5.9 Crystal Growth:

Crystal growth is a major stage of a crystallization process, and consists in the addition of few atoms, ions, or polymer strings into the characteristic arrangement of crystalline Bravais lattice. The growth typically follows an initial stage of either homogeneous or heterogeneous nucleation, unless a seed crystal purposely added to start the growth

Solidification (or) Crystallization

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

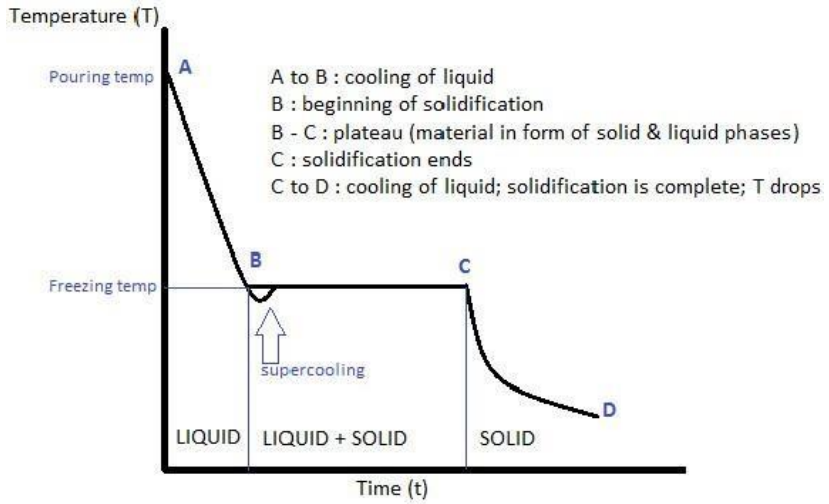

Fig 5.9.1-Transformation of material from liquid to solid state

When a liquid solidifies (solidification process), the energy of each atom is reduced. This energy is given out as latent heat. For a pure metal it occurs at a fixed temperature. During solidification, the disordered structure of the liquid transforms to the orderly arrangement depending upon the time of solidification. This solidification process greatly influences the crystal growth.

Crystal Growth Techniques

Crystal can be grown using the following basic growth techniques. They are,

- Low temperature solution growth
- High temperature solution growth (Flux growth)
- Melt growth
- Hydrothermal growth
- Gel growth
- Vapour phase growth

Melt Growth

Melt technique is the process of crystallization by fusion and re-solidification of the starting materials from the melt. The growth from melt is further sub-grouped into two techniques.

- ❖ Czochralski technique
- ❖ Bridgman technique (high temperature technique)

Czochralski Method or Crystal Pulling Method

Principle:

Crystals are grown by a gradual layer by layer condensation of melt. It is based on liquid-solid phase transition initiated by a seed crystal.

Description

1. The pure material which is to be grown in the form of single crystal is taken in the crucible.
2. The material is heated above the melting point using induction heater.
3. Thus melt is obtained in the crucible. A small defect free single crystal, called seed crystal is introduced into the melt.
4. The seed crystal is attached to a rod, which is rotated slowly.
5. The seed crystal is rotated and gradually pulled out of the melt.

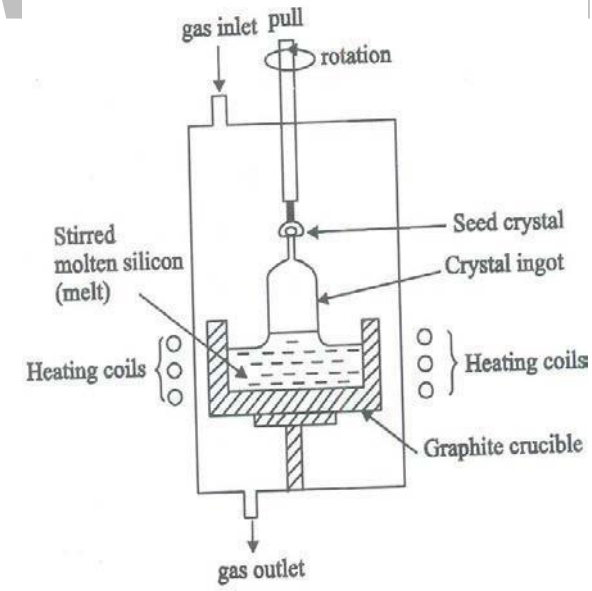

Fig 5.9.2-Czochralski Method

7. A single crystal grows on the seed crystal.
8. The seed crystal acts as i) a nucleation centre

ii)heat sink through which the latent heat of solidification escapes.

