CHAPTER – 3

DEPOSITION OF ZnX (X=Se, Te) THIN FILMS

3.1 INTRODUCTION

In recent years thin film science and related technology has grown world-wide. Generally, thin films are denoted as films with the thickness of less than some micrometers and which are produced in the vacuum through evaporation or cathodic sputtering. Thin films of metals were proposed first in 1857 by Michael Faraday. In the initial stages, use of thin films was centered on the preparation of anti-reflecting for lenses, multilayer interference filters, automobile headlights and decorative coatings. In the beginning of the 20th century, industrialization of thin film technology has been started. Thomas Edison invented a procedure of metallizing wax molds using cathode sputtering. He applied in 1903 for US patents No. 767216. This patent proposed a bell jar installation with two vacuum which containers which are evacuated with a pump stand (without valve) to sputter alternatively. The first use of thin films for optical purpose can be dated to exactly 1912, when Paul and Pringsheim published their work about production of mirrors.

In recent years the importance of coatings and synthesis of new materials for industries have resulted in a tremendous increase of innovative thin film processing technology. Currently this development goes hand-in-hand with the explosion of scientific and technological break-through in microelectronics, optics and nanotechnology [1, 2]. A second major field comprises process technologies for films with thickness ranging from one to several microns, which are essential for multitude of production areas such as thermal barrier coatings and wear protection [3, 4]. Presently, rapidly changing needs for thin film materials and devices are creating new processes materials and technologies as shown in figure-3.1.
Therefore, basic research activities will be necessary in the future, to increase knowledge, understanding and to develop predictive capabilities for relating fundamental physical and chemical properties to the microstructure and performance of thin films in various applications. In basic research, special model systems are needed for quantitative investigations of the relevant and fundamental processes in thin film materials science. In particular, these model systems enable the investigation regarding thin films i.e. nucleation and growth processes, solid state reactions, the thermal and mechanical stability of thin film systems and phase boundaries. Results of combined experimental and theoretical investigations are a prerequisite for the development of new thin film systems and the tailoring of their microstructure and performance.
3.2 REVIEW OF THIN FILM PREPARATION TECHNIQUES

Thin films play an important role in present technological development and depending on application and material properties there could be more than one technique for deposition of thin films. The overview of classification of thin film deposition techniques is shown in figure 3.2.

![Thin Film Deposition Techniques Diagram]

**Figure 3.2** Various thin film deposition techniques.
3.2.1 VACUUM EVAPORATION

This technique is one of the oldest techniques for deposition of thin films. Today also it is very widely used in research laboratories as well as industries. There are three basic stages of this technique.

1. generation of vapor from condensed phase
2. transfer of vapor from the source to the substrate in vacuum
3. condensation of vapor molecules on the surface of the substrate

When evaporation is made in vacuum, the evaporation temperature is considerably lowered and the incorporation of oxides and other impurities is reduced strongly.

3.2.1.1 THERMAL EVAPORATION

There are different methods of evaporating the materials to be deposited. The commonly used method for evaporation of many materials is the thermal evaporation technique, in which the boat or the container of material has high melting point and very low vapor pressure. The materials used are tungsten, tantalum, molybdenum, etc. They are heated electrically during the process of evaporation. The boat and wire helix sources are shown in figure 3.3.

Figure 3.3   Evaporation sources (A) Boat and (B) Wire helix.
Even though new and modified techniques have been developed, electrical resistive heating sources are still used extensively to prepare thin films of elements [5-11], oxides [12-16], dielectrics [17] and semiconductor compounds [18-24].

3.2.1.2 FLASH EVAPORATION

In this technique, a small quantity of material to be evaporated are dropped in powder form on to the source (boat) which is hot enough to ensure the evaporation process be taken place instantly. This technique is useful in the preparation of multi-component alloys or compounds that tend to distill fractionally. Varieties of methods (ultrasonic, mechanical, electromagnetic etc.) are used to drop the powder on the boat.

A disadvantage of this technique is the difficulty in pre-outgassing the evaporant powder. Degassing the powder can be accomplished to some extent by vacuum storage for 24 – 36 hours prior to deposition. Otherwise a large quantity of the material is to be used during evaporation process.

