Chapter III
GROWTH

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Section - I

Crystal Growth of SnSe, $SnSe_2$ and SnSe-$SnSe_2$ eutectic

I. INTRODUCTION :

The intrinsic properties of a solid, i.e the properties that result from its specific structure, can be largely modified by crystallographic and chemical defects. The formation of these defects is governed by heat and mass transfer conditions which prevail on and near a crystal-nutrient interface during crystallization. Hence, both the growth of highly perfect crystals and the preparation of samples having predetermined defect-included (extrinsic) properties require a thorough understanding of the reaction and transport mechanisms that govern crystallization from vapours, solutions and melts. Crystal growth, as a science, is therefore mostly concerned with the chemistry and physics of heat and mass transports in the relevant phase transitions. Transport concepts are largely built upon equilibrium considerations, i.e., on thermodynamics and phase equilibrium concepts. Crystal growth can be called in a wide sense, the science and technology of controlling phase transitions that lead to (single crystalline) solids.

Semiconductor crystals are the basic materials for fabrication of most of the electronic and optoelectronic devices. These crystals are used either as substrates for the growth of epitaxial layers or directly for electronic applications. The performance of these devices depends on the purity, perfection, and homogeneity of the bulk crystals.

There are various methods of crystal growth.

1. Crystal growth from Solid State.
2. Crystal growth from vapour phase.
   (i) Sublimation,
   (ii) Vapour transport,
   (iii) Gas phase reaction.
3. Crystal growth from aqueous solution.
4. Gel Method.
5. Hydrothermal growth.
6. High temperature solution growth.
7. Melt growth.

Crystal growth from melt is normally used for the binary alloys and compounds.

The method is described briefly below :

II. GENERAL DISCUSSION :

Crystal growth is a complex process which usually takes place by a phase change. The melt growth of a crystal takes place by a phase change from liquid to solid. The solid and the liquid states are the two condensed states of matter. As such, they have many properties which are rather similar ; for example, the electrical conductivity properties. All exhibit a discontinuity on melting of the solid or solidification of the melt. However, the change in these properties as in many others may be small.

The fundamental difference between the melt and the crystal is the change in symmetry. The environment of an atom in a crystal possesses crystalline symmetry, the environment of an atom in a melt does not. The liquid-solid transformation is unlike the vapour-liquid tranformation in this aspect. Although many physical properties can change significantly during a vapour-liquid tranformation, the symmetry normally does not change; and so there are paths in temperature and pressure such that the properties can change continuously upon vapour to liquid tranformation. On the other hand, the melt to crystal transformation cannot take place continuously because of the difference in symmetry. Crystalline symmetry is either there or not. So the solid ⇆ liquid phase change must be discontinuous.

The crystalline phase is an ordered array of atoms or molecules. The atoms are in thermal motion with an amplitude at the melting point of the order of about 15 % of the interatomic distance. The melting point, or more properly, the equilibrium temperature, is the temperature at which the two phases co-exist in equilibrium at normal pressure. Many compound semiconductors contain volatile constituents which evaporate rapidly from the melt if no precaution is taken. Hence in growing cyrstals of such type of compounds a closed system is taken either in a completely sealed set-up or by encapsulation of the melt. (1)

One of the widely used methods for growing the bulk crystals of II-VI and IV-VI compounds is the Bridgman - Stockbarger technique (2,3). Vertical and horizontal versions of the Bridgman techniques exist. In the vertical Bridgman techniques, the compound/alloy is melted in a vacuum sealed ampoule placed inside a vertical furnace. The furnace consists of two halves. A baffle is used to separate these two halves. The upper part of the furnace is kept at a higher temperature more than the melting point of the material, as compared with the temperature of the lower one. The baffle serves to nearly isolate the temperature profiles of the two halves from each other. Hence a steep temperature gradient is usually achieved. In the growth process, the melt in the lower tip of the crucible is frozen first by lowering it slowly through the temperature gradient (4), defining the effective change from the liquid to solid and it is here that the single nucleation event is required to grow subsequently as a single grain through the rest of the material as it solidifies. The tapered narrow end of the crucible or ampoule favours single nucleation or one of the nuclei to survive and grow further as the lowering of the ampoule and gradual solidification of the melt proceeds.

One of the advantages of the vertical Bridgman technique is that one can get the crystal of required shape and dimensions pre-determined by means of the crucible/ampoule configuration. A major drawback of the technique is that there is no control over the orientation of the resulting crystals. In many cases the size of the grown crystal is relatively massive and there is sufficient material to enable a specimen crystal of the required orientation to be cut from the bulk. If a particular orientation is required in the complete as-grown crystal, seeding of the melt is necessary(5). The melt and crystal are in contact with the walls of the crucible during crystallization. So during cooling process, due to the difference in thermal expansion coefficients of the crystal and the crucible, the crystal may build up internal stresses.

Garg et al(6) have grown single crystals of p-type SnSe using the Bridgman technique. The grown crystals were approximately 3 cm long and 1.5 cm in diameter. Elkorashy et al(7) and Valiukonis et al(8) have also grown these crystals using the same technique. Yu et al(9) have grown single crystals of SnSe about one cm long and one cm in diameter by vapour transport technique.

$SnSe_2$ single crystal by iodine vapour transport method was grown by Domingo et al(10), for which temperatures of 500°C at the ingot position and 400°C in the region of crystal growth provided the best temperature gradient. The duration of growth period varied from 2 to 72 hrs. in this method. The crystals exhibited cleavage parallel to growth axis. Bertrand et al(11) have also grown these crystals by vapour phase growth.

W.Albers et al(12) have grown the $SnSe-SnSe_2$ p-n multilayered structures from an eutectic melt. They have shown that the lamellar $SnSe-SnSe_2$ eutectic exhibited 10^3 to 10^4 p-n heterojunctions per cm of length. A simple chemical vapour etching techniques is discussed which enables the components of the eutectic to be identified. Bhatt et al(13) have grown SnSe and $SnSe_2$ crystals using Bridgman-Stockbarger technique.

III. EXPERIMENTAL RESULTS :

3. Crystal growth of SnSe :

A mixture of very pure (99.999%) Sn and Se (supplied by Nuclear Fuel Complex, Hyderabad) in the atomic rato Sn/Se=0.99 (1% excess Se) were taken in a quartz tube 12 to 15 cm long and about 1.8 cm in diameter which was sealed under a vacuum of 5×10^{-5} torr. Then it was put into another quartz tube having a little larger diameter and was sealed under a vacuum of 5×10^{-5} torr in order to prevent oxidation of the material since the latter expands, like GaAs and GaP, during solidification, frequently causing the inner tube to break as the temperature reaches about 500°C.

The ampoule was kept in the alloy mixing unit (Ref. Chapter 2) at a constant temperature 100° above the melting point, 860°C, for a period of two days. During this period, the ampoule was continously rotated and rocked and then cooled slowly to 500°C. This temperature was then maintained for further 24 hrs. Then the temperature was lowered to room temperature over about 24 hrs. The temperature was measured and controlled with a proportional temperature controller (within ± 5°C)[Thyristor Power Controls Pvt. Ltd., Banglore - 86].

