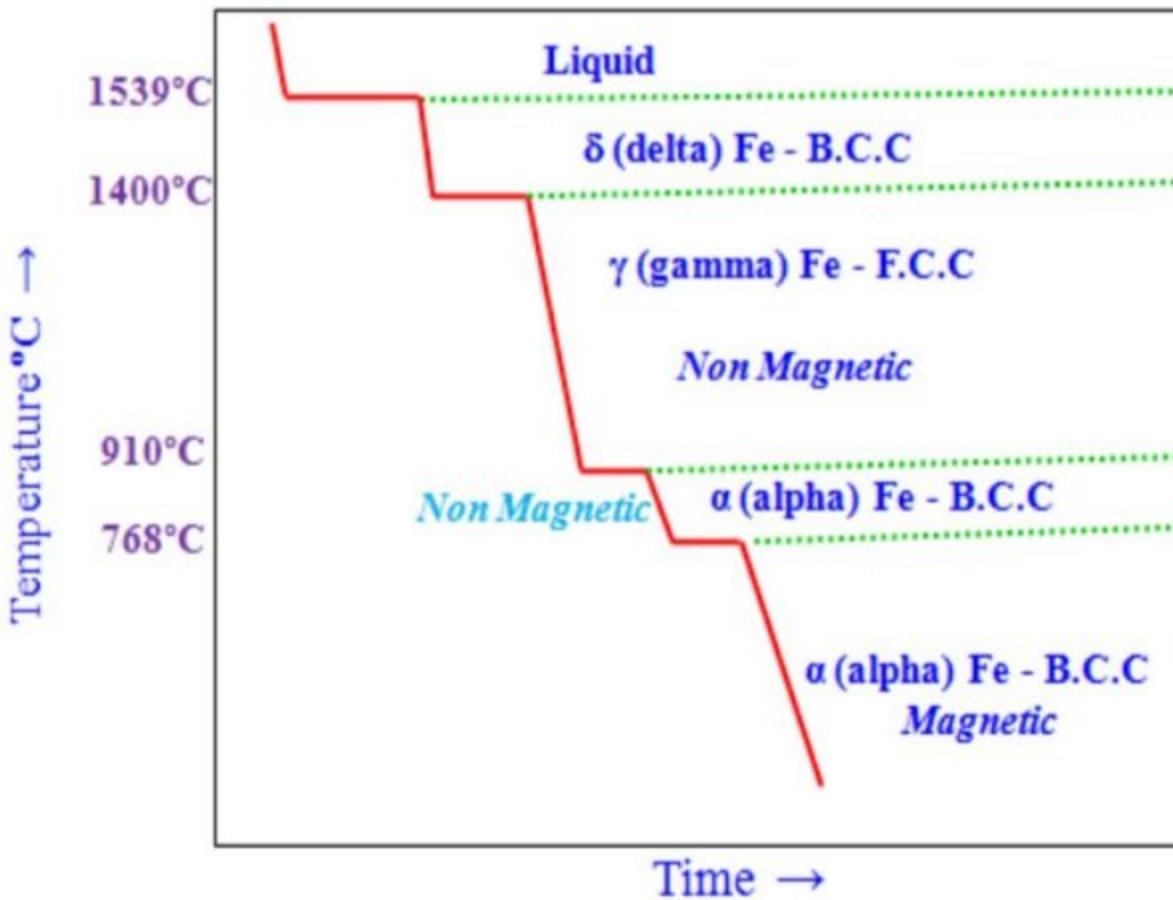
Iron – Carbon Phase Diagram

Cooling Curve for Pure Iron

Key Points:

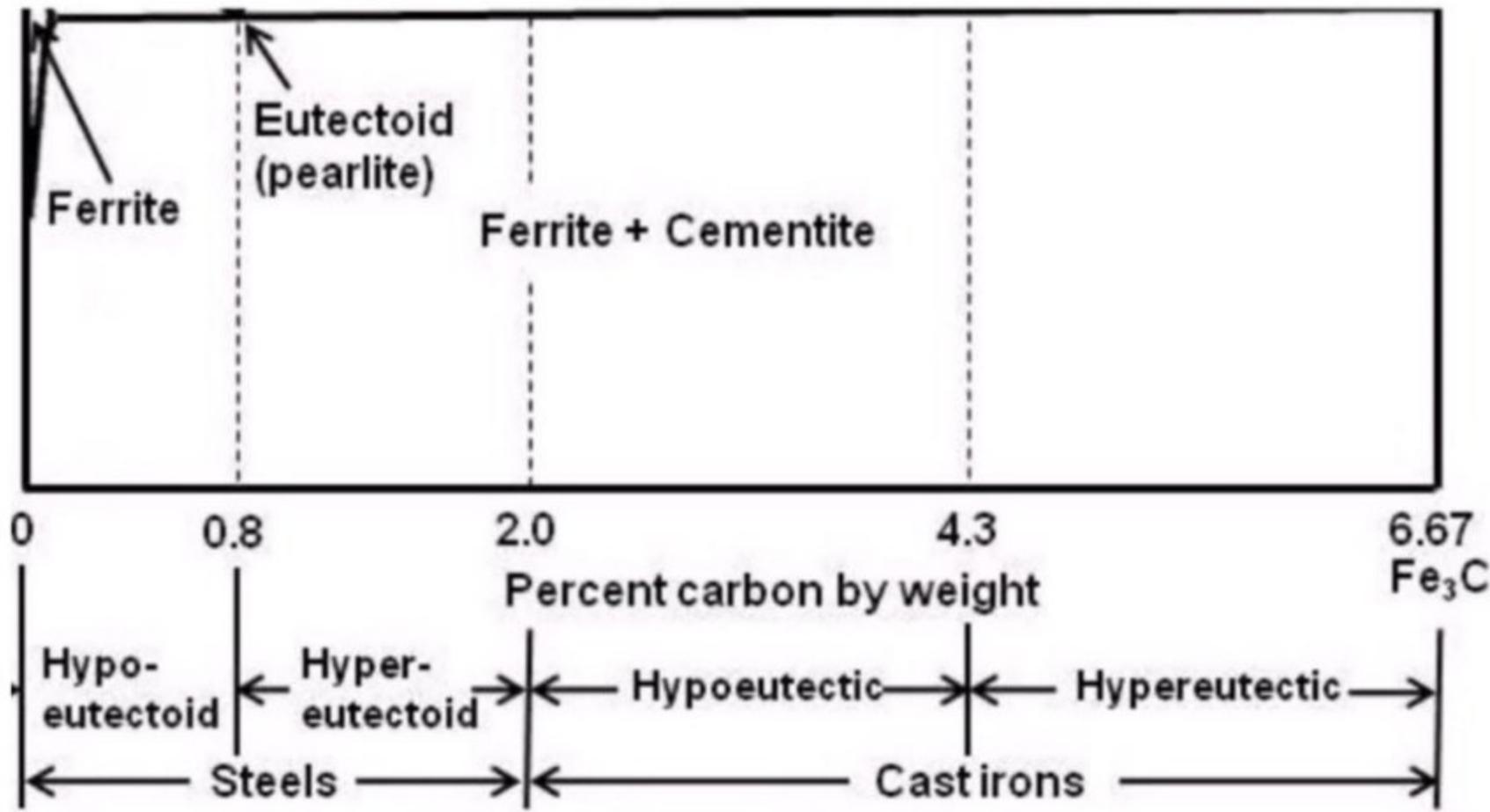
- Pure Iron melts or solidifies at 1559°C and from 1539 to 1394°C it remains in BCC structure.
- Upon further cooling from 1394°C , iron atoms rearrange themselves to convert into Face Centred Cubic (FCC) which is non-magnetic.
- At 910°C another phase change occurs from Face-Centered Cubic (F.C.C) nonmagnetic to Body-Centred Cubic (B.C.C) nonmagnetic iron
- Finally at 768°C , iron becomes magnetic without a change in lattice structure





Allotropic Transformations in Iron

Iron is an allotropic metal which exists in more than one type of lattice structure depending upon temperature.
 A cooling curve for pure iron is shown above



0.05% - 0.25%

Low carbon steel

0.26% - 0.54%

Medium carbon steel

0.55% - 0.95%

High carbon steel

0.96% - 2.20%

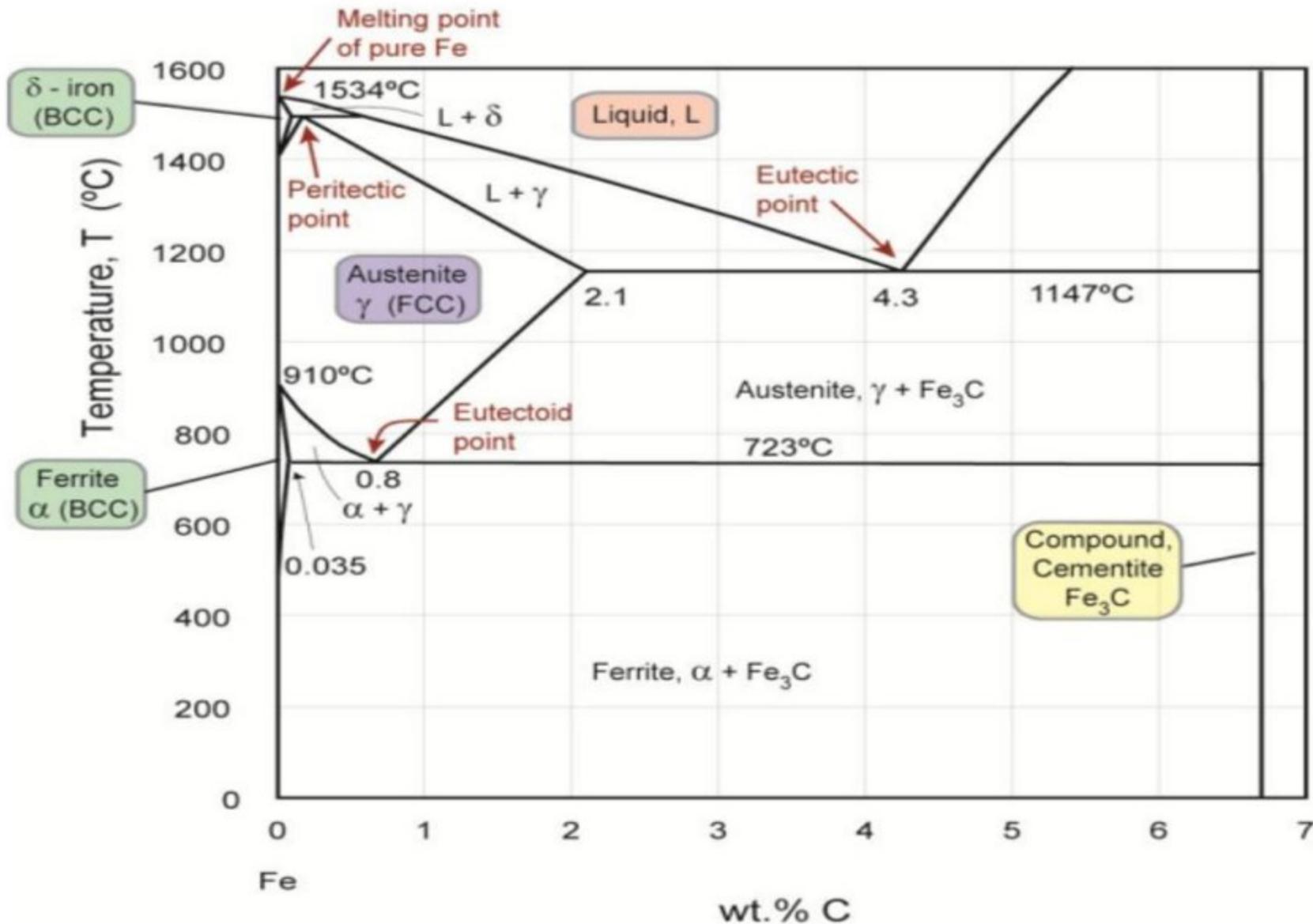
Very high carbon steel

| | |
|---------------|------------------------|
| 0.05% - 0.25% | Low carbon steel |
| 0.26% - 0.54% | Medium carbon steel |
| 0.55% - 0.95% | High carbon steel |
| 0.96% - 2.20% | Very high carbon steel |

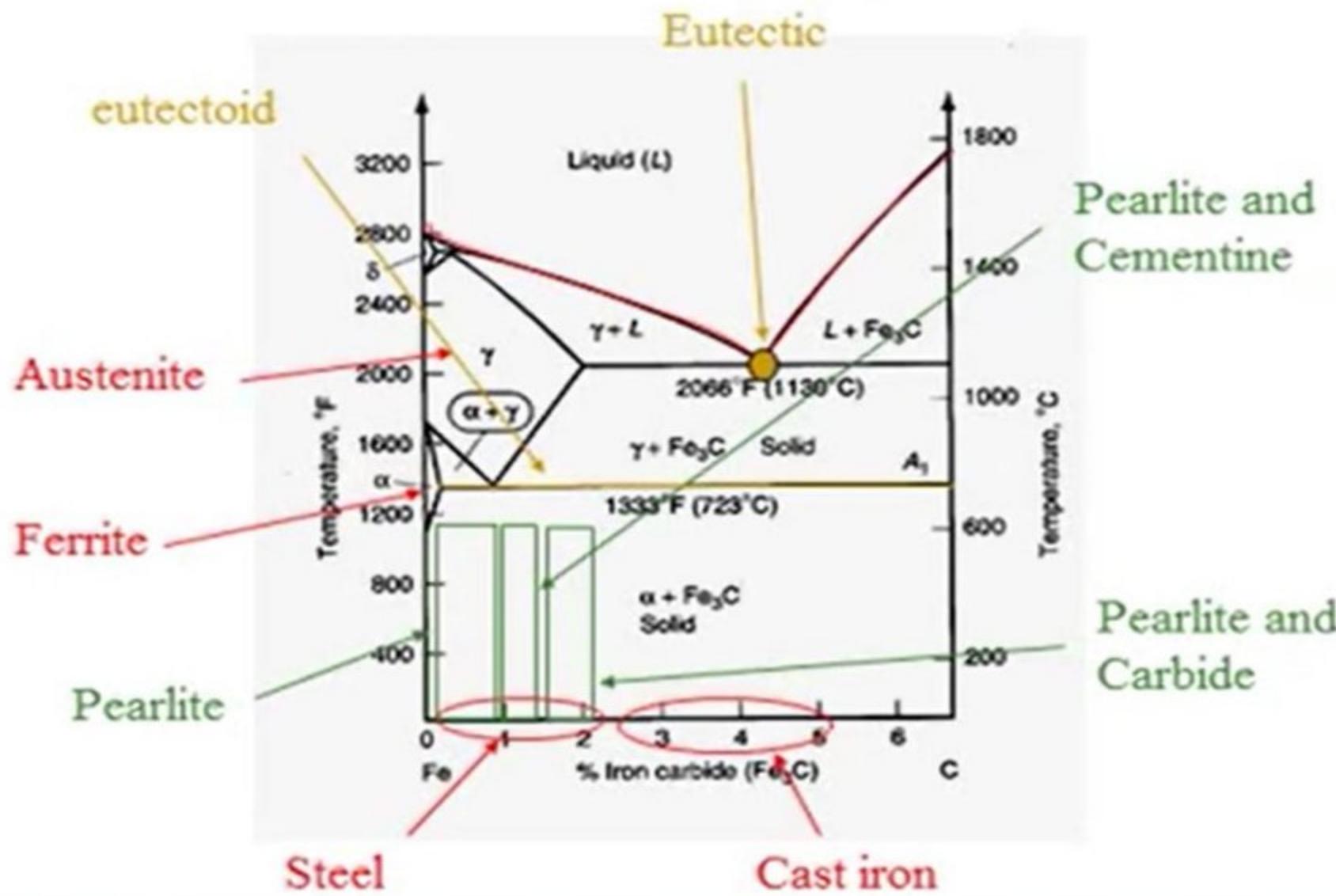
Iron Carbon Diagram

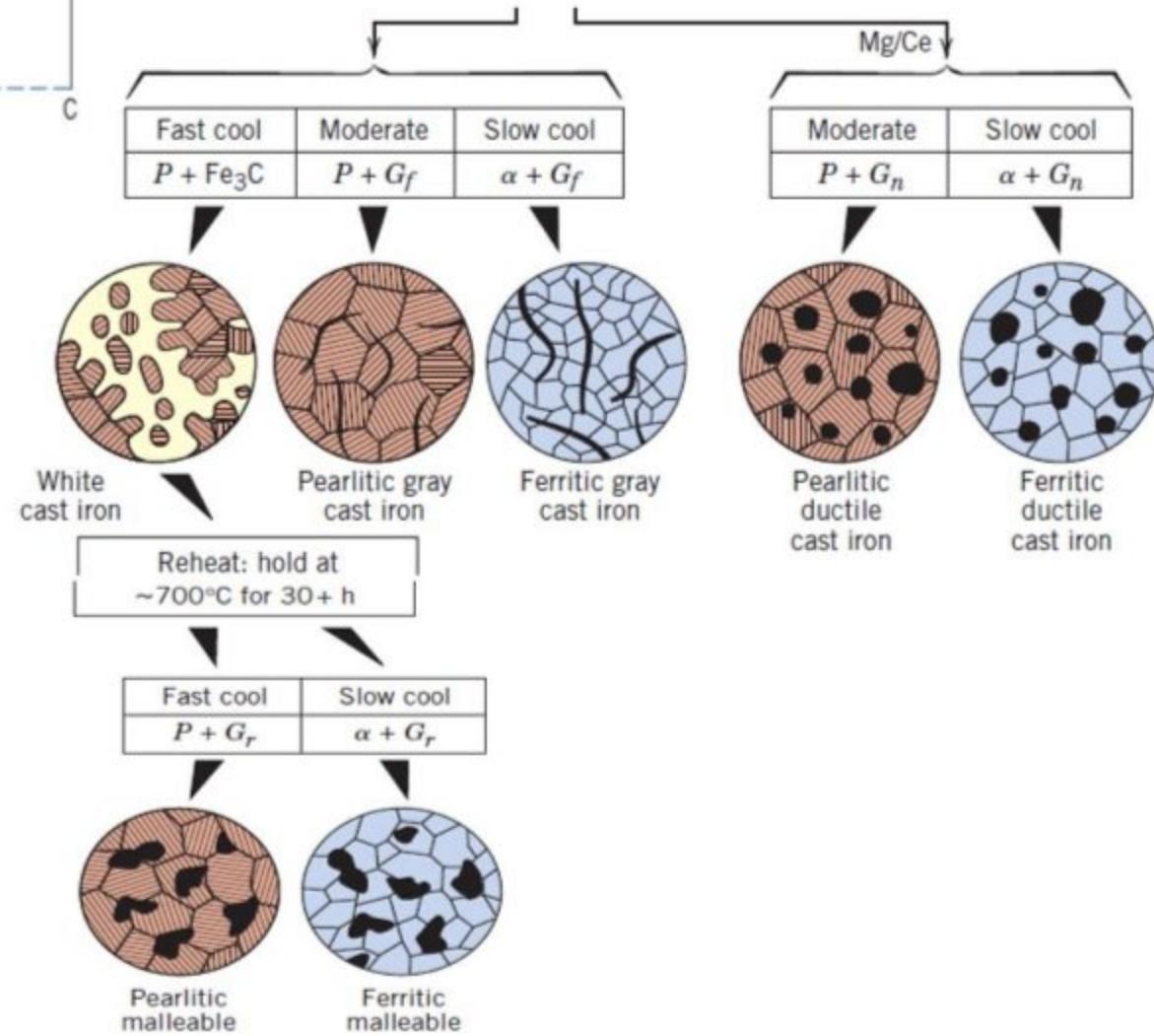
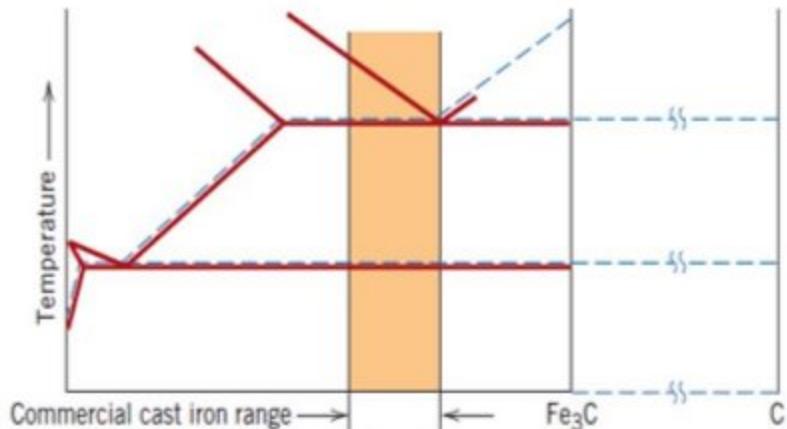
- In their simplest form, steels are alloys of Iron (Fe) and Carbon (C)
- The study of the constitution and structure of iron and steel start with the iron-carbon phase diagram
- It is also the basis of understanding of the heat treatment of **steels**

IRON-CARBON PHASE DIAGRAM



IRON-CARBON DIAGRAM





The Iron-Iron Carbide Diagram

- A map of the temperature at which different phase changes occur on very slow heating and cooling in relation to Carbon, is called Iron-Carbon Diagram.
- Iron-Carbon diagram shows
 - the type of alloys formed under very slow cooling,
 - proper heat-treatment temperature and
 - how the properties of steels and cast irons can be radically changed by heat-treatment.

Three Phase Reactions

- Peritectic, at 1490 deg.C, with low wt% C alloys (almost no engineering importance).
- Eutectic, at 1130 deg.C, with 4.3wt% C, alloys called cast irons.
- Eutectoid, at 723 deg.C with eutectoid composition of 0.8 wt% C, two-phase mixture (ferrite & cementite). They are steels.