3.2.1.3 ELECTRON BEAM EVAPORATION

In this technique, a stream of electrons is accelerated by a voltage of 5 to 10 kV. This stream of electrons is focused on to the surface of the material which is contained in a water cooled crucible. Because of that the kinetic energy of the electrons is transferred very rapidly in to thermal energy, and the material starts melting at the surface and evaporates.

With this method, high purity films can be prepared. The rate of evaporation is comparatively higher than that of resistive heating.

This technique is rather expensive and complicated so its use is not justified if the easier way of evaporation like thermal evaporation is available and suitable for the particular material.
3.2.1.4 LASER EVAPORATION

A laser beam is used as a thermal source to vaporize the evaporant material. The source of power for evaporation is kept outside the vacuum system. The vaporized material is deposited on to the substrates placed in the front of the source material inside the vacuum chamber.

Laser is clean and it introduces minimal contamination from the heat source. Film contamination from the support material is reduced by this method because of surface evaporation characteristics of the beam. Moreover, high melting point materials can be vaporized at high deposition rates using this technique.

3.2.1.5 ARC EVAPORATION

In this method, an arc is struck between two electrodes, producing a temperature high enough to evaporate the materials of the conducting electrodes. This method is not easily reproducible.

3.2.1.6 RADIO FREQUENCY HEATING

In this method, suitable arrangement of r.f. coils levitation and evaporation can be achieved, thereby eliminating the possibility of contamination of the film by support crucible. The evaporant can also be contained in a crucible, which is surrounded by the r.f. coils.

This is not a commonly used technique for preparation of thin films because of the difficulty in positioning the coil and the sample properly, for effective coupling in a vacuum system. Also the method requires relatively expensive and bulky r.f. heating equipments.

3.2.2 SPUTTERING

Sputtering has become one of the most versatile techniques in thin film technology for preparing thin solid films of almost any material. When the surface is bombarded with high velocity positive ions, it is possible to cause the ejection of the surface atoms. This process of the ejecting atoms from the surface by the bombardment of positive ions usually
inert is commonly known as sputtering. The ejected atoms can be made to condense on a substrate to form a thin film. Several works on the subject of sputtering and sputtering processes of thin films deposition are available.

Some advantages of sputtering technique for thin film deposition are:
1. High uniformity of thickness of films
2. Good adhesion to the substrate
3. Better reproducibility of films
4. Ability to deposit and maintain the stoichiometry of the original target composition

3.2.2.1 GLOW DISCHARGE DC SPUTTERING

It is a simplest system for the deposition of thin films by sputtering. Here the plate of the material to be deposited is connected to a negative d.c. voltage supply (1 to 5 kV) and the substrate is mount on the anode facing the target. A glow discharge is initiated by introducing a neutral gas such as argon, in to the vacuum chamber to a pressure of $10^{-1}$ to $10^{-2}$ Torr. When the glow discharge starts, the positive ions strike the target plate, removing mainly neutral atoms from the surface of the target, which eventually condense as a thin film on a substrate.

The main drawback of this technique is the contamination of the deposited film by the inert gas used to produce the discharge.

3.2.2.2 TRIODE SPUTTERING

An alternative method to increase the ionization and sustain the discharge at low pressure is to supply additional electrons from source other than the target cathode. In triode sputtering the electrons are provided in the discharge from an independent source. A hot cathode emits electrons through thermionic emission. This cathode is used to inject electrons in to discharge system. A supported discharge system like this allows operation at much lower pressure ($10^{-3}$torr).
The main limitation of this technique is the difficulty in producing uniform sputtering from very large flat target. Also the supported discharge is hard to control, for reproducible results.

3.2.2.3 RADIO FREQUENCY SPUTTERING

In the sputtering technique described earlier, the target used should be a conductor. If this metal target is replaced by an insulator, a surface positive charge is built up on the front surface of the insulator during ion bombardment and prevents further ion bombardment. It has been shown that this charge builds up can be prevented by simultaneously bombarding the insulator with ions and electron beams. In r.f. sputtering system the r.f. potential is applied to the metal electrode placed behind the dielectric plate target. At r.f. potential the electrons oscillating in the alternating field have sufficient energies to cause ionizing collision and the discharge will be self sustained. Since the electrons have much higher mobility than ions, many more electrons will reach the dielectric target surface during positive half cycle and target will become self–biased negatively. 13.56 MHz frequency is normally used for r.f. sputtering.

