4.1. WHAT IS A THIN FILM

A thin film can be simply defined as a very thin layer of any material. It can be either a solid, liquid or gaseous layer. In case of our study under the designed optoelectronic system, we regard only the solid layer of material as a thin (solid) film, and deal with only thin solid films.

A thin solid film may be classified to be belonging to mainly three subgroups, depending on how thin a film is purely numerically. Those thin films which are very thin so that their “Average Thickness” is less than about 100Å or so are called Ultra-Thin thin films. In these films one can define only an “Average Thickness” as the film consists of a number of islands randomly distributed and unconnected, thus making it difficult to measure its “Geometric Thickness”. The second category belongs to those thin films whose thicknesses lie between about 500Å and about 5000Å or so. These films are simply called as thin films. This is the most extensively studied range thicknesses of the films because of the fact that in this thickness range of thin films, the film thickness is comparable to one or more of some of the characteristic lengths of the film material like the mean free path of the charge carriers or the de Broglie wavelength of the carriers in the case of electrical properties of thin films or the wavelength of the incident light or other radiation incident on the film in the case of optical properties, or the superconducting (magnetic field) penetration depth in case of superconducting properties. Further this thickness range
is also amenable to structural studies, at least in a limited range upto 1000Å or so. The third category of thin films is called the thick films and the thicknesses of these films are above about 1µm or so, i.e. 10,000Å. Even though not much scientific interest can be expected in this category of thin films because of the fact that in this range, the thickness of the film is no longer a parameter which changes the properties of the films, these thick films are of technological importance in devices like solar cells, other photovoltaic devices, corrosion resistant layers, heat absorbing layers, optical multilayer coatings, printed circuit boards (PCB) etc. and hence, are of equal interest, if not scientifically, then technologically\textsuperscript{1,2}.

4.2. THE CHARACTERISTICS OF THIN FILMS:

The main characteristic feature of thin films is their thickness. Depending on the thickness of a thin film, the property of the thin film will change, and will be, therefore, different for films of different thicknesses. In addition to thickness, one more feature that influences the properties of a thin film is the grain or micro-crystallite size in the thin film if the film is polycrystalline. Of course, if the film is single crystalline, or it is an amorphous one, the properties will also on this fact of single crystallinity or the amorphous nature of the thin film.

Another important feature of a thin film is its surface area – both the external, apparent surface area and the internal grain boundary surface area. The surface area of the thin film per unit volume of the material is also a function of the film thickness, and increases as the film thickness decreases. Thus, the smaller thickness film will have a larger surface area than a larger thickness film, if unit
volumes of the film materials are considered. This has important consequences and affects those properties which depend sensitively on surface area. Also, these properties will be thickness dependent because of the large surface to volume ratio.

4.3. IMPORTANCE AND PRACTICAL APPLICATIONS OF THIN FILMS:

The two important features of thin films are its thinness as its thickness is very very small when compared to its lateral dimensions, and its very large surface area to volume as compared to the same ratio in the bulk solid state. In addition, one more important feature in the case of polycrystalline thin films is the very small size (a few hundred Angstroms) of the crystallites or grains of the thin films. In addition, the number of various defects in thin films is very large as compared to the bulk solid material, and hence a careful control of the quality and nature of the defects in thin films can also control the properties of thin films$^{3-5}$.

These four features make the thin film eminently suitable for applications in areas where interactions with surfaces and also absorption only up to very small depths in the materials used are required. Further, dependence of the thin film properties on their thickness, grain size, surface to volume ratio and the defect structure enables one to tailor-make thin films to the desired requirements of their properties, if one has the knowledge of the various parameters influencing the properties and also the ability to control them adequately to the desired accuracy. In addition, just because of its thinness, it can be used in a number of devices and circuits simply, only to save space by miniaturization. Another feature of thin films is
their shine and glitter if they are metallic. This feature can be made use of for decorative purposes on variously shaped objects by what are called decorative coatings. As a matter of fact, because of these unique features, thin films find wide application in diverse and varied fields.