Principal phases of steel and their Characteristics

| Phase | Crystal structure | Characteristics |
|-----------|---|---------------------------------------|
| Ferrite | BCC | Soft, ductile, magnetic |
| Austenite | FCC | Soft, moderate strength, non-magnetic |
| Cementite | Compound of Iron & Carbon Fe ₃ C | Hard & brittle |

Important Metallurgical Phases and Micro-constituents

| Phase | Crystal Structure | Characteristics |
|-------------------------------------|----------------------|---|
| Ferrite(α -iron) | BCC | Relatively soft low-temperature phase; stable equilibrium phase |
| δ -ferrite (δ -iron) | BCC | Isomorphous with α -iron; high-temperature phase; stable equilibrium phase |
| Austenite (γ -iron) | FCC | Relatively soft medium-temperature phase; stable equilibrium phase |
| Cementite (Fe ₃ C) | Complex orthorhombic | Hard metastable phase |
| Graphite | Hexagonal | Stable equilibrium phase |
| Pearlite | | Metastable micro constituent; lamellar mixture of ferrite and cementite |

INDUSTRIAL APPLICATION

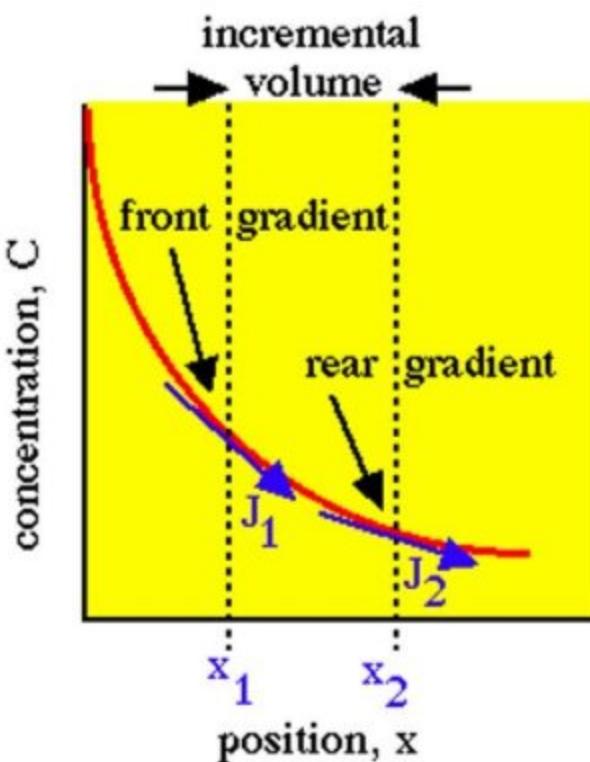
Designing a Test to Separate Ni-based Stainless Steel Scarp

- For efficient recycling of stainless steel scrap, the high-nickel stainless steel and the low-nickel stainless steel are to be separated.
- Performing a chemical analysis on each piece of scrap is tedious and expensive.
- Sorting based on hardness might be less expensive; however, because of the different types of treatments - such as annealing, cold working, or quench and tempering - the hardness may not be related to the steel composition.
- **The high-nickel stainless steels are ordinarily austenitic, whereas the low-nickel alloys are ferritic or martensitic. An ordinary magnet will be attracted to the low nickel ferritic and martensitic steels, but will not be attracted to the high-nickel austenitic steel.**
- This simple and inexpensive magnetic test for separation process is more useful.

FICK'S LAWS

Fick's first law states that the rate of diffusion of a substance across unit area (such as a surface or membrane) is proportional to the concentration gradient.

Fick's second law of diffusion describes the rate of accumulation (or depletion) of concentration within the volume as proportional to the diffusivity (or local curvature of the concentration gradient).



Interconnects in IC chips

- In integrated circuits (ICs), interconnects are structures that connect two or more circuit elements (such as transistors) together electrically.
- The design and layout of interconnects on an IC is vital to its proper function, performance, power efficiency, reliability, and fabrication yield.
- Interconnections may be composed by conductive lines, vias, wires, pads, and joints.
- Each of these components can be formed by different materials.
- Metals and theirs alloys are usually the best choice for building interconnections, due to their high electrical conductivity.

FRACTURE MECHANICS

Fracture mechanics is the field of mechanics concerned with the study of the propagation of cracks in materials.

BRITTLE FRACTURE - Griffith Theory

The **Griffith theory** states that a crack will propagate when the reduction in potential energy that occurs due to crack growth is greater than or equal to the increase in surface energy due to the creation of new free surfaces.

This theory is applicable to elastic materials that fracture in a brittle fashion.

FRACTURE TOUGHNESS

“Fracture toughness” is the **resistance of brittle materials to the propagation of flaws under an applied stress**.

Longer the flaw, lower is the stress needed to cause fracture.

The ability of a flaw to cause fracture depends on the fracture toughness of the material.

EMBRITTLEMENT PHENOMENA

Various metal alloys experience a significant reduction in ductility and tensile strength when atomic hydrogen penetrates into the material known as *hydrogen embrittlement, hydrogen-induced cracking or hydrogen stress cracking*.

It is a type of failure in response to applied or residual tensile stresses resulting brittle fracture.

Atomic hydrogen diffuses interstitially through the crystal lattice most often can lead trans-granular cracking, based on the interference of dislocation motion by the dissolved hydrogen.

Pickling of steels in sulfuric acid, electroplating, elevated temperatures such as during welding and heat treatments are examples where hydrogen embrittlement may occur.

Hydrogen sulfide, probably the most aggressive poison, is found in petroleum fluids, natural gas, oil-well brines, and geothermal fluids retard the formation of molecular hydrogen and thereby increase the residence time of atomic hydrogen on the metal surface, ie. Embrittlement.

EMBRITTLEMENT PHENOMENA (... continued)

- High-strength steels are susceptible to hydrogen embrittlement, and increasing strength tends to enhance the material's susceptibility.
- Martensitic steels are especially vulnerable to this type of failure; bainitic, ferritic, and spheroiditic steels are more resilient.
- FCC alloys (austenitic stainless steels, and alloys of copper, aluminum, and nickel) are relatively resistant to hydrogen embrittlement, mainly because of their inherently high ductilities.
- However, strain hardening these alloys will enhance their susceptibility to embrittlement.
- Some of the techniques commonly used to reduce the hydrogen embrittlement include reducing the tensile strength of the alloy via a heat treatment, removal of the source of hydrogen, "baking" the alloy at an elevated temperature to drive out any dissolved hydrogen, and substitution of a more embrittlement-resistant alloy.

FATIGUE

The behavior of materials under repeated cycles of stress or strain which cause a deterioration of the material that results in a progressive failure.

CREEP

Creep (cold flow) is the tendency of a solid material to move slowly or deform permanently under the influence of persistent mechanical stresses.

Occurs as a result of long-term exposure to high levels of stress that are still below the yield strength of the material.

Creep is more severe in materials that are subjected to heat for long periods and generally increases as they near their melting point.

STRENGTHENING MECHANISMS

The relation between dislocation motion and mechanical behavior of metals / alloys is important to understand the strengthening mechanisms.

The macroscopic plastic deformation corresponds to the motion of large numbers of dislocations.

In other words, the ability of a metal to plastically deform depends on the ability of dislocations to move.

By reducing the mobility of dislocations, the mechanical strength may be enhanced.

Restricting or hindering dislocation motion renders a material harder and stronger.

The ease with which a material is capable of plastic deformation is a function of dislocation mobility - that is, restricting dislocation motion leads to increase in hardness and strength.

Single phase metals may be strengthened by grain size reduction, solid-solution alloying, and strain hardening methods.

STRENGTHENING MECHANISMS (... continued)

Strengthening by Grain Size Reduction

Grain boundaries are barriers to dislocation motion for two reasons: When crossing a grain boundary, a dislocation's direction of motion must change. There is a discontinuity of slip planes within the vicinity of a grain boundary. A metal that has small grains will be stronger than one with large grains because the former has more grain boundary area, and, thus, more barriers to dislocation motion.

For most metals, yield strength depends on average grain size.

Solid-Solution Strengthening and Dispersion Strengthening

The strength and hardness of a metal increases with increase of concentration of impurity atoms that go into solid solution (both substitutional and interstitial).

Solid-solution strengthening results from lattice strain interactions between impurity atoms and dislocations; these interactions produce a diminishment in dislocation mobility.

Solid-solution strengthening is due to the point defects created which restrict dislocation motion and hence cause strengthening.

STRENGTHENING MECHANISMS (... continued)

The effects of several alloying elements on the yield strength of copper is shown.

Nickel and zinc atoms are about the same size as copper atoms, but beryllium and tin atoms are much different size from copper atoms.

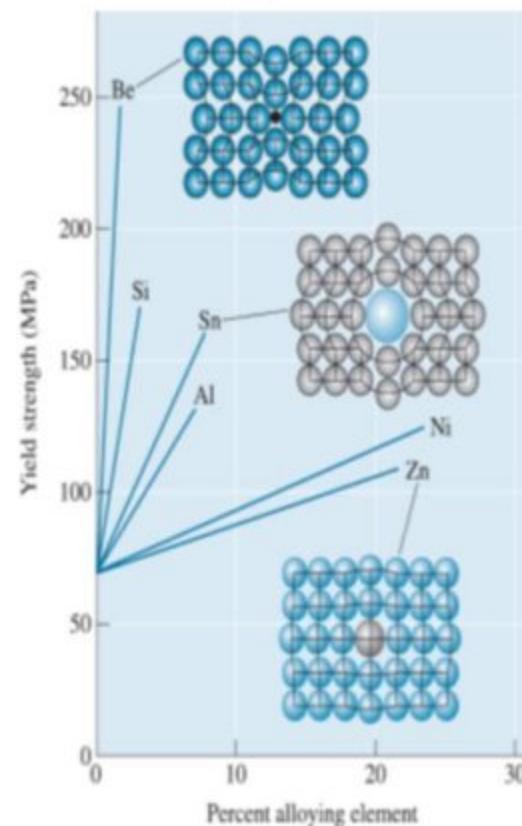
Increasing both atomic size difference and amount of alloying element increases solid-solution strengthening.

A larger size difference produces a greater disruption of the initial crystal structure, making slip more difficult.

Also, increase in amount of such alloying atoms increases the strength.

If very high amount of a large or small alloying atom is added, the solubility limit may be exceeded and dispersion strengthening mechanism may come in to play.

In dispersion strengthening, the interface between the base metal phase and alloy metal phase resists dislocation motion and contributes to strengthening



Strain Hardening

The strengthening obtained during cold working by increasing the dislocation density is called strain hardening or work hardening.

Strain hardening is the enhancement in strength (and decrease of ductility) of a metal as it is plastically deformed.

During plastic deformation

- (1) dislocation density increases,
- (2) the average distance between adjacent dislocations decreases,
- (3) repulsive — dislocation mobility becomes more restricted and thus, the metal becomes harder and stronger.

To develop *strain hardening* the specimen is stressed beyond the yield strength before the stress is removed.

Now the specimen has a higher yield strength and tensile strength, but lower ductility.

By repeating the procedure, the strength continues to increase and the ductility continues to decrease until the alloy becomes very brittle.

Age-hardening or Precipitation Hardening

Age hardening, or precipitation hardening, is produced by a sequence of phase transformations that leads to a uniform dispersion of nano-sized, coherent precipitates in a softer, more ductile matrix.

Used to increase the yield strength through simple heat treatments and without creating significant changes in density.

There are three steps in the age-hardening heat treatment:

- (1) Solution treatment: The Al alloy (4% Cu) is first heated above the solvus temperature and held until a homogeneous solid solution α is produced,
- (2) Quench: The alloy which contains only α in its structure is rapidly cooled or quenched and the structure is a **supersaturated solid solution** (α_{ss}) and
- (3) Age: It is heated to aging temperature below the solvus temperature and is held for sufficient time. This produces the equilibrium $\alpha+\theta$ structure.

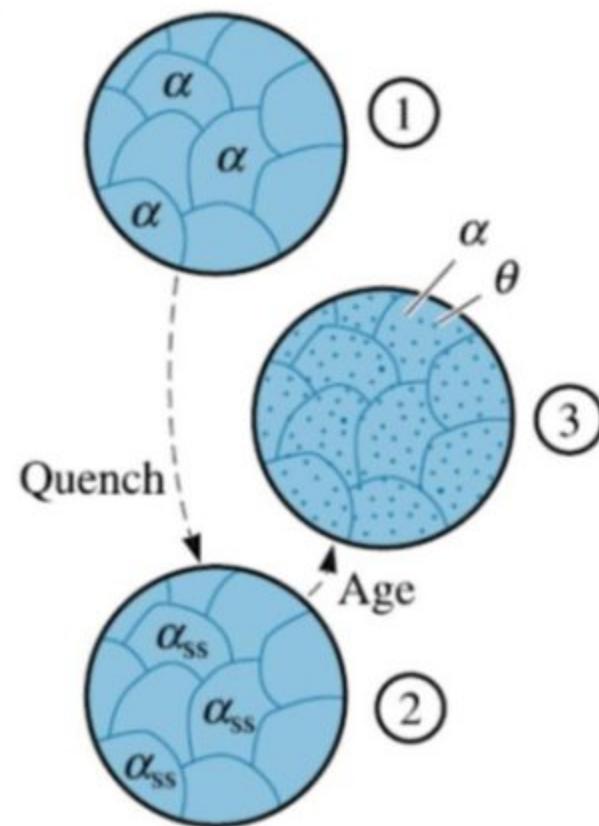
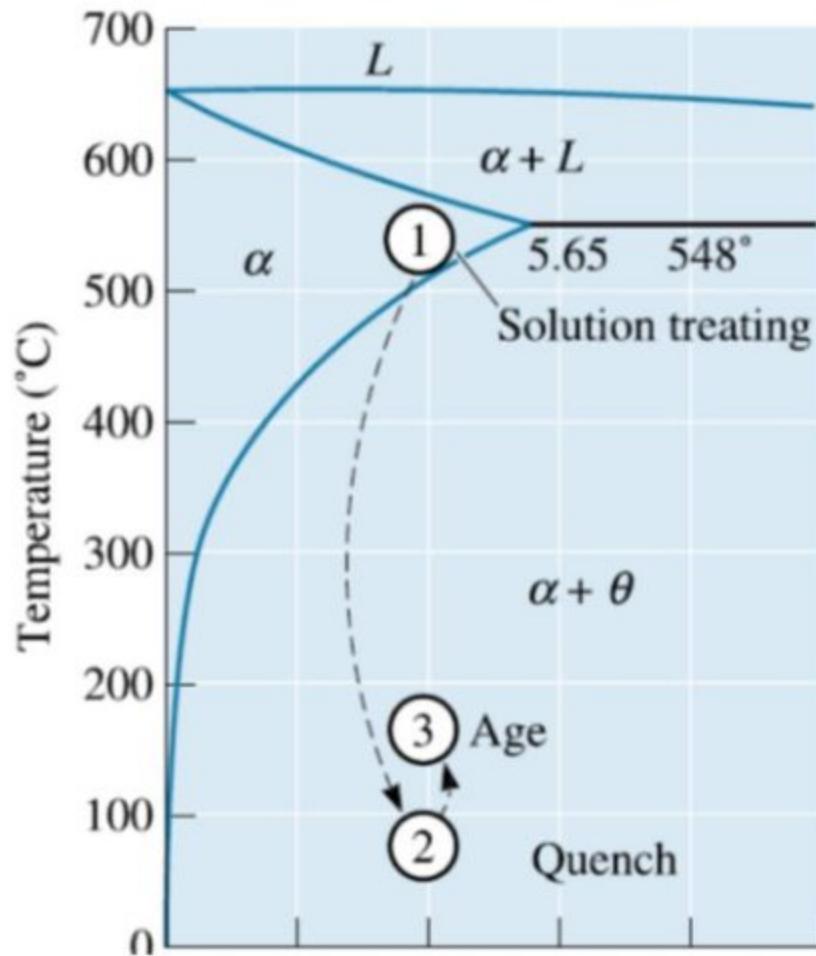
INDUSTRIAL APPLICATIONS: Aluminium alloys used in aircraft structures are strengthened by this method (Figure).

Nickel-based super alloys, Titanium alloys, Stainless steels etc. are age-hardened for various applications.

Al and Al-Li alloys (age-hardening strengthened during friction stir welding) for aerospace and aircraft applications.

The three-step heat treatment age hardening produces the θ phase in the form of ultrafine uniformly dispersed second-phase precipitate particles.

This is termed as effective precipitation strengthening.



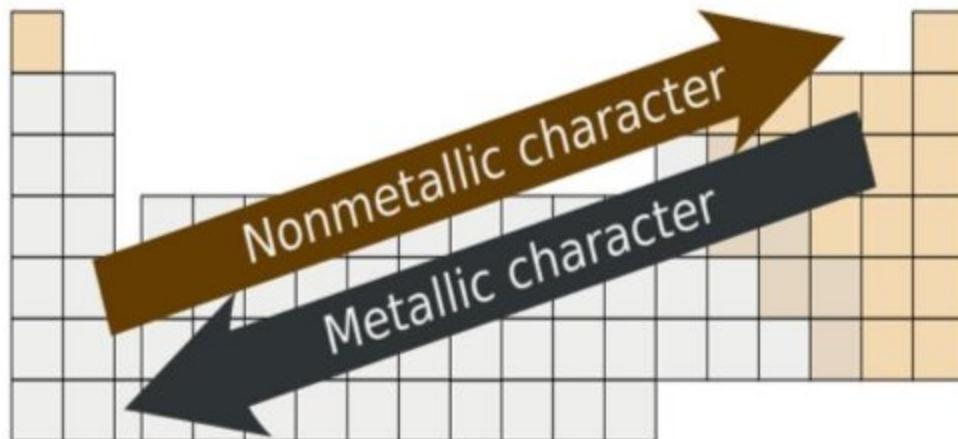
Physical Properties of Metals

- Metals are lustrous, malleable, ductile, good conductors of heat and electricity.
- **State:** Metals are solids at room temperature with the exception of mercury, which is liquid at room temperature (Gallium is liquid on hot days).
- **Luster:** Metals have the quality of reflecting light from their surface and can be polished e.g., gold, silver and copper.
- **Malleability:** Metals have the ability to withstand hammering and can be made into thin sheets known as foils. For example, a sugar cube sized chunk of gold can be pounded into a thin sheet that will cover a football field.
- **Ductility:** Metals can be drawn into wires. For example, 100 g of silver can be drawn into a thin wire about 200 meters long.
- **Hardness:** All metals are hard except sodium and potassium, which are soft and can be cut with a knife.
- **Valency:** Metals typically have 1 to 3 electrons in the outermost shell of their atoms.

- ❑ **Conduction:** Metals are good conductors because they have free electrons. Silver and copper are the two best conductors of heat and electricity. Lead is the poorest conductor of heat. Bismuth, mercury and iron are poor conductors
- ❑ **Density:** Metals have high density and are very heavy. Iridium and osmium have the highest densities whereas lithium has the lowest density.
- ❑ **Melting and Boiling Points:** Metals have high melting and boiling points. Tungsten has the highest melting and boiling points whereas mercury has the lowest. Sodium and potassium also have low melting points.

Chemical Properties of Metals

- ❑ Metals are electropositive elements which form *basic* or *amphoteric* oxides.
- ❑ **Electropositive Character:** Metals tend to have low ionization energies, and *typically lose electrons (i.e. are oxidized) when they undergo chemical reactions*. They normally do not accept electrons.



| Metals | Non-metals | Metalloids |
|---------|------------|------------|
| Gold | Oxygen | Silicon |
| Silver | Carbon | Boron |
| Copper | Hydrogen | Arsenic |
| Iron | Nitrogen | Antimony |
| Mercury | Sulfur | Germanium |
| Zinc | Phosphorus | |

Extraction of Iron

| | | |
|--|-------------------|---|
| 1. Concentration of Ore | (i) Hematite | Fe_2O_3 |
| 2. Extraction of metal from concentrated Ore | (ii) Magnetite | Fe_3O_4 |
| | (iii) Limonite | $\text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ |
| 3. Purification of the metal | (iv) Iron pyrites | FeS_2 |
| | (v) Siderite | FeCO_3 |

Extraction of iron begins with **Concentration, Calcination and Smelting** in blast furnace to get cast iron.

Concentration by gravity separation removes unwanted earthy materials like clay, sand, etc.

Calcination in a reverberatory furnace with limited air removes moisture and other volatile impurities such as S, P, As and carbonates.

The calcined ore, coke and limestone (CaCO_3) are mixed (8:4:1 ratio) and fed into the blast furnace from the top.

Blast Furnace

The purpose of a **Blast Furnace** is to reduce the concentrated ore chemically to its liquid metal state.

A blast furnace is a gigantic, steel stack lined with refractory brick where the concentrated **iron ore, coke, and limestone** are dumped from the top, and a blast of hot air is blown into the bottom.

All the three ingredients are crushed into small round pieces and mixed and put on a hopper which controls the input.

Hot air is blown from the bottom and coke it burned to yield temperatures up to about 2200K.

At such high temperatures, coke reacts with the oxygen in the hot air to form carbon monoxide (CO).

The CO and heat now move upwards and meet the raw material running down from the top.

The temperature in the upper parts of the Blast Furnace is considerably lower than the 2200K at the bottom.

In this part, Haematite (Fe_2O_3) and Magnetite (Fe_3O_4) are reduced to Ferrous Oxide (FeO).

BLAST FURNACE

Cast Iron or pig iron:

2 to 4.5% C and little S, Si, P, Mn etc. Most impure iron, brittle, NOT weldable.

Wrought Iron:

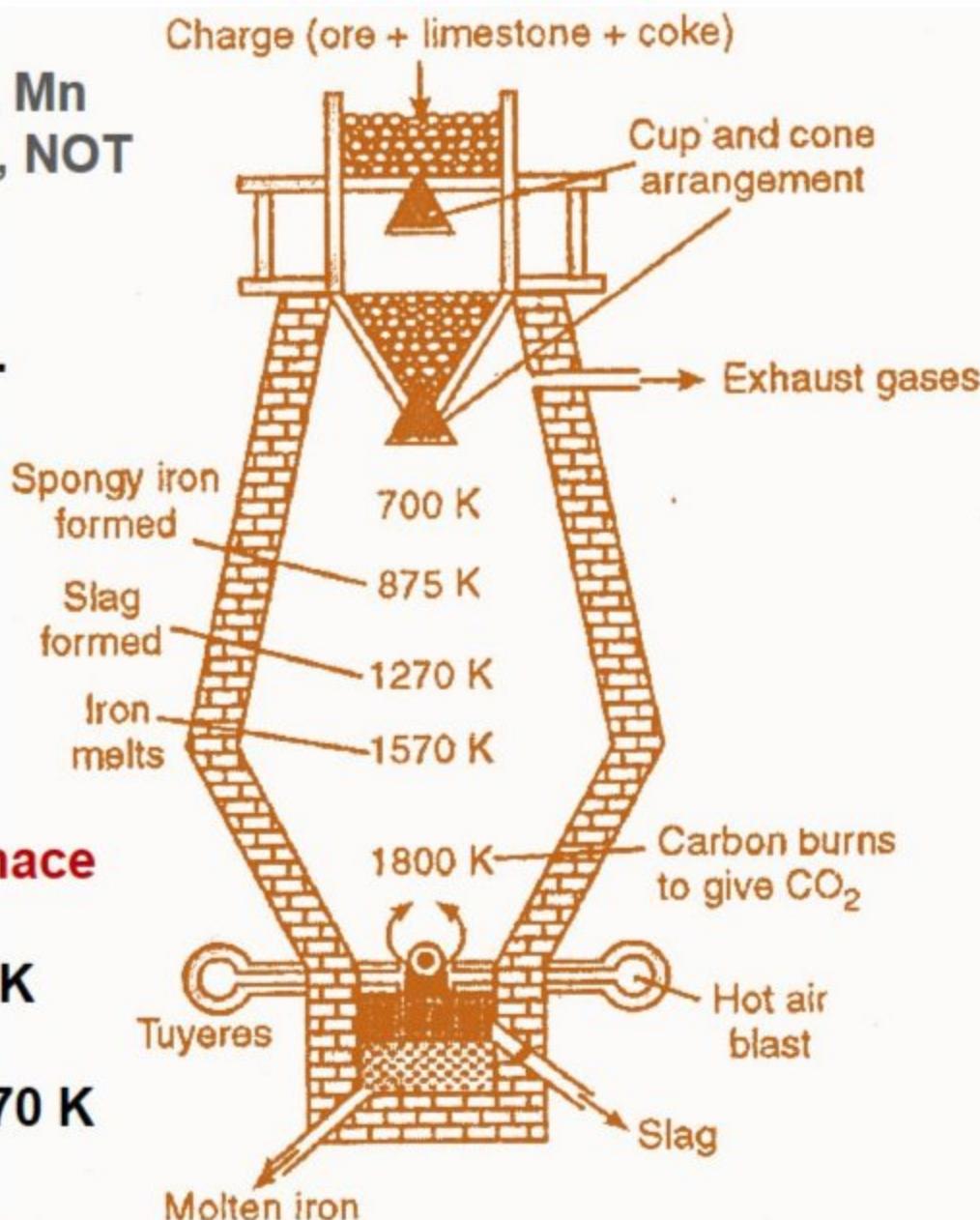
< 0.5% C & others. Pure iron.
Malleable and weldable.