The main advantage of this technique is to do more economical deposition of high melting point oxides and refractories on to large substrates of large areas.

3.2.2.4 ION BEAM SPUTTERING

Ion beam sputtering is one more useful technique for film deposition, offering independent control of the ion beam energy, as well as the current density of the bombarding ions. Here the ion beam generated at an ion source is extracted in to high vacuum chamber and directed at the target material, which is sputtered and deposited on to the nearby substrate.

In this system the directionality of the beam allows the angle of incidence (target) and angle of deposition (substrate) to be varied. Other advantage is low background pressure and the greater isolation of the substrate from the ion source.
3.2.2.5 MAGNETRON SPUTTERING

This technique has made significant progress since it is developed for the high rate of deposition of metal, semiconductor and dielectric films. In comparison to conventional diode sputtering, magnetron sputtering apart from obtaining high deposition rates at lower operating pressure, make it possible to obtain high quality films at low substrate temperatures. For a simple planer magnetic system, a planer cathode is backed by a permanent magnet that provides a toroidal field with field lines forming a closed path over the target surface (cathode). The secondary electrons generated are trapped in cycloid on it near the target and present self heating on the surface. The confinement of the plasma and the resultant intense plasma allow magnetron sputtering system to operate at much lower pressure and lower target voltages than are possible for r.f. diode sputtering. Here the deposition rates are higher and cover large deposition area.

3.2.3 CHEMICAL METHODS FOR FILM DEPOSITION

As discussed in the previous sections, the physical methods of preparation of thin films involve evaporation or ejection of material from a source. Similarly chemical methods of thin film deposition entail a definite chemical reaction.

These methods use simpler equipments and are more economical than physical methods, although the former methods are complex and difficult to control.

3.2.4 CHEMICAL VAPOR DEPOSITION

This is an important and well known technique for the preparation of thin films of elements as well as compounds on various substrates. Using this technique, films of high purity and quality, with required composition and doping level can be obtained. Since many reactions can be accomplished at ambient pressures, the need for high vacuum equipments can be avoided.
Here constituents from a vapor phase react to form a solid film on the substrate surface, which is maintained at a suitable temperature. The chemical reaction itself is an important characteristic of all CVD processes. The reactions basic to CVD include the thermal decomposition (pyrolysis), reduction, oxidation, nitriding and carbide formation and chemical transferred reactions. Flow rate, input concentration, deposition temperature, pressure and reactor geometry are process variables controlling the deposition of films.

In short, a basic CVD system for the deposition of thin films involves three main steps:
1. Transportation of the reactants to the site of reaction
2. Provision of activation energy for reaction
3. Removal of by-product gases and vapors

**3.2.5 ELECTROLESS DEPOSITION**

Deposition of films directly by chemical reaction without the application of any electrode potentials is known as electrode less deposition. The chemical vapor reaction can take place with or without catalyst. Catalytic reactions using activators also are taken as electrode less deposition.

Electroless deposition is a simple technique that does not require high temperatures and is very economical. Large area depositions are also possible with this technique.

**3.2.6 ELECTRODEPOSITION**

This is the process of depositing a substance by the passage of electric current through the conducting medium (called the electrolyte), producing a chemical change (electrolysis). The system used for electrodeposition consists of an anode and cathode immersed in a suitable electrolyte. When the electric current is passed, the material is deposited on cathode. The properties of electrodeposited films depend upon the electrolyte, the electrodes and the current density. Several semiconductor films have been prepared using this technique [25-29].
3.2.7 ANODIC DEPOSITION

The electrodeposition process discussed above is concerned with catholic reactions, while the anodization process relies on anodic reactions. Here when the electric current is passed, an oxide coating forms at the surface of the metallic anode. It means an anode reacts with negative ions from the electrolyte and forms an oxide coating. Aluminum, tantalum, silicon and zirconium are few of metals that give useful films by this simple method. This method produces amorphous continuous films, but film thickness is limited.