One of the single most important applications of thin and thick films from the point of view of the severe energy crunch faced globally nowadays is in the photovoltaic devices and other solar cells which convert directly the energy of the solar radiation from the sun to useful electrical energy or at least thermal energy. In photovoltaic devices, the use of thin films, or rather thick films not only enable materials costs saving but also the fabrication of large area devices at a comparatively low cost. Further, one can actually tailor-make materials of the desired properties for use in these devices in the thin film state much more efficiently, cost-effectively and with comparative ease.

Another important application is in the area of electronic devices and circuits to effect miniaturization and thereby save space. As a result of thin film and thick film technology, many very bulky electronic types of equipment can now be made so compact that we can handle these miniature equipments with ease. Making of active devices like p-n junction rectifiers, transistors, LEDs, ICs (both VLSI and LSI) have been possible by the use of photo and electron-beam lithographic techniques to micro-pattern thin films in processing and fabrication.

Another area of application of thin films is in optical and optoelectronic devices. For example, thin films are widely used in making reflection
coatings, anti-reflection coatings, transmission coatings, band pass filters, band stop filters, thin film wave guides, light emitting devices etc. Amorphous thin film switches of suitable materials can be in a high resistive non-conducting state at lower applied voltages to the device or when the current density in the device is lower than a critical value. When the applied voltage or the current density in the device exceeds a critical value, the switch goes over to the second, low resistive high-conducting state. This switching of the state of the device from the high resistive state to the low resistive state can also be brought about by other means, e.g., by means of a light pulse of proper strength. But, the most important feature of this switch is that its state can be changed reversibly and hence this device acts as a switch indefinitely during its lifetime. Thus, thin film can be used to make Thin Film Micro Switches.

Thin films of proper materials can also be used as data-storing devices either by optical or magnetic means. They can also be used as optical image storing devices also by what is called the ‘optical phase change recording’. Thin films of the magnetic materials find application in the situations when perfectly rectangular, slim hysteresis curve of the magnetic material is required so that there is no loss of energy during cycling the material through an alternating magnetic field because of the low value of coercivity and also in bistable devices where switching from one state to the other can be done in an extremely short time interval of the order of a couple of microseconds to nanoseconds. Thin films also find applications in mechanical and chemical purposes. Thin films of suitable materials can serve as hard coatings on objects at the place where there is likely to be a lot of wear and tear so that, that particular region is protected from rapid wear due to this hard coating Thin film coatings can also be used as protective coatings against chemical reagents of various
types. The inner surfaces of boilers, pipes and other containers or flow tubes or apparatus for chemical reaction of chemicals can be coated with protective coating of thin films of suitable materials so that they can last longer and sustain the attack of corrosive liquid or gaseous reagents. Thin films, particularly the discontinuous films, can also be used in electrochemical devices like strain gauges which are used to measure the strain in a beam or any other object by the large change in resistance of the thin film, specially in discontinuous thin films, that is produced as a result of the bending of the thin film strain gauge which is attached to the specimen under investigation. Also certain thin films, particularly columnar thin films, can also be used as gas detecting transducers which can quantitatively determine the amount of gas absorbed by the thin film transducer.

4.4. VARIOUS METHODS OF THIN FILM DEPOSITION:

Thin films can be prepared from a variety of materials such as metals; semi-conductors, insulators etc. and various techniques have also been developed as per need for the preparation of different types of films. The different methods of film preparation are:

1. PHYSICAL VAPOUR DEPOSITION:

Physical Vapour Deposition is the most common technique which involves physical processes. In Thermal Evaporation Technique, the materials are vapourized in a vacuum by means of resistive heater. The condensation of
the vapour onto a cooler substrate yields thin films. One can evaporate from two or more sources in order to deposit a multilayer film or an alloy/compound film.

2. CHEMICAL DEPOSITION TECHNIQUES:

In the chemical deposition techniques, a film is formed on a surface by certain chemical reactions in a solution in which the surface to be coated is kept. An appropriate chemical reaction can be produced by the passage of an electric current as in electroplating. In some processes, the chemical reaction can be activated through an external agency, such as application of heat, RF field, light or X-rays, an electric arc or glow discharge, electron bombardment etc.