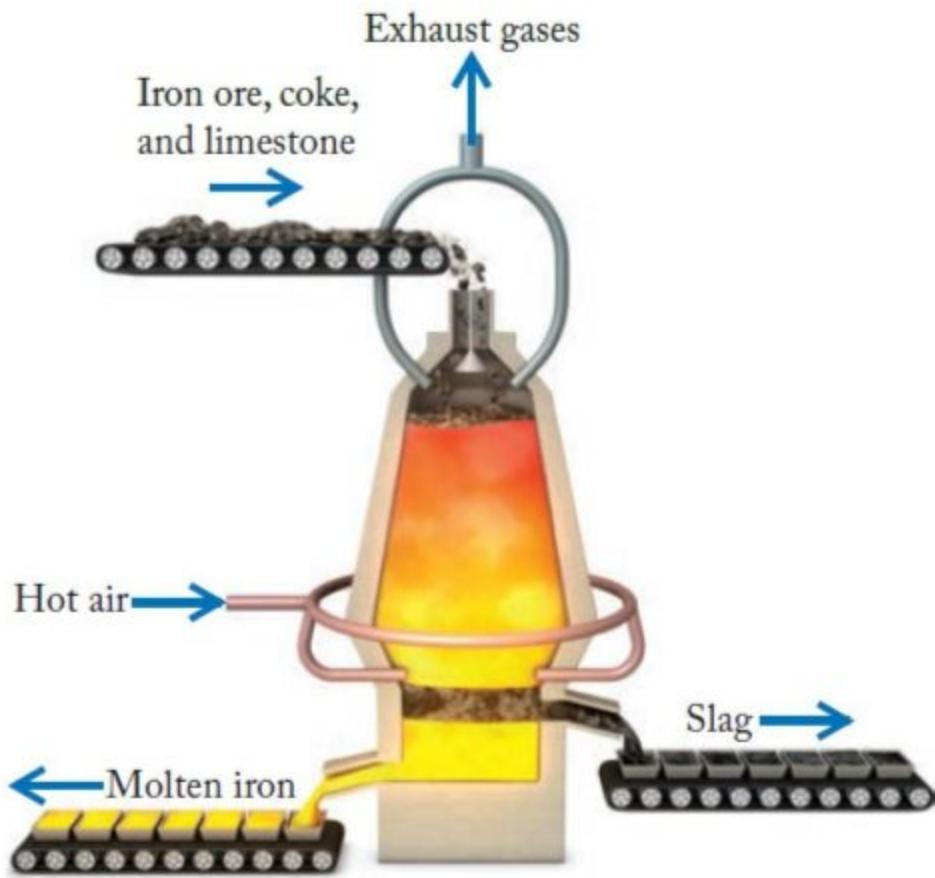
Steel:

0.5 to 1.5% C and Mn, Cr, Ni

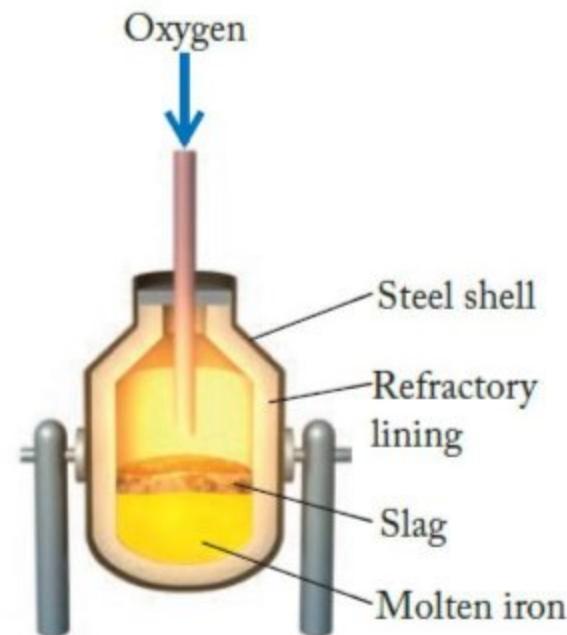
Reaction zones in Blast Furnace

- (0) Hot air burning, 2200 K
- (i) Combustion zone, 1800 K
- (ii) Fusion zone, 1570 K
- (iii) Slag formation zone, 1270 K
- (iv) Reducing zone, 875 K





(a) Blast furnace



(b) The basic oxygen process

(a) Blast furnaces operate continuously at temperatures near 1600°C to convert iron ore into iron. Blasts of hot air inject $\text{O}_2(g)$ into the furnace, which converts C to CO. Limestone is added to react with Si and P impurities. The products of these reactions become part of the slag layer. **(b)** Molten iron from a blast furnace is further purified in a **second furnace**, where pure $\text{O}_2(g)$ is injected instead of air.

Pig Iron & Cast Iron

The limestone also decomposes to CaO which removes the silicate impurity of the ore in the form of Slag.

It can be easily separated out of molten iron.

The **Pig iron** which is hard and brittle manufactured in Blast Furnaces contain about 3 – 4 % of C and smaller quantities such as S, Si, etc.

To further reduce the carbon content of pig iron, it is melted again with scraps of iron and coke and subjected to the blast of hot air.

The **Cast Iron** formed has a carbon content 2 – 3 %.

Wrought Iron/ Malleable Iron

Wrought iron is the purest form of iron available commercially and is prepared by heating cast iron in a furnace lined with Fe_2O_3 .

The Haematite (Fe_2O_3) reacts with C in the cast iron to give pure iron and carbon monoxide gas which escapes.

Limestone is then added as flux, and it creates the slag. Impurities such as S, Si pass into the slag and the slag later can be easily separated to yield pure iron. **Magnesium powder** blown through the molten iron reacts with sulphur forms MgS which goes into slag.

Birth of the Steel Industry

- The revolution in steel production that provided cheaper, higher quality material, was recognized by many businessmen of the day as an investment opportunity.
- Capitalists of the late 19th century, including Andrew Carnegie and Charles Schwab, invested and made millions (billions in the case of Carnegie) in the steel industry.
- Carnegie's US Steel Corporation, founded in 1901, was the first corporation ever valued at more than \$1 billion.

Bessemer Process and Modern Steelmaking

- The growth of railroads during the 1900s put great pressure on the iron industry, which still struggled with inefficient production processes.
- Steel was still unproven as a structural metal and production was slow and costly, until 1856.
- Henry Bessemer came up with a more effective way to introduce oxygen into molten iron to reduce the carbon content (Bessemer Process).

Bessemer Process and Modern Steelmaking

- He designed a pear-shaped receptacle - a converter - in which iron is heated while oxygen is blown through the molten metal.
- As oxygen passed through the molten metal, it reacts with carbon, releasing carbon dioxide and producing a more pure iron.
- The process is fast and inexpensive but removal of carbon much more and oxygen addition to very high levels are the problems.
- Bessemer began adding a compound of iron-carbon-manganese in correct amount to his conversion process with great success and solved the problems.
- Also, limestone (a basic flux) added to Bessemer Process removes phosphorus from pig iron into the slag.
- Steel prices dropped drastically (1880s), initiating growth of the world steel industry.

The Open Hearth Process

- This process enhanced steel production from pig iron in large shallow furnaces.
- Using high temperatures to burn off excess carbon and other impurities in a heated brick chambers below the regenerative hearth furnaces.
- Larger quantities (50-100 metric tons in one furnace) of steel produced.
- periodic testing of the molten steel can be carried out to meet particular specifications.
- Scrap steel can be used as the raw material.
- Although the process itself was much slower, by 1900 the open hearth process had largely replaced the Bessemer process.

Oxygen Steelmaking

- The majority of global steel production - about 66 % - is produced in basic oxygen facilities.
- The **basic oxygen furnace** was developed to separate oxygen from nitrogen on an industrial scale in 1960s.
- Basic oxygen furnaces blow oxygen into large quantities of molten iron and scrap steel and can complete a charge much more quickly than open-hearth methods.
- Large vessels holding up to 350 metric tons of iron can complete conversion to steel in less than one hour.
- The cost efficiencies of oxygen steelmaking made open-hearth factories uncompetitive open-hearth operations began closing.

Electric Arc Furnace Steel Making

- Electric arc furnace (EAF) was designed to pass an electric current through charged material, resulting in exothermic oxidation above 1,800°C, more than sufficient to heat steel production.
- Initially used for specialty steels, EAFs grew in use and during World War II used for the manufacturing of steel alloys.
- The low investment cost involved in setting up EAF mills, especially in carbon steels or long products.
- Cold ferrous or 100% scrap feed, less energy per unit of production, stopping & starting operation with little cost are advantages in this process.
- For these reasons, production via EAFs has been steadily increasing for more than 50 years and accounted for about 33 percent of global steel production, as of 2017.

The Modern Steel Production Process

- Modern methods of steel production are still based on the same premise as the original Bessemer Process, which uses oxygen to lower the carbon content in iron.
- Today, steel production makes use of recycled materials as well as traditional raw materials, such as iron ore, coal, and limestone.
- Primary steelmaking has two processes, **basic oxygen steel-making (BOS)** and **electric arc furnaces (EAF)**, account for virtually all steel production.
- Ironmaking, the first step in making steel, involves the raw inputs of iron ore, coke, and lime being melted in a blast furnace.
- The resulting molten iron / hot metal has 4 to 4.5% carbon and other impurities that make it brittle.
- The BOS method adds recycled scrap steel to the molten iron in a converter.
- At high temperatures, oxygen is blown through the metal, which reduces the carbon content to between 0-1.5%.

The Modern Steel Production Process

- The EAF method feeds recycled steel scrap through high-power electric arcs (with temperatures of up to 1,650°C) to melt the metal and convert it into high-quality steel.
- Secondary steelmaking involves treating the molten steel produced from both BOS and EAF routes to adjust the steel composition.
- This is done by adding or removing certain elements and/or manipulating the temperature and production environment.
- Depending on the types of steel required, the following secondary steelmaking processes can be used:
 - Stirring
 - Ladle furnace
 - Ladle injection
 - Degassing

The Modern Steel Production Process

- ❑ CAS-OB (composition adjustment by sealed argon bubbling with oxygen blowing) process.
- ❑ Continuous casting sees the molten steel cast into a cooled mold, causing a thin steel shell to solidify.
- ❑ The shell strand is withdrawn using guided rolls, then it's fully cooled and solidified.
- ❑ Next, the strand is cut depending on application—slabs for flat products (plate and strip), blooms for sections (beams), billets for long products (wires), or thin strips.
- ❑ In primary forming, the steel that is cast is then formed into various shapes, often by hot rolling, a process that eliminates cast defects and achieves the required shape and surface quality.
- ❑ Hot rolled products are divided into flat products, long products, seamless tubes, and specialty products.

The Modern Steel Production Process

Finally, it's time for manufacturing, fabrication, and finishing. Secondary forming techniques give the steel its final shape and properties.

These techniques include:

- Shaping (cold rolling), which is done below the metal's recrystallization point, meaning mechanical stress - not heat - affects change.
- Machining (drilling)
- Joining (welding)
- Coating (galvanizing)
- Heat treatment (tempering)
- Surface treatment (carburizing)

FURNACE

- An **industrial furnace (a direct heater / direct fired heater)** is a device used to provide heat for an industrial process, above 400°C.
- They are used to provide heat for a process or can serve as reactor which provides heats of reaction.
- Furnace designs vary as to its function, heating duty, type of fuel and method of introducing combustion air.
- Heat is generated by an industrial furnace by mixing fuel with air or oxygen, or from electrical energy.
- The residual heat will exit the furnace as flue gas.
- These are designed as per international codes and standards.
- Types of industrial furnaces include batch ovens, vacuum furnaces, and solar furnaces. Industrial furnaces are used in applications such as chemical reactions, cremation, oil refining, and glasswork.

FLUX

- ❑ In metallurgy, a **flux** is a chemical cleaning agent, flowing agent, or purifying agent added to molten metals to bond with impurities (gangue) that can be readily removed.
- ❑ Fluxes may have more than one function at a time.
- ❑ They are used in both extractive metallurgy and metal joining.
- ❑ Examples: sodium carbonate, potash, charcoal, coke, borax, lime, lead sulfide and certain minerals containing phosphorus.
- ❑ Iron ore was also used as a flux in the smelting of copper.
- ❑ These agents served various functions, the simplest being a reducing agent, which prevented oxides from forming on the surface of the molten metal, while others absorbed impurities into the slag, which could be scraped off the molten metal.
- ❑ Fluxes are also used in foundries for removing impurities from molten nonferrous metals or for adding desirable trace elements.
- ❑ As cleaning agents, fluxes facilitate soldering, brazing, and welding by removing oxidation from the metals to be joined.
- ❑ In some applications molten flux also serves as a heat-transfer medium, facilitating heating of the joint by the soldering tool or molten solder.

SLAG

- ❑ Slag is a by-product formed in smelting, welding, and other metallurgical and combustion processes from impurities in the metals or ores being treated.
- ❑ Slag is an impure residue with large amount mixed oxides of calcium, magnesium silicate, iron, aluminum, etc. derived during the process of pig iron and steel production and during the smelting of metals
- ❑ ash and products formed in their reactions with furnace linings and fluxing substances such as limestone.
- ❑ Slag floats on the surface of the molten metal, protecting it from oxidation by the atmosphere and keeping it clean.
- ❑ Slag forms a coarse aggregate used in certain concretes; it is used as a road material and ballast and as a source of available phosphate fertilizer.
- ❑ Molten ash formed upon combustion of coal in some high-capacity boiler furnaces is also sometimes termed slag.

Steel is basically up of an alloy of carbon and iron, where the presence of carbon may vary from 0.25% to 1.5% and hence steel is primarily classified as low carbon steel (around 0.25% carbon), medium carbon steel (between 0.25% to 0.75% carbon) and high carbon steel (0.75% to 1.5% carbon).

Steel may be manufactured through the following principle methods:

Cementation Process

Crucible Process

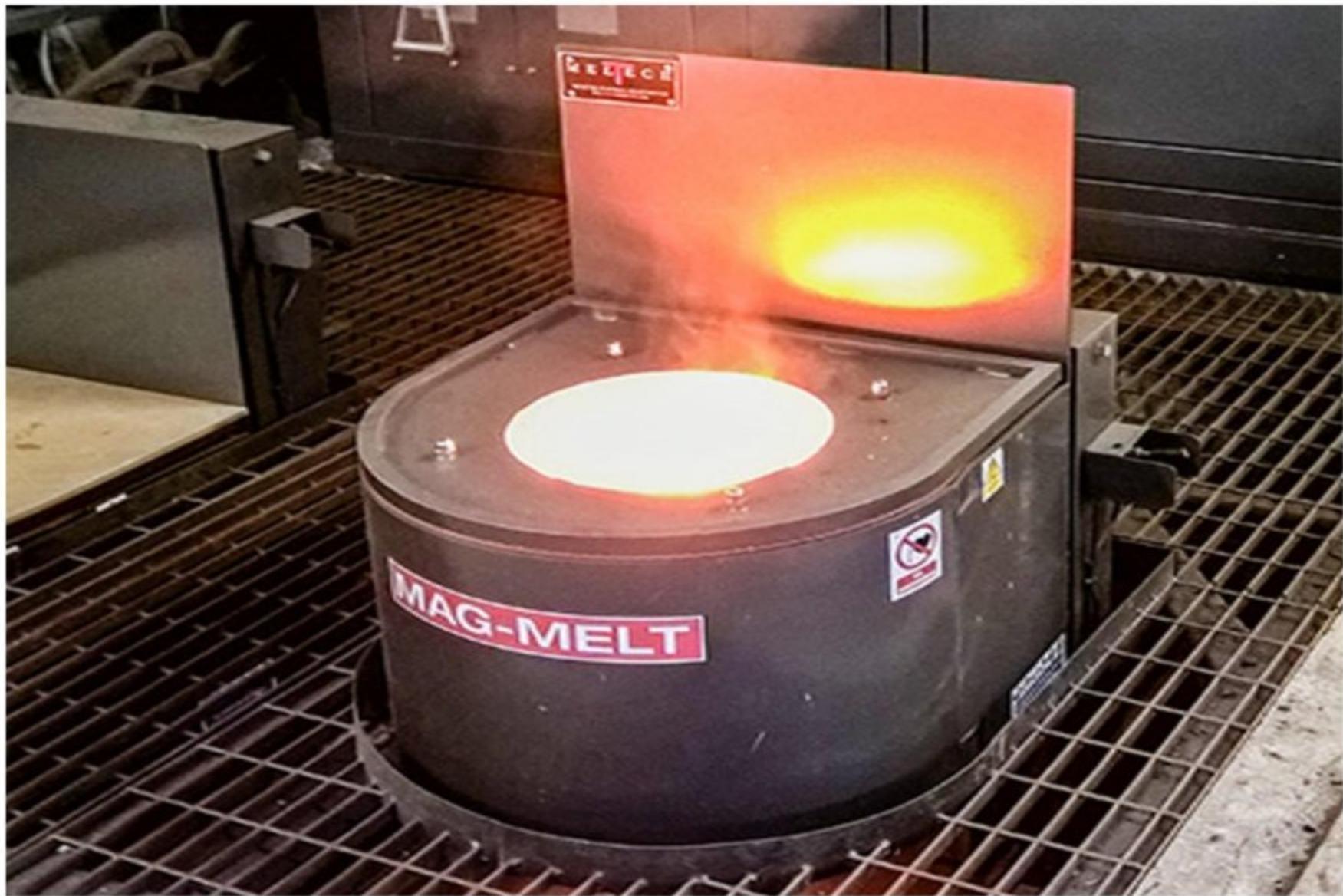
Bessemer Process

Open-hearth Process

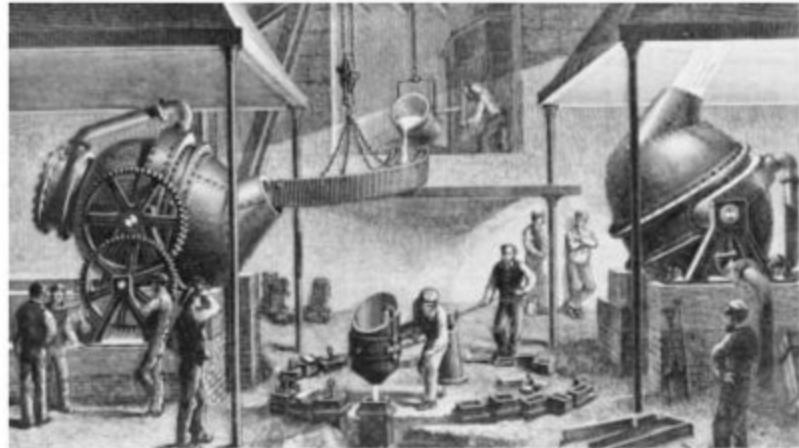
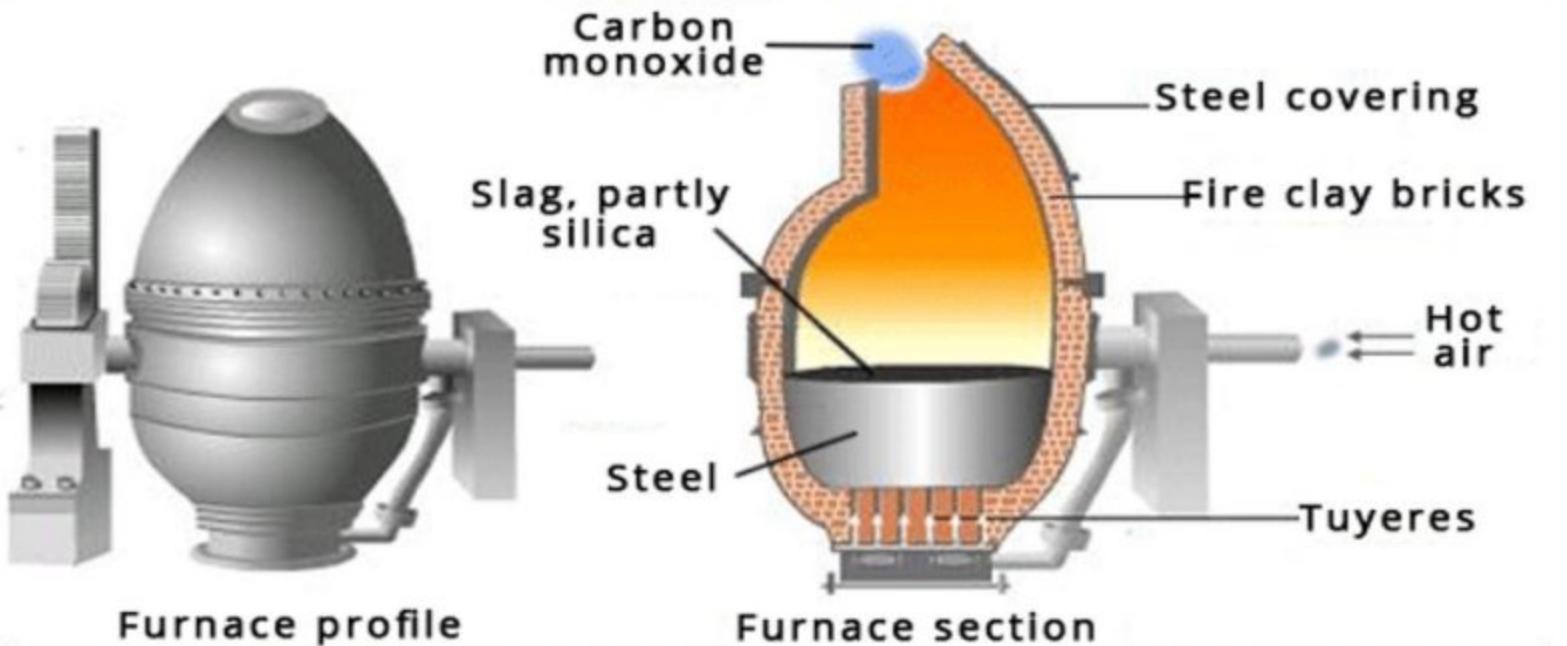
Electric Process

Cementation Process: In this process wrought iron bars are introduced in a furnace in between powdered charcoal layers and are subjected to a very high temperature – about 7000 Celsius for about a week to fortnight depending upon the required quality of the steel. The conditions slowly diffuse carbon into iron and cause the carbon to become dissolved in the iron, raising the carbon percentage. Steel obtained from this process is called “blister steel” due to the blister-like marks formed on the surface due to the evolved gases during the manufacturing process. The carbon amount here is usually around 0.75% to 1.5%.