3.3 CHOICE OF THIN FILM DEPOSITION TECHNIQUE FOR ZnX (X = Se, Te)

A wide literature survey regarding the deposition of the thin films of ZnSe and ZnTe has been done to optimize and to choose the thin film deposition technique for the present work. It has been observed that ZnSe films can be grown by several techniques like Vacuum Evaporation [30-33, 40-43, 52-60, 62-65], Chemical Vapor Deposition (CVD) [34,45], MOCVD [35], Brush Plating[61], Electro deposition [36,49,68,69], Pulse electro deposition[67], Photochemical deposition[37], Chemical bath deposition[38,50], Pulse laser deposition (PLD)[39,44,70], MBE [44,51], solution growth[46], Spray pyrolysis [47,48,66], and atomic layer deposition[24]. ZnTe thin films can also be grown by several deposition techniques like Vacuum evaporation [71-77, 79-84, 86-88], Electro deposition [78,91,92,98-107], Closed space vacuum sublimation[85,95], R.F. Sputtering[93,94], Electron beam evaporation[89], Hot wall evaporation[96], MBE[97] etc.

The deposition technique can be chosen depending upon the type of the material, its physical properties and its intended use for various applications. Generally physical vapor deposition technique is the simplest and widely used technique for deposition of semiconducting thin films. Therefore for deposition of ZnSe and ZnTe materials, this technique has been used. Among the physical vapor deposition techniques, thermal evaporation technique has been used in the present investigations.

Thermal evaporation method is the most common method in producing thin films because of its suitability, reproducibility and high deposition rate. It is well known that the
structural and optical parameters of the deposited films are very much dependent on method of deposition. Various deposition parameters are easier to maintain and control in case of thermal evaporation technique in comparison with other techniques. This simplicity and lower cost makes this technique more common and attractive for thin film deposition. Looking to all these advantages, the physical parameters of the selected compounds (ZnSe & ZnTe) and availability of the infrastructure, it is found that in our case, the thermal evaporation technique for thin film deposition is the most suitable method.

Thermal evaporation can be carried out in two ways. Resistive and electron beam evaporation. Both of them have their own properties and merits. In present investigations thermal evaporation technique by resistive heating process is adopted for deposition of ZnSe and ZnTe thin films

### 3.4 REQUIREMENTS OF THERMAL EVAPORATION TECHNIQUE

A complete thermal evaporation technique can be divided in to three major stages as mentioned below.

1. Transformation of solid material, to be deposited in to the gaseous form
2. Transport of the created material to the substrate from the evaporation source in the form of vapor stream
3. Deposition of material on the substrate and growth of the film

According to the need of the above three stages, the requirements of the thermal evaporation technique can be discussed.

In the first stage, the transformation of the material takes place from solid to gaseous state. If this is to be accomplished by resistive heating , (as in this investigation) than it is always essential that the material of the evaporation source (boat) should withstand very high temperature (grater than the melting point or oiling point of the material to be evaporated) to be reached during the whole process. Also the power supply used for such heating of source should be capable of delivering enough amount of current.
In the second stage, the transport of gaseous form of the material from evaporation source to the substrate takes place. This process can be accomplished properly only if the mean free path of the evaporated gas molecules is greater than the distance between the evaporation source and substrate. If this condition is not fulfilled, then the vapor molecules will get diverted from the normal path due to collision with ambient gas molecules or among themselves. Therefore the pressure of the order of $10^{-5}$ torr is always necessary.

In the third stage, the nucleation and condensation of vapor molecules take place to form a continuous film. This process can be optimized if the rate of the incidence of vapor molecules at the substrate is kept less than the rate of the nucleation. Besides to minimize the incorporation of impurities in the thin film, it is always essential that the rate of impingement of the ambient gas molecules should be very small compared to that of the evaporated molecules.

Thus it is obvious that, for the deposition of thin films by thermal evaporation method, one must have high vacuum conditions in the system so as to precisely control stages 2 and 3 discussed above. To achieve a vacuum pump, capable of creating vacuum of the required amount can be created.

Also the tantalum boat was used as evaporation source, because its melting point is higher compared with that of the evaporation materials (ZnSe and ZnTe).