3. SPUTTERING TECHNIQUES:

When the surface of a solid i.e. the target, is bombarded with high energy particles, the atoms of the material are ejected. The ejected or sputtered atoms are allowed to condense on a substrate and a thin film is formed. The ions required for bombardment is usually obtained by maintaining a glow discharge due to an applied electric field within the vacuum chamber. For the glow discharge, the sputtering gas Argon or Helium is admitted into the vacuum chamber.
4. ELECTRON BEAM EVAPORATION:

In electron beam evaporation, an electron beam is accelerated through a potential of 5 to 10KV and focused on the material which is to be evaporated. At the high temperature produced by electron beam, most of the refractory metals and compounds can be evaporated.

5. MOLECULAR BEAM EPITAXY (MBE):

The deposition of single crystal (epitaxial) films by the condensation of one or more beams of atoms and/or molecules from Knudsen sources under UHV conditions is called Molecular Beam Epitaxy. MBE makes it possible to deposit epitaxial films of compound semiconductors like GaAs, CdTe etc. having precisely controlled properties.

6. PLASMA ACTIVATED REACTIVE DEPOSITION:

If the evaporated material is transported through plasma of a reactive gas (Oxygen, Acetylene etc.), the deposition technique is called Activated Reactive Deposition. The technique has been mainly used to deposit highly adherent films of oxides and carbides (SnO₂, In₂O₃, TiC etc.). For example, if Ti metal vapour is passed through acetylene plasma, TiC is formed and is deposited on the substrate.
4.5. VARIOUS METHODS OF THIN FILM THICKNESS MEASUREMENT:

The main characteristic feature of thin films is their thickness. Various thin film thickness measurement techniques are based on different principles such as the mass difference, light absorption, interference effect, conductivity, capacitance, etc. of the films with increasing thickness. The measurements can either be in dynamic or static condition\textsuperscript{10}.

Different methods of measuring film thickness along with their useful ranges of accuracies are given below in brief. The accuracy is given in terms of a unit of length for thinner films and in terms of percentage in case of thicker films. Quite often the useful ranges and accuracies vary depending on the materials being examined, the operator’s ability, the instrumentation used and some other factors\textsuperscript{11,12}.

1. MULTIPLE BEAM (FIZEAU) INTERFERENCE METHOD:

The range of this method is 30-20,000Å and the accuracy or precision is in between 10-30Å. In this process a step and evaporated reflecting coating is necessary.

2. MULTIPLE BEAM (FECO) INTERFERENCE METHOD:

The range of this method is 10-20,000Å and the accuracy is 2Å in each step. In this technique, an evaporated reflecting coating is needed. Although the technique is very accurate it is quite time consuming.
3. **MICHELSON INTERFEROMETER:**

The range of this method is between 300 and 20,000Å and the accuracy is 150 to 300Å in each step. The technique requires a step over the thin film.

4. **POLARIZATION INTERFEROMETER:**

The range of this method is between 300 and 20,000Å and the accuracy is from 150 to 300Å. This technique also requires a step over the thin film.

5. **COLOUR COMPARISON:**

The range of this method is between 500 and 15,000Å and the accuracy is from 100 to 200Å. These values are useful only for SiO₂ on Si materials. The technique is limited to transparent films on substrate such that the reflectivities at the two interferences are not too much different.

6. **STYLUS METHOD:**

The range of this method is 20Å to no limit with an accuracy of several angstroms to greater than 3%. In this method a step is required over
the film and the film should be sufficiently hard to resist deformation by stylus. The method is simple and rapid.

7. VAMFO METHOD:

The range of this method is from 800 to 1,300Å and 2,300Å to 10µ. The accuracy is from 0.02 to 0.05%. The technique is useful for transparent films on reflective substrates. This is a non-destructive method and the technique can be used to measure the number of moles (n) of the material. The lower limit of the measuring range can be extended to 400Å and the 1,300Å to 2,300Å gap can be removed by using a detector system at shorter wavelength.

8. CARIS METHOD:

The range of this technique is from 400Å and 20µ and the accuracy is 10Å to 0.1%. The technique is useful for transparent films. This method is also non-destructive, but to measure film thickness one has to know the refractivity if only one angle of incidence is used.

9. ELLIPSOMETRY METHOD:

The range of this method is of few Å to a few microns. The accuracy is around 1Å to 0.1%. The method is useful for transparent films.
The mathematics of calculation involved in this method is quite complicated, especially with thicker films.