The ampoule with the ingot was kept in the vertical Bridgman furnace for single crystal growth. The length of the core of the furance was about 45 cm. A baffle was kept at 23cm. Inner diameter of the core was 5 cm. The upper furnace was kept at a higher temperature than the lower. The maximum temperature was set to 925°C in the upper furnace and the lower furnace was set to 325°C. The temperature profile of the double furance is shown in Figure - 1 together with the starting position of the ampoule. The ampoule was kept steady for 24 hrs in the upper hot zone of the furnace and was unidirectionally solidified. For this, different growth rates in the range of 4 mm/hr. to 15mm/hr. and temperature gradients from about 30°C/cm to 70°C/cm were tried out. The growth rate and the temperature gradient, 4mm/hr. and 55°C/cm, respectively, were found most suitable for obtaining fairly good crystals of SnSe. The crystals obtained were about 2.5 cm in length and 1.5 cm in diameter. Crystals could be easily cleaved. Microscopically the cleavage plane of the crystal was found to be fairly planar. The growth axis was found to have an inclination of 45° to 60° with (001), the cleavage plane.

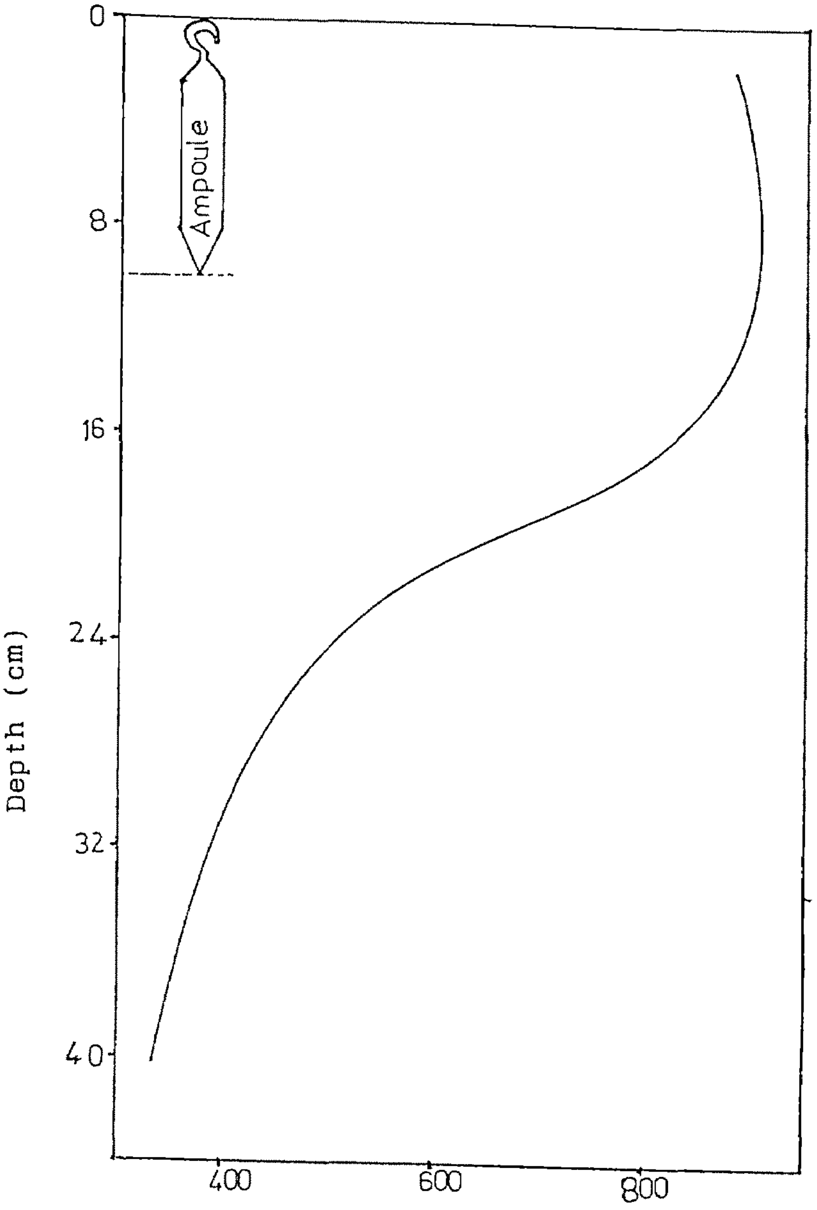

FIGURE - 1 : Temperature profile of furnace.
(SnSe crystal growth)

The single crystalline character was checked by Laue back reflection pattern. A typical Laue photograph obtained for a cleavage slice about 1.2 mm thick is shown in Figure - 2. The photograph was obtained with tungsten white radiation and an exposure time of 1½ hr. The distance between the film and the sample was 2 cm. The photograph displays the four fold symmetry indicating the cleavage plane to be (001). The sharpness of Laue spots shows good perfection of the crystal.

Powdered samples from different parts of the crystal were used to obtain X-ray diffractograms. The X-ray diffractometer, Philips PM 8203 with pw 1390 channel control and pw 1373 goniometer was used. It was operated at 35 to 40 KV and 20 mA with Cu target (λ =1.5418 Å). A typical diffractogram is shown in Figure - 3. The peaks observed were found to correspond to SnSe. They were indexed on the basis of lattice parameters: a=4.33 Å, b=3.98 Å and c=11.18 Å (14). The indices are also shown in the figure. The observed and calculated d values are given in Table-1 for comparison. It can be seen that there is a fairly good agreement between the two. It is further important to note here, that there is no $SnSe_2$ peak observed in the diffractogram, indicating the material to be single phased. In the report by W.Albers (12), the sample was observed to contain $SnSe_2$ precipitates, while in the present case there is no observable evidence of such precipitates. This may be because of the compound preparation method and a much lower growth rate used in the present study.

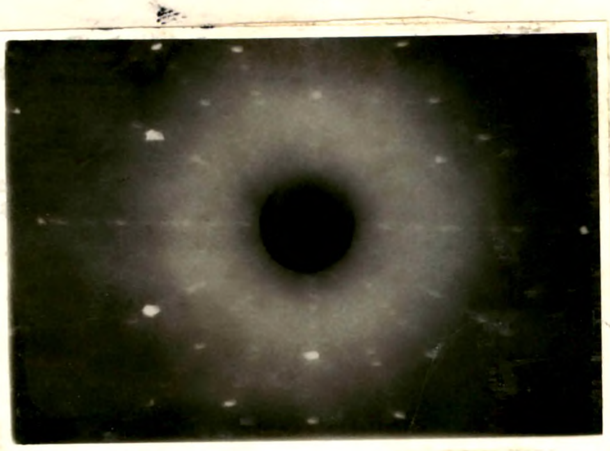

FIGURE - 2 : Laue back reflection pattern of the cleavage plane (001) of SnSe single crystal.