3.5 VACUUM COATING UNIT
3.5.1 VACUUM GENERATION

As discussed earlier, to get good quality of thin films by thermal evaporation technique, the enough high vacuum in the vacuum chamber is required. To obtain this vacuum in the chamber, vacuum pumps are used. Basically there are two different principles on which these vacuum pumps are used. The first one is removal of gases from vessel and exhausting the gas load to the outside atmosphere. The second is the condensation or trapping gas molecules on some part of the inner surface of vessel without discharging. Thus there are two types of vacuum pumps used here, the rotary pump and the diffusion pump.
The rotary pump can create a vacuum up to about $10^{-3}$ torr while remaining level of vacuum ($10^{-6}$) can be achieved using the diffusion pump.

**Oil–sealed rotary pump**

It belongs to the class of mechanical pumps. The working principle of rotary pump is the removal of gases by the cyclic motion of a system of mechanical parts (rotors). The basic construction of rotary pump is shown in figure 3.4. The system consists of acentrically positioned rotor which fits tightly in a cylindrical seal provided by stator. Two spring loaded vanes sliding in opposite slots keep fitting tight. The stator, rotor and vans are immersed in oil to minimize the friction between them. A crescent shaped volume is encased between rotor and stator. Normally the exhaust is closed by a pressure valve leading in to an oil reservoir. During the motion of rotor, air is drawn from the inlet in to the crescent shaped volume which is then compressed. This exerts pressure on the outlet valve and opens it thereby permitting the gas to go out. Vacuum up to $10^{-3}$ torr can be obtained by this pump.

![Figure 3.4 Rotary pump.](image-url)
Diffusion Pump

It works on a principle of momentum transfer from streaming oil molecules to diffusing gas molecules. The basic elements of a diffusion pump are shown in figure 3.5. The working fluid (diffusion oil—generally silicon oil) is heated by electrical heater and the hot vapor rises in the stack. These vapor molecules exert pressure on jet cap and the vapor come out through the nozzle pointing away from the high pressure side. The direction of the vapor molecules gets reversed. Gas molecules from vacuum chamber diffuse through the throat and collide with the oil vapor molecules. Because of this collision, the momentum is transferred from streaming to diffusing molecules and the direction of the momentum is towards the fore-vacuum side. These gas molecules are pumped out by rotary pump. Thus a region of reduced gas pressure is generated near the nozzle and more gas molecules from vacuum chamber diffuse towards this region. The outer walls are eaten cooled to retain the oil vapor.

Figure 3.5 Diffusion pump.
The diffusion oil must satisfy the following requirements:

1. It should be stable against thermal decomposition at operating temperatures
2. It should have minimum oxidation at operating temperature
3. It should have low vapor pressure near room temperature

High grade silicon oil is used as diffusion oil

3.5.2 VACUUM MEASUREMENT

It is always required that the reduced gas pressure (vacuum) attained in the vacuum system should be properly measured. Various vacuum measurement gauges are available. There are two basic principles, on which these gauges work.

1. The direct measurement of pressure based on force exerted by the gas per unit area.
2. The measurement of some physical property which is function of the gas density

The vacuum gauges based on the first principle can measure vacuum up to about 1 torr, while those based on second principle, can measure vacuum up to $10^{-3}$ to $10^{-6}$ torr. Usually two vacuum gauges are used in any vacuum system; they are thermocouple gauges and penning ionization gauges.

**Thermocouple Gauge**

It works on the principle of the change in the amount of heat conducted away from metallic heated wire with the gas pressure. A typical gauge is shown in figure 3.6. The thermocouple is spot welded to the centre of the filament wire, which receive constant energy from an external power supply. When the constant current source feeds the filament, it gets heated up. Heat loss from filament takes place through conduction and radiation. The amount of heat loss depends on the number of gas molecules. If the number of gas molecules is more, heat loss is also more. As the system is continuously pumped, the pressure is gradually reduced and the number of gas molecules is also reduced. At lower pressure the mean free path of molecule is large. The conduction of heat from hot too cold object is linearly proportional to gas pressure. At low pressure heat loss from filament is also low and hence temperature increases and emf produced by
thermocouple also increases. This change of temperature can be calibrated in terms of pressure and can be measured using moving coil ammeter.