10. LIGHT-SECTION MICROSCOPE SYSTEM:

The range of this system is from 1 to 400 micron with an accuracy of 0.2 micron to 2%. In this system a step is required on opaque films. It is a non-destructive system with transparent films. For this system the refractivity of the material must be known.

11. GRAVIMETRIC SYSTEM:

The range of this system is from a few angstroms to no limit. The accuracy or the precision is greater than 1Å to 0.1%. This technique gives an average value over the system. For this method one must know the film density.

12. X-RAY ABSORPTION:

The range of X-ray absorption method is in between 0.1 to 1000 micron. The accuracy is ± 5%. In this method, the substrate must produce characteristic radiation.
13. X-RAY EMISSION:

The range of X-ray emission method is in between 20 to 10,000Å with an accuracy of ± 2%. In this method the substrate must not contain any of the elements in the film. The multi-component films can be measured by this system.

14. BETA BACKSCATTERING METHOD:

The range of this method is 0.1 to 50 micron and the accuracy is ± 5%. In this technique, the film and the substrate must have large differences in atomic number.

Detailed descriptions of various methods mentioned above are available in the literature^13-19

4.6. THIN FILM THICKNESS MEASUREMENT UNDER THE DESIGNED OPTOELECTRONIC SYSTEM:

4.6.1. THE THEORY INVOLVED:

Optical phenomena such as absorption, transmittance and reflectance of light and related interference effects can be utilized to monitor the growth of thin films during chemical deposition and vacuum deposition. The choice of the quality to be measured depends on the type of the substrate and the film to be monitored. Metal
films, for example, may be observed by transmittance measurements, provided they are deposited onto transparent substrates. However, the amount of transmitted light decreases rapidly with thickness so that sensitive measurements are limited to rather thin films\(^7\).

When light passes through a transparent medium, the transmitted intensity \(I\) is related to the maximum intensity \(I_0\) by Lambert - Beer law, which is given by,

\[
I = I_0 \exp(-\alpha d)                       \quad 4.01
\]

The same equation can also be written in terms of transmittance \(T\) as,

\[
T = T_0 \exp(-\alpha d)                       \quad 4.02
\]

Where, \(\alpha = 4\pi k/\lambda\) and \(k = \) extinction co-efficient, \(\lambda = \) wavelength of the light used and \(d\) is the film thickness.

4.6.2. THE THICKNESS MEASURING TECHNIQUE UNDER THE DESIGNED SET-UP:

For thin film thickness measurement under our designed optoelectronic system, we have used transmitted mode of the incident laser light passing through the thin films under study\(^{20-23}\). For thickness measurement of thin films by optoelectronic method, earlier workers Baruah et. al. had reported that film thickness can be
measured by scanning the object horizontally with a fixed laser light at the edge of the thin film which results a voltage signal of varying amplitude depending on the thickness of the film. In our present technique, instead of scanning the film, we have kept the film in static condition and have used a mechanical chopper for modulating the fixed incident laser light beam. The modulated transmitted laser beam passing through the thin film produces square wave like voltage signals from the output of the photo-detector. Since, we have used phototransistor (L14G1/ L14G2) which has a very fast response to even high frequency signals, thus the step input pulse produced by the chopped laser light after passing through the static film has produced similar square wave like as shown in Fig. 4.03-4.07. The unduly nature of the output pulse is due to the non-uniformity of thickness throughout the film. In this context, we have used laser light of wavelength 632 nm. Also, our designed technique is characterized by simple, fast and non-destructive nature compared to stylus and other mechanical methods of studying film thickness. At the same time accuracy of measurement is also found to be high (±5%).

Thin films of different thicknesses grown under the same deposition conditions have been used for studying the thickness dependence of voltage signal transmitted by the film. Films grown under the same deposition condition are assumed to have same optical conditions and parameters. Since the transmittance of light is dependent on absorption co-efficient of the film material, therefore, we have used films of high transmission to the wavelength used. For the observations under the designed optoelectronic system, various thin films of Zinc-Selenide (ZnSe) and Cadmium-Sulfide (CdS) have been taken (Photograph 4.01-4.02). The CdS films of various thicknesses were deposited by Chemical Method onto suitably cleaned glass
substrates held at room temperature and ZnSe films of various thicknesses were deposited by Thermal Evaporation method from ZnSe powder (99.99%) onto suitably cleaned glass substrates held at a temperature of 150°C under a vacuum (Better than $10^{-4}$ to $10^{-5}$ Torr) using Vacuum Coater (Model No.VC-12). Several films deposited under similar conditions were taken for observation. The thicknesses of ZnSe films were determined to an accuracy of 15Å with the help of Multiple Beam Interferometric method developed by Tolansky². For chemically deposited CdS films, thicknesses were measured by Gravimetric method.

