CHAPTER II

CRYSTAL GROWTH TECHNIQUES
2.0 INTRODUCTION

Crystal growth is an important area of material science that involves chemists, metallurgists, chemical engineers, geologists, physicists, crystal growers and simulation experts. Single crystals contain atoms or group of atoms arranged in perfect symmetry. The development in both theoretical and technical aspects of crystal growth has been stimulated by the increasing commercial importance of the field. The spectacular growth of solid state electronics depends critically on crystal growth and perfection. Good single crystals are essential for a wide variety of scientific and technological use. They are needed for the scientific appraisal of crystallography, topography and tensor properties of all crystalline materials. It is practically impossible to grow a single crystal free from defects partly due to thermodynamic reasons [1] and due to practical limitations of the crystal grower. However, the crystal grower is growing single crystals controlling physical and chemical properties such as (a) defects like non-stoichiometry, dislocations, density etc. (b) impurity concentrations (c) concentrations of dopants and (d) geometry to suit specific purpose.

Today high quality compound crystals are being produced with the advanced crystal growing techniques such as liquid encapsulation [2], molecular beam epitaxy (MBE) [3], metal organic vapour phase epitaxy (MOVPE) and liquid phase epitaxy (LPE) [4]. To grow a desired single crystal an extensive first-hand experience is needed more than bookish knowledge. Therefore, it is not uncommon that a growth apparatus performs effectively only to its master [5, 6]. Thus crystal growth is still considered as an ART rather than SCIENCE. The growth mechanism and the dependence of growth parameters on quality of the crystals have been studied extensively. In the following sections the growth and various parameters, which influence the performance of growth apparatus are presented.
2.1 NUCLEATION

Nucleation and growth process are two important aspects of crystal growth. Nucleation is the precursor of crystal growth. The condition of super saturation / super cooling alone is not sufficient for crystallization. In many cases, there must exist minute bodies known as centers of crystallization / seeds / critical embryos / critical nuclei. The formation of such critical entities is nucleation. Nucleation may occur either spontaneously or inductively. The spontaneous formation of critical nuclei in the interior of the parent medium unaided by a foreign particle is known as homogeneous nucleation. If the critical nuclei form around ions, impurities, molecules, dust particles, dislocations or other imperfections then it is called heterogeneous nucleation. The necessary and sufficient condition for any spontaneous process to take place is $\delta \mu \delta n < 0$ while for reversible process $\delta \mu \delta n = 0$, where $\delta \mu$ is the change in the chemical potential and $\delta n$ is the number of monomers. In solid phase, the chemical potential $\mu$ of a monomer is less than that of liquid or gas. While forming a solid phase, the structure with the least chemical potential is preferred and hence the diversity in structures. The chemical potential $\mu$ is given by

$$\mu = [\partial \mathcal{G} / \partial n]_{T,P} = [\partial (H-TS) / \partial n]_{T,P}$$

Where $H$ is the enthalpy, $S$ is the entropy and $G$ is Gibb's free energy and all these have more than two when applied to the condensation of monomers. These contributions can be from surface volume, configuration and strain. In the early stages of formation of embryo, the volume contribution is proportional to the cube of its dimensions and dominates over the surface term which is proportional to the square of its dimension. This leads to a reversible polymerization till the nuclei attain a critical size. Afterwards, the volume term becomes insignificant and hence solidification or crystallization begins. The formation and rate formation of critical nuclei have been studied for different shapes of monomers [7].
2.2 GROWTH PROCESS

Critical nucleus with anisotropic bonding with the neighbours leads to a variety of surfaces for monomers to select and occupy. The fast growing surfaces or planes leave the facets of slowly growing planes to remain which are normally found as facets in the habit [8]. Growth phenomenon is then linked with equilibrium process and the activation energies. Theoretical growth rates based on activation energies of monomers in homogeneous nuclei on smooth surfaces did not agree with the experimental growth rates and hence the role of dislocations in general and screw dislocations in particular was invoked by Frank [9]. This acquired unprecedented reputation [10] and served as basis for the equilibrium growth theory of Burton, Cabrera and Frank known as BCF theory [11] of crystal growth. The main outcome of this theory is that the growth rate varies as the square of the super saturation / super cooling at very low super saturations and varies linearly with super saturation / super cooling at higher limits. This agreed quantitatively with the observed higher rates even at lower saturations and thereby removed the discrepancy between the experimental and prevailing theoretical concepts [12]. Stochastic approach is also in vogue for nucleation and growth studies. In this approach loss of memory or trajectory or random access is invoked in computers to stimulate both nucleation and growth kinetics using Monte Carlo techniques [13-14] and to compare experimental results.