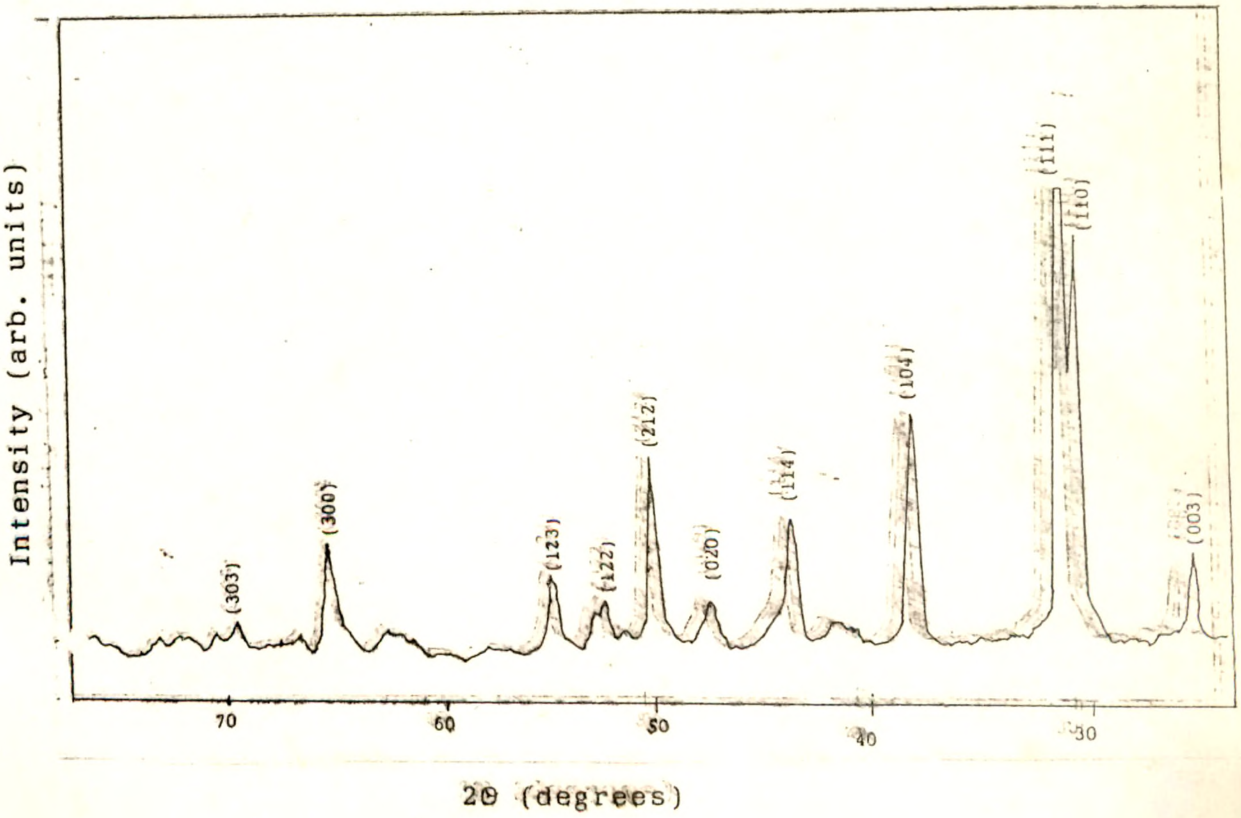

FIGURE - 3 : XRD plot of SnSe powder

TABLE - 1

Observed and calculated 'd' values

(SnSe powder)

hkl	d (Å) observed	d (Å) calculated.
003	3.801	3.727
110	2.936	2.930
111	2.869	2.834
104	2.329	2.348
114	2.004	2.022
020	1.898	1.990
212	1.796	1.800
122	1.703	1.720
123	1.625	1.627
300	1.409	1.443
303	1.341	1.346

3.2 Crystal growth of $SnSe_2$:

For the crystal growth of $SnSe_2$, Sn and Se were mixed in atomic ratio 1 : 2 with 1% excess Sn(12) and both were sealed together under a vacuum of the order of 5×10^{-5} torr in a quartz tube of about 12 to 15 cm in length. Mixing and homogenization was carried out by the same method as used for SnSe. The maximum temperature of the upper Bridgman furnace in this case was 700°C (m.p. of $SnSe_2$ is 657°C) while it was 150°C in the lower half. The temperature profile of the double furnace is shown in the Figure - 4. The ampoule containing the charge was lowered in the vertical Bridgman furnace at different rates and temperature gradients. After various trials, it was found that with 4mm/hr rate and 33°C/cm gradient, good crystals can be obtained. The crystal size obtained was about 2.5cm x 1.5cm. The growth axis was found almost invariably to be perpendicular to (0001) and consequently the cleavage plane which is (0001) had horizontal orientation. The crystals exhibited very easy, but at the same time, quite soft cleavage, very often yielding a bent or severely deformed cleavage slice. A back reflection Laue photograph (tungsten white radiation) of a cleavage slice is shown in Figure - 5. The six fold symmetry of the pattern confirms the cleavage plane to be (0001). The blurred elongated nature of spots is a result of cleavage induced deformation of the specimen.

XRD obtained from powdered samples taken from different parts of the ingot confirmed the formation of the compound

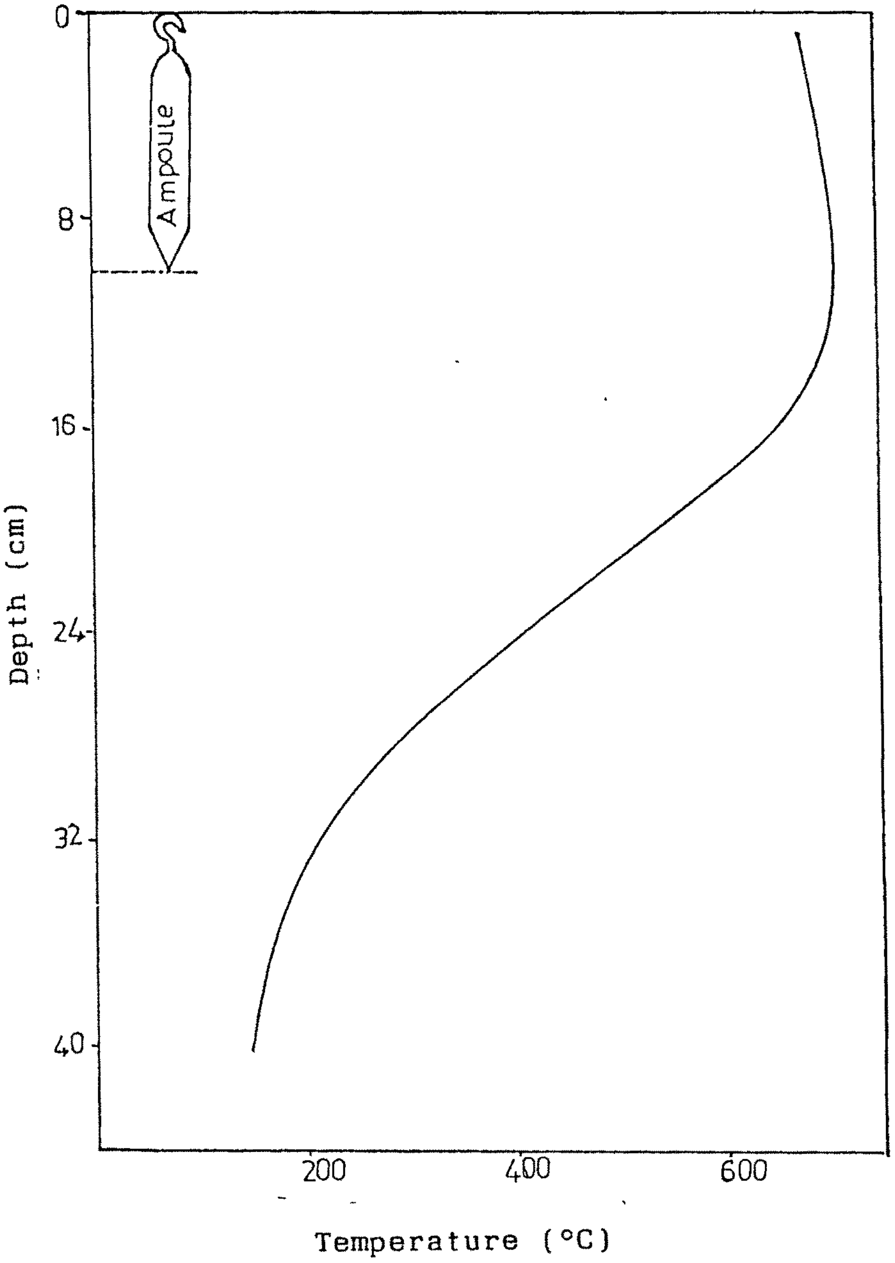

Figure - 4 : Temperature profile of the furnace.
(SnSe Crystal growth).