While different types of thin films (CdS and ZnSe) were probed with focused pulsed laser under the designed optoelectronic system in transmitted mode, various square wavelike time varying signals have been obtained. Fig. 4.03-4.07 shows the square wave like signals obtained while scanning thermally evaporated ZnSe thin films of different thicknesses and Fig. 4.08-4.12 shows the square wave like signals obtained while scanning chemically deposited CdS thin films of different thicknesses under the designed optoelectronic system. Surface area of approximately 5mm×5mm of each film has been covered while probing under pulsed laser.

For the confirmation of the ability of the designed system in measuring thickness of thin films, films of different thicknesses were taken under observation. The average value of voltage signal heights developed at different points of each thin film relative to the probed position of the film has been measured with the help of CRO. The average voltage signal heights for different thicknesses of ZnSe and CdS thin films under study and their corresponding thicknesses are given in Table 4.01 and Table 4.02 respectively.
A graphical plot of voltage signal height against film thickness yields the thickness monogram of thin film. The graphical plot gives the average voltage developed while laser light passes through the film i.e. the relation between the intensity of laser light penetrating through the thin film vs. thickness of the thin film at the respective points. Plotting of voltage signal vs. thickness co-ordinates of respective thin films, a calibration curve between the voltages developed at the detector (phototransistor) by transmitted laser light and the thickness of respective films within a certain limit of thickness is obtained which is found to be linear in nature. While plotting the monograms, the average value of the voltage developed has been considered as the thicknesses of the films under study are not uniform at all the points as observed from the unduly nature of the detector signals (Fig. 4.03-4.12). The thickness monograms of ZnSe and CdS thin films obtained by curve fitting in Microsoft Excel are shown in Fig. 4.13 and 4.14 respectively. These monograms or calibration curves are used to determine the thickness of unknown thin film from the corresponding voltage signal obtained by chopping technique.

From the thickness monograms of the studied thermally evaporated ZnSe thin films and chemically deposited CdS thin films, separate linear equations relating the film thickness (x) and voltage (y) are obtained as following:

For thermally evaporated ZnSe thin films, the linear equations relating the film thickness (x) and voltage (y) is found to be,

\[ y = -6E - 06x + 0.0264 \]  ........................................... 4.03
For same equation for chemically deposited CdS film is found to be,

\[ y = -0.0001x + 0.9838 \] ................................. 4.04

From the above equations, one can find out the thickness of an unknown film by putting the value of \( y \) (i.e. the voltage developed by the photodetector by probing particular thin films by pulsed laser light). Similar equations can be obtained for different types of thin films by plotting of thickness monograms as discussed above.
Fig. 4.01. ZnSe thin films studied for thickness measurement

Fig. 4.02. CdS thin films studied for thickness measurement
Detector signals obtained from ZnSe thin films of various thicknesses:

Fig. 4.03

Fig. 4.04

Fig. 4.05

Fig. 4.06

Fig. 4.07
Detector signals obtained from CdS thin films of various thicknesses:

Fig. 4.08

Fig. 4.09

Fig. 4.10

Fig. 4.11

Fig. 4.12
Table: 4.01

<table>
<thead>
<tr>
<th>Thin film No.</th>
<th>Film thickness in Å</th>
<th>Voltage in milli-volts</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1700</td>
<td>0.017</td>
</tr>
<tr>
<td>2</td>
<td>2000</td>
<td>0.015</td>
</tr>
<tr>
<td>3</td>
<td>2500</td>
<td>0.012</td>
</tr>
<tr>
<td>4</td>
<td>3000</td>
<td>0.01</td>
</tr>
<tr>
<td>5</td>
<td>3600</td>
<td>0.006</td>
</tr>
</tbody>
</table>