2.3 METHODS OF CRYSTAL GROWTH

A large number of existing crystal growing techniques crystal growing techniques with overlapping working principles make it difficult to draw discrete boundaries for various methods. However, Pamplin has to some extent pictorially represented the classifications of these techniques [1, 15] as shown in the Fig. 2.1.
Fig. 2.1 Pictorially represented classifications of crystal growth techniques by Pamplin [1]
Depending on the growth phase, the techniques may be classified broadly as

(i) growth from solid phase,
(ii) growth from solutions,
(iii) growth from melt and
(iv) growth from vapour phase

It is not possible to give a detailed account of all these methods here. While, in the context of the present work, the principles of these techniques are briefly given in the following sections with more emphasis laid on vapour phase growth and Bridgman technique.

2.3.1 GROWTH FROM SOLID PHASE

The material in the solid state either amorphous or polycrystalline is kept at sufficiently high temperature but well below its melting / sublimation temperature. The atoms in the solid due to their high mobility try to occupy the symmetry position. This natural driving force drives an amorphous state into polycrystalline state and then to single crystal form. This method is suitable only for the materials, which are stable at high temperature with appreciable diffusion coefficient. This technique is better known in the names of annealing, heat treatment, sintering and quenching which are employed in metallurgy in tailoring the properties of materials. Single crystals of several metals like lead [16], tungsten [17] molybdenum [18] and some oxides like YIG, BeO, Al₂O₃ and ZnO [19,20] have been grown and even some solid solutions of mixed III-V and II-VI compounds have been successfully grown [21-22].
2.3.2 SOLUTION GROWTH

This is a simple and inexpensive technique. In this method, growth occurs from super saturated solutions in about a few hours to a few months. The solvents could be water, molten metals or mixture of metals, molten salts etc, depending upon the material to be grown. To achieve appreciable growth rates, high temperatures and pressures are also used in specific cases [23]. The main drawbacks in this method are multinucleation, smaller growth rates, non-availability of suitable solvents. However, this method is still preferred for growing incongruently melting / evaporating materials and also for low temperature crystalline phases which cannot be grown at high temperatures. Organic and inorganic gels are used to control the flow of sparingly soluble solute into the solution [24]. In hydrothermal growth water insoluble materials are forced to dissolve at elevated temperatures and pressures in autoclaves [25]. Flux growth method is used to grow many multicomponent materials and high $T_c$ superconducting oxides using molten oxides and salts and solvents [26]. Many technologically important epilayers of a micron thick have been grown by liquid phase epitaxy [27]. Elwell and Scheel [28] have published a book giving a comprehensive and critical review on this technique.

2.3.3 MELT GROWTH

Melt growth is most widely used technique for growing large single crystals of higher perfection and it is the fastest of all crystal growth methods. It has been used to grow single crystals of metals, semiconductors, ionic crystals, homogenously doped semiconducting crystals and a few organic compounds. This method is applicable to non-volatile and congruently melting materials only.

In this technique, either a seed crystal or one of the many nuclei formed will be in contact with the melt and the growth interface is moved out of the melt at the rate of the crystal growth so that the melt grows into a single crystal with the
orientation of the seed. The heat of crystallization is quickly removed from the growing crystals by coolants or by attaching conducting rods. Theoretical and experimental aspects of melt growth have been dealt with in several books [29-31]. Depending on the design of crucible, seed holder, melting methods etc, melt growth technique can be broadly classified as a) freezing methods b) crystal pulling c) flame fusion methods.