CRUCIBLE FURNACE



Bessemer Process



Crucible Process: The process involves heating of blister steel fragments or short lengths of wrought iron bars mixed with charcoal inside fire clay crucibles. The resulting molten steel is allowed to run through iron moulds. "Cast iron / Cast steel" formed is extremely hard and perfectly homogenous and specifically used for cutting tools & finest cutlery items.

Bessemer Process: Pig iron is melted in a cupola and poured into Bessemer converter which is pear shaped and has a steel shell lined with refractory material. It's pivoted on trunnions so as to facilitate tilting, pouring or charging.

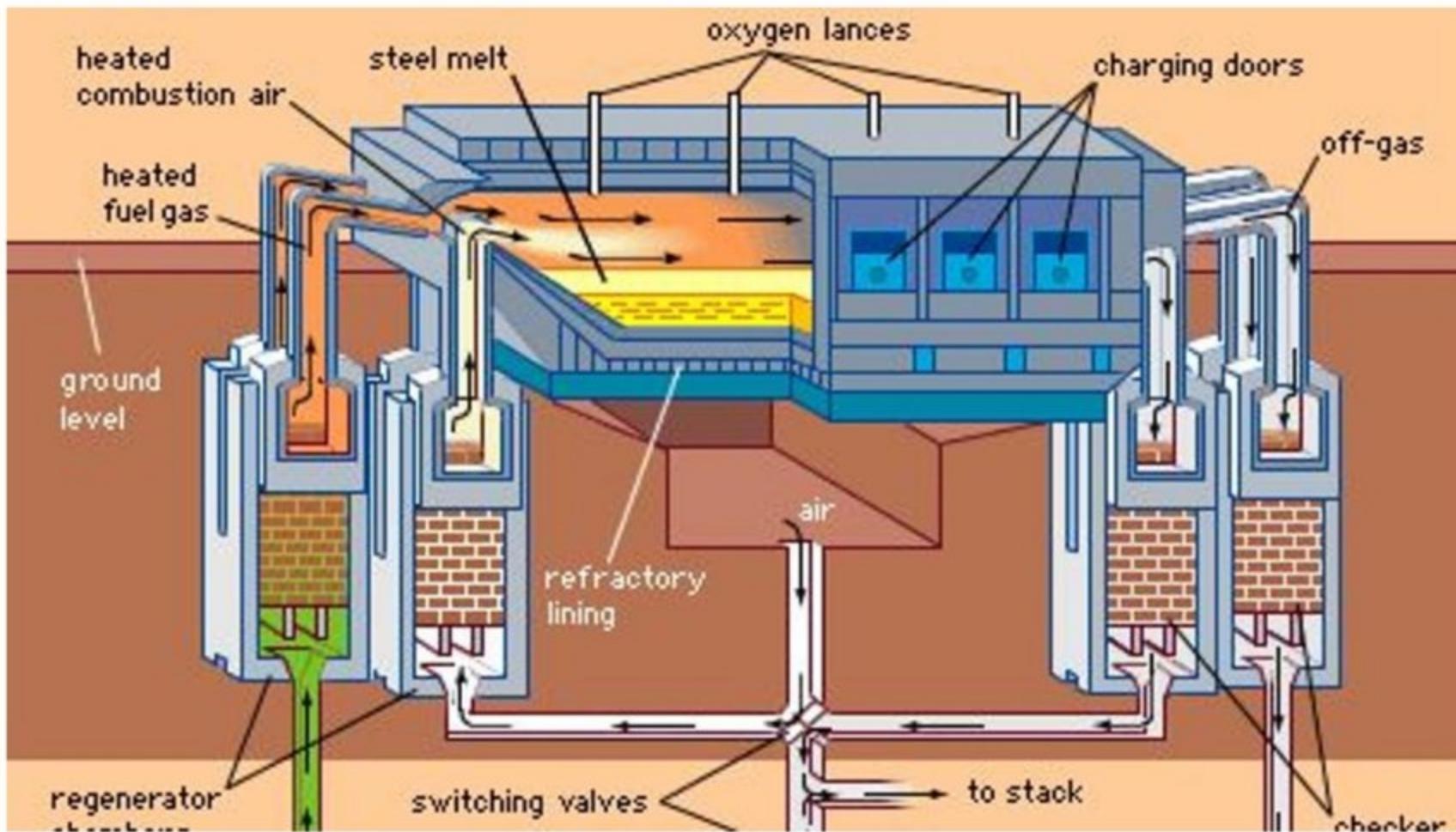
Once the above converter is charged with molten pig iron, a strong thrust of air is blasted across the molten mass for about 20 minutes through nozzles provided at the bottom of the vessel. All traces of carbon and silicon present are oxidized, leaving the converter with pure iron.

After this the blasting of air is stopped and the specified amount of ferro-manganese is added to it for including the recommended content of carbon and manganese to the steel.

The air blasted procedure is again initiated for some time, ensuring perfect mixing of the alloy.

The converter is then tilted so that the molten material can be discharged into the ladles. In the final step the molten alloy is shifted into rectangular moulds where it's obtained in the form of solid ingots.

The Open-Hearth Furnace (OHF)



Open Hearth Process: The specialty of open-hearth furnaces is the extreme heat that can be obtained from them due to their regenerative process.

The charge of pig iron, steel scrap, iron ore, and flux are together kept in a shallow container with a flame burning above it.

The process is initiated inside reverbaratory gas-fired regenerative furnaces for greater efficiency.

Regenerators are placed below the furnace and positioned in two pairs. The pairs are heated alternately through the passage of hot gases given out from the furnace in their route to the chimney. This heat is retained by the regenerators and is reversed and given back to the furnace.

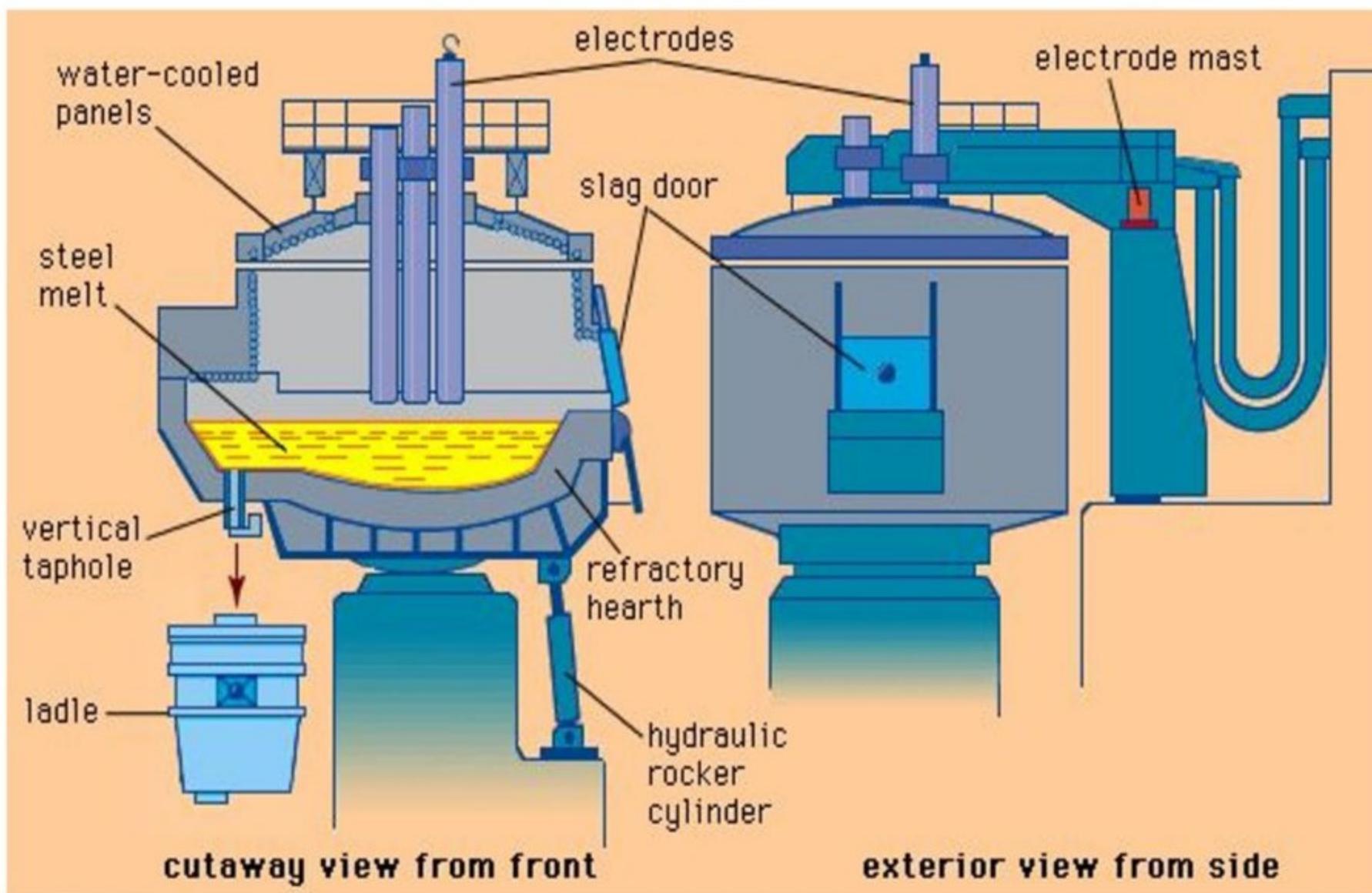
This heat exchange procedure helps the furnace to maintain high temperatures even with less fuels.

Once the furnace is charged with pig iron, pure oxidizing ores like haematite are added to it from time to time, which helps oxidation and the removal of impurities like silicon, carbon, and manganese in the pig iron.

Spiegel is also introduced when the carbon content becomes less than 0.1%, and ferro-manganese after the metal is tapped out into the ladle.

Ferro-manganese becomes important for restoring malleability and also for carburizing the iron.

ELECTRIC ARC FURNACE



Electric Process: In this process electric arc or electric high frequency furnaces are used.

In **electric arc furnaces**, high voltage electric arc struck between carbon electrodes and the charge becomes the source of a very high temperature.

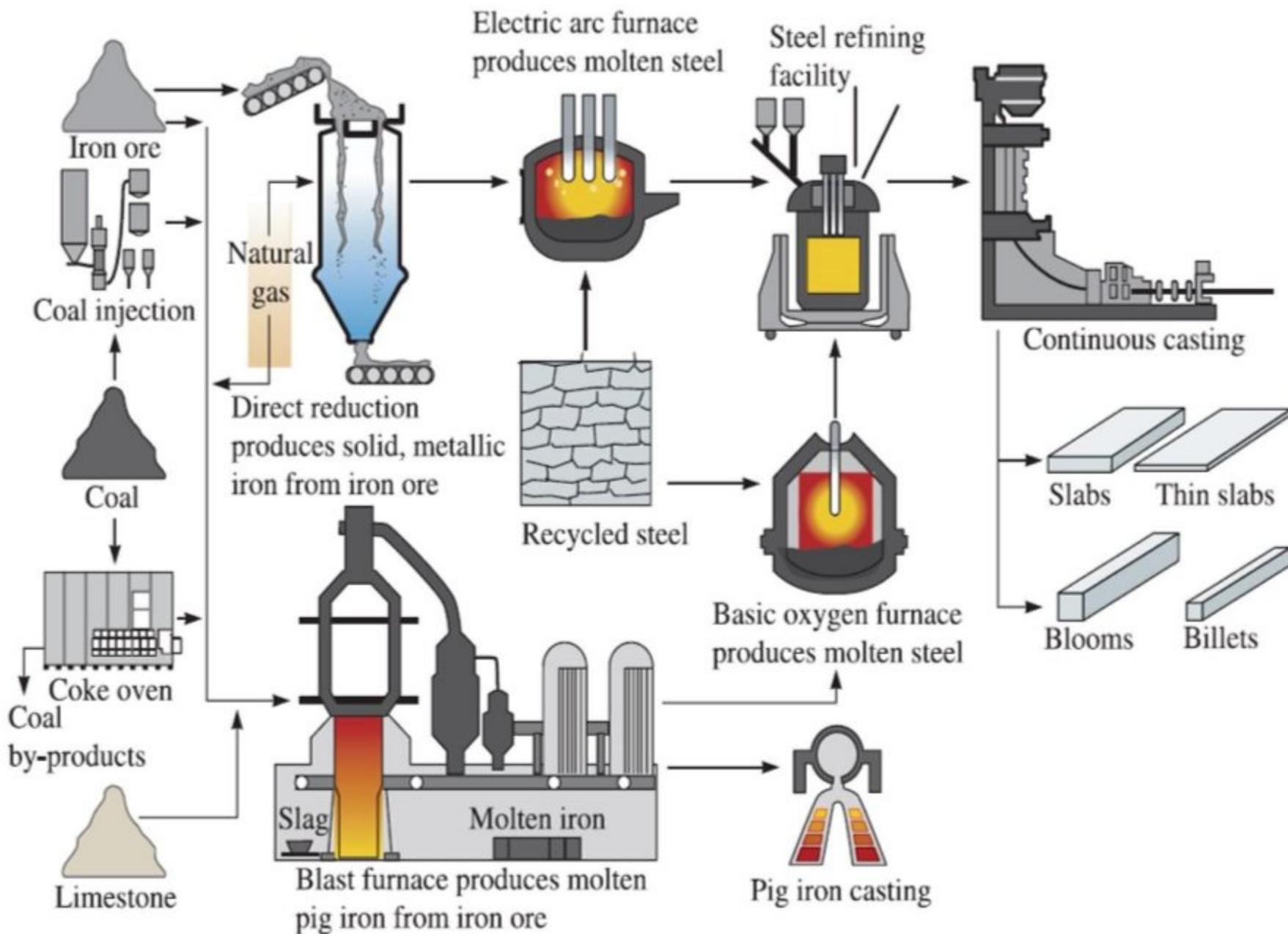
The charge is collected directly from an open hearth furnace, the intense arc heat keeps the charge in its molten state, and the impurities are removed in the form of slag.

The high frequency furnace is based on the principle that when high frequency alternating current is applied to steel, eddy currents starts flowing in them.

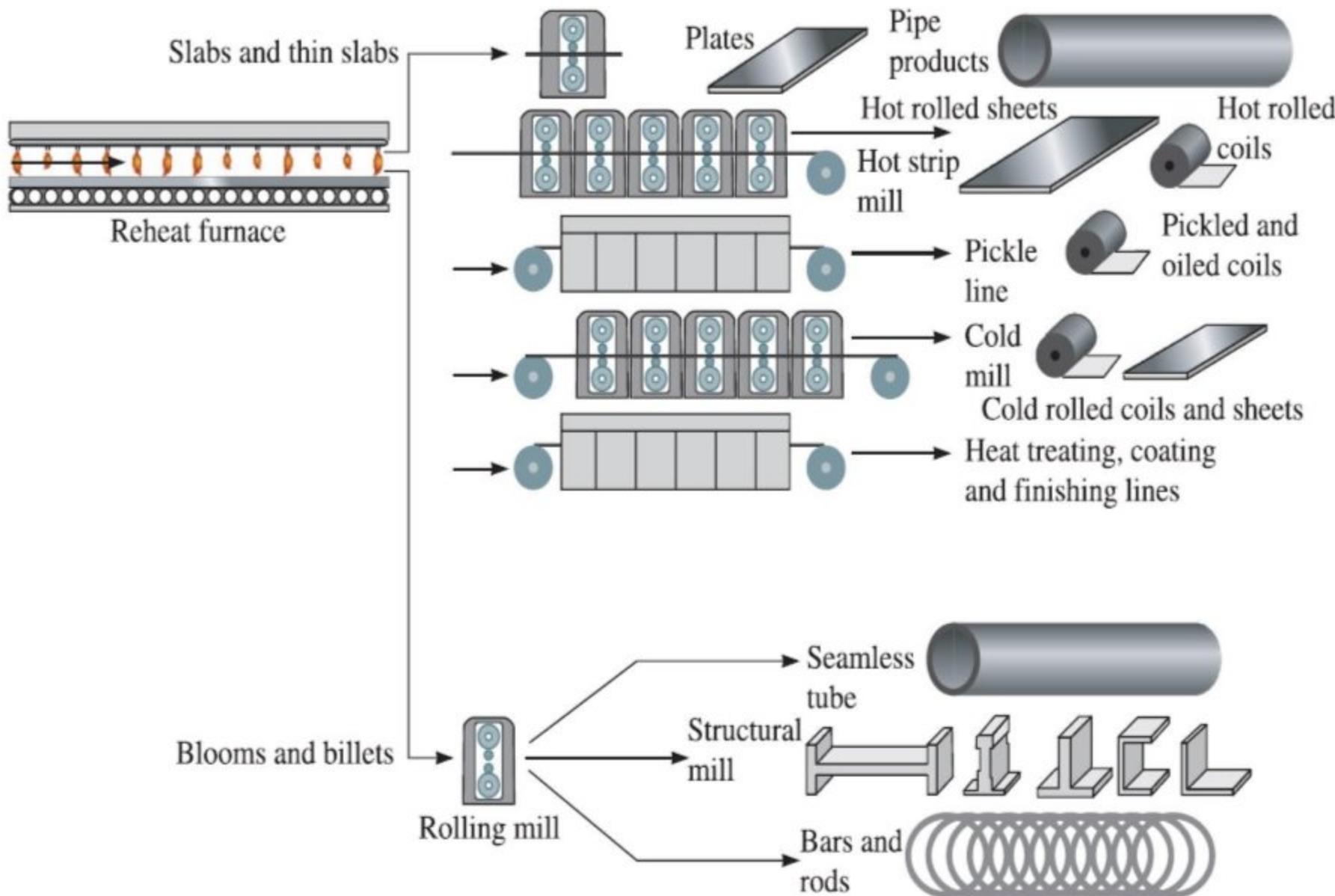
If this induction is made very strong, it can heat up the steel and melt it.

Electric furnaces are more advantageous compared to the other steel manufacturing processes due to the absence of evolving gases, fumes, etc., which normally become a major problem with fuel operated furnaces.

Extraction of steels using iron ores, coke & limestone



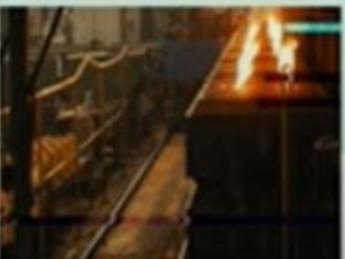
Secondary Processing Steps for Steels & Alloys



METAL FABRICATION PROCESSES

Manufacturing process.

- Manufacturing process:-Shape metal, machine them & join them to produce different components parts.

| casting | forming | Machining | Joining | S/T Finishing |
|---|--|--|---|---|
| <ul style="list-style-type: none">Sand castingShell moldPlaster moldInvestmentPermanentdie | <ul style="list-style-type: none">RollingForgingDrawingExtrusionSheet metal. | <ul style="list-style-type: none">Facing.Turning.Milling.GrindingBoringTappingDrillingReaming | <ul style="list-style-type: none">WeldingBrazingSolderingPressingAdhesive bondingRiveting. | <ul style="list-style-type: none">GrindingPolishingHoningLappingbuffing |
|  |  |  |  |  |

METAL FABRICATION PROCESSES

FORGING

Forging is a common practice for intricate metalwork. The process involves using forced compression to achieve the desired shape.

Done by hand, forging involves heating metal to the point that it can be bent, shaped, and formed with the use of a hammer or die strikes.

One of the oldest forms of metal fabrication processes in existence, forging is still widely used by blacksmiths and fabricators for detailed and artistic work to this day.

CASTING

Whereas forging metal is something that's done by hand, casting is the process of pouring molten metal into a mold. From there, it's cooled to the point that it becomes hard.

Casting is widely accepted as the go-to process for mass-produced products where the same mold can be used over and over again with minimal variance in the final outcome.

METAL FABRICATION PROCESSES

DRAWING

This fabrication process utilizes tensile force to pull metal into a tapered die. This stretches the metal into a thinner shape.

Drawing is most commonly used in sheet metal fabrication processes where the final product is a box-like or cylindrical vessel.

FORMING

Just as the name would suggest, **forming** is the process of bending metal to the desired angle. This is most often accomplished with a brake press that applies pressure to metal so that a crease is formed.

Other forms of folding can be done with the use of a hammer with clamp bars that hold the metal in place.

METAL FABRICATION PROCESSES

MACHINING

Machining is a subtractive shaping process that removes unwanted material to create a form.

Within this substrate, there are several different forms including drilling, turning, and milling.

Turning uses a lathe to rotate metal at a speed that allows cutting tools to remove the metal in a circular fashion. A CNC machine is the most common tool used for turning and often involves precise measurements.

Just like it sounds, drilling uses a bit to protrude a hole in the metal. Simple enough.

Lastly, milling is very common among the metal fabrication processes by which multi-point cutting tools remove metal from the workpiece. CNC machines are also used for this process which normally functions as a final finish process to create a desired texture or appearance.

METAL FABRICATION PROCESSES

EXTRUSION

During extrusion, metal is forced between an opened or closed die. Here, the piece of metal is reduced to the cross-section of the die. As a result, a cavity is formed.

The end result is usually a cylindrical shape that's common in piping or wiring.

CUTTING

One of the most versatile metal fabrication processes is cutting. On the surface, the process is nothing more than cutting large pieces of metal into smaller ones.

Cutting is quite intricate and involved, however. Technology like lasers, waterjets, power scissors, and plasma are all utilized to create exacting cuts.

Depending on the project, cutting is often the first, if not only, process used in fabrication.

PUNCHING

This process creates holes in a workpiece. Punch presses have specially designed turrets that hit the metal through a die to produce holes in specific locations.