Thin film thickness measurement observations of ZnSe films

Table: 4.02

<table>
<thead>
<tr>
<th>Thin film No.</th>
<th>Film thickness in Å</th>
<th>Voltage in milli-volts</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3600</td>
<td>0.50</td>
</tr>
<tr>
<td>2</td>
<td>3800</td>
<td>0.48</td>
</tr>
<tr>
<td>3</td>
<td>4200</td>
<td>0.42</td>
</tr>
<tr>
<td>4</td>
<td>4700</td>
<td>0.36</td>
</tr>
<tr>
<td>5</td>
<td>5100</td>
<td>0.30</td>
</tr>
</tbody>
</table>

Thin film thickness measurement observations of CdS films
Fig. 4.13. Thickness monogram of ZnSe thin films

Fig. 4.14. Thickness monogram of CdS thin films
4.7. THIN FILM SURFACE STUDY:

Topographic study of thin films refers to the study of the surface roughness, presence of unwanted speckles, micro-holes and cracks over a thin film surface\textsuperscript{27-29}. Thin solid films are required for fabrication of various semiconductor devices, ICs and other components of electronics and instrumentation system. However, the surface properties i.e. roughness and other defects such as pin-holes, cracks etc. may drastically degrade their performance and application in such devices. Therefore, study of surface profiles and detection and measurement of various defects such as pin-holes, cracks etc. plays an important role for enhanced performance and application of various thin film devices\textsuperscript{30,31}.

4.7.1. REVIEW OF SURFACE STUDY TECHNIQUES:

Various types of techniques and instrumentation have so far been applied for measuring surface roughness for various wavelength regions\textsuperscript{32}. For smooth surfaces, optical profilers produce line profiles or maps of an area and light scattering methods give statistical information about the surface properties such as r.m.s. roughness or power spectrum but not the surface topography. An optical profiler is non-contact in nature and does not damage the film surface. The highest sensitivity of the interferometer based instruments are in the sub nanometer range. Optical profilers have certain limitations. Their lateral resolutions are limited by the properties of the optical system and wavelength of the incident beam of light illuminating the surface and the maximum step height that can be measured is less than half of the incident wavelength\textsuperscript{11,23,27}.
Stylus Instruments are so often chosen for surface roughness measurement in engineering industries. As a complement to these instruments, optical instruments have been developed which have the advantage of being non-contacting, rapid and have the ability of measuring in a production environment. The main drawback of optical methods is their difficulty to attain parameters.

The optical microscope can give pictures of surfaces and sometimes quantitative information only. Scanning probe microscope can be used to produce topographic maps of surfaces on an atomic scale both laterally and vertically\textsuperscript{33,34}. The scanning electron microscope (SEM) and the scanning transmission electron microscope (STEM) are excellent for giving pictures of thin film surfaces or their cross-sections. The SEM requires steep surface slopes to produce an image with good contrast. The disadvantages of the above techniques are the need of vacuum chamber and difficulty to operate in production conditions\textsuperscript{22}.

A differential interface contrast or Nomarski\textsuperscript{35} (light) microscope is far superior to a SEM for observing roughness of structure on smooth surface. Lower magnifications from about 100X to 400X are generally better because surface slopes are larger and the contrast in the image is better. A review of various earlier methods for studying thin films and surfaces is given by Bennett\textsuperscript{11}.

Conventional scanning optical microscope (Type 1 microscope), as designed by earlier workers Baruah et.al., offers capability of surface roughness measurement from around 4000Å upwards\textsuperscript{36}.

Confocal (Type 2) scanning microscopy is another alternative for producing topographic maps of surfaces whose heights are large compared to the focal range (depth of focus) of a microscope objective i.e. more than about 1 micrometer\textsuperscript{37,38}. 

103
4.8.1 SURFACE ROUGHNESS STUDY OF THIN FILMS UNDER THE DESIGNED OPTOELECTRONIC SYSTEM:

For thin film surface study under the designed optoelectronic system, we have used reflected mode of the incident laser beam for thin film roughness study\(^{32,39}\). Unlike in the case of film thickness measurement, for thin film roughness measurement, the film under study is scanned horizontally keeping the incident laser beam fixed and moving the specimen along the horizontal direction\(^{40}\). Spatially reflected light from the film under study is focused onto another photo-detector (phototransistor) positioned in front of the specimen holder\(^{29}\) kept within a very small angle (\(<5^\circ\)). The spatially reflected light from point to point scanning of the object carries the roughness information to the detector. From the output voltage signal of the detector the roughness profile of the studied thin films are obtained.