TABLE - 2

Observed and calculated 'd' values

($SnSe_2$ powder)

hkl	d (Å) observed	d (Å) calculated
001	6.128	6.137
002	3.071	3.069
110	2.899	2.907
112	2.248	2.247
003	2.049	2.046
111	1.816	1.820
004	1.530	1.534

$SnSe_2$. The diffractogram is shown in Figure - 6. The diffraction peaks could be indexed on the basis of the lattice parameters :

a = 3.811 Å, c = 6.137 Å, (Garg et.al) (6). The observed and calculated d values are compared in Table - 2.

3.3 SnSe - $SnSe_2$ Eutectic Crystal Growth :

The phase diagram of the Sn-Se system also exhibits the eutectic between the compounds SnSe and $SnSe_2$ at 640°C and at 61 at. % Se. To obtain this crystal, a mixture of very pure Sn and Se (5 N) were taken in the 39:61 atomic ratio (12) together in a quartz tube of 12 to 15cm in length and about 1.8cm in diameter. The tube was evacuated to a vacuum of the order of 5×10^{-5} Torr and was sealed. This evacuated tube with the material was kept in the alloy mixing furnace and was continuously rocked and rotated, for 48 hrs. Then the molten charge was slowly cooled to room temperature over a period of two days. The ingot was then subjected to Bridgman crystal growth. The temperature profile used is shown in Figure - 7. The crystals were grown at various rates and under various temperature gradients. While in most of the cases it was difficult to obtain single crystals, at a rate of 4mm/hr and 50°C/cm temperature gradient, crystals having grains of large size could be obtained and a sizeable part of the ingot was found to be a single crystal.

Powdered samples from different parts of the ingot were used for obtaining X-ray diffractogram. The X-ray diffractogram

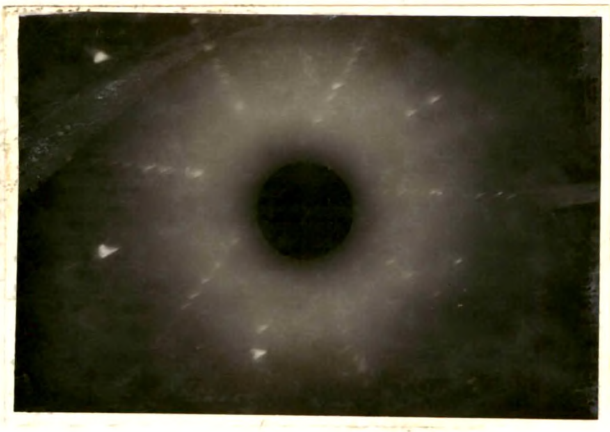

Figure - 5 : Laue back reflection pattern of the cleavage plane (0001) of $SnSe_2$ single crystal.