Punching is used in both light and heavy metalwork.

METAL FABRICATION PROCESSES

STAMPING

Akin to punching, stamping involves creating indentations in metal, instead of holes with the use of a press or handheld hammer.

The most common applications for stamping are creating letters, shapes, and images.

WELDING

Welding is the art of adjoining two or more pieces of metal through a mixture of heat and pressure. This process is most common in large scale or industrial fabrication projects.

It's popular because various pieces of metal can be conjoined no matter their shape or size.

Depending on the project, the most common types of welding include MIG, stick and arc, TIG, and flux-cored arc.

METAL FABRICATION PROCESSES

Shearing. For sheets of metal that require long cuts, the process is known as shearing. In some cases, the sheet is fed horizontally through a metal-cutting machine.

In other applications, a cutting tool is applied vertically against the length of a flat metal sheet. A third method involves placing the metal over the edge of an open cutter and lowering the blade, much like the paper cutters found at copy facilities.

Shearing is often applied to trim down the edge of a metal sheet, but the process may be done anywhere along the metal.

Folding. One of the more complicated processes involves folding, where a metal surface is manipulated to shape at a certain angle.

With certain folding applications, the intent is to make the metal surface fold at a 90-degree angle, or something else that's either more or less blunt. However, folding may only be performed in facilities that are equipped with specific, high-tech equipment due to the complexity of the whole process.

In many cases where a fold is needed, the joining of two metal panels at select angles would be the more practical alternative.

FORGING

Forging is the oldest metal working process.

Requires heating and hammering of metals.

Forging operations are:

Drawing down or swaging:

Upsetting:

Coining (closed-die forging):

Heading (open-die forging):

Punching:

Cogging:

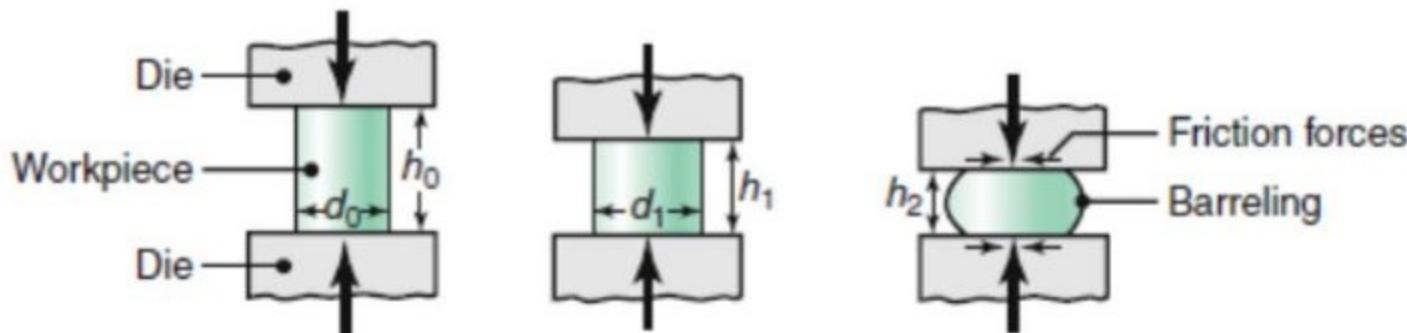
Fullering and Edging:

Roll Forging:

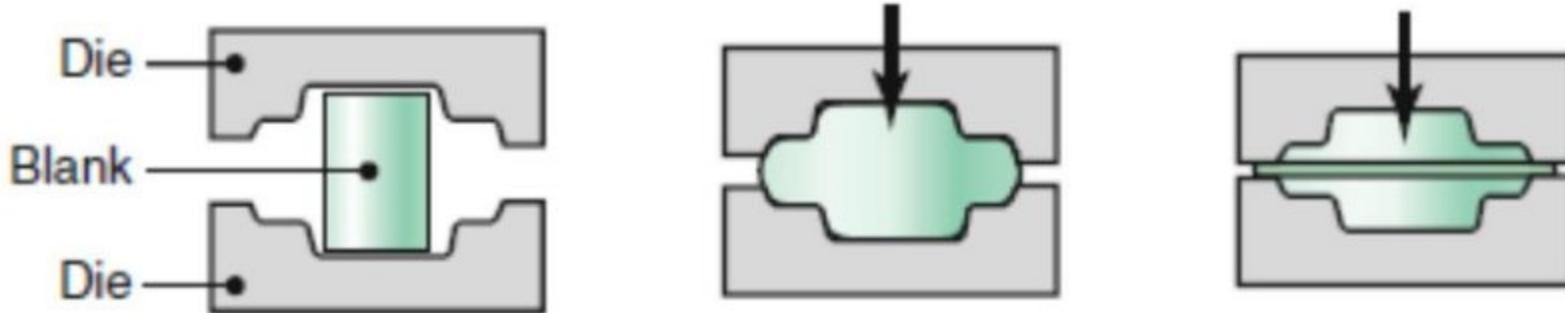
Skew Forging:

Classification of Forging

1. Open-die Forging (Hand or Power Forging)
2. Impression-die Forging
3. Closed-die Forging.

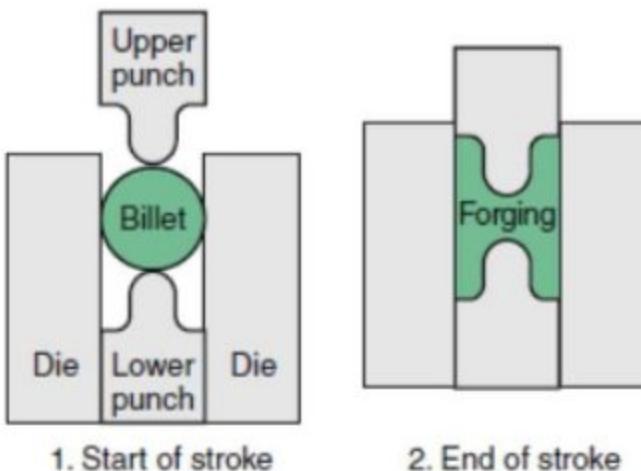


Open-die Forging



Impression-die Forging

Classification of Forging



Closed-die Forging

Applications of Forging:

Typical parts made by forging are crankshafts and connecting rods for engines, turbine disks, gears, wheels, bolt heads, hand tools, and many types of structural components for machinery and transportation equipment.

ROLLING

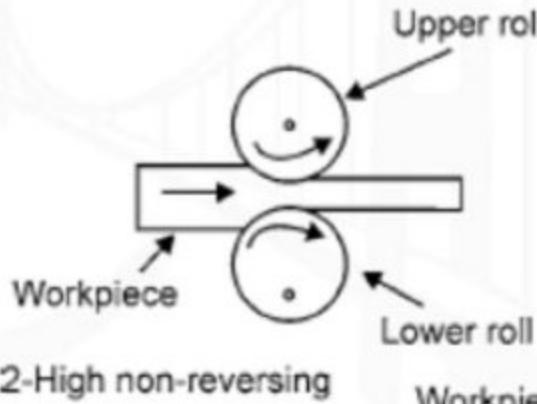
Reducing the thickness or changing the cross-section of a long workpiece by compressive forces applied through a set of rolls.

One effect of the hot working rolling operation is the grain refinement brought about by recrystallization

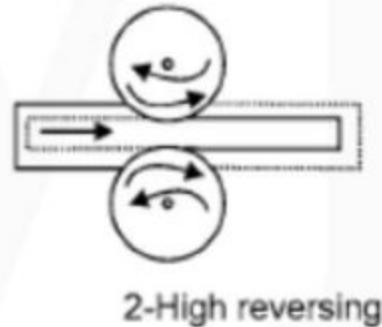
Coarse grain structure is broken up and elongated by the rolling action.

Recrystallization starts immediately and small grains begin to form and grow rapidly until recrystallization is complete.

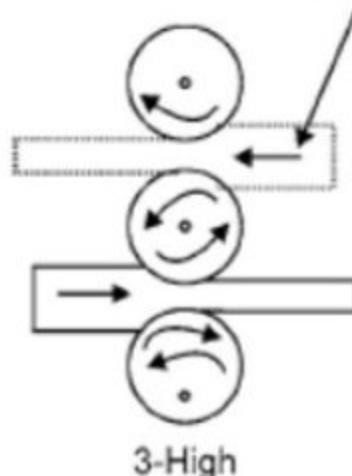
Hot rolling of metals (Two-high rolling mill, Three-high rolling mill) Cold rolling of metals (Four high rolling mill, Cluster rolling mill)



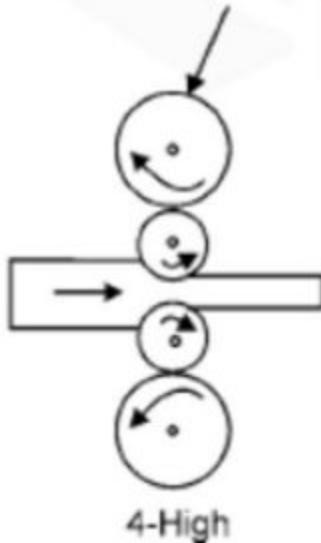
2-High non-reversing



2-High reversing

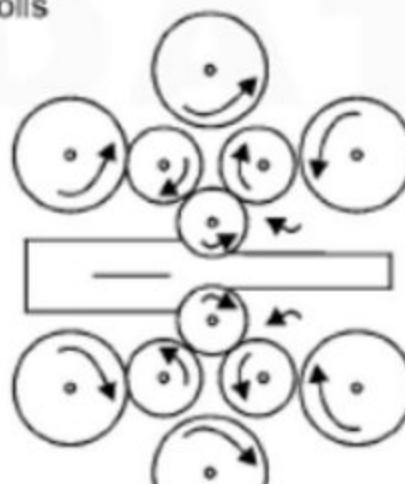


3-High



4-High

Backing rolls



Cluster

The roll pass sequences are:

- 1. Breakdown passes:** These are used for reducing the cross-sectional area nearer to what is desired. These would be the first to be present in the sequence.
- 2. Roughing passes:** In these passes also, the cross-section gets reduced, but along with it, the shape of the rolled material comes nearer to the final shape.
- 3. Finishing passes:** These are the final passes which give the required shape of pass follows a leader pass

Applications of Rolling:

To produce components having constant cross-section throughout its length.

- (a) **Structural shapes or sections:** This includes sections like round, square, hexagonal bars, channels, H and I beams and special sections like rail section.
- (b) **Plates and sheets:** These are produced of varying thickness.
- (c) **Special purpose rolled products:** These include rings, balls, wheels and ribbed tubes

Hot Rolling

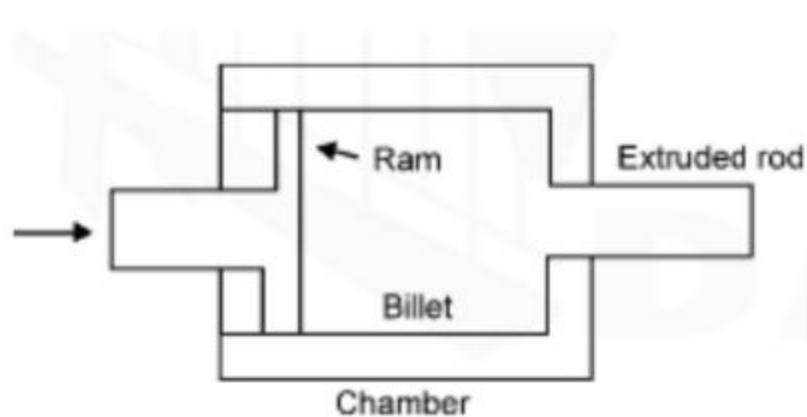
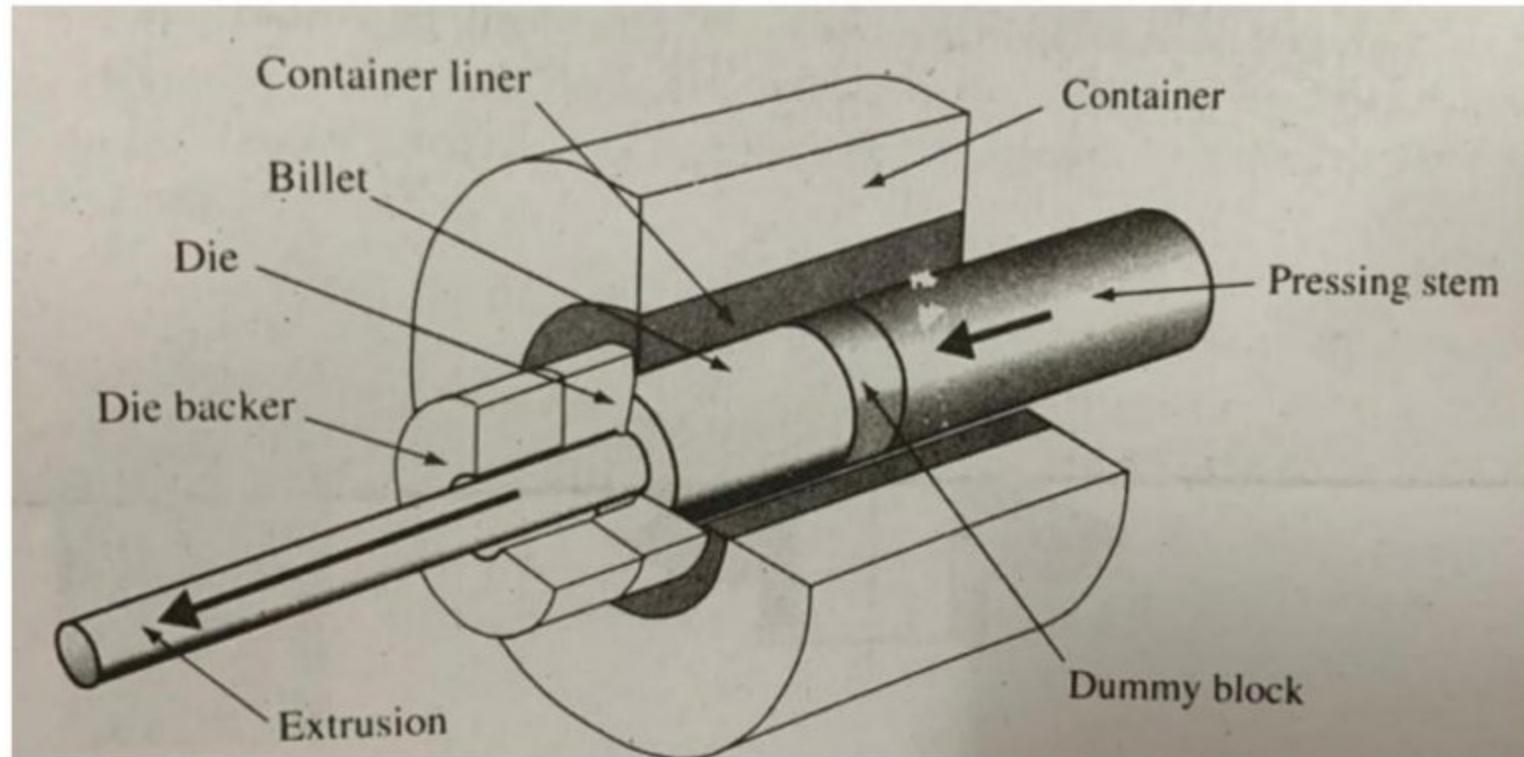
1. Metal is fed into rolls after being heated above recrystallization temperature.
2. Coefficient of friction between rolls and Metal is higher.
3. In Hot Rolling Process heavy reduction in cross-sectional area is possible.
4. In Hot Rolling, Close dimensional tolerances cannot be obtained.
5. Poor surface finish
6. Hot rolled metal does not show a work hardening effect.

Cold Rolling

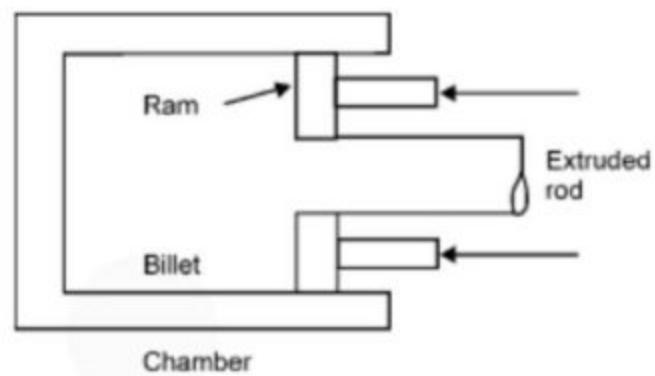
1. Metal is fed into rolls when its temp. is below recrystallization temperature.
2. Coefficient of friction between rolls and Metal is lower.
3. Heavy reduction in cross-sectional area is not possible.
4. In Cold Rolling, Section dimensions can be finished to close tolerances
5. The smooth and oxide-free surface can be obtained
6. Cold rolled metal hardening effect.

EXTRUSION

- Extrusion is the process that forces metal to flow through a shape-forming die.
- The metal is plastically deformed under compression in the die cavity.
- Extrusion produces only compressive and shear forces in the stock without any tensile force, which makes high deformation possible without tearing the metal.
- It is a hot-working process which, like forging, rolling, etc., uses the good deformability of heated metallic materials for shaping them.
- A metal billet heated to the appropriate temperature is fed into the cylindrical container of the extrusion press.
- Then it is forced by the action of a ram through a steel die whose orifice has the desired shape to produce the solid or hollow section.



Direct Extrusion



In-direct Extrusion

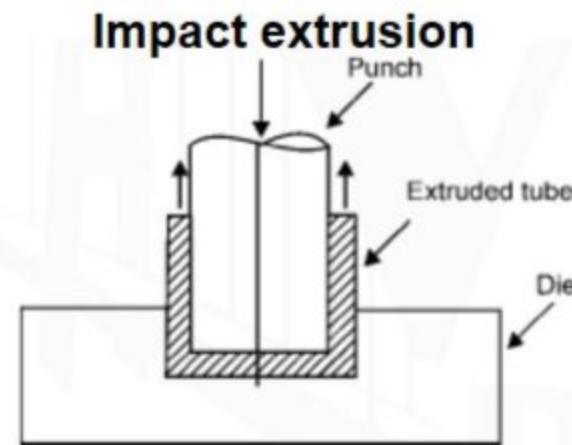
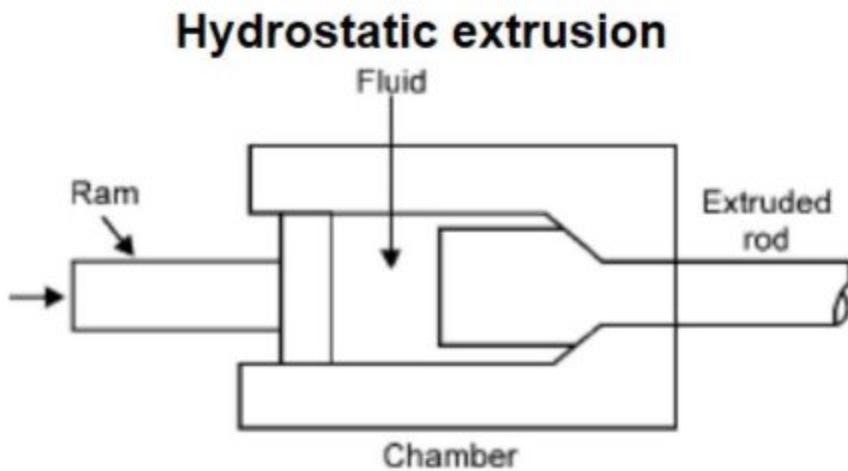
Direct (forward) extrusion

Indirect (reverse, inverted or backward) extrusion

Combined extrusion

Hydrostatic extrusion

Impact extrusion



SHEARING

Before a sheet-metal part is made, a blank of suitable dimensions first is removed from a large sheet (usually from a coil) by shearing.

CASTING

Casting is a fabrication process whereby a totally molten metal is poured into a mold cavity having the desired shape.

Upon solidification, the metal assumes the shape of the mold but experiences some shrinkage.

Casting techniques are employed when:

- (1) the finished shape is so large or complicated that any other method would be impractical,
- (2) a particular alloy is so low in ductility that forming by either hot or cold working would be difficult, and
- (3) in comparison to other fabrication processes, casting is the most economical.

Furthermore, the final step in the refining of even ductile metals may involve a casting process.

A number of different casting techniques are commonly employed, including sand, die, investment, lost foam, and continuous casting.

CASTING

Sand Casting

- A two-piece mold is formed by packing sand around a pattern that has the shape of the intended casting.
- A *gating system* is usually incorporated into the mold to expedite the flow of molten metal into the cavity and to minimize internal casting defects.
- Sand-cast parts include automotive cylinder blocks, fire hydrants, and large pipe fittings.

Die Casting

- The liquid metal is forced into a mold under pressure at high velocity and allowed to solidify with the pressure maintained.
- A two-piece permanent steel mold/die is clamped together to form the desired shape.
- The die pieces are opened, cast piece is ejected after the metal solidified completely.
- Rapid casting rates are possible and a single set of dies may be used for thousands of castings as an inexpensive method..

CASTING

Investment Casting

- For investment (sometimes called *lost-wax*) casting, the pattern is made from a wax or plastic that has a low melting temperature.
- Around the pattern is poured a fluid slurry, which sets up to form a solid mold or investment; Plaster of Paris is usually used.
- The mold is then heated, such that the pattern melts and is burned out, leaving behind a mold cavity having the desired shape.
- This technique is employed when high dimensional accuracy, reproduction of fine detail, and an excellent finish are required (jewelry and dental crowns and inlays).
- Blades for gas turbines and jet engine impellers are investment cast.

CASTING

Lost Foam Casting

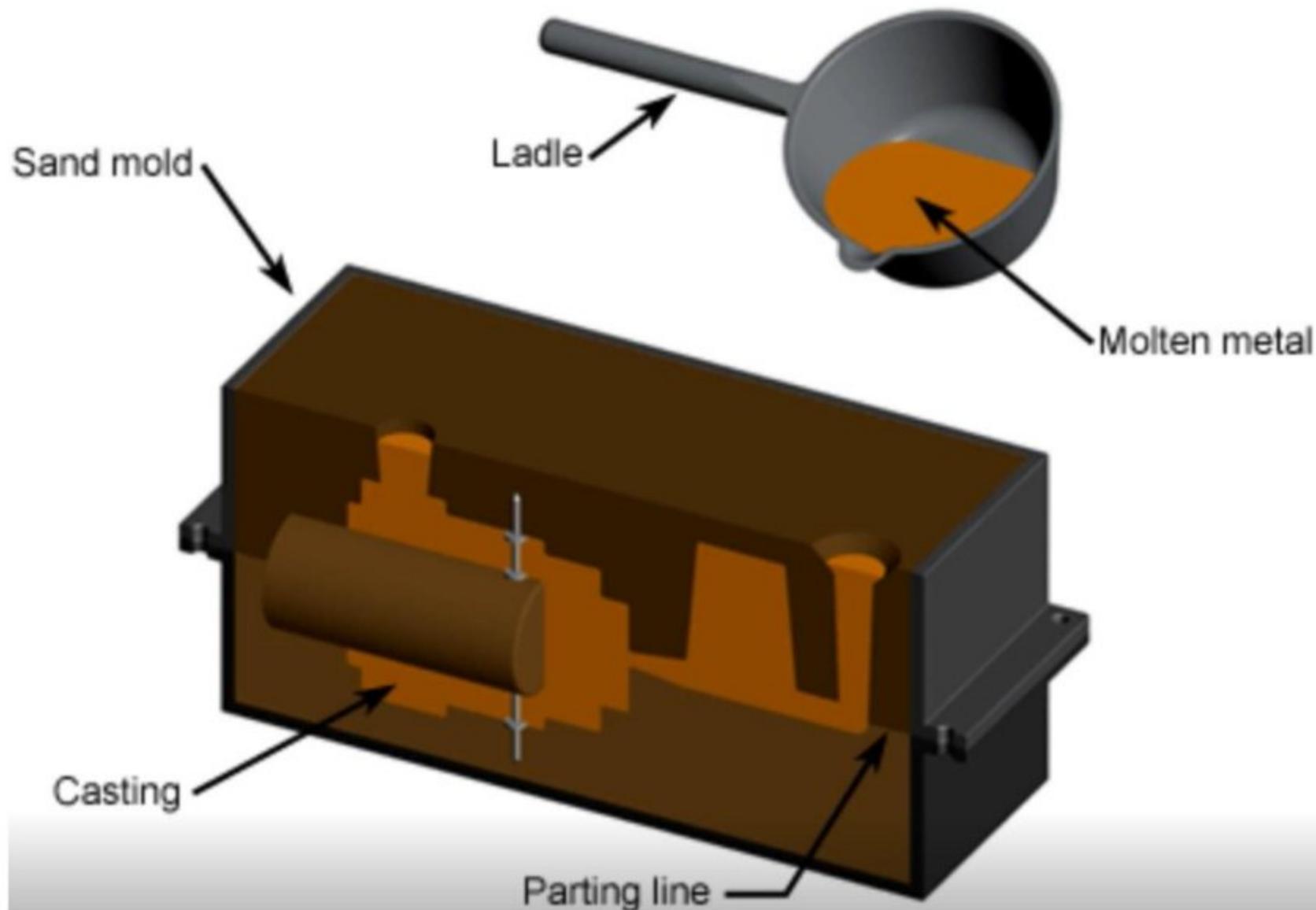
- An expendable pattern foam is formed by compressing polystyrene beads into the desired shape and then bonding them together by heating.
- Alternatively, pattern shapes can be cut from sheets and assembled with glue.
- Sand is then packed around the pattern to form the mold.
- As the molten metal is poured into the mold, it replaces the pattern, which vaporizes.
- The compacted sand remains in place, and, upon solidification, the metal assume the shape of the mold.
- With lost foam casting, complex geometries and tight tolerances are possible and is a simpler, quicker, and less expensive process.
- Metal alloys that most commonly use this technique are cast irons and aluminum alloys.
- Applications include automobile engine blocks, cylinder heads, crankshafts, marine engine blocks, and electric motor frames.

CASTING

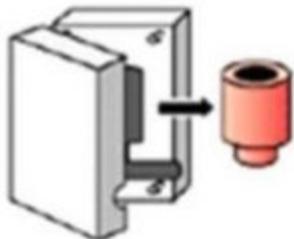
Continuous Casting

- The casting and rolling steps may be combined by a *continuous casting* (sometimes also termed *strand casting*) process.
- Using this technique, the refined and molten metal is cast directly into a continuous strand that may have either a rectangular or circular cross section
- Solidification occurs in a water-cooled die having the desired cross-sectional geometry.
- The chemical composition and mechanical properties are more uniform throughout the cross sections for continuous castings than for ingot-cast products.
- Furthermore, continuous casting is highly automated and more efficient.

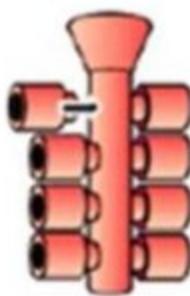
SAND CASTING



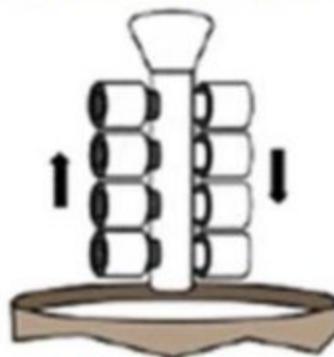
The Basic Steps in the Investment Casting Process



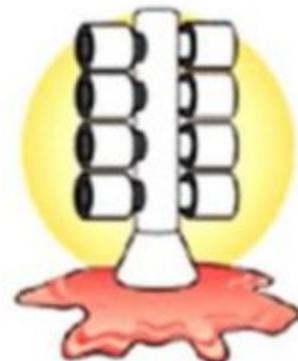
Wax Injection



Assembly



Shell Building



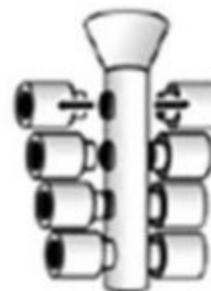
Dewax/Burnout



Gravity Pouring



Knock Out

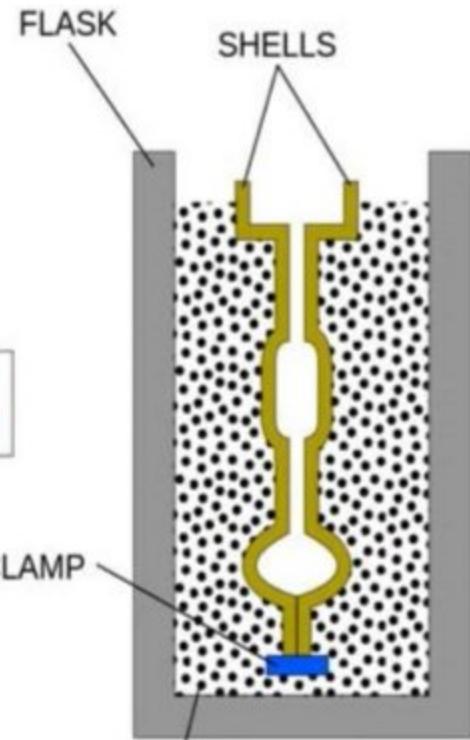
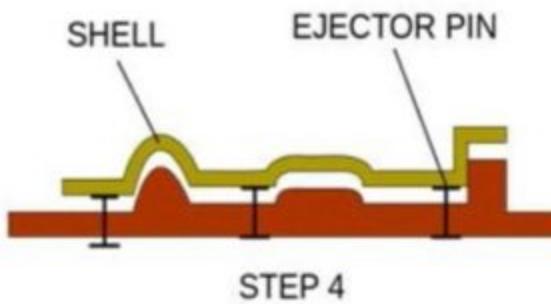
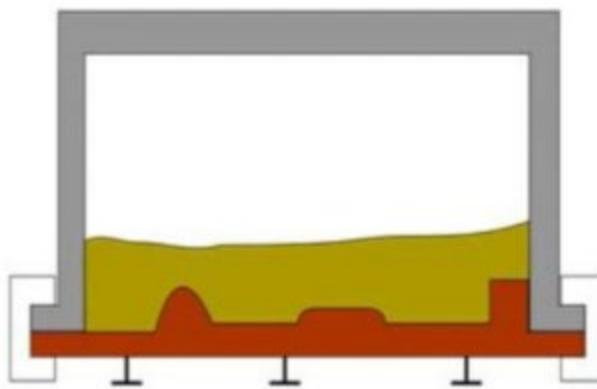
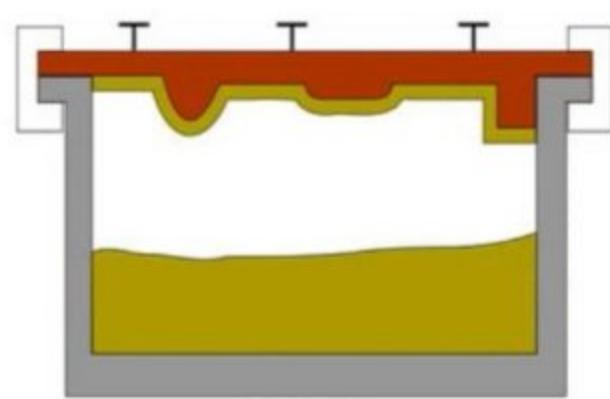
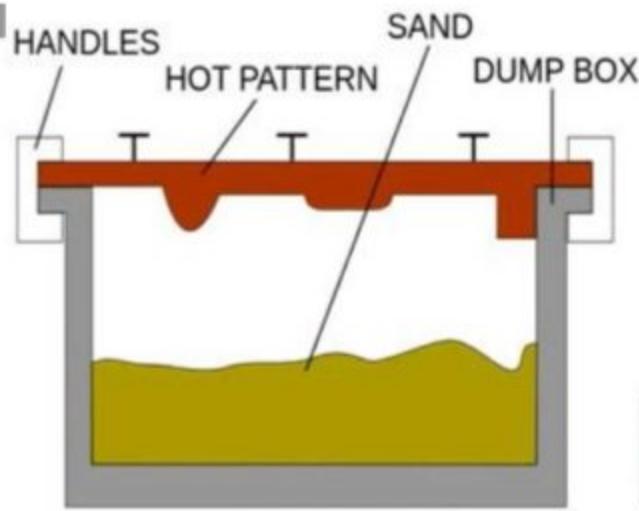


Cut-off



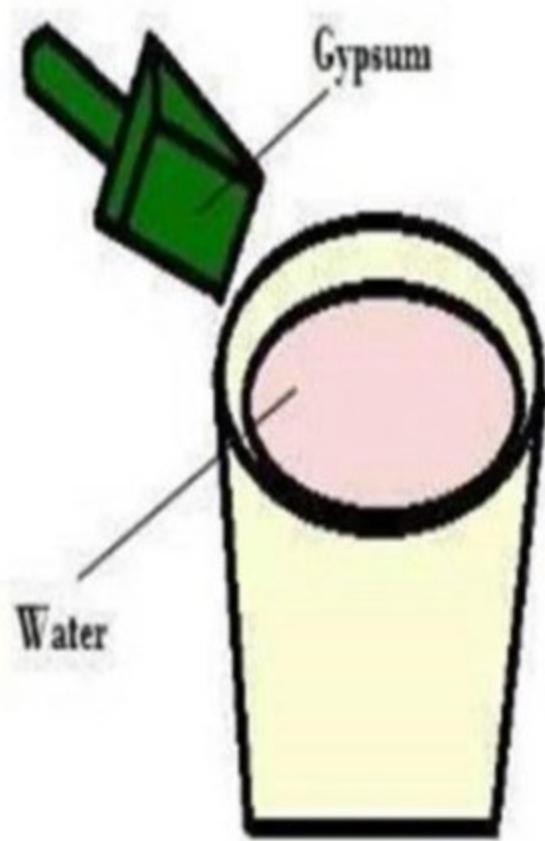
Finished Castings

Investment Casting

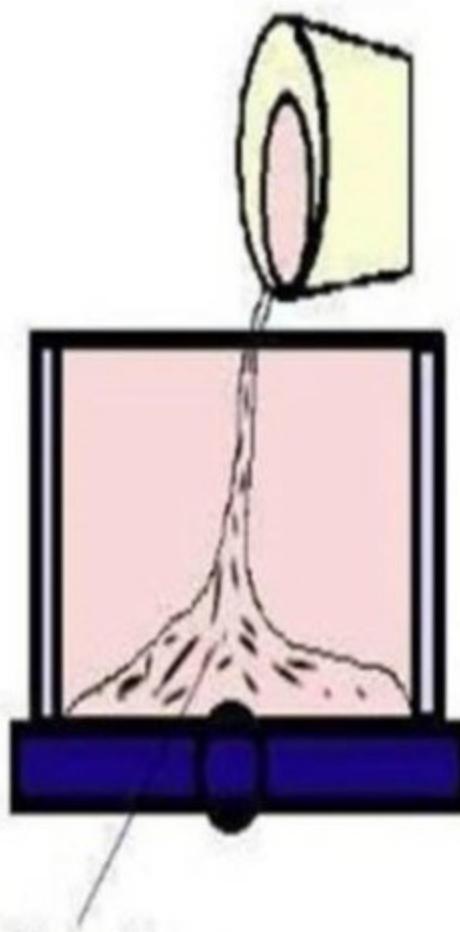


Shell Casting

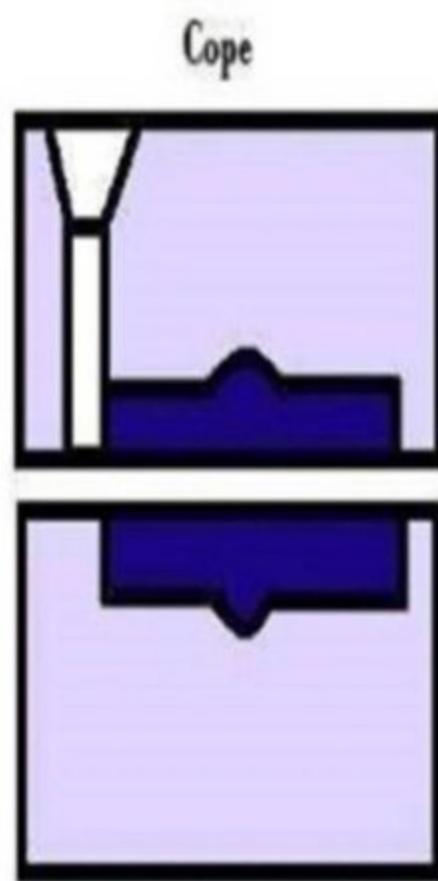
PLASTER MOULD CASTING



Making of Plaster Slurry

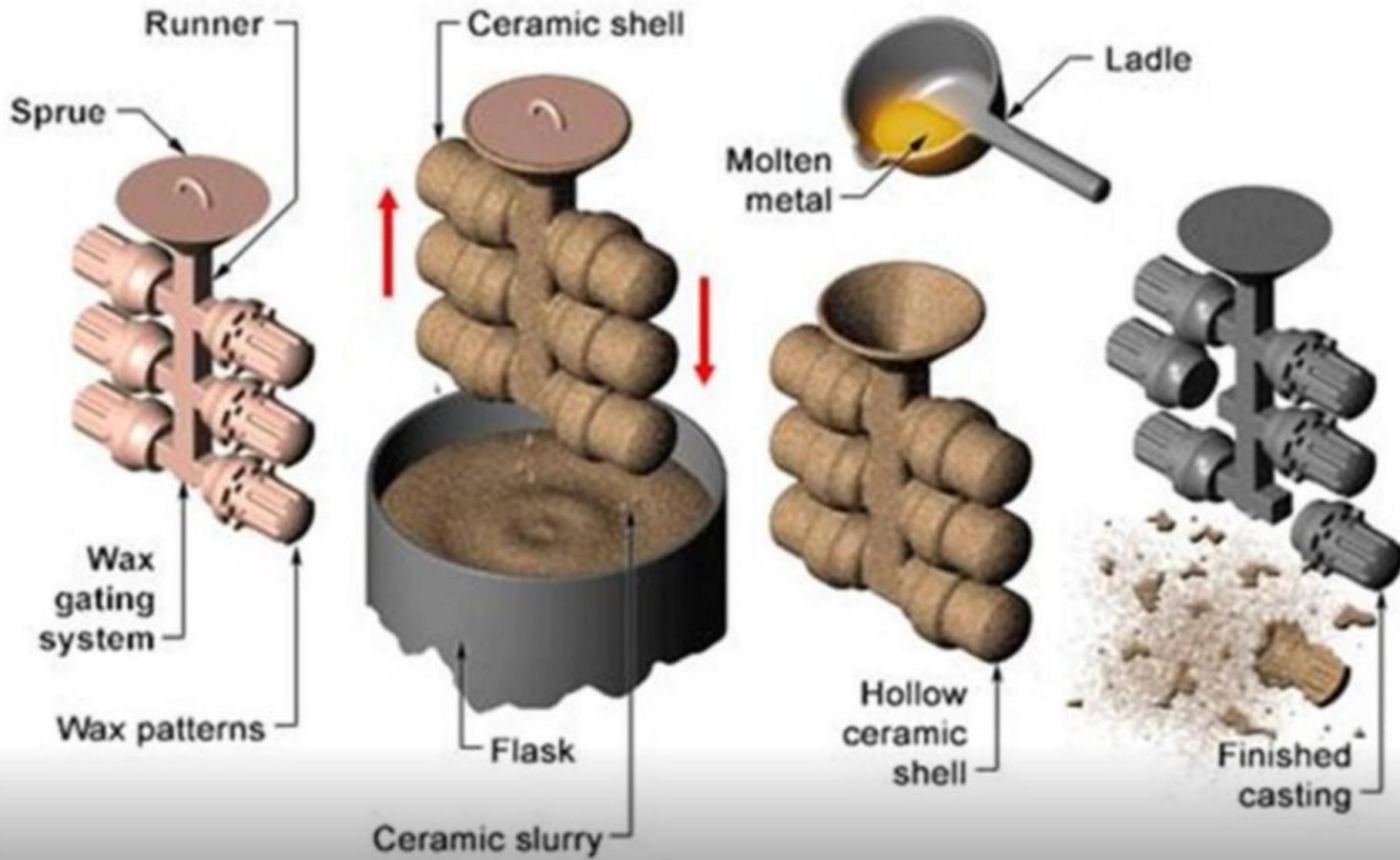


Ceramic Mould Making

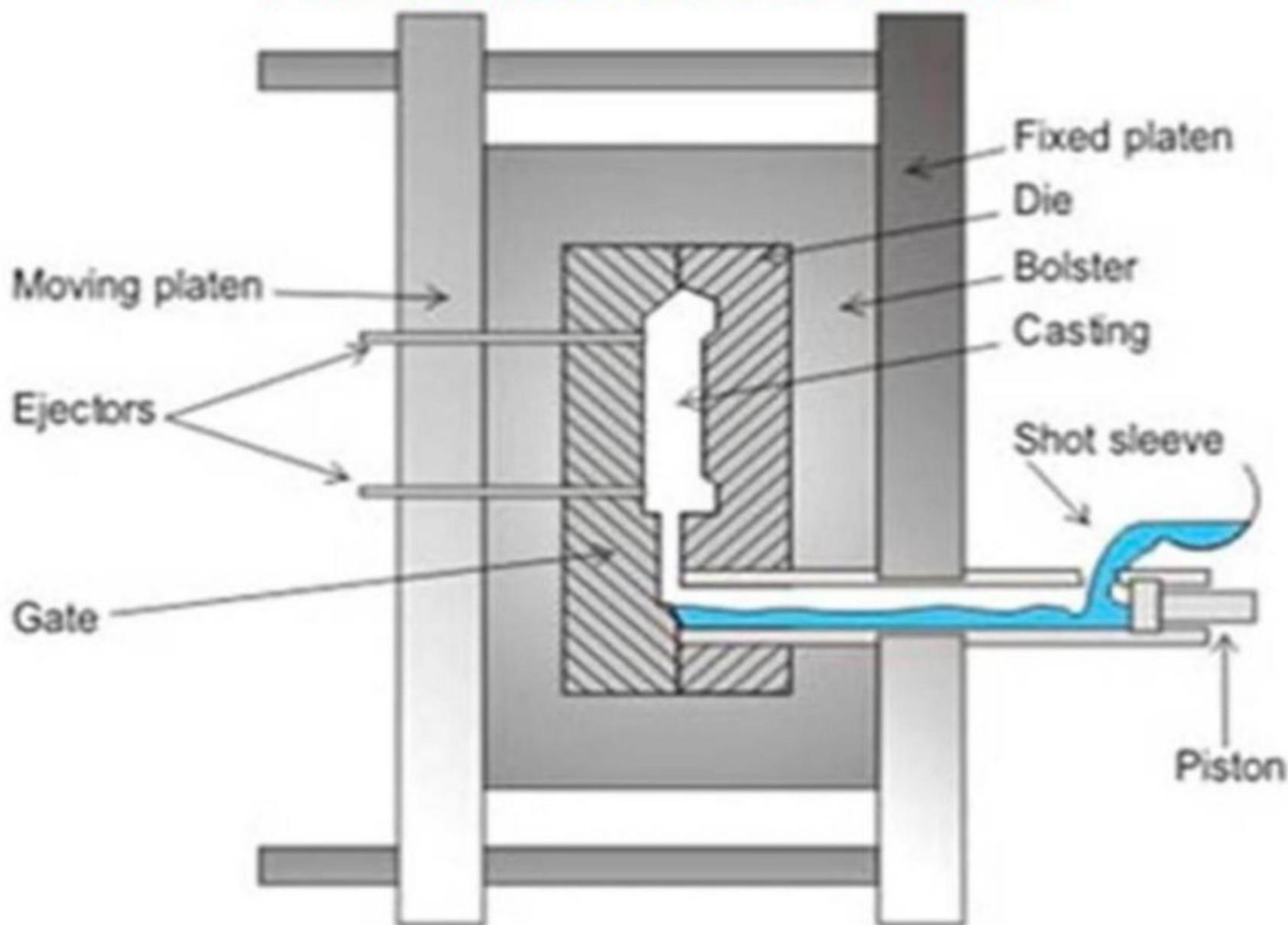


Final Part Production

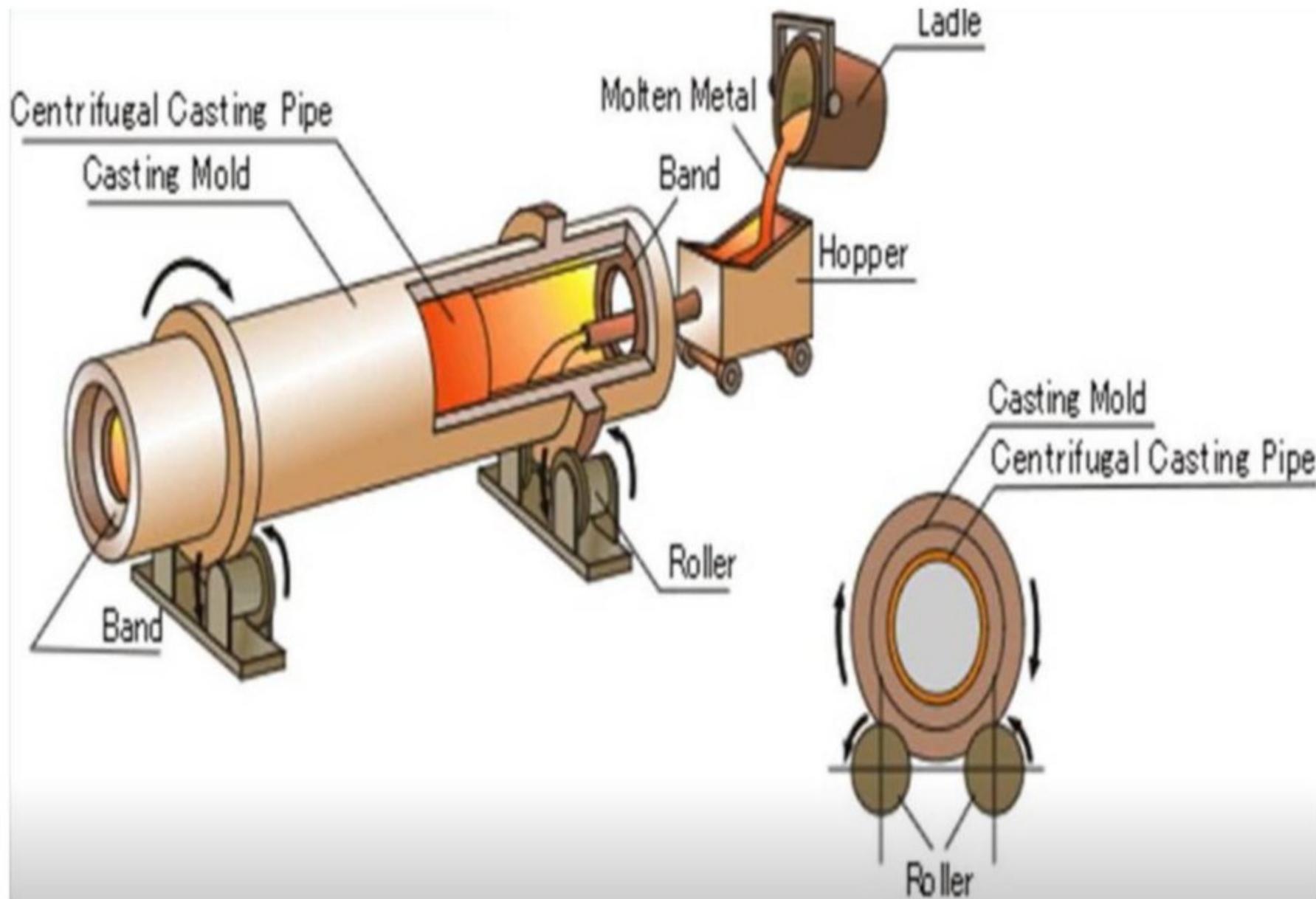
CERAMIC MOULD CASTING



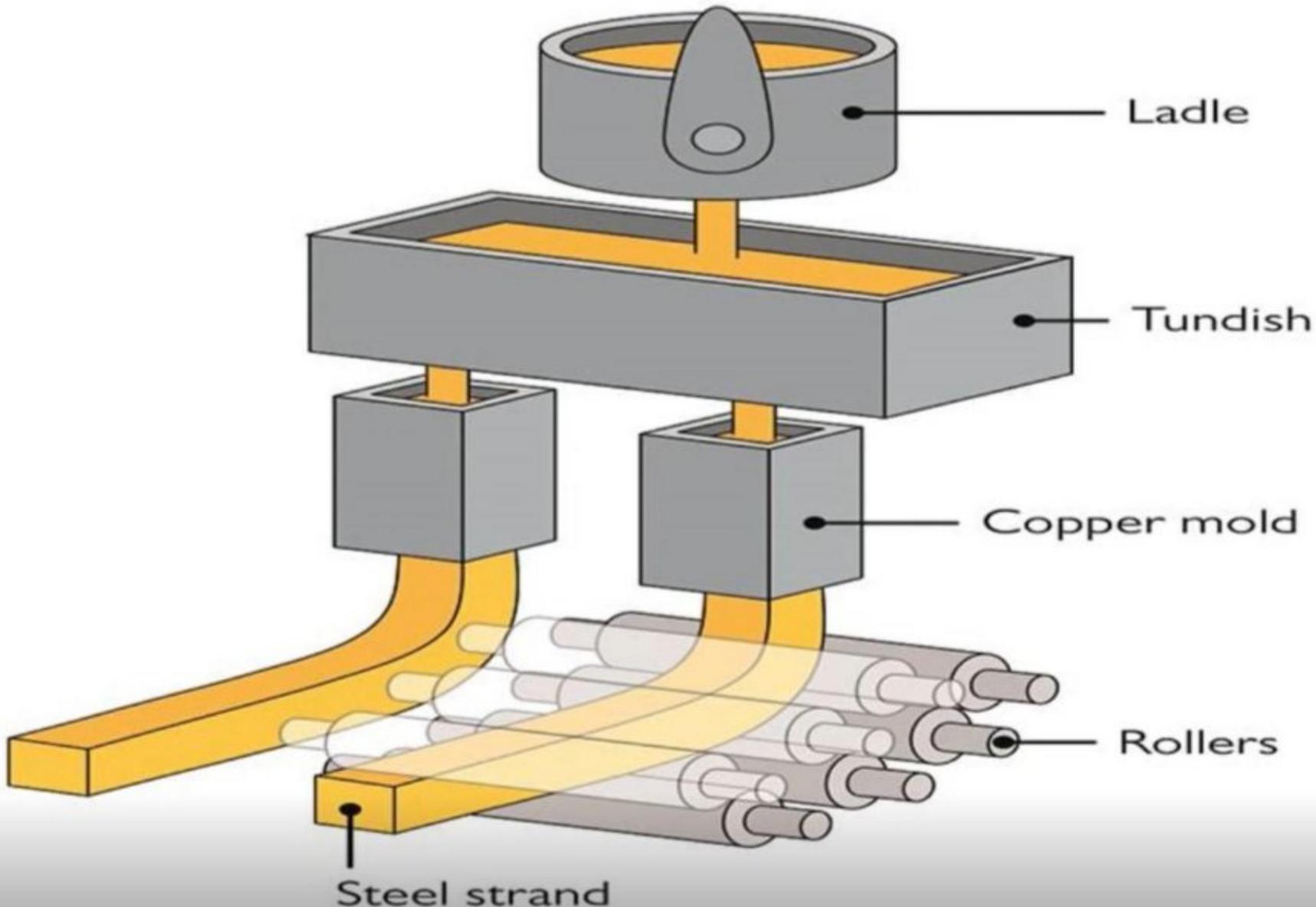
PRESSURE DIE CASTING



CENTRIFUGAL CASTING



CONTINUOUS CASTING



LOST FOAM CASTING



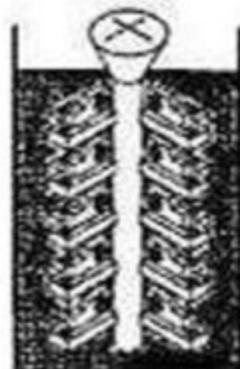
PATTERN
MOLDING



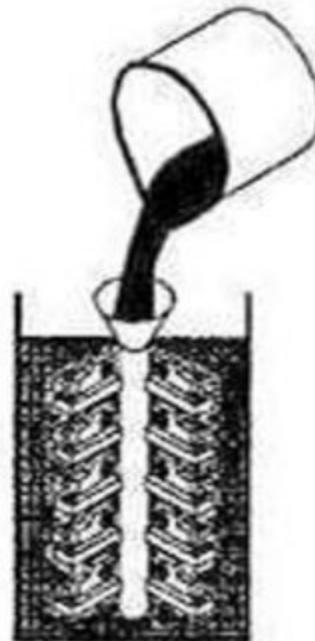
CLUSTER
ASSEMBLY



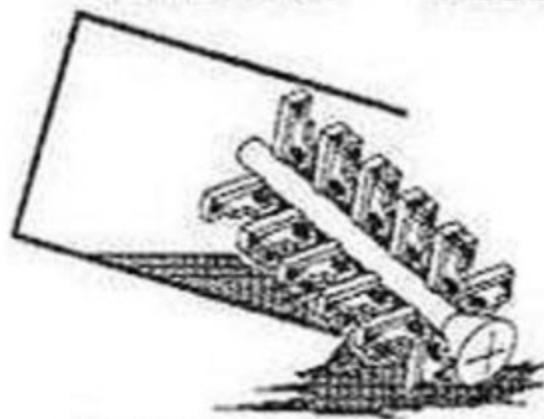
COATING



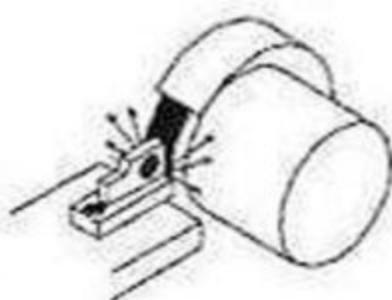
INVESTMENT
IN SAND



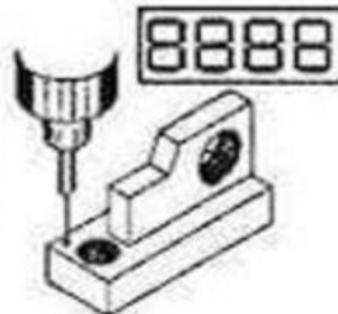
CASTING



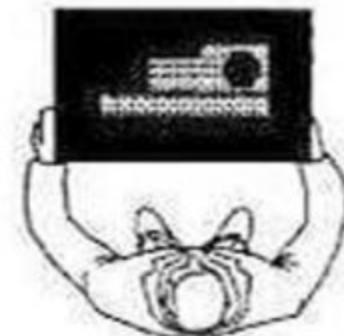
SHAKE-OUT



FINISHING



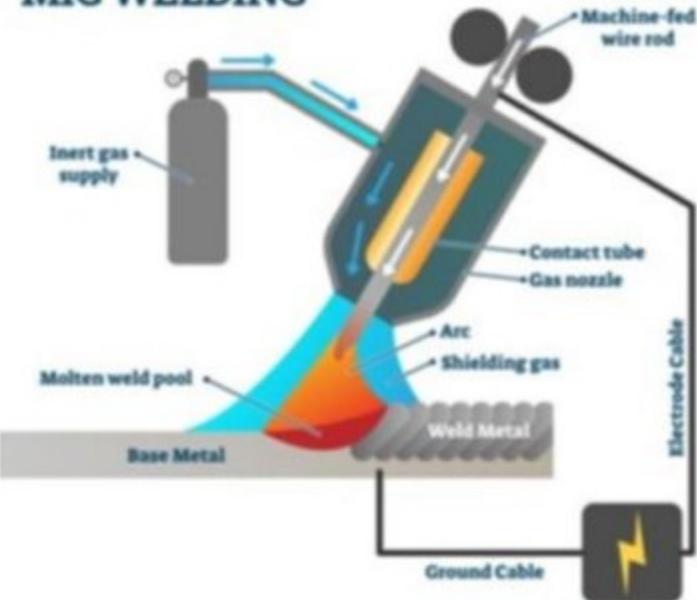
INSPECTION



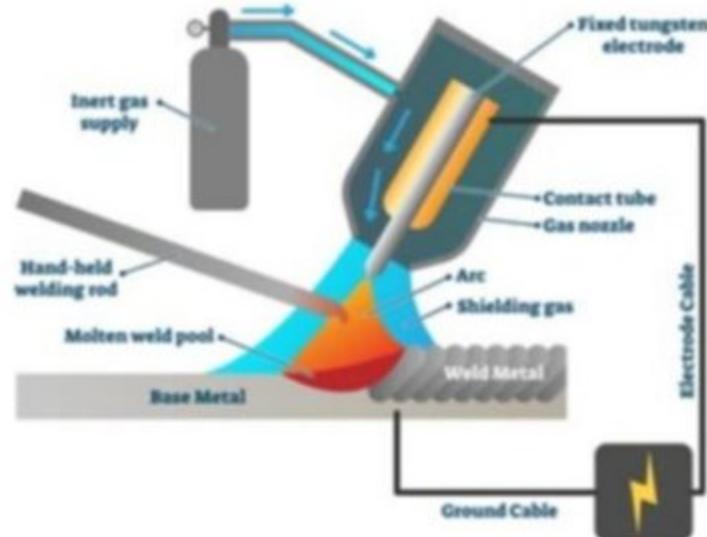
WELDING

- **MIG Welding - Gas Metal Arc Welding (GMAW)**
- **TIG Welding - Gas Tungsten Arc Welding (GTAW)**
- **Stick Welding - Shielded Metal Arc Welding (SMAW)**
- **Flux Welding - Cored Arc Welding (FCAW)**
- **Energy Beam Welding (EBW)**
- **Atomic Hydrogen Welding (AHW)**
- **Gas Tungsten-Arc Welding.**
- **Plasma Arc Welding.**

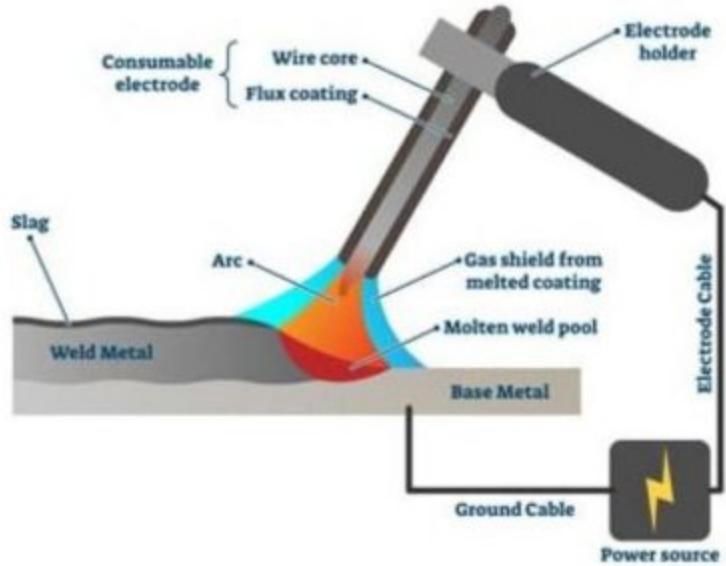
MIG WELDING



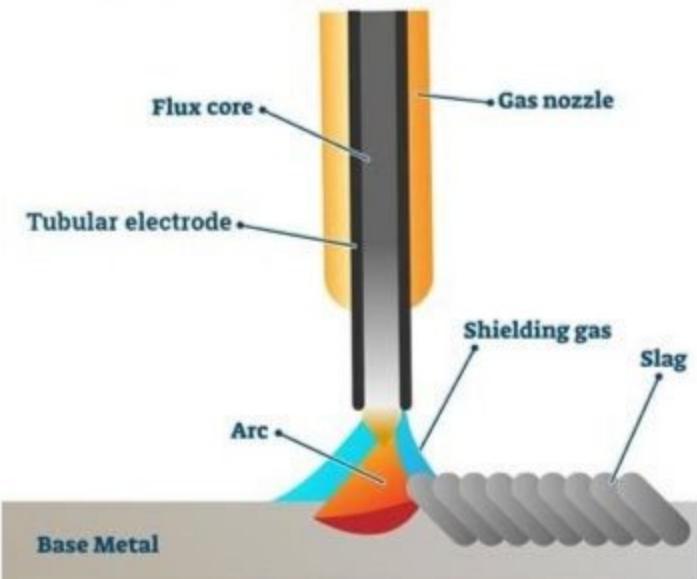
TIG WELDING



STICK WELDING

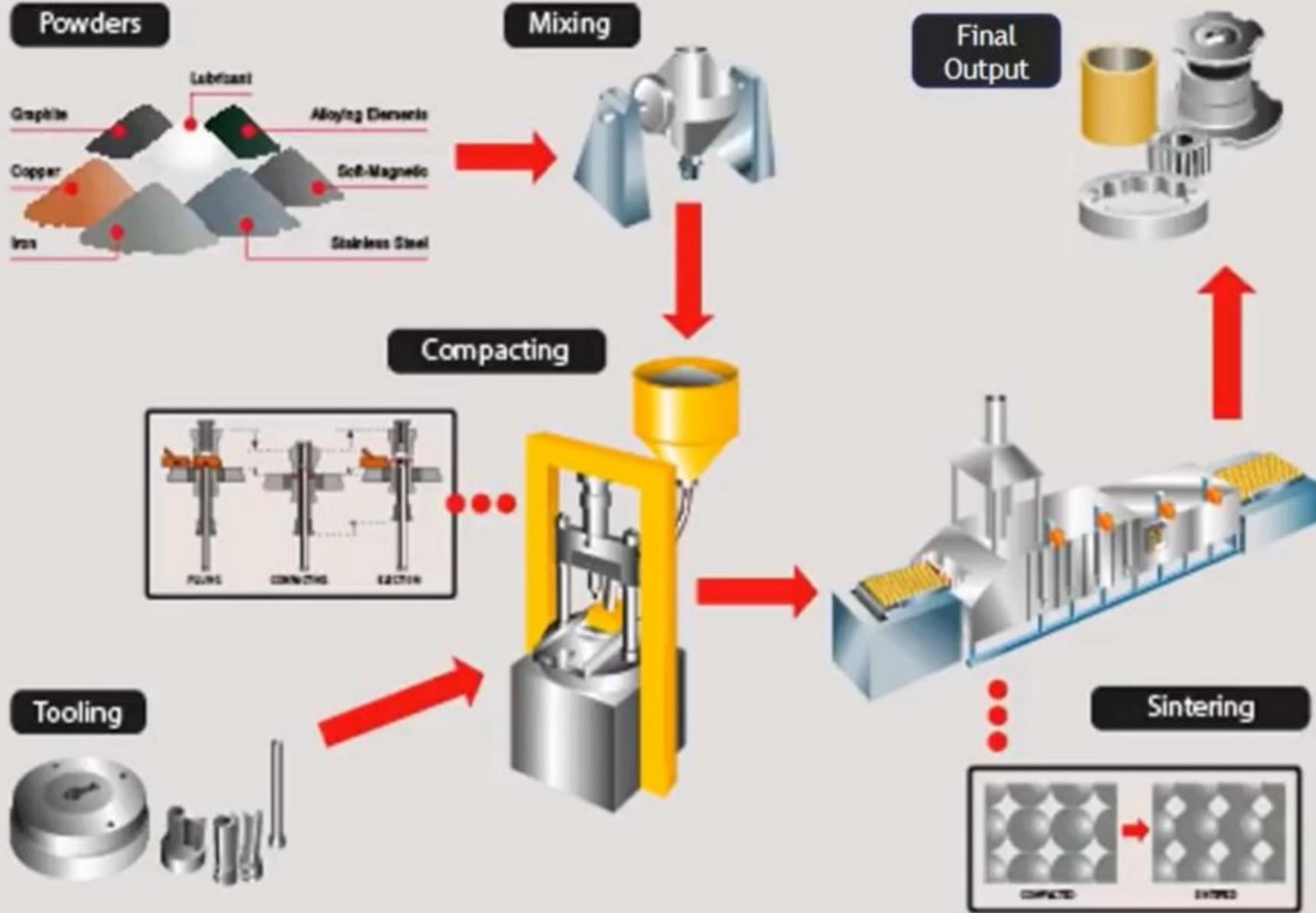


FCAW WELDING



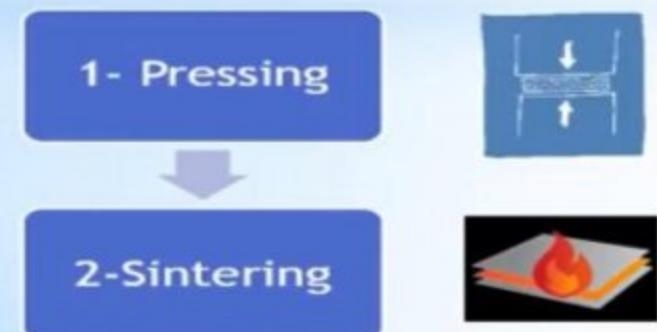
| | | |
|---|--|---|
| 1 | MIG – Gas Metal Arc Welding (GMAW) | Also called Metal Inert Gas Welding (MIG). Provides the most consistent weld results. Some feel this is probably the easiest place to start for beginners. |
| 2 | TIG – Gas Tungsten Arc Welding (GTAW) | Also called Tungsten Inert Gas Welding (TIG). One of the neatest (cleanest) types of welding because it doesn't create splatter. |
| 3 | Flux Cored Arc Welding (FCAW) | A great solution for tanks, boilers, and pipes that have thick plate metal. |
| 4 | Submerged Arc Welding (SAW) | Hot materials don't splash, or splatter and the flux keeps high levels of radiation from escaping into the air. |
| 5 | Stick – Shielded Metal Arc Welding (SMAW) | This is also known as Stick Welding. Doesn't require the gas shielding that TIG and MIG require and is very portable. It also works great outdoors for gates farm machinery, etc. |
| 6 | Thermit Welding | Allows for the fast joining of two dissimilar metals without requiring a power supply. |
| 7 | Oxyacetylene (Gas) Welding | Easy to learn and perform as well as being a low-cost solution. |
| 8 | Forge Welding | Best option for blacksmithing. |
| 9 | Resistance Welding | Is able to translate to very thin metals (0.1 millimeters) to this metals (20 millimeters). |

POWDER METALLURGY



- Powder Metallurgy (PM) is a metal processing technology where metallic powders are produced and used for part production.

- Main PM production sequence:



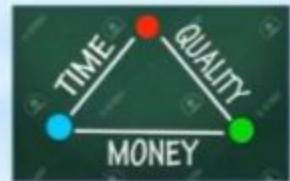
Advantages

PM process wastes much less material.

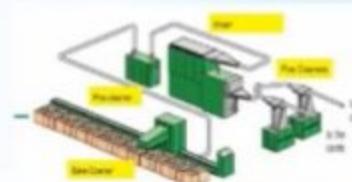
97%
of Starting
Powder used



Complex features and good dimensional accuracy can be achieved.



PM parts can eliminate the need of subsequent machining.



Process steps ?



1- Blending and Mixing of powder composition



2- Pressing of Powder



3- Sintering of the Part



1- Blending and Mixing of powder composition



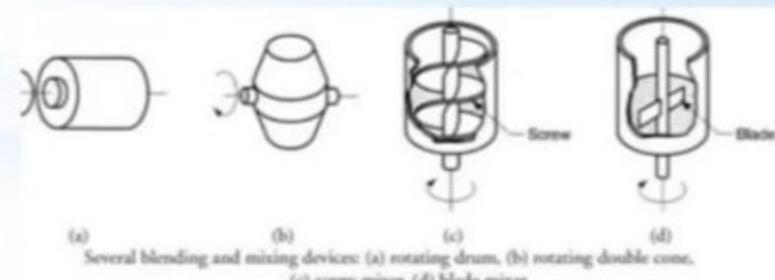
Bronze = Copper + Tin

Sterling Silver = Silver + Copper

Brass = Copper + Zinc

* It is important to make a uniform homogenous mixture of powders composition. By doing this we get better results in the subsequent operations.

* Different devices can be used, it depends on the composition to be mixed.

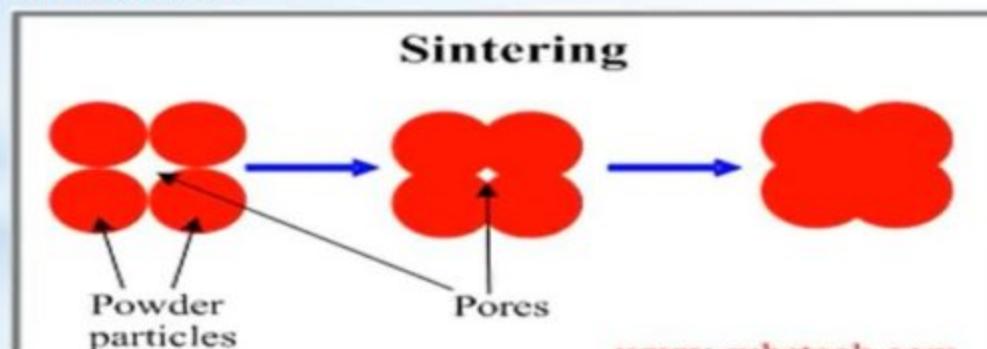


2. Pressing of Powder

- * Isostatic Pressing
- * Cold Isostatic Pressing
- * Hot Isostatic Pressing
- * Powder Injection Molding
- * Powder Rolling
- * Powder Extrusion
- * Powder Forging

- * It is a heat treatment process in which ranges between 70% to 90% of the materials melting temperature.
- * This step is done to achieve the maximum possible hardness and strength needed in the final product.

3. Sintering of Parts



Gear Manufactured by
Powder Metallurgy

VS

Gear Manufactured by
Traditional Process



- High strength achieved.
- Helical gear with angle of 33 degree is possible.
- Maximum density of 7.2 g/cm³.
- Bear usually high pressure up to 4 Gpa.



- 10% greater ultimate tensile strength and yield strength.
- 15-20% greater rotating bending fatigue properties.
- Mirror like surface finish.

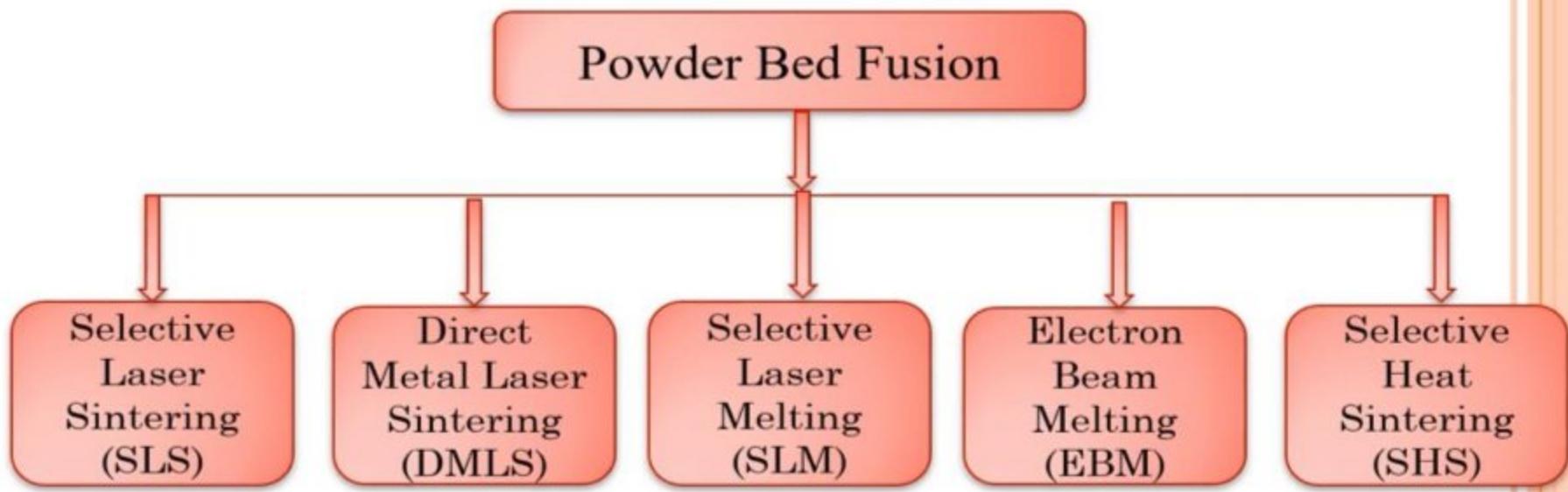


POWDER METALLURGY

- Eliminates or minimizes machining and scrap.
- Impractical or even impossible complex and unique shapes can be made.
- Improved quality rates through excellent part to part repeatability.
- Reduced waste by improved material usage.

3-D PRINTING

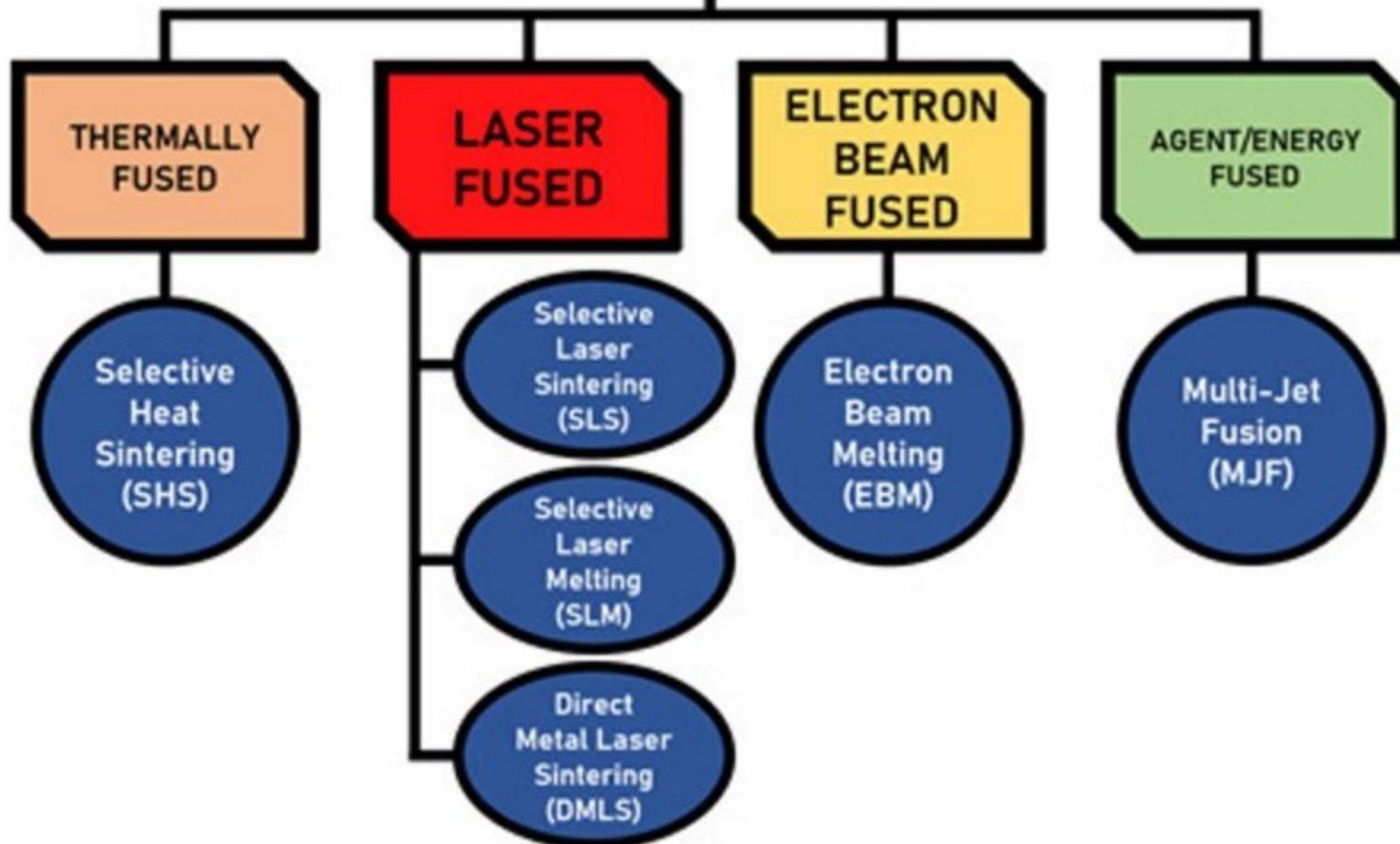
TYPES OF PBF PROCESS



The Powder Bed Fusion process commonly used printing techniques:

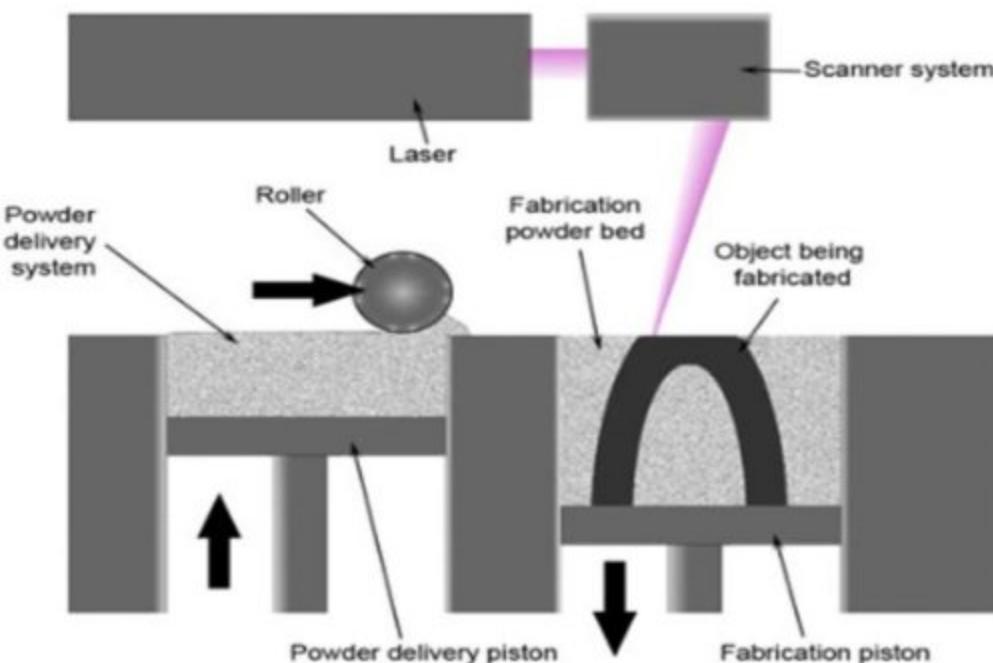
- **Direct metal laser sintering (DMLS),**
- **Electron beam melting (EBM),**
- **Selective heat sintering (SHS),**
- **Selective laser melting (SLM) and**
- **Selective laser sintering (SLS).**

POWDER BED FUSION (PBF)



SELECTIVE LASER SINTERING (SLS)

1. A continuous layer of powder is deposited on the fabrication platform
2. A focused laser beam is used to fuse/sinter powder particles in a small volume within the layer
3. The laser beam is scanned to define a 2D slice of the object within the layer
4. The fabrication piston is lowered, the powder delivery piston is raised and a new layer is deposited
5. After removal from the machine, the unsintered dry powder is brushed off and recycled



ADVANTAGES, DISADVANTAGES & APPLICATIONS

○ Advantages

- Wide array of structural materials beyond polymers
- Cheaper than EBM
- One of two technologies that allow complex parts in metals

○ Disadvantages

- The quality of metal parts is not as high as with EBM

○ Applications

- Structural components

Powder bed fusion (PBF)

It is an additive manufacturing process.

Works on the basic principle in that parts are formed through adding material rather than subtracting it through conventional forming operations such as milling.

The PBF process begins with the creation of a 3D CAD model, which is numerically 'sliced' into several discrete layers.

For each layer, a heat source scan path is calculated which defines both the boundary contour and some form of fill sequence, often a raster pattern since the heat source is typically an energy (laser) beam.

Each layer is then sequentially bonded on top of each other.

PBF processes spread powdered material over the previously joined layer, ready for processing of the next layer hence the manufacturing is discrete rather than continuous (though each layer is fully consolidated to adjacent layers).

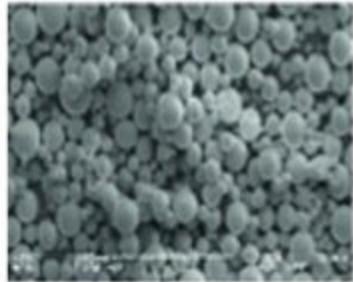
A hopper supplies the powdered material which is then spread uniformly over the powder bed build platform area via a roller or blade.

The optimal thickness of each layer of spread powder is dependent on the processing conditions and material used.



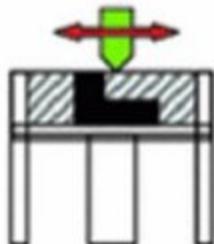
3D-CAD model
in slices

+

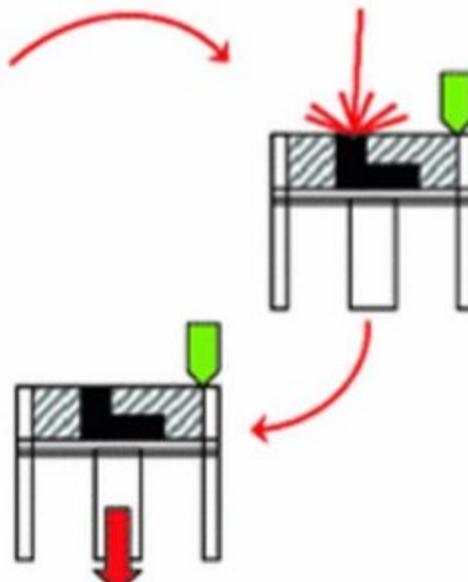


Powder

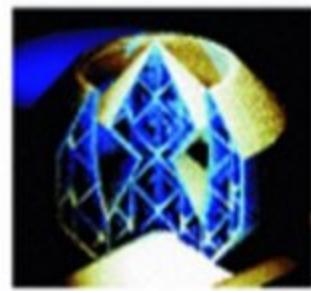
Deposition of a
powder layer



Melting of the powder
by a laser beam

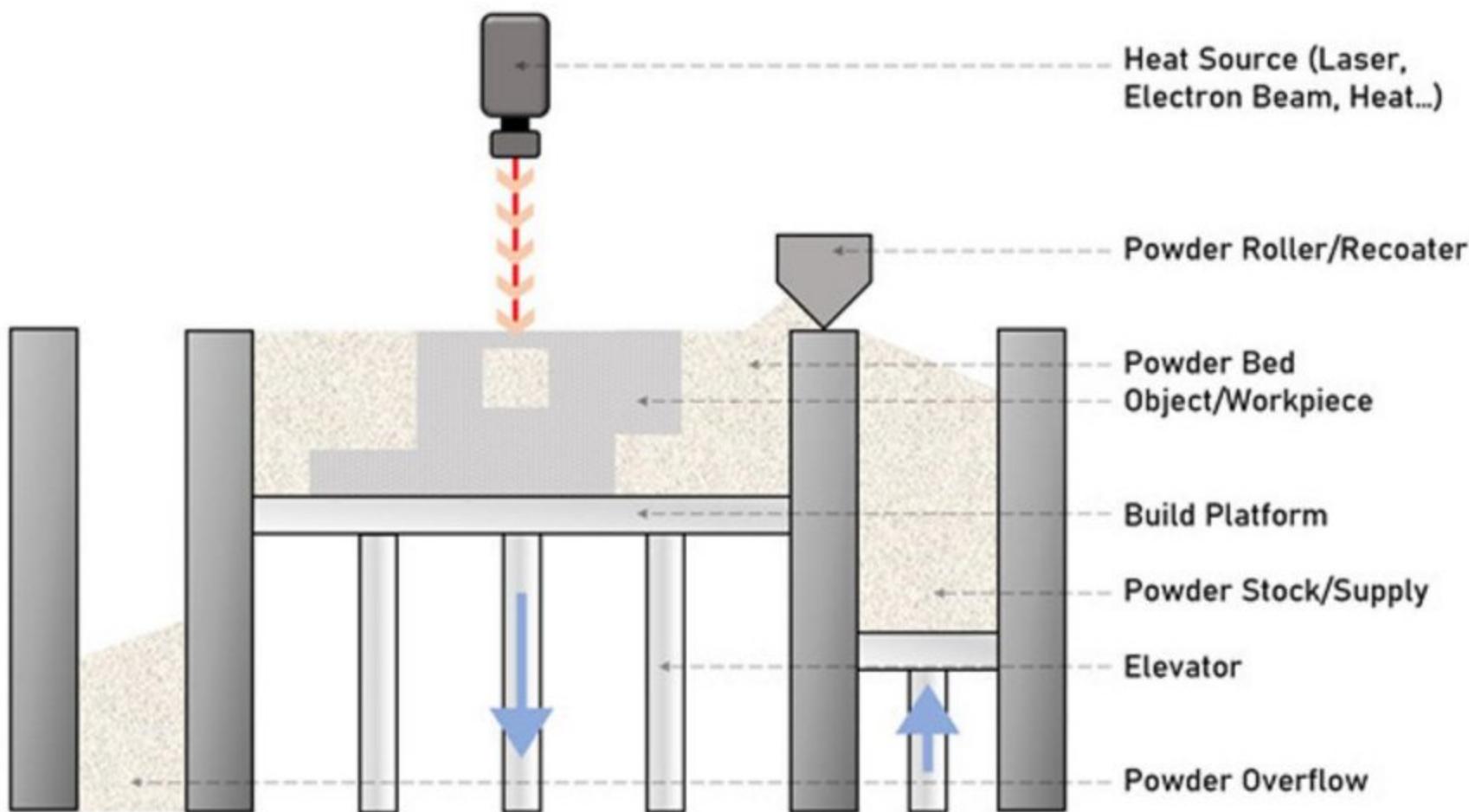


Lowering



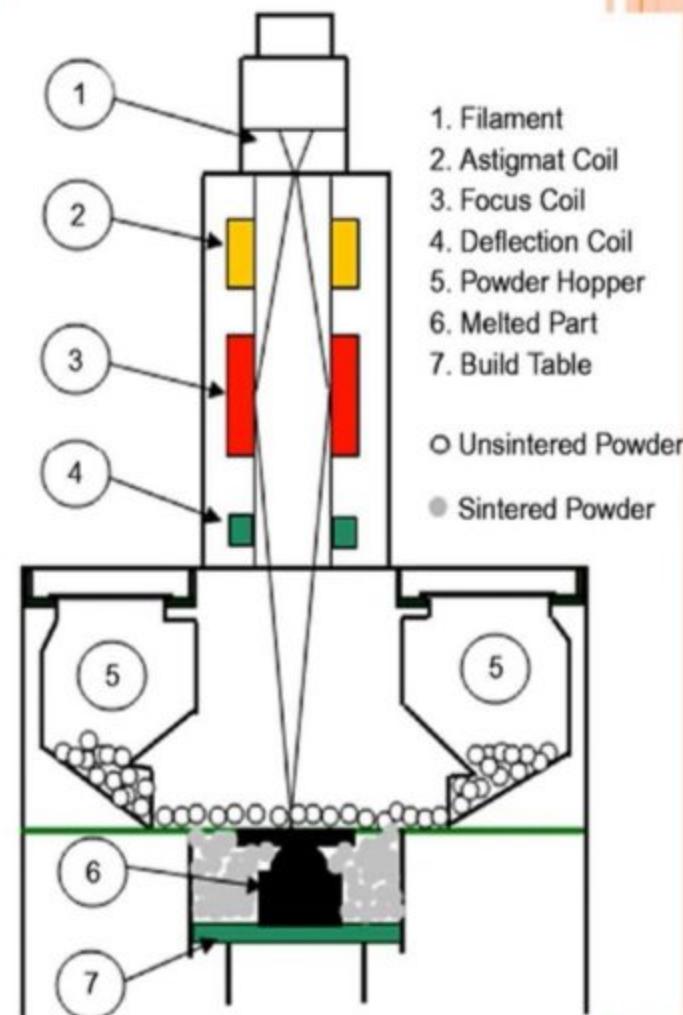
Novel geometries of
serial-materials

Powder Bed Fusion Work



ELECTRON BEAM MELTING (EBM)

- In EBM, a heated tungsten filament emits electrons at high speed which are then controlled by two magnetic fields, focus coil and deflection coil as shown in
- Focus coil acts as a magnetic lens and focuses the beam into desired diameter up to 0.1 mm whereas deflection coil deflects the focused beam at required point to scan the layer of powder bed .
- When high speed electrons hit the powder bed, their kinetic energy gets converted into thermal energy which melts the powder.
- In preheating stage, a high current beam with a high scanning speed is used to preheat the powder layer in multiple passes.



Power bed fusion advantages include:

- Reduced material wastage and cost (superior buy-to-fly ratio)
- Improved production development times
- Enablement of rapid prototyping and low volume production
- Capable of building functionally graded parts
- Fully customised parts on a batch by batch basis, eliminating fixed designs
- Good resolution when compared to other additive manufacturing processes
- Efficient recycling of un-melted powder
- Ability to join many material grades, including ceramics, glass, plastics, metals and alloys
- Elimination of the need for machining fixtures

PBF has applications in:

Rapid prototyping of parts/assemblies

Commercial aerospace components

Medical implants, hearing aids, and dental equipment

Military technologies

Automotive engines and components

Highly complex and delicate objects

Consumer products

Visual prototypes/high finish parts.

Aerospace, Oil and Gas, Marine, Defense,

Automobile, Medical, Manufacturing Tools and much more.

Sources:

1. [Powder Bed Fusion | Additive Manufacturing Research Group | Loughborough University \(lboro.ac.uk\)](#)
2. [Powder bed fusion - DMLS, SLS, SLM, MJF, EBM | Make \(3ds.com\)](#)
3. [What is Powder Bed Fusion? Process Definition and Advantages - TWI \(twi-global.com\)](#)
4. [What is powder bed fusion and what are the pros and cons? \(engineeringproductdesign.com\)](#)
5. [The Complete Guide to Laser Powder Bed Fusion in 3D Printing - 3Dnatives](#)
6. [What is Selective Heat Sintering \(SHS\)? \(goprint3d.co.uk\)](#)
7. [DMLS vs SLM 3D Printing for Metal Manufacturing | Element](#)
8. [What is Selective Laser Sintering? | Live Science](#)
9. [Multi Jet Fusion \(MJF\) | 3D Printing | Protolabs](#)
10. [Jet Fusion Technology for 3D Printing \(sculpeo.com\)](#)
11. [Additive manufacturing technologies: An overview | 3D Hubs](#)