a) Freezing methods

Bridgman [32], Stockbarger [33], Zone melting [34] Kyropoulos [35] methods come under freezing methods. In Bridgman and Stockbarger techniques the whole charge in molten state passes through smooth and steep temperature gradients respectively. In Zone melting method on the other hand, only a part of the charge in the molten state moves from one end to the other end of the whole charge. In Kyropoulos technique, the melt in contact with the seed is force cooled and the melt temperature is slowly decreases so that the melt grows into a single oriented crystal. In this method instead of a seed, a favourable site for nucleation is provided then it is called normal freezing methods.

b) Crystal pulling methods

Methods of pulling crystals from melt have been developed Czochralski [36] and Kyropoulos [35]. Here essentially, a seed is dipped into the surface of the melt and slowly withdrawn. For uniformity and homogeneity, the seed is also rotated slowly. This was first used by Czochralski for the crystallization of metals [36]. This technique has been employed for an extremely wide range of materials with melting points ranging from 25°C (Ga) to 2135°C (MgAl2O3). Even dissociative materials like III-V semiconductors are now commercially grown using liquid encapsulation like Be2O3. Automatic size or weight controllers have become part of the current day crystal pullers.
c) **Fame fusion or Verneuil method**

The Verneuil [37-38] is the earliest of crucible less techniques, originally designed for the preparation of artificial gem-stones. In this method, a molten zone is formed on the tip of the seed crystal with an oxygen-hydrogen flame or r.f. heating. Powder of the material is fed to the zone and growth proceeds by lowering the seed. Verneuil's technique is used to produce rubies and sapphires for watch bearings and similar applications and for the growth of refractory oxide crystals like corundim.

**2.3.4 VAPOUR PHASE GROWTH**

In vapour phase growth atoms or molecules that are produced from charge either by sublimation or vapourization at a high temperature zone is transferred to a suitable low temperature region where the reverse occurs slowly and the crystal grows after proper nucleation [39-41]. If the rates of vapourization and crystallization are not suitably controlled, non-equilibrium condensation leads to the formation of amorphous or polycrystalline phases. Use of ultra high vacuum and proper temperature gradients controls atomic beams and also favourable sites for nucleation. In some cases chemicals like iodine and chlorine are used as agents to transport vapours from a high temperature region to low temperature region. Control on nucleation in vapour phase growth is achieved by the following rules [42].

(i) Super saturation must be controlled to the level of subcritical nuclei so that spurious nucleation is avoided. (ii) The ampoule must be translated through temperature gradient at the rate of equal to the linear growth rates of the crystal without drastically changing the super saturation condition.
2.3.5 EPITAXIAL GROWTH TECHNIQUES

Epitaxial layers are two dimensional single crystalline layers. As the actual device performance of single crystalline materials is limited to only a few micron lengths in one direction methods of producing and using such layers drastically reduces the required quantity of the material and production costs. Liquid phase epitaxy, Molecular beam epitaxy (MBE), Vapour phase epitaxy, organo-metallic vapour / molecular beam epitaxy are widely used in electronic devices. There are several controls in these techniques and even non-equilibrium processes can be achieved. The materials so produced have all the desired quantities except equilibration. Epitaxial techniques are also useful the preparation of buffer layers of required lattice constant to avoid performance of the devices due to mismatch.

2.4 CRYSTAL GROWTH APPARATUS

For crystal growth, a furnace, a crucible, temperature measuring instrument / programmer and a suitable environment / vacuum and the required material are essential. Normal joule heating materials with insulation blankets used in furnaces are Nichrome (400°C), Kanthal Al (1375°C), super Kanthal (1700°C), Silicon carbide (1600°C), Platinum (1550°C), rhodium (1800°C), thorium oxide with Lanthanum or Calcium oxide (2000°C), and ZrO₂ (2400°C) over longer periods of time from a few weeks to a few months. For still higher temperatures, Molybdenum (2400°C), Tantalum (2600°C) and Graphite (2600°C) are normally used in inert / hydrogen / argon atmosphere [15, 28]. For selective heating without electrical contact, induction heating methods are widely used whenever the required temperature is above 1600°C. In special cases, heating could be done by direct flames, plasma, LASER and electrons.