This gauge can operate between 1 to $10^{-3}$ torr. Below $10^{-3}$ torr pressure this gauge cannot be used properly as the numbers of air molecules are too small to transfer the heat. Using this gauge a continuous measurement can be obtained.

![Figure 3.6 Thermocouple Gauge.](image)

**Penning Ionization Gauge**

The basic principle of ionization gauge is shown in figure 3.7. A pair of cathode plates is placed on either sides of a ring-shaped anode. A d.c potential of about 2KV is maintained between anode and cathode. Because of the stray radioactivity, a few electrons are librated within the space between anode and the cathode. These electrons ionize some gas
molecules and produce positive and negative ions which further accelerate and ionize the gas. As a result a self sustaining glow discharge is obtained. The number of ion pairs produced per centimeter path of the electron at a given pressure will depend on electron energy and the nature of gas. Inversely keeping electron energy and gas fixed, the nature of ion-electron pairs produced will be directly proportional to the pressure. To ascertain sufficient ionization, at low pressure, one has to increase the length of flight of electrons. This is done by confining charged particles in a magnetic field of approximately 400 Oe. Due to this electrons travel in long helical paths before reaching the anode and to make ionizing collision. However positive ions are not much more affected by magnetic field and go to the cathode directly. The total discharge current consisting of positive ions and electron-emission currents is used as a measure of gas pressure.

Figure 3.7   Penning ionization gauge.
This gauge measures pressure between $10^{-3}$ to $10^{-6}$ torr. Because of the ionic oscillations, it tends to suffer to start a discharge at low pressure. The simple and rugged construction of the gauge makes it insensitive to exposure to air.

### 3.5.3 COMPLETE VACUUM SYSTEM

The schematic of actual vacuum coating unit used for deposition of thin films of ZnSe and ZnTe is shown in figure 3.8.

![Vacuum Coating Unit](image)

**Figure 3.8** Vacuum Coating Unit.
The assembly in the vacuum chamber is as required for thin film deposition by resistive heating method. A substrate stand is provided to hold substrate. Substrate heater was kept on its top to control substrate heating. Crystal monitor was kept near the substrate to know the rate of deposition as well as the thickness of the film. The evaporation source and the substrate are kept at straight and at the distance, less than the mean free path of the molecules of material which is being deposited. A penning ionization gauge was connected to the chamber to measure the vacuum. An air-inlet valve was provided to leak the vacuum present in chamber. A Dom made up of Pyrex glass was used and gasket was seated on the base plate. A thin layer of silicon grease was applied on the surface of gasket to avoid air leakage.

Diffusion pump was directly connected below the vacuum chamber by a baffle valve. A rotary pump was attached via roughing valve. Both, diffusion and rotary pumps were connected by a backing valve. A thermocouple gauge was connected near the rotary pump to measure the rough vacuum created by rotary pump. The air inlet valve protects the rotary oil from entering in the chamber when pump is closed.

### 3.5.4 THICKNESS MONITOR

It is important to control and know the thickness of the deposited film during the deposition process. Thickness monitor contains crystal oscillator. The thickness of the film can be measured even after removing the film from the chamber, by mainly two methods.

1. In the first method, the mass of the material deposited is measured which is along with the known density of the material used to estimate the thickness.
2. In the second method, the multiple beam interference is used for the exact estimation of thickness. The method of in-situ measurement of thickness allows both monitoring and controlling of the deposition rate and film thickness. This is one of the most appropriate methods which have been used in the most of the vacuum systems. In the present investigation, the control and measurement of the film thickness has been done by crystal oscillator fitted in vacuum system.
A quartz crystal oscillator was first used in 1957 to determine small quantities of deposited matter. This is based on the measurement of change in resonant frequency of the quartz crystal oscillator with loading by evaporated film when operated in a particular mode or vibration. The transducers required to monitor film thickness have simple construction and are practically unaffected by the mechanical shocks and external vibrations. Hence, crystal oscillators are most widely used for measuring and monitoring thin film deposition. These crystal oscillators utilize piezo-electric property of quartz. A thin X-cut crystal wafer is connected on its two surfaces and made part of an oscillator circuit. X-cut wafer forms an angle of about 35° from the vertical axis of the quartz crystal. The resonance frequency of the wafer varies with temperature. Since the frequency change due to temperature fluctuations affect the accuracy of mass determination, the quartz crystal are cut in an orientation where the frequency change due to this effect is very small. This is achieved in the X-cut position. The instruments required operating an X-cut quartz crystal for thickness monitoring and measurement usually consists of an oscillator and a stable frequency meter. To have better accuracy in thickness measurement, a reference oscillator – a second crystal with fixed frequency is used to generate a different frequency. This is then mixed with the signal from variable frequency generator. The advantage of this is that, it permits operation in the most sensitive frequency range regardless of the thickness of the film formed on the crystal. The output signal of this arrangement is in the audio frequency range and drives an appropriate counter circuit with a suitable display to give the thickness of the deposited film.