9. The temperature difference between the melt and the seed crystal establishes the desired growth condition.
10. The seed is slowly with-drawn from the melt by pulling and rotating such that the crystal diameter is gradually increased to the desired value.
11. The seed crystal is dipped into a melt held at a temperature slightly above the melting point.
12. The shape of the crystal is initially in the form of thin neck and then the diameter of the crystal is increased to the required size. This is known as necking procedure.
13. By using necking procedure and pulling mechanism, bulk single crystals can be obtained by Czochralski method.

Advantages

- It produces defect free crystal.
- It produces large single crystal.
- It allows convenient chemical composition of crystal.
- The dopant distribution in the crystal will be uniform.

Limitations

- High vapour pressure of the materials can be used.
- Possibility of liquid phase encapsulation occurs during solidification.
- It may produce contamination of melt by the crucible.

5.10 Bridgman Techniques

Principle

The selective cooling of the molten material is used to form single crystal by solidification along a particular direction. It is classified into two types. They are,

- Vertical Bridgman Technique
- Horizontal Bridgman Technique

In both techniques, the melt in a sealed crucible is progressively frozen from one end to other end.

Description

1. The material to be grown in the form of a single crystal which is taken in a cylindrical crucible.

2. Crucible is made of platinum and tapered conically with pointed tip at the bottom.
3. The crucible is suspended in the upper furnace until the material in the crucible in completely melted into molten liquid.

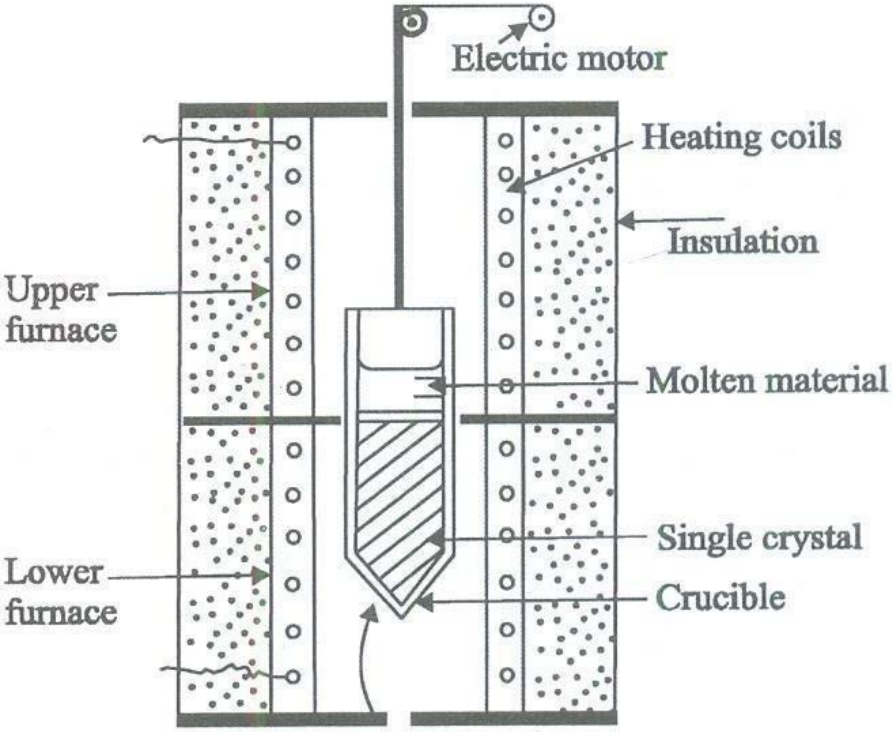

Fig 5.10.1Bridgman Technique

4. Crucible is then slowly lowered from upper furnace into lower furnace with the help of an electric motor.
5. Temperature of the lower furnace is maintained below the melting point of the material inside the crucible.
6. Thus, a bulk single pure crystal can be grown in the crucible by lowering the crucible at steady rate (1 – 30 mm/hr.).

Advantages

- ❖ It is relatively cheaper than other pulling techniques.
- ❖ Simpler technology
- ❖ Melt composition can be controlled during the growth.
- ❖ It enables easy stabilization of temperature gradient

Disadvantages

- ❖ Growth rate is very low.

- ❖ Sometimes instead of single crystals, polycrystals may grow.
- ❖ Since the material is in contact with the walls of the container for long period, it leads to dislocations of the nucleus.

- ❖ This technique can't be used for materials which decompose before melting.