Crucibles generally used are made of pyrex (400°C), quartz (1200°C), alumina (1800°C), zirconia (2300°C) and graphite in reducing atmosphere (3000°C),
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platinum (1500°C), tungsten (2500°C) °C, tantalum oxide (3000°C), rhenium (2380°C) and iridium (2200°C). The temperatures in brackets are the higher limits of safe usage of crucibles. The reactivity of growth materials restricts the use of specific containers [15, 28]. Pyrex and quartz in particular are widely used as they can be easily used and moulded.

2.4.1 TEMPERATURE MEASURING DEVICES

Most common temperature measurement and control units are based on the use of thermocouples. Since thermo electric power of a variety of metal and alloy combinations is accurately known, single junction can be used by electronically controlling the other junction. Thermocouples like Iron/Constantan (650°C), Chromel/Alumel (100°C), Pt/Pt94%-Rh6%, Pt/Pt70%-Rh30% (1500°C), Pt/Pt 90%-Rh10% (1600°C), Ir/Ir40%-Rh60%, W/W-Ir (2000°C), Re/Re-Ir (2000°C), W/W97%-Re3%, W/W75%-Re25%(2200°C), and W/W-Re (2200°C) are widely used for continuous temperature monitoring. In very high temperature regions, not accessible to thermocouples, optical / radiation pyrometry is used for temperature measurement. The most frequently used temperature controllers are based on thyristor powered proportional band integral and differential (PID) controllers [28]. Temperature programming is done using PID controllers with either phase angle firing or fast cycling. Growth atmospheres vary from ultra high vacuum (10⁻¹⁴ Torr) to continuous flow of suitable gases at atmospheric pressure.

2.5 GROWTH METHODS APPLICABLE FOR II-VI DMS

All the II-VI based Diluted Magnetic Semiconductors (DMS) have high melting temperature (>1000°C) and they evaporate disassociatively and congruently well below their melting points. Though this limits the application of melt growth techniques, high temperature autoclaves have been used successfully [43-46]. Of all these compounds, ZnTe has the highest melting point and it is difficult to grow the
crystals of ZnTe based DMS by melt growth techniques. Hence thick walled graphitized quartz tubes are used in this technique, so that breakage of growth tube is avoided. High temperature solution growth was employed with limited success. Hydrothermal growth though promising has yet to gain momentum. The serious problems associated with growth techniques when applied to II-VI based DMS, and the congruent and appreciable vapourization of II-VI based DMS have led to the revival of the vapour phase growth technique from time to time. However, the growth runs are longer, extending from several days to weeks, as compared to those of III-V based DMS, which have still higher vapour pressures but dissociate incongruently. Platelets, needles and flakes are quite common in this technique due to difficulties in controlling multinucleation. Though vapour phase growth is not a fast technique, still this is the only successful economic technique evolved as a result of experimentation extending over several decades.

**2.5.1 CHOICE OF THE GROWTH TECHNIQUE FOR THE PRESENT WORK**

Various methods of growing single crystals have been reviewed briefly in the preceding sections and a brief account has been given in the last section about the growth methods for II-VI based DMS. When more than one type of growth method is applicable, the factors that should be considered are the nature of the material to be grown, purity of crystal, size of the crystal, economy and last but not the least is the applicability of the technique for a variety of materials.

Vapour phase and Bridgman growth techniques have been developed and used for the growth of Zn_{1-x}Cr_{x}Te crystals in the present investigation. The fabrication details of furnaces, temperature programming profile, environment maintained and the crystal puller developed are described in the following sections along with a general description procedure of growth established at after great deal of trail and error.
By experience, it has become clear that mere selection of the appropriate method does not guarantee its successful application to the growth of a single crystal. Whatever may be the method, exploratory experiments, modifications in the growth apparatus, some alterations in the growth parameters and finally 'trial and error' would be required for the reproducible growth of single crystals. May be it is for this reason that crystal growth is still considered as an ‘ART’.

2.6 DETAILS OF THE GROWTH TECHNIQUES USED IN PRESENT WORK

2.6.1 VAPOUR PHASE GROWTH TECHNIQUE

A modified vapor phase growth technique has been developed and a sketch of the same is shown in the Fig 2.2 along with the Piper-Polich set up [39]. A photograph of vapour phase growth set up is shown Fig. 2.3.

![Fig 2.2 A sketch of modified vapor phase growth technique](image)
Fig. 2.3 Photograph of Vapour phase growth set up
The following modifications have been introduced in the present set up:

- In order to increase the sublimation, the plateau region of the temperature profile has been extended.

- By incorporating a capillary in the growth tube a better nucleation has been provided. The capillary also serves as a leak for volatile materials and excess elements and helps to remove the heat of crystallization.

- In order to protect the costly charge in case of any breakdown of the growth tube an extra protective quartz surrounds the growth tube. In the present set up the restriction of choosing two snugly fitting quartz tubes as in the Piper-Polich technique has been removed.

Extended temperature profile, incorporation of capillary for better nucleation and vent for volatile impurities / materials are improvements made to the present set up. A thick walled protective quartz tube is used to avoid the bursting of growth tube and for better growth atmosphere. The entire assembly along with the charge is kept in vacuum is slowly move horizontally through the temperature gradient. Nucleation and growth takes place in the capillary.

2.6.2 BRIDGMAN GROWTH TECHNIQUE

One of the most successful methods for the growth of high quality crystals is the Bridgman method [47-50]. It is essentially a normal freezing technique with low unidirectional cooling of the molten material. This technique produces a single solid-liquid interface by carrying on crystallization process in a steep temperature gradient. The conically tapered region of the growth tube acts nucleation centre and the pulling through the temperature gradient of furnace aids the growth of unstrained
crystal. A sketch of the Bridgman growth set up used in the present investigation is shown in the Fig. 2.4. A photograph of the experimental set up is shown in the Fig. 2.5.

![Fig. 2.4 A sketch of the vertical Bridgman growth set up](image)

**2.6.3 DESIGN DETAILS OF THE TECHNIQUES**

(a) **The furnace**

A furnace of 50x50x70 cm³ dimensions, which can be used in two configurations (both horizontal vapour phase and vertical Bridgman growth techniques), has been fabricated and used in the present study. As the temperatures used are near the breakdown regions of the heating elements, the furnace selection for any growth run is only a matter of convenience. The break down of heating
Fig. 2.5 Photograph of Vertical Bridgman growth set up
elements is quite frequent and is a matter of great concern. The furnace runs interruptedly using 10 KVA (M/s Kirloskar, India) generator in the event of power failures and regular power shutdowns. Zirconia wool is used as blanket surrounding the heating elements for thermal insulation. With all the precautions temperatures above 1300°C could be maintained more than 7 days. The temperature gradients of 10 - 30°C/cm are used.

(b) Temperature controller

Controlled programmer is used for temperature controller. The complete programming of a growth runs without any human intervenes. The temperature sensors used are inevitably Pt-PtRh 13% thermo couples as the temperature involved are very high. The longitudinal temperature gradient inside the mullite tube is obtained by measuring the temperature across the length of the mullite tube. A uniform temperature region of about 10 cm is obtained at ~1100°C. A typical temperature profile obtained in the present work at 1100°C is shown in Fig. 2.6.

(c) Growth atmosphere

The present set up has provision to grow crystals in a high vacuum / desired inert gas pressure. The high vacuum system (M/s Avac, India) consists of a three stage 4” O.D oil diffusion pump with 300 lit/sec pumping speed in conjunction with a 200 lit/min two stage directly driven rotary pump. As the vapour pressure of silicon oil (DC 704) is as low as 10⁻⁷ Torr it has been used as the charge for the diffusion pump. Pirani and Penning gauges are used for measuring low and high vacuum respectively. A vacuum of 2x10⁻⁶ Torr could be obtained with this system in about an hour.
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Fig. 2.6. Temperature profile of the furnace at 1100°C

(d) Crystal puller

Two crystal puller systems capable of providing continuous pulling rates ranging from a fraction of mm/day to a few cm/hour have been designed and fabricated. An ac synchronous motor (M/s ECIL, India) of 25 RPM having peak torque of 10 Kg-cm is used in conjunction with a reduction gear boxes. The speed reduction is obtained using three matched 32-teeth pinions along with the corresponding three racks. These racks and pinions are arranged alternatively in aluminium box fixed over the motor assembly. The stepped iron pulleys with increasing step diameters could be attached to any of three stages of the gear reduction system and hence a desired pulling rate can be selected.

(e) Growth system

The growth system consists of growth tube and an outer protective tube. The shape and geometry of the tube are most significant factors. Care should be taken in designing the growth tube to provide proper nucleation center, a suitable leak for eliminating volatile impurities and a heat sink to remove the heat of crystallization. Transparent quartz tubes of thickness 1.5mm (M/s INFUSIL, India) are used, as the
vitreous temperature of quartz is about 1200°C and also can be blown to the desired shape. In the present work, quartz tube of 0.7 and 1.0 cm diameters and 20 cm long closed at one end and the other end drawn to a capillary of length of 15-20 cm are used in vapour phase. A similar other tube with one end closed and the other end tapered into conical shape is used in Bridgman growth. The growth tubes are placed in a protective tube of about 1 m long with one closed and the other end fixed to B-24 ground joint fused to a high vacuum stopcock is used to connect / isolate the growth tube from the vacuum / inert gas filling system.

(f) Preparation of the growth tube

Cleanliness of the ampoule is one of the most important aspects in the growth of crystals. If the ampoules are not cleaned properly, the impurities may get into growing crystal and spoil its quality completely. The procedure adopted for cleaning the ampoules is as follows: The quartz tubes are first cleaned with commercial liquid soap or detergent powder. Then the ampoules are filled aquaregia, 1 HNO₃+3 HCl, and are kept for more than 24 hours. After pouring out the acid, the ampoules are cleaned again in ultrasonic bath with double distilled water. The ampoules are finally rinsed with acetone and then kept in a oven at 700°C, for a few hours. These ampoules are used as growth tubes in crystal growth experiments. To increase the tolerance and strength of quartz tubes near softening temperature, graphitization must be used. In the present work, clean growth tubes re rinsed with carbon tetrachloride and heated directly with oxy-acetylene flame. For uniform graphitization, continuous rotation and sliding are to be maintained. This minimizes the nucleation sites on the walls of the ampoule and also impedes impurities like Si [51-53].
2.7 PREPARATION OF CrTe

The stable structure of CrTe is hexagonal (NiAs structure) [54], the required zincblende phase is not stable and is to be prepared by a specific method and used in-situ [55]. Pure Cr and Te are weighed in a microbalance (M/s SICO, India) to 10μg accuracy. These materials are physically mixed and ground thoroughly in an agate mortar with a pestle for physical homogeneity. The mixed powders are tightly packed with the help of a quartz rod into graphitized tune of length of 20 cm and 0.7 cm diameter. The tube was sealed in a vacuum of $2 \times 10^{-6}$ Torr. The sealed quartz tube is kept at a temperature of 1000°C for about five minutes and is then quickly removed to yield the desired zinc blende polycrystalline CrTe.

2.8 PREPARATION OF Zn$_{1-x}$Cr$_x$Te CRYSTALS

Appropriate quantities of freshly prepared CrTe and ZnTe (99.99% M/s Sigma Aldrich) are weighed, mixed and ground thoroughly in an agate mortar with a agate pestle to ensure homogeneity. The powder is packed tightly in the growth tube with the help of a clean quartz rod. One end of the tube is drawn into a fine capillary of 20 cm length leaving a space of 4-5 cm. The growth tube is carefully introduced in well cleaned protective quartz tube, evacuated to $2 \times 10^{-6}$ Torr, and it is placed in a furnace such that, the capillary drawn end is at the lowest temperature of the furnace in vapour phase growth while the conically tapered region is kept at the highest temperature in the Bridgman technique. The temperatures of the furnace are slowly raised to 1050°C and 1300°C in a period of about 24 hours for vapour phase and Bridgman growth respectively. The crystal pullers are connected to protective tubes and pulled at the rate of 0.6 mm/hour for about 3-5 days. At the end of the growth run the pulling motor is switched off and the furnace is slowly cooled to room temperature in about 72 hours using temperature programmer. The growth tube is carefully removed from the furnace and the crystal bowl is taken out by breaking the growth tube.
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