3.6 DEPOSITION OF ZnSe AND ZnTe THIN FILMS

The vacuum coating unit – Hind High Vac, Bangalore, model 124A is used in the present investigation to deposit thin films of ZnSe and ZnTe.

First of all the components in the vacuum chamber were properly cleaned using acetone as well as ultrasonic cleaner, where required. The boat of tantalum was also properly cleaned with acetone and fixed in the filament holder inside the chamber. ZnSe compound powder (Fluka make), having purity of 99.99% was loaded in the boat. As a substrate, ultrasonically cleaned glass slides were used. A specially designed substrate rotor which can
contain five glass slides at a time was used to deposit five thin films at a time with various substrate temperatures. A substrate heating was achieved with power controlled halogen lamp inside the chamber near the substrate rotor. A temperature indicator cum controller was used to control and monitor the substrate temperature during deposition process. The rough vacuum of about $10^{-3}$ was achieved using rotary pump with only roughing valve open. After that diffusion pump was switched on. Now the roughing valve was closed and backing valve was opened. After some time (15-20 minuets) the baffle valve was opened. When a sufficient vacuum of $10^{-6}$ torr was attained in the chamber, the temperature controller was switched on with temperature setting of 448K. After achieving this temperature, the power source to the boat terminals was switched on and slowly increased. These causes the ZnSe powder in the boat to evaporate and began to deposit on the glass substrate heated at 448K temperature. The rate of deposition was fixed at 5Å/Sec. and thickness was achieved 1000Å. Thus a thin film of thickness 1000Å and substrate temperature 448K was deposited. The power to the boat was slowly reduced. The rotor now rotates and the next glass substrate came in front of the evaporation source. The temperature was now set to 373K. After attaining this temperature, a film was deposited and the film of thickness 1000Å and substrate temperature 373K was prepared. The same way another film of the same thickness with substrate temperature 303K was prepared.

After the completion of this process, a baffle valve was closed and diffusion pump was switched off. When the diffusion heater cooled down to the room temperature, the backing valve was closed. The rotary pump was switched off and the air inlet valve to the rotary pump was opened to restrict rotary oil from entering to the system.

An air inlet valve to the chamber was slowly opened to open the chamber. The deposited films (three) were taken out of the chamber and properly marked on the backside and stored in the vacuum sealed container.

In the same manner thin films of ZnSe with thickness 2000Å, 3000Å and 5000Å were deposited at substrate temperatures of 303K, 373K and 448K.

The whole vacuum chamber was then properly cleaned with all the components inside it. Another tantalum boat was taken and properly cleaned and heated prior to use. Boat and cleaned glass substrates were installed at their places inside the chamber and ZnTe
(Fluca make, 99.99% pure) powder was loaded in the boat. The chamber was then properly closed and the rotary pump was switched on.

The whole process was repeated as in earlier case of ZnSe powder. Thin films of ZnTe with thickness 1000Å, 2000Å, 3000Å and 5000Å were deposited at substrate temperatures of 303K, 373K and 448K. These films were also carefully stored in the proper container.

In this way the deposited films of ZnSe and ZnTe compounds, of various thicknesses (1000Å, 2000Å, 3000Å and 5000Å) deposited at various substrate temperatures (303K, 373K and 448K) were utilized for various characterizations.
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