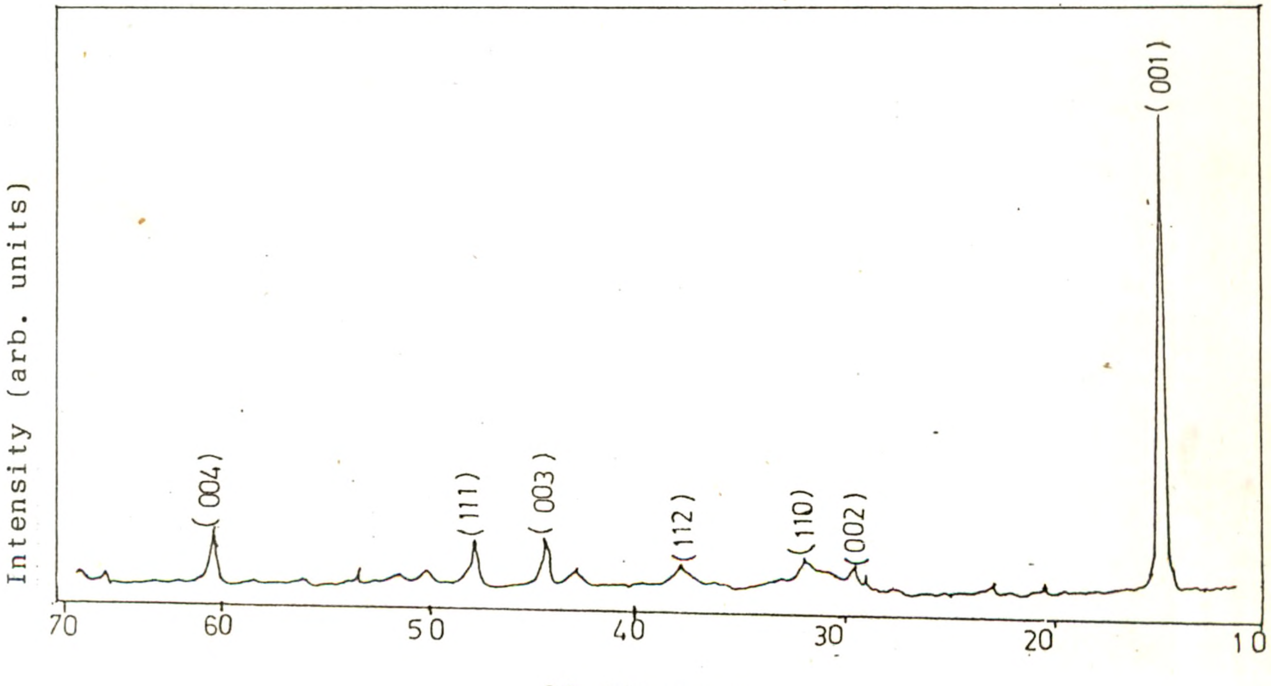

Figure - 6 : XRD plot of $SnSe_2$ powder

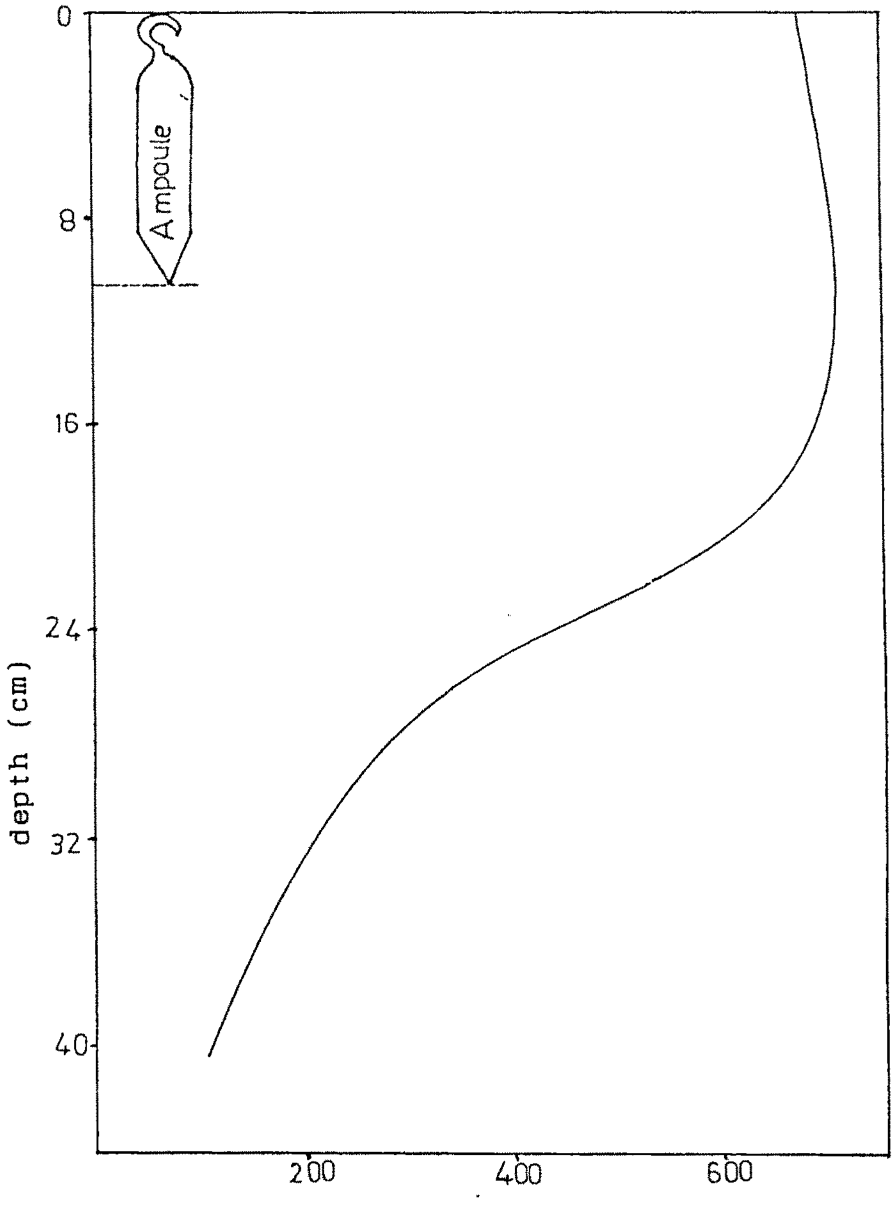

Figure - 7 : Temperature profile of the furnace. ($SnSe$-$SnSe_2$ eutectic crystal growth).

is shown in Figure - 8. A comparison of the calculated and observed d values, as given in the Table - 3, shows the presence of SnSe and $SnSe_2$. The eutectic has been reported to have a lammellar structure consisting of alternate n and p type layers, that is SnSe and $SnSe_2$(12). The mutual structure orientation has been reported to be as illustrated in the Figure - 3 of the Chapter 1. Thus it is likely that the crystals grown in the present study also may have similar structure since the diffractogram indicates the presece of these two compounds only.

Section - II

Thin Films :

1. INTRODUCTION :

The dramatic development of electronics favours continuous progress and improvement of knowledge in the field of material science applied to thin films used in electronic devices. The most important requirements of the growth processes for obtaining semiconductor films are that they provide a film of uniform thickness in a wide interval of doping and stability with time. The substrate plays an important role in the epitaxial growth of the films, their structure orientation and properties. The term 'epitaxy', meaning "arrangement on", was first introduced by Royer(15) to denote the phenomenon of oriented over growth of one crystal on another. In the present study different substrates like glass, mica and NaCl crystals had been used for $SnSe_2$ films in

TABLE - 3

Observed and calculated 'd' values

($SnSe - SnSe_2$ eutectic powder)

hkl	$SnSe/SnSe_2$	d (Å) observed	d (Å) calculated
001	$SnSe_2$	6.140	6.137
120	SnSe	3.449	3.493
002	$SnSe_2$	3.011	3.068
111	SnSe	2.869	2.834
004	SnSe	2.783	2.795
113	SnSe	2.319	2.303
003	$SnSe_2$	2.058	2.047
020	SnSe	2.001	1.990
111	$SnSe_2$	1.889	1.820
212	SnSe	1.807	1.800
200	$SnSe_2$	1.650	1.650
124	SnSe	1.511	1.518
300	SnSe	1.435	1.443
121	$SnSe_2$	1.227	1.223
114	$SnSe_2$	1.195	1.195

particular for improving their electrical characteristics.

2. GENERAL DISCUSSION :

Royer(15,16) put forward three rules of epitaxy. One of the important rules is that when there exists a parallelism of lattice planes (one of the substrate and the other of the deposit) which have identical or nearly identical atomic network and have similar lattice spacings, an oriented overgrowth can take place. The difference between the network spacing of the substrate surface and the deposit plane is generally expressed in terms of percentage misfit which can be defined by the expression (in percentage) 100 (b-a)/a. Here 'a' and 'b' represent the network spacings in the substrate and in the film, respectively. Frankand Van der Merwe in 1949 treated the substrate as a two dimensional network(17) and theoretically derived the maximum limit to be 15 % misfit permissible for the formation of the oriented growth.

Recently, Goswami(18) and Pashley(19) have worked on epitaxial growth which indicated that an oriented overgrowth can be obtained even when the misfit is as large as 50%. This questions the validity of the Van der Merwe's conclusions. Many theories and explanations have been put forward about the oriented over-growth. It is a fact that none of the existing theories is general or applicable to all the cases consistently. However, one fact becomes conclusive that the growth of a first few layers is governed by the substrate surface and hence will be epitaxial (with respect to substrate) and successive epitaxial overgrowth will be controlled by the substrate-deposit pair, lattice fitting, substrate temperature, deposition rate etc. It is probable that the orientation of first few layers would determine the final orientation of the over-growth.

Now-a-days the attention of a number of leading laboratories in this field is diverted to the studies on epitaxial growth by new techniques like organometallic method(20,21) and defect characterization of the composite materials.

Marianio and Chopra(22) studied polymorphosim in some IV-VI compounds induced by high pressure and thin epitaxial growth. According to them, epitaxial growth, on rock salt, of thin films of normally orthorhombic SnS, SnSe and $PbSnSe_2$ induces NaCl structure of the film. There exists close correlation between the lattice constants of the corresponding NaCl and Orthorhombic polymorphic structures of Pb and Sn compounds.

According to Fernandez-Samuel et al(23), $SnSe_2$ exhibits the $2HCdI_2$ structure in bulk (single crystal) form. The as-deposited films are amorphous in nature; on annealing at 200°C, it undergoes an amorphous to crystalline transformation. If the annealing temperature is raised to about 300 to 500°, it gives rise to several other phases.

3. EXPERIMENTAL RESULTS :

Film preparation of SnSe, $SnSe_2$ and SnSe-$SnSe_2$: The homogeneous compound/alloy prepared as mentioned earlier was used for the thin film preparation. Thermal evaporation by resistive heating method had been employed with a molybdenum boat as the resistive heat source. During the evaporation the pressure was reduced to 10^{-5} torr. Glass, freshly cleaved NaCl and Mica were used as substrates. Prior to deposition the substrates were thoroughly cleaned. Preliminary cleaning of glass slides was done by rinsing them with detergent, washing in distilled water and then they were hot-air-dried. NaCl and Mica substrates were obtained as freshly cleaved crystal surfaces. The substrates were then heated to a temperature of about 200°C in the vacuum chamber at a reduced pressure of 10^{-5} torr to remove the volatile contaminants, if any. The deposition rate was about 50 Å/Sec. The deposition rate and the thickness of the film being deposited were measured in-situ by the use of quartz crystal oscillator as described in Chapter 2. Films at different substrate temperatures and of different thicknesses and geometries were deposited for various measurements.

3.1 SnSe thin film :

Films of various thicknesses were deposited at various temperatures on glass substrates, from the SnSe compound. The comparative study of the X-ray diffractograms indicated that there is an increase in intensities of the peaks in the case of films deposited at a substrate temperature of 200°C. This indicates that the crystallinity of the film increases with substrate temperature. A typical X-ray diffactogram is shown in Figure - 9. The only strong and prominent peak observed corresponds to (111) plane (indexed in terms of orthorhombic SnSe lattice). The d values are listed in Table - 4.

3.2 Solid State reacted SnSe film :

For preparing SnSe films, the method of solid reaction(24) of multilayer alternate deposits of Sn and Se was also used. Tin and Selenium were taken in two separate molybdenum boats. They were alternately evaporated onto a glass substrate at room temperature at a reduced pressure of 10^{-5} torr. Using the multifilament turret, alternate layers of Sn and Se were deposited with number of layers ranging from 3 to 9. Each layer was of equal thickness. The deposition rate of each layer was maintained at about 50 Å/Sec to achieve the desired layer thickness. The thickness was measured in-situ by a quartz crystal thickness monitor. Since selenium has a high vapour pressure, among the 3,5,7 and 9 layers in the film, the first and the last layers were of tin to minimize sublimation loss of Se during heat treatment. After completion of deposition, the temperature was raised from room temperature to 200°C at a rate of about 5°C/min. The temperature was then maintained constant for a duration of 2 to 3 hrs to achieve the reaction(2,3). Then the film was cooled gradually down through 2 hours to room temperature. X-ray diffractograms of the films of various total

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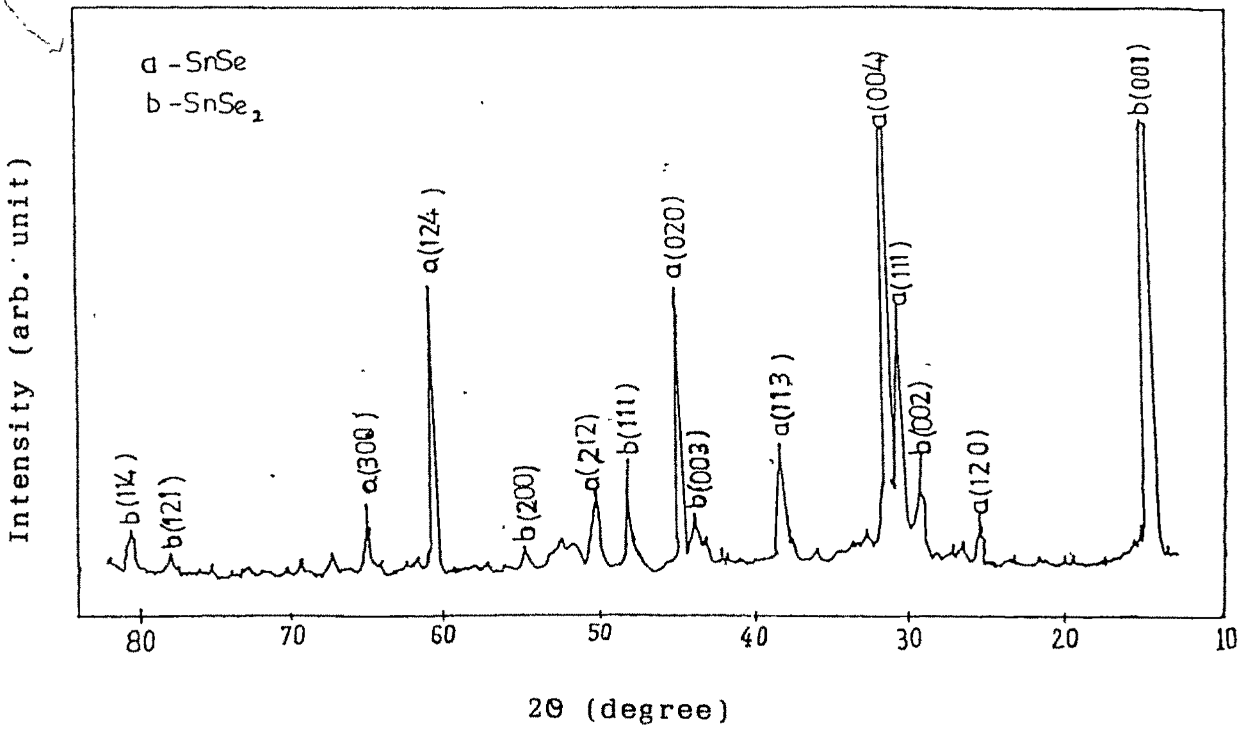

Figure - 8 : XRD Plot of SnSe-SnSe$_2$ eutectic powder.

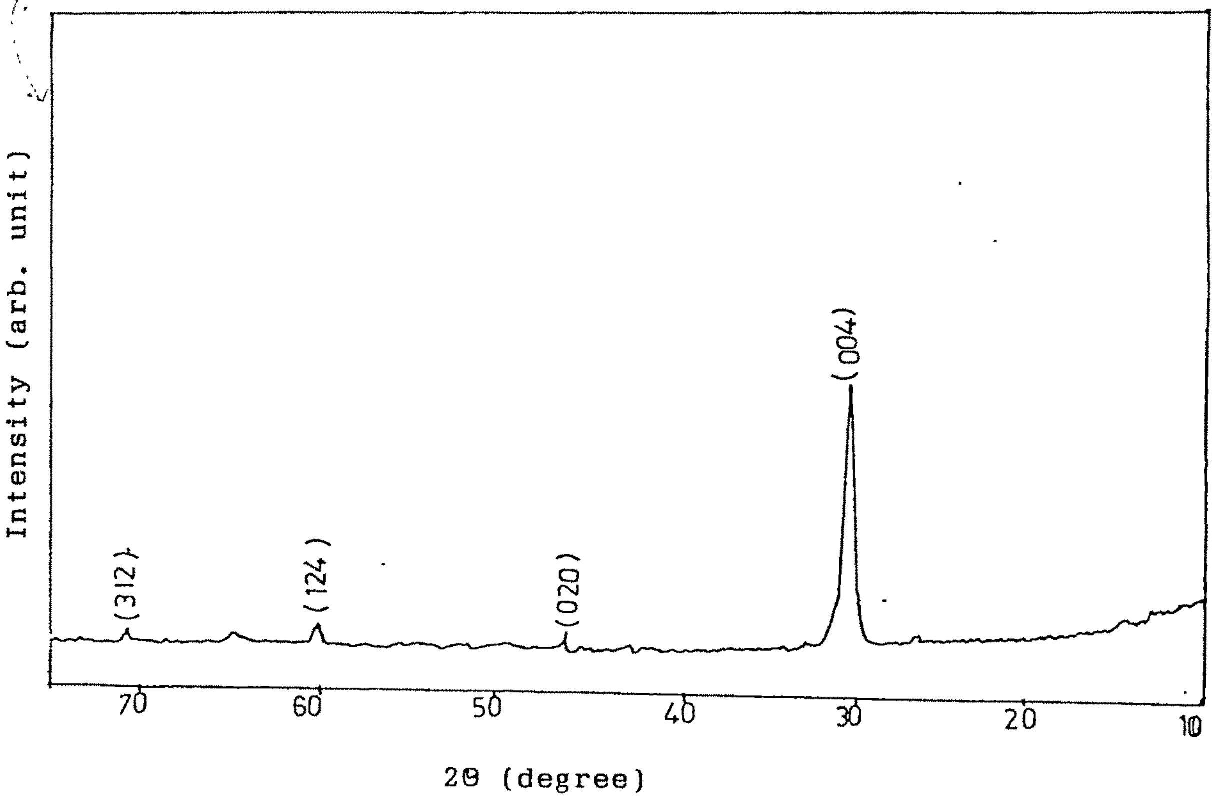

Figure - 9 : XRD plot of SnSe thin film directly evaporated from the compound. (Substrate temperature, 200°C)

TABLE - 4

Observed and calculated 'd' values

(SnSe film)

hkl	d (Å) observed	d (Å) calculated
004	2.783	2.795
020	1.996	1.990
124	1.515	1.518
312	1.318	1.318

TABLE - 5

Observed and calculated 'd' values

(SnSe; synthesis by solid state reaction)

hkl	d (Å) observed	d (Å) calculated
011	3.739	3.749
111	2.835	2.834
222	1.419	1.417
311	1.344	1.346
132	1.235	1.237

thicknesses were obtained. The peaks corresponded to the SnSe d values within 0.02 Å. The observed and calculated d-values are listed in Table - 5.

A typical X-ray diffractogram for the films of thickness 1200 Å obtained as described above is shown in Figure - 10. The most prominent peak observed in the diffractogram and corresponding to 110 indicates that the multilayer solid state reacted SnSe films have a preferential texture of (111).

3.3 $SnSe_2$ thin films :

Using above techniques, films of various thicknesses were deposited on glass substrate at various temperatures ranging from room temperature (30°C) to 200°C. From the X-ray diffractogram study, it was observed that in the case of as-grown films, there is no noticeable peak, indicating amorphous nature of the films. For the films deposited at 200°C, well defined peaks were obtained (Figure 11). The peaks are indexed in terms of the hexagonal $SnSe_2$ lattice. The observed and calculated d-values are shown in Table - 6. The films were also deposited on mica and NaCl substrates in order to possibly improve their electrical conductivity (Chapter - 4).

3.4 Films of SnSe-$SnSe_2$ Eutectic :

Films of different thicknesses were deposited at different substrate temperatures ranging from room temperature

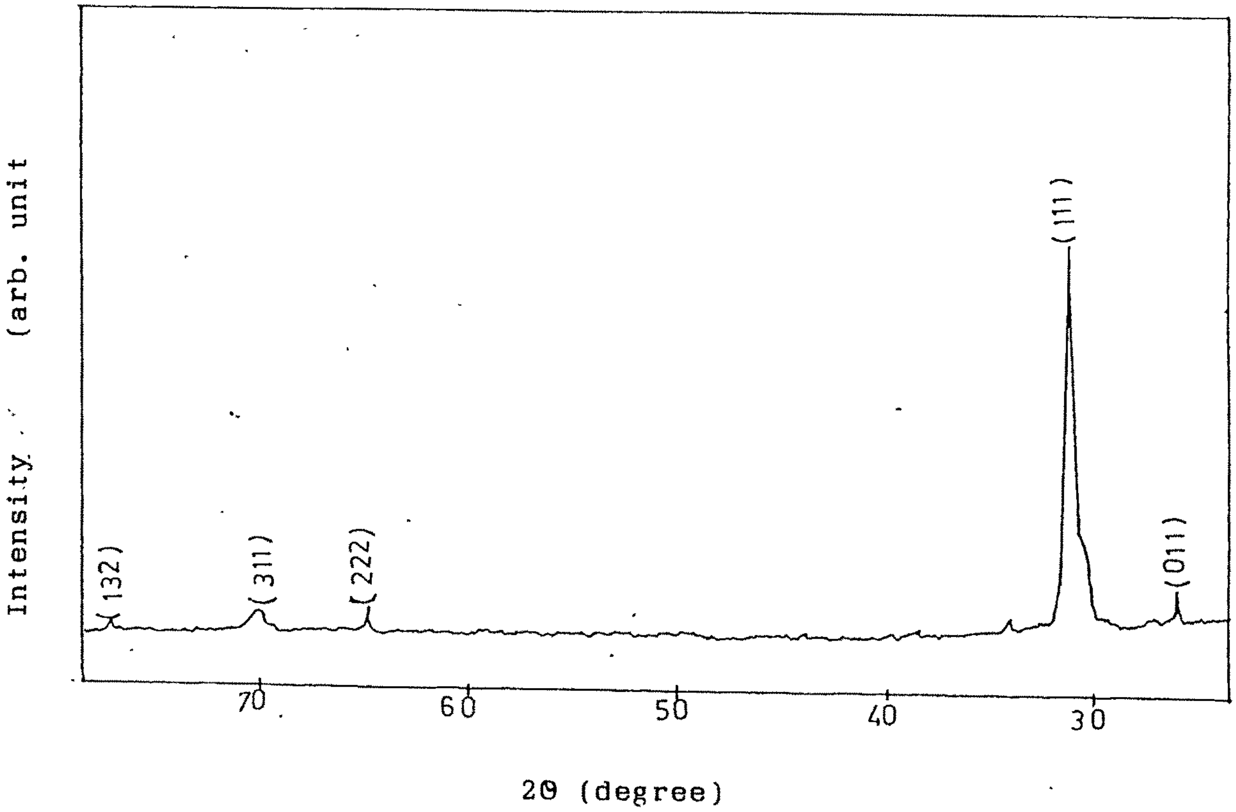

Figure - 10 : XRD plot of SnSe film obtain by solid state reaction.

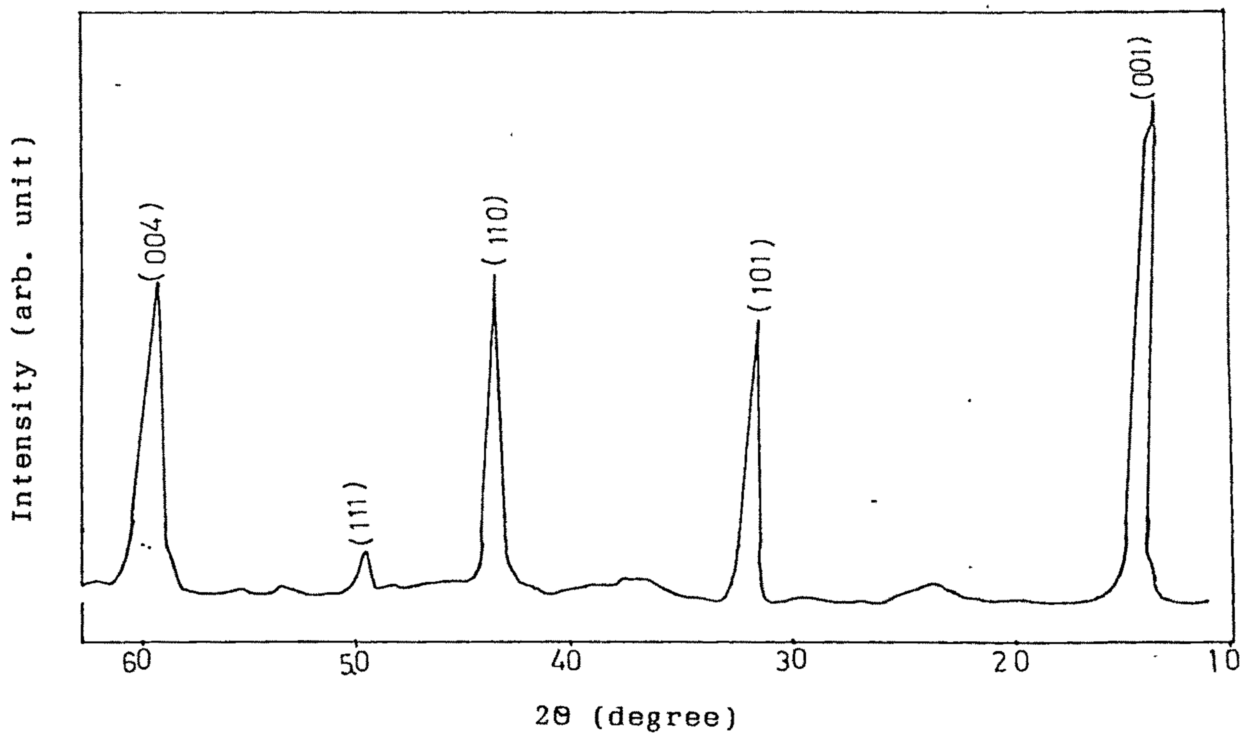

Figure - 11 : XRD Plot of $SnSe_2$ film.
(Substrate temperature 200°C)

to 150 °C. The X-ray diffractions of the films were found to contain the peaks corresponding to both SnSe and $SnSe_2$. The observed and calculated d-values are listed in Table - 7. The corresponding diffractogram is also shown in Figure - 12.

4. CONCLUSIONS :

Crystals :

Fairly large, about 2.5 cm x 1.5 cm., and good quality crystals of SnSe can be obtained by the Bridgman-Stockbarger technique at a lowering rate of 4 mm/hr and under a temperature gradient of about 55°C/cm. Crystals have fairly plane and easy cleavage along (001).

Similarly good quality $SnSe_2$ single crystals can be grown by the Bridgman-Stockbarger technique at a lowering rate of 4 mm/hr and under a gradient about 33°C/cm. They have an easy but a very soft cleavage along (0001).

Growth of single crystals of SnSe-$SnSe_2$ eutectic is difficult and at the most, large crystallities can be obtained by the Bridgman - Stockbarger technique at a lowering rate of 4 mm/hr and under a temperature gradient of 50°C/cm.

THIN FILMS :

Thin films of SnSe obtained by evaporation of the compound at a pressure of 10^{-5} torr exhibit increasing

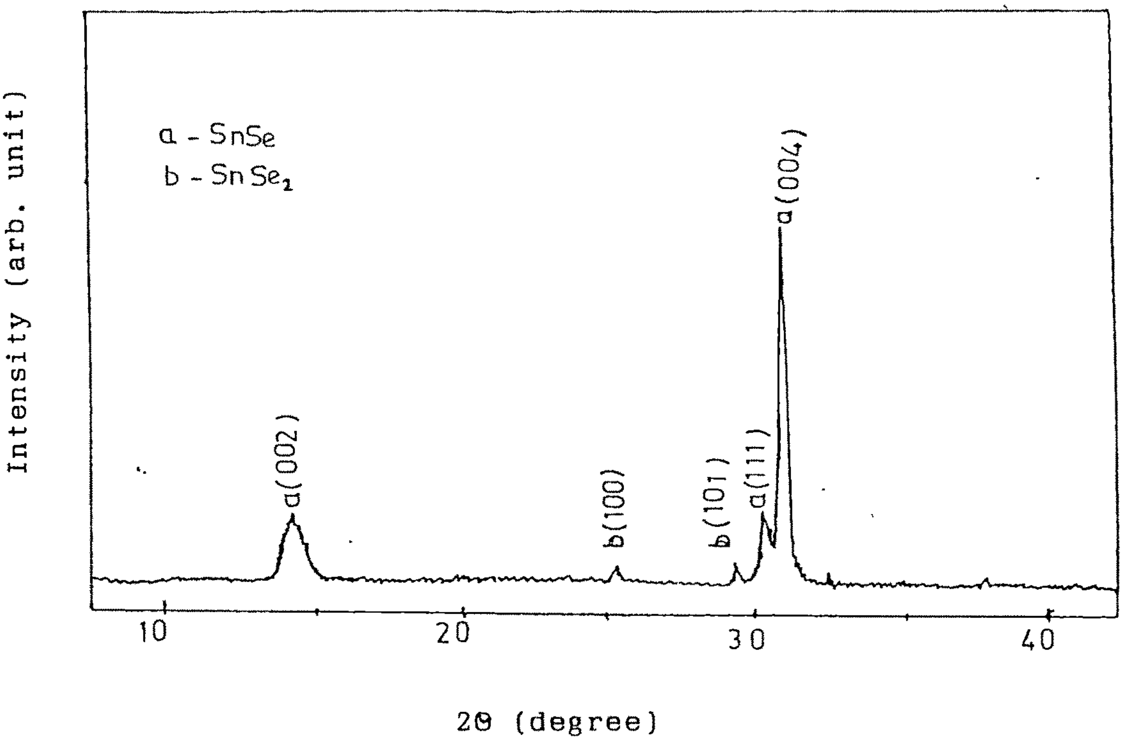

Figure - 12 : XRD plot of films obtained from evaporating SnSe-SnSe$_2$ eutectic. (Substrate temperature 150°C)

TABLE - 6

Observed and calculated 'd' values

($SnSe_2$ film)

hkl	d (Å) observed	d (Å) calculated
001	6.139	6.137
101	2.905	2.907
110	1.909	1.906
111	1.809	1.819
004	1.538	1.534

TABLE - 7

Observed and calculated 'd' values

(SnSe-$SnSe_2$ eutectic film)

hkl	SnSe/$SnSe_2$	d (Å) observed	d (Å) calculated
002	SnSe	5.584	5.590
100	$SnSe_2$	3.308	3.301
101	$SnSe_2$	2.908	2.907
111	SnSe	2.839	2.834
004	SnSe	2.795	2.795

crystallinity with increasing substrate temperature upto 200°C at which they have (111) texture. Similarly the $SnSe_2$ films exhibit well defined XRD peaks if deposited at 200°C.

Snse films can also be successfully prepared by solid state reaction of alternate elemental layers.

Films of $SnSe-SnSe_2$ eutectic can be obtained by evaporation of the alloy at 10^{-5} torr. Good crystalline films can be obtained at a substrate temperature of 150°C.

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