CHAPTER 2

2.0 INSTRUMENTATION

2.1 Introduction:

The thin films can be prepared by various techniques such as thermal evaporation, sputtering, screen printing, electro plating etc. which are briefed in the previous chapter. The thermal evaporation method is cost effective and efficient. In this method, the films will be prepared in the vacuum chamber and are free from contamination. The substrate is placed straight above the source and hence the films will be of uniform thickness. In the present work, the ZnTe thin films of different thickness were prepared by thermal evaporation method. The fabricated films were characterized for their optical property using UV-VIS spectrophotometer. The instruments that are used in the present study are discussed in this chapter.

2.2 The Vacuum Coating Unit

The simplest and efficient method to fabricate thin film is the thermal evaporation method. A Vacuum Coating Unit manufactured by M/s Hind High Vacuum Company, Bangalore, Model 12A4D was used to fabricate the ZnTe thin films. The instrument is as shown in Fig 2.1. Sahay et al. (2007); Pradip et al.(2000) and several other researchers have used the 12A4D vacuum coating unit to prepare the thin films of CdS, CdTe, ZnSe and ZnTe materials. The basic unit consists of a Vacuum Chamber, Rotary Drive, Rotary Vacuum Pump, etc. The details of which are discussed in the following sections.
2.2.1 Vacuum chamber:

The chamber is fabricated from electrochemically-polished stainless steel. Three circular glass windows enable visual inspection of the coating process. When the chamber is placed on the base plate it makes a vacuum tight seal with the base plate by means of an ‘L’ type neoprene gasket. A cooling water pipe line is coiled on the outer wall of the chamber to prevent overheating, and to reduce the out gassing by circulating the water. The chamber is evacuated by a diffusion pump Model-114D and backed by 250 liters per minute, double stage, direct driven, rotary vacuum pump, Model ED-15 with an overload protection
2.2.2 Rotary Drive

The rotary drive is useful for deposition of materials uniformly on large plane surface substrates. This comprises of a rotating work holder, which as a useful diameter of 6 inch. The work holder ring is supported by three equally spaced ball bearings one of which is spring loaded, acting on the rim of the work holder. The work holder is rotated by a variable speed electric motor situated on a platform inside the coating unit cabinet. The speed of the rotary drive motor is controlled by a rider control fixed on the left side of the cabinet.

2.2.3 Ion Bombardment:

This is used in conjunction with the rotary drive. This high tension discharge cleaning system consists of a super pure Aluminum electrode bar. The bar is shielded to avoid electron contamination of the substrate during discharge cleaning.

2.2.4 Source Shutter Plate:

The source shutter plate is designed to cover any one of the off sector filament holders meant for sequential evaporation when a rotary drive is used. The shutter plate is attached to a standard source shutter shaft when a rotary derive is used.

2.2.5 Rotary Vacuum Pump:

HINDHIVAC Rotary pumps are spring loaded sliding vane type, with vanes placed in the slots of the rotor. They are mounted eccentrically both in the first and second stage with inter connecting ports as shown in the Fig 2.2.
Both, the first stage which creates primary vacuum and second stage which creates the low pressure, are isolated with the introduction of an isolator in between and the two rotors are mounted eccentrically within respective stators. The first stage end plate has a bearing and an oil seal for isolation from atmosphere. The second stage end cover also has a bearing for locating the shaft, which will give the closest possible tolerance and free movement within the stator and rotor for efficient performance of the pump. The oil pump housing is mounted on the rear end plate of the stator. This oil pump provides the lubrication to the pump and the oil flow to the stator. The pump functions in a similar manner as the vacuum pump. It has a vane, mounted in an exocentric position in the bore and drives the oil under pressure. In the event of the pump stopping a spring loaded flap valves is activated to seal the oil port of the oil pump, thereby preventing any back streaming of oil in to the chamber because of difference in pressure. A filter is provided on the oil pump housing through which oil is sucked in, this prevents any dirt or fibrous tissues from entering the pump and causing reduction in flow of oil and thereby seizure of moving parts.

Fig. 2.2 Construction & Working of Rotary pump
CHAPTER 2 Instrumentation

Working principle of Rotary Pump:

During operation, the rotor vanes sweep the volume of the gas or air trapped in the crescent shaped gap formed by the rotor which is mounted eccentrically in the stator. As each vane passes the inlet port opening a known quantity of gas is introduced and subsequently trapped and compressed by the next vane following it and ejected via the exhaust flap valve mostly and via the interconnecting port to the second stage partially, when the inlet pressure is near atmospheric pressure. As the inlet pressure drops, the first stage exhaust flap valve closes and all the air or gases pass to the second stage, where it is further compressed and discharged to atmosphere.

2.2.6 Oil Diffusion Pump:

These pumps are available with nominal bore sizes from a range of 1 inch to 36 inch diameter. While inch pump gives 10 liters per second pumping speed the 36inch pump gives 40-45000 lit/sec. with two to five stages (depending on size). These water cooled oil diffusion pumps are fabricated either from mild steel or electroplated Aluminum or stainless steel. Diffusion pumps are employed in the pressure range $10^{-1}$ to $10^{-7}$ mbar and even lower pressure using the correct technique.

Main body:

The diffusion pump body is cylindrical in shape which has housing for the jet assembly and the charging fluid. This is provided with a metallic flange at the top which is either made of stainless steel or MS electro plated with suitable ‘O’ ring groove and securing bolt holes. This may be either fitted to the bottom flange or a suitable baffle valve or to the system to be evacuated.

Generally, all pumps are fitted with a baffle valve or other type of baffles at the opening to avoid back streaming i.e., the migration of working fluid molecules into the system.
which ultimately lead to complete loss of the working fluid and contamination of the whole system. Cooling water coil is soldered n the outer wall of the pump to cool the pump body.

**Heaters:** This consists of a set of metallic nozzles which are assembled around a central rod and housed in the main body of the pump. All the nozzles are oriented downwards at a certain angle towards the water cooled pump body and designed to give the best performance and maximum efficiency. The water circulated on the outer wall of the main body cools the vapor molecules which emerge from these nozzles striking the inner wall of the pump body.

**The Jet:** Jet may be of a single or multi-stage depending on the size of the pump. In multi-stage the number of nozzles will be of more than one. The jet accessories are made of stainless steel or electroplated aluminum. Diffusion pumps are manufactured in two types called the standard diffusion pump and Fractionating Diffusion pump. The main difference between these two is in the fabrication and arrangement of the nozzles. The working of these two explained in the section “working principles” separately.

**Boiler:** This is the part of the main body where the working fluid is heated and evaporated. The boiler is designed to hold a certain specified quantity of the recommended working fluid to give maximum efficiency.

**Working principle of the Diffusion Pump**

The working principle of both the standard and the fractionating type of diffusion pumps are almost the same except with minor modifications. The working principle of the diffusion pump is as shown in Fig. 2.3. The oil in the boiler is heated by the heater and converted into vapor. This rises in the concentric columns and is limited by the jets due to the comparative high pressure existing above the boiler in the jet system. The vapor is
forced through jet aperture where it is deflected downwards by the jet deflectors while the tabular side jet discharges vapor into the backing system. The molecules issuing from the jet engulf gas molecules; diffuse into the vapor streams not being able to diffuse back due to the downwards deflected vapors. The gas molecules are finally removed to the atmosphere by the backing pump. The oil vapor impinging on the water cooled pump wall condenses and drains to the boiler where it is re-evaporated.

In the conventional oil vapor pump the ultimate vacuum achieved is little less than the fractionating type. This is due to comparatively high volatility of the light fractions which arrive at the top jet and prevents achieving an ultimate vacuum lower than the vapor pressure of these fractions, where as, in the fractionating type diffusion pump the condensed oil returning to the boiler for re-evaporation on its path to the centre of the boiler evaporates progressively. The light fractions or low molecular weight fractions of high vapor pressure travel to the side jet as its temperature rises rapidly and evaporates before it reaches the centre of the jet. The medium fractions of little lower vapor pressure travels still further towards the centre but vaporize before they reach centre and feed the

Fig. 2.3 Construction & Working of Diffusion pump

In the conventional oil vapor pump the ultimate vacuum achieved is little less than the fractionating type. This is due to comparatively high volatility of the light fractions which arrive at the top jet and prevents achieving an ultimate vacuum lower than the vapor pressure of these fractions, where as, in the fractionating type diffusion pump the condensed oil returning to the boiler for re-evaporation on its path to the centre of the boiler evaporates progressively. The light fractions or low molecular weight fractions of high vapor pressure travel to the side jet as its temperature rises rapidly and evaporates before it reaches the centre of the jet. The medium fractions of little lower vapor pressure travels still further towards the centre but vaporize before they reach centre and feed the
CHAPTER 2

Instrumentation

intermediate jets. Finally the oil composed mainly of the heaviest and the lowest vapor pressure fraction reaches the pump centre and vaporizes, feeding the top jet. The characteristic which is termed as fractionating pumps thus allow only stable and heaviest fractions of the pump fluid to reach first stage which results in the high performance of the pump.

2.2.7 Mini Penning Stabilized Gauge Model – STP 4M –D

The HINDHIVAC mini penning stabilized gauge model STP4M –D is a pressure control instrument designed with the cold cathode type gauge head (Sensor). This provides all necessary circuit and signal conditioned analog output. This instrument can accept a range from $1 \times 10^{-3}$ to $1 \times 10^{-5}$ (Top scale) and from $2 \times 10^{-5}$ to $1 \times 10^{-6}$ (Bottom scale). This gauge covers a pressure range of $1 \times 10^{-3}$ to $1 \times 10^{-6}$ mbar in two ranges with instant range changing provided by a toggle switch. No manual adjustment is required and pressure is indicated in a clear front meter directly calibrated in mbar. The specifications are;

<table>
<thead>
<tr>
<th>Electricity supply</th>
<th>230V AC 50Hz (± 10%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Output voltage</td>
<td>2.2 KVDC (± 5%)</td>
</tr>
<tr>
<td>Pressure range</td>
<td>Range-1: $1 \times 10^{-3}$ to $1 \times 10^{-5}$ (top scale)</td>
</tr>
<tr>
<td></td>
<td>Range-2: $2 \times 10^{-5}$ to $1 \times 10^{-6}$ (bottom scale)</td>
</tr>
<tr>
<td>Fuse</td>
<td>500 ma size 5Dia×20mm</td>
</tr>
<tr>
<td>Gauge head</td>
<td>HINDHIVAC Model PNG-2</td>
</tr>
<tr>
<td>Control</td>
<td>Input to penning controller model PNGC-1 for pressure control</td>
</tr>
<tr>
<td>Size in MM</td>
<td>98H×98W×180D (Approximately)</td>
</tr>
</tbody>
</table>
2.2.8 Mini Pirani Stabilized Gauge: Model – A6STM-D

The HINDHIVAC mini pirani stabilized gauge model A6STM-D is a pressure control instrument designed with the thermal conductivity type gauge head (Sensor). This provides all necessary bridge circuits and signal conditioned analog outputs. This instrument can accept range from 0.5 mbar to 0.001 mbar using a direct reading meter. This instrument works on the principle of change in resistance of material with a change in temperature. This can be used to measure vacuum from 0.5 mbar to 0.001 mbar anywhere in the vacuum system with a suitable adopter. This gauge is very much essential in the pirani controller to control vacuum (0.5 mbar to 0.001 mbar). Two gauge heads can be directly connected to this gauge to read fore vacuum and roughing vacuum of a vacuum system.

The mini pirani gauge model A6STM is a modular front panel construction and forms 1/4 size of a standard 19” rack. This can be used as a bench standing type or as a panel mounting type by using an appropriate rack adaptor. The specifications are:

- **Electricity supply**: 230V AC (+ OR - 10%)
- **Bridge output voltage**: 2V D.C. (+ OR - 0.5%)
- **Pressure range**: 0.5 m.bar to 0.001 m.bar
- **No. of gauge heads**: TWO HINDHIVAC MODEL PR-3
- **Fuse**: 250 mA SIZE: 5×20mm
- **GH1, GH2**: Input to pirani controller Model PRGC-1 for pressure
- **Size in mm**: 98H×98W×180 D (approximately)
2.2.9 Digital Thickness Monitor: Model-DTM-101

Working principle of crystal oscillator:

Certain crystalline materials, namely, Rochelle salt, quartz and tourmaline exhibit the piezoelectric effect that is, when we apply an a.c. voltage across them, they vibrate at the frequency of the applied voltage. Conversely, when they are compressed or placed under mechanical strain to vibrate, they produce an a.c. voltage. Such crystals which exhibit piezoelectric effect are called piezoelectric crystals.

Frequency of crystal:
Each crystal has a natural frequency like a pendulum. The natural frequency $f$ of a crystal is given by;

$$ f = \frac{K}{t} \text{ Hz} $$

Where $K$ is a constant that depends upon the cut and $t$ is the thickness of the crystal. It is clear that frequency is inversely proportional to crystal thickness. A quartz crystal monitor in the deposition chamber monitors the deposition rate of the films and their final thickness. It has a resonant quartz crystal with one surface exposed to the deposition source. As material is deposited onto the surface of the device the resonant frequency changes because of the added mass of the deposited film. The monitor then uses the known parameters of density and acoustic impedance of the source material to determine the deposition rate and thickness

**Thickness Set Point Shutter Control (TL-1 & TL-2)**

The thickness set point establishes the film thickness at which the shutter closes. As described above, depressing the START button zeros the thickness display and opens the shutter. The shutter is then automatically closed when the Thickness Display equals or
exceeds the thickness set point. The shutter can also be closed manually by depressing the STOP button. In this way complete manual control of the shutter, as may be required for servicing the source, is available through use of the START and STOP button. This eliminates the need for a separate OPEN, CLOSE, AUTO switch and eliminates the possibility of leaving such a switch in the open or close position when it should be in the auto position. If auto control of the shutter is not desired the thickness set point parameter can be programmed at a value much greater than can reasonably be achieved. The thickness set point -2 may be used to activate a second shutter relay to allow two materials to be sequentially deposited to two present thicknesses or to operate other electrical or electrical-mechanical devices.

**Crystal Test**: When the TEST button is depressed the normal displays are replaced with crystal test information. The normal rate display is replaced with a three digit number the leftmost digit of which is 6 indicating the type of sensor crystal the monitor is set up for. The monitor is compatible with 6 MHz sensor crystals. Crystal health is indicated as a percentage of crystal life remaining. A new crystal will have a health of 98 to 99%. The health decreases as material is deposited on the crystal sensing surface.

The normal thickness display is replaced with a display of the current operating frequency in MHz of the sensor crystal.

The crystal test display reverts to normal after the TEST button is released. The crystal test function does not affect the normal operation of the monitor. In particular, both thickness and rate continue to be calculated and the normal operation of the thickness set point is not affected.

**Tooling Factor (TFC)**: The tooling factor parameter compensates for geometric factors in the deposition system which results in a difference between the deposition rate on the substrates and the rate on the sensing crystal. This parameter is entered in percent units
and 100% corresponds to equal rates at the substrate and at the sensing crystal. For initial approximation the tooling factor can be calculated using the following equation.

\[
\text{Tooling \%} = \left(\frac{d_{\text{cry}}}{d_{\text{sub}}}\right)^2 \times 100\%
\]

Where,

- \(d_{\text{cry}}\) = distance from the source to the crystal.
- \(d_{\text{sub}}\) = distance from the source to the substrate.

**Density (DNT):** The density parameter provides the Monitor with the density of the material being deposited so that it can calculate and display the physical film thickness. If the film density is known, it should be used. A list of the more commonly used film densities is presented in the above. As a first approximation, bulk material density can be used in programming, this parameter.

**Acoustic Impedance (ACI):** The shear wave acoustic impedance of the deposited film is required by the monitor in order to accurately establish the sensor scale factor when the sensor crystal is heavily loaded. If the acoustic impedance of the film material is known, it can be entered directly in units of 100.000 gm/sq.cm sec. in most cases the acoustic impedance of the bulk material can be used and can be obtained from the Handbook of physics or other sources of acoustic data. The shear wave acoustic impedance can be calculated from the shear modulus or the shear wave velocity and the density by using the following equations:

\[
\text{Acoustic impedance} = PC = PG
\]

Where \(P\) = density (gm/cubic cm.)

- \(C\) = Transverse (shear) wave velocity (cm/sec)
- \(G\) = shear modulus (dynes/sq.cm)
CHAPTER 2 Instrumentation

In many cases, and particularly if the sensor crystal is not heavily loaded, sufficient accuracy can be achieved by using the acoustic impedance of quartz: \(8.83 \times 10^0.000\) qm/sq.cm sec.

2.3 UV-VIS Spectrophotometer

The optical properties like absorbance, transmittance and optical band gap of the prepared thin films were measured using UV-VIS Spectrophotometer (Shimadzu, Japan Model UV-1650PC). Absorption spectroscopy refers to a range of techniques employing the interaction of electromagnetic radiation with matter. In absorption spectroscopy, the intensity of a beam of light measured before and after interaction with a sample is compared. One ray is passed through sample and other is transferred through the reference sample.

2.4 Working Principle of UV-VIS Spectrophotometer

The working principle of the UV-VIS spectrometer is schematically shown in Fig. 2.4. The UV-Visible spectrophotometer uses two light sources, a deuterium (D\(_2\)) lamp for ultraviolet light and a tungsten (W) lamp for visible light. After bouncing off a mirror (mirror 1), the light beam passes through a slit and hits a diffraction grating. The grating can be rotated allowing for a specific wavelength to be selected. At any specific orientation of the grating, only monochromatic (single wavelength) successfully passes through a slit.
A filter is used to remove unwanted higher orders of diffraction. The light beam hits a second mirror before it gets split by a half mirror (half of the light is reflected, the other half passes through). One of the beams is allowed to pass through a reference (empty glass slide), the other passes through the sample thin film coated with ZnTe. The intensities of the light beams are then measured at the end.

**Beer-Lambert Law**

The ratios of incident and transmitted intensities are measured; the optical coefficient \(a\) is calculated using Beer–Lambert law:

\[
\ln \left(\frac{I_o}{I} \right) = 2.303 \quad Abs = \alpha \cdot d
\]

Where, \(Abs\) = Absorbance

\(I_o\ & I\) = Intensities of the incident & transmitted radiation respectively

\(\alpha\) = Molar absorption coefficient

\(d\) = Path length of the absorbing solution (Thickness of the films in cm)
The plots of absorption, transmittance and reflectance versus wavelength are obtained. Further band gaps of the prepared films will be calculated.

The Specifications of the Shimadzu Model UV-1650PC are;

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Wavelength range</strong></td>
<td>190.0 to 1100.0 nm</td>
</tr>
<tr>
<td><strong>Wavelength display</strong></td>
<td>Readable to 0.1 nm</td>
</tr>
<tr>
<td><strong>Wavelength accuracy</strong></td>
<td>±0.5 nm (Automatic wavelength correction)</td>
</tr>
<tr>
<td><strong>Wavelength slew rate</strong></td>
<td>About 6000 nm/min.</td>
</tr>
<tr>
<td><strong>Data bunching interval</strong></td>
<td>Automatic selection of 2.0, 1.0, 0.5, 0.2, 0.1 nm</td>
</tr>
<tr>
<td><strong>Stray light</strong></td>
<td>Less than 0.05 % (at 220.0 nm and 340.0 nm)</td>
</tr>
<tr>
<td><strong>Photometric range Absorbance</strong></td>
<td>-0.5<del>3.999 Abs. Transmittance: 0.0</del>300 %</td>
</tr>
<tr>
<td><strong>Photometric reproducibility</strong></td>
<td>±0.002 Abs. at 1.0 Abs. ±0.001 Abs. at 0.5 Abs.</td>
</tr>
<tr>
<td><strong>Baseline flatness</strong></td>
<td>±0.002 Abs.</td>
</tr>
<tr>
<td><strong>Light source</strong></td>
<td>50 W halogen lamp and deuterium lamp</td>
</tr>
<tr>
<td><strong>Detector</strong></td>
<td>Silicon photodiode</td>
</tr>
<tr>
<td><strong>Interface ports</strong></td>
<td>RS-232C port for UV-1650PC</td>
</tr>
<tr>
<td><strong>Ambient requirements</strong></td>
<td>Temperature: 15<del>35 °C Humidity: 45</del>80 %</td>
</tr>
<tr>
<td><strong>Spectral bandwidth</strong></td>
<td>2 nm</td>
</tr>
<tr>
<td><strong>Wavelength setting</strong></td>
<td>0.1 nm increments (1 nm increments for wavelength slewing)</td>
</tr>
<tr>
<td><strong>Wavelength reproducibility</strong></td>
<td>±0.1 nm</td>
</tr>
<tr>
<td><strong>Wavelength scanning speed</strong></td>
<td>About 3200 nm/min. to about 160 nm/min.</td>
</tr>
<tr>
<td><strong>Lamp interchange wavelength</strong></td>
<td>295.0~364.0 nm (340.8 nm)</td>
</tr>
<tr>
<td><strong>Photometric system</strong></td>
<td>Double-beam optics</td>
</tr>
</tbody>
</table>
CHAPTER 2

Instrumentation

<table>
<thead>
<tr>
<th>Feature</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photometric accuracy</td>
<td>±0.004 Abs. at 1.0 Abs. (Tested with NIST 930Dfilter) ±0.002 Abs. at 0.5 Abs.</td>
</tr>
<tr>
<td>Baseline stability</td>
<td>Less than ±0.001 Abs./hour</td>
</tr>
<tr>
<td>Baseline correction</td>
<td>Automatic with computer memory, in two stages of coarse and fine</td>
</tr>
<tr>
<td>Monochromator</td>
<td>Aberration corrected concave blazed holographic grating</td>
</tr>
<tr>
<td>Sample compartment</td>
<td>Inner dimensions: W110.0 X D230.0 X H105.0 mm (partly 105 mm deep) Distance between light beams: 100.0 nm Installation: Fixed with 2 screws Beam size: 10 X 1 mm</td>
</tr>
<tr>
<td>Power requirements</td>
<td>100, 120, 220, 240 VAC, 50/60 Hz, 160 VA.</td>
</tr>
<tr>
<td>Dimensions and weight</td>
<td>W550 X D470 X H200 mm, 18 kg</td>
</tr>
</tbody>
</table>

Employing the vacuum coating unit, ZnTe thin films were prepared with different deposition parameters like thickness and substrate temperatures. Details of fabrication of the thin films are outlined in the next chapter.