For surface roughness study, various chemically deposited CdS thin films and thermally evaporated ZnSe thin films grown under different conditions have been used. Figures 4.15-4.18 show the roughness profiles of some of the thin films studied by our designed optoelectronic system.

The shape of the trace is found to be nearly same for a number of observations if all the design parameters and measuring parameters are kept constant. The sensitivity of roughness measurements are found to be dependent on adjustment of the laser beam within numerical aperture (NA) of the optical fiber. Depending on the type of roughness, various forms of signals are obtained as seen in the figures.
Thin film surface roughness study:

Fig. 4.15. Rough and Wavy surface

Fig. 4.16. Highly Rough Surface
Fig. 4.17. Less Rough Surface

Fig. 4.18. Smooth but Wavy Surface
4.8.2 STUDY OF MICRO-HOLES IN THIN FILMS UNDER THE DESIGNED OPTOELECTRONIC SYSTEM:

For detection and measurement of size of micro-holes present in thin films by our designed optoelectronic system, we have used transmitted mode of the incident laser light passing through the thin films under study. In this case, we have kept the film in dynamic condition. The thin film under study is scanned horizontally keeping the incident laser beam fixed and moving the specimen along the horizontal x-direction. Spatially transmitted light from the film under study is focused onto another photo-detector (phototransistor) positioned behind the specimen holder. The transmitted laser beam passing through the thin film produces voltage signals from the output of the photo-detector. From the spatially transmitted light from point to point scanning of the film carries the information regarding the presence and position of any micro-hole from the photo-voltage signal generated by the detector corresponding to each and every point along the scanned line. Corresponding to any pin-hole at any point on the film, a sharp spike is observed in the voltage signal. This is due to the direct transmission of the incident light which produces a high photocurrent signal corresponding to the position of the pinhole at that point. From the observed signal the position of such a pin-hole can easily be determined with the help of CRO under the designed optoelectronic system.

Presence of such micro-holes on various thin films under study by our optoelectronic system has been shown in figures 4.19-4.21. The sizes of such micro-holes have also been found out by measuring the width of the spikes by fitting of calibration curves with the help of known pinholes of different sizes.
Pin-holes present in various thin films:

Fig. 4.19

Fig. 4.20

Fig. 4.21
To calibrate the designed optoelectronic system for measurement of pin-hole size, several standard pinholes of different known sizes have been made from dark, hard but light plastic material and cut to the size of the specimen holder to fit into it. The size (diameter) of each of the standard pin-holes has been measured by optical microscope (Table 4.03). Putting inside the specimen holder, each of the standard pin-holes is scanned horizontally keeping the incident laser beam fixed and moving the specimen along the horizontal direction. Spatial transmitted light from the pin-holes is focused onto the photo-detector positioned in front of the specimen holder. The transmitted laser beam passing through each of the standard pin-holes gives a sharp spike of different width on the CRO screen (Fig. 4.22-4.27). The widths of the spikes are measured at the of the time-base scale for all the standard pinholes. By plotting the pinhole size vs. width of the spikes in time-base scale of CRO of respective standard pin-holes a calibration curve (Fig. 4.28) is obtained which is found to linear in nature. From the calibration curve, a linear equation relating the pin-hole size \( x \) and the spike width \( y \) is obtained which can be written as:

\[
y = 0.0136x + 0.0819
\]

This equation is used to measure the size of unknown micro-holes present in a thin film from the corresponding spike width as measured from the time-base scale. Results of measurement of the sizes of some unknown micro-holes (Fig.4.19-4.21) as observed while scanning various thin films under the designed system are incorporated at the end of this chapter.
SPIKES OBSERVED WITH PINHOLE OF KNOWN SIZE:

Fig.4.22. Pinhole-1

Fig.4.23. Pinhole-2

Fig.4.24. Pinhole-3

Fig.4.25. Pinhole-4

Fig.4.26. Pinhole-5

Fig.4.27. Pinhole-6
MEASUREMENTS OF THE STANDARD PINHOLES MADE WITH OPTICAL MICROSCOPE:

Table – 4.03

<table>
<thead>
<tr>
<th>Serial No.</th>
<th>Pinhole No. (size-wise in descending order)</th>
<th>Microscope Divisions</th>
<th>Microscopic Diameter (in μm)</th>
<th>Spike width in time-base scale of CRO (in ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Pinhole 1</td>
<td>66</td>
<td>660</td>
<td>9</td>
</tr>
<tr>
<td>2</td>
<td>Pinhole 2</td>
<td>58</td>
<td>580</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>Pinhole 3</td>
<td>52</td>
<td>520</td>
<td>7</td>
</tr>
<tr>
<td>4</td>
<td>Pinhole 4</td>
<td>46</td>
<td>460</td>
<td>6.5</td>
</tr>
<tr>
<td>5</td>
<td>Pinhole 5</td>
<td>37</td>
<td>370</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>Pinhole 6</td>
<td>24</td>
<td>240</td>
<td>3.5</td>
</tr>
</tbody>
</table>
SIZE MEASUREMENT OF UNKNOWN MICRO-HOLES WITH CALIBRATION CURVE:

\[ y = 0.0136x + 0.0819 \]

Fig. 4.28. Pin-hole width calibration curve
**Microscopic Constant:**

\[ 6 \text{ OD} = 6 \text{ S D} \]

\[ \therefore \text{Known distance between two lines} = 6 \times 0.01 = 0.06 \text{ mm.} \]

Microscopic Constant = known dist. between two lines on SM / No. of div. on ocular micrometer.

\[ \therefore \text{Microscopic Constant} = \frac{0.06}{6} = 0.01 \text{ mm} = 10 \mu\text{m}. \]

**Magnification of Microscope:**

Magnification = Magnification of objective \( \times \) Magnification of Eye-piece

\[ = 10 \times 15 \]

\[ = 150. \]

**SIZE MEASUREMENT WITH CALIBRATION CURVE:**

If, \( x = \) pinhole size in micron,

and \( y = \) Width of the spike in time base scale in ms,

Then from the calibration curve \( x \) and \( y \) are found to be related by the equation,

\[ y = 0.0136x + 0.0819 \]

\[ \therefore x = \frac{y - 0.0819}{0.0136} \]

\[ \therefore x = 4.06 \]
Results of measurement of widths of some unknown micro-holes
Using equation 4.06 are given below:

Pinhole 1 (Fig. 4.19):

Width of the observed spike = 2 ms.

∴ Width of the respective pinhole,

\[ x = \frac{y - 0.0819}{0.0136} \]

= 141.03 micron.

Pinhole 2 (Fig. 4.20):

Width of the observed spike = 2.5 ms.

∴ Width of the respective pinhole,

\[ x = \frac{y - 0.0819}{0.0136} \]

= 177.8 micron.

Pinhole 3 (Fig. 4.21):

Width of the observed spike = 4 ms.

∴ Width of the respective pinhole,

\[ x = \frac{y - 0.051}{0.0441} \]

= 288.09 micron.
4.9. CONCLUSION:

The designed optoelectronic system is found to be excellent for observing thin film thickness pattern at its various points when used in transmitted mode of operation. The method can be used as PC based method for rapid analysis of film thickness by calibrating the output voltage of the detector with known thickness of some films of same material deposited under identical condition. The known thickness has to be measured with a standard accurate measuring technique like Multiple Beam (Fizeau) Interference Method. The voltage vs. thickness curve is found to be linear up to a certain range of thickness (within 1000Å to above 5000 Å).

The designed system is also found to be suitable as surface roughness evaluation technique when used in reflection mode of operation. The technique directly provides visual display about the nature of surface roughness in magnified form on the screen of CRO/ PC monitor. The surface roughness can be measured if compared with standard deviation of the signal trace with that of a known surface.

The method is also found to be suitable for detecting the presence of micro-holes on the surface of thin films and the size of such hole can be measured in micro-meter range by comparing with holes of known size. The width of the spike directly provides the size of the micro-hole as they are related by a linear equation.

Thus the designed system is found to be simple, rapid and non-destructive due to non-contacting nature for thin film study.
References:


