CHAPTER (IV)

THIN FILM THICKNESS MEASUREMENT AND TOPOGRAPHIC STUDY BY THE DESIGNED SOM.

4.1 INTRODUCTION:

The technology of making thin solid film forms the vital part of developing solid state semiconductor and microelectronic devices. Thin films at present find wide industrial applications in fabrication of transducer, solar cells, integrated circuits etc. Thin films are used extensively in various fields like IC chip making. In addition to the commercial products, thin films are also used to obtain information about various basic properties of solids, as it is more convenient to use thin films than any other form of a solid.

The ideal superconductors for the current flow at high temperature (HTSC) are also prepared as thin film. Thin films of metals, semiconductors and dielectrics, inorganic and organic materials play an increasingly important role in the development of microelectronics, optical coatings, integrated optics and opto-electronic devices, quantum engineering, metallurgical coatings, surface engineering, solar energy conversion devices etc. In microelectronics, deposited films are widely used in the production processes of modern VLSI (Very Large Scale Integration), ULSI (Ultra Large Scale Integration) and integrated circuits. Conductive films are required to provide interconnection between contacts on devices and between devices and the outside world. Most of the solar cell devices using semiconductor junction consist of one material in thin film form. Thin films are used in solar cells with promise of high conversion efficiency (over 15%) and low cost. Various important applications of thin films in the field of optics are as anti-reflection coatings, reflection-increasing coatings and as interference filters.
Some of the photosensitive thin films are used as radiation detector from far infrared region to high frequency nuclear particles. They are also used in contact less remote acting devices or as light sensitive switches (as photodiode). Thin films of photoconductors are used in videcon pick-up tube to convert light from a scene into electric signals. Thin film phototransistors constitute another active photo-device in optoelectronics.

Surface characterization is also an increasingly important area of research in industrial applications in monitoring thin film surface of any material in general and semiconductor material in specific. Surface roughness can take many forms. Often it consists of tiny scratches in random directions remaining after polishing.

The area of surface study is mostly involved in performance characteristics of electronics, photonics and optoelectronic devices. The accurate measurement of the surface profile helps in designing such devices with higher performance capability in detection, to make microelectronic circuits and to achieve higher information storage density in optical compact discs and magnetic disks and many related applications. The existing methods for finding surface roughness have been discussed in this chapter.

4.2 DEFINITION OF THIN FILM:

Thin solid films are two-dimensional micro-materials consisting of layers with plane parallel faces. Mathematically an ideal film can be regarded as bounded by two parallel planes extended infinitely in two directions but is restricted along the third direction known as thickness ($t$). The volume of thickness can vary from the limit $t \to 0$ to any other values but always remaining much less than those of other two dimensions. As in case of thin film, the material thickness varies in the dimension of thickness and becomes comparable or less than the mean free path of electrical conduction process.
penetration depth of light, effective deBroglie wavelength, diffusion length etc. In additional effects will appear. That is why in almost all optical applications of thin films require coatings of well defined and precisely controlled thickness.

4.3 THEORY OF THIN FILM DEPOSITION:
The first evaporated thin film was probably the deposited, which Faraday obtained in 1857 when he exploded metal wires in an inert atmosphere. Further experimentation in the nineteenth century was stimulated by interest in the optical phenomena associated with thin layers of materials and by investigations of the kinetics and diffusion of gases. The possibility of depositing thin metal films in a vacuum by Joule heating of platinum wires was discovered in 1887 by Nahrwold and a year later adapted by Kundt for the purpose of measuring refractive indices of metal films.

The vapour deposition, sputtering method and chemical deposition are common and popular in depositing thin films over suitable substrate. In vapour deposition and sputtering method the emission of atoms from the specific material and its deposition over the substrate is considered to be uniform, as the substrates are planar and parallel to the plane of the emitting surface.

The amount of material which condenses on an opposing surface depends on the position of the receiving surface with regard to the emission source. The material contained in an evaporant beam of solid angle \( d\omega \) covers an area, which increases with distance as well as with the angle of incidence \( \theta \). The element of the receiving surface which corresponds to \( d\omega \) is \( dA_r = r^2 d\omega / \cos \theta \). Therefore, the mass deposited per unit area is

\[
\frac{dm}{dA_r} = r^2 \frac{d\omega}{\cos \theta}
\]

\[
\text{(72)}
\]
\[
\frac{dM_r(\rho, \theta)}{dA_r} = \left[ \frac{M_a}{\pi r^2} \right] \cos \rho \cos \theta.
\] (4.01)

For a receiving element \(d A_r\) within the solid angle \(d\omega\), the dependence on source distance and the beam direction is the same as for a small surface source, \(dA_r = r^2 d\omega / \cos \theta\), and therefore the amount of deposit received from a point source is

\[
\frac{dM_r}{dA_r} = \left[ \frac{M_a}{4\pi r^2} \right] \cos \theta.
\] (4.02)

The receiving surface of uniform deposit is a sphere with the point source in the centre, so that \(\cos \theta = 1\) and \(r = \text{constant}\). The equation 4.02 is a function of source-to-substrate distance and angle of incidence. Hence, thickness profiles can be derived for substrate areas of any given shape or position relative to the source. Normally the substrates are planar and parallel to the plane of the emitting surface. But in actual case, each and every points of the substrates are not equally parallel to the plane of the emitting surface. That is also clear from the equation 4.02. Which shows that the distribution of atom over the substrate is dependent of the atomising angle \((\cos \theta)\).
4.4 REVIEW OF METHODS FOR THICKNESS MEASUREMENT:

Traditionally, monitoring devices have been constructed by individual investigators to suit their particular purposes. Several types of monitors have become commercially available, after sufficient operational experience have accumulated as regards ways to ensure ruggedness, various ranges of application and desired practical designs.

The best technique for a specific application or process depends upon the film type, the thickness of the film, the accuracy desired, and the use of the film. These criteria include such properties as film thickness, film transparency, film hardness, thickness uniformity, substrate smoothness, substrate optical properties, and substrate size. In many cases there is no single best technique, and the particular one chosen will be determined by the personal preferences of the investigator.

Since thin film thickness is generally of the order of a wavelength of light, various types of optical interference phenomena have been used to measure film thickness. There are also some other optical techniques like ellipsometry and absorption spectroscopy, which can be used to measure thickness. In addition to the optical techniques, there are mechanical, electrical, and magnetic techniques too, which have been used for film-thickness measurements.

Different system of measuring film thickness, together with their useful ranges and accuracies are given below. In some cases, only the precision is listed under accuracy because the absolute accuracy is not available. The accuracy is given in terms of a unit of length for thinner films and in terms of a 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 other factors. Some techniques are omitted because their dependence on particular details to be listed in such a general form.
Multiple-beam (Flzeau) method: The range of this method is 30 - 20,000 Å° and the accuracy or the precision is in between 10 - 30 Å°. In this process a step and evaporated reflective coating is necessary.

Multiple-beam (FECO) method: The range is 10 - 20,000 Å° and the accuracy is 2 Å° in each step. In this technique, an evaporated reflective coating and a spectrograph is needed. It is a very accurate system but time-consuming.

Michelson interferometer: The range of this method is in between 300 to 20,000 Å° and the accuracy is 150 to 300 Å°. The technique require a step over the thin film.

Polarisation Interferometer: The range is 300 to 20,000 Å° and the accuracy is 150 to 300 Å°. This technique also require a step over the thin film.

Colour comparisons: The range of the system is in between 500 to 15,000 Å° and the accuracy is 100 to 200 Å°. This values are useful only for SiO₂ on Si materials. The technique is limited to transparent films on substrate such that reflectivities at the two interferences are not too different.

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

CARIS Method: The range of this technique is 400 Å° to 20 μ and the accuracy is 10 Å° to 0.1 %. The method is useful for transparent films. This method is also non-destructive, but to measure thickness of the thin film, one must know the n if only one angle of incidence is used.
Ellipsometry method: The range of this method is of few A° to few microns. The accuracy is around 1 A° to 0.1 %. This method can be use for transparent films. The mathematics of calculation for this technique is a bit complicated, especially with thicker films. PC based data acquisition and analysis of thin films by Spectroscopic Ellipsometer has been reported by J.V.Khedkar et.al. 10

Light - section microscope system: The range of this system is 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 technique the n must know.

Gravimetric system: The range is from few armstrong to no limit. The accuracy or the precision is greater than 1 A° to 1%. This technique gives an average over sample. For this technique one must know the film density.

Stylus method: The range of this method is 20 A° to no limit with an accuracy of several armstrong 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.

X-ray absorption: The range of X-ray absorption method is in between 0.1 to 1,000 micron. The accuracy is ± 5%. In this method, the substrate must produce characteristic radiation.

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

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 11.
4.5 THICKNESS MEASUREMENT UNDER SOM SET-UP:

4.5.1 The theory involved:

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

$$T_r = T_0 \exp (-\alpha d)$$  

(4.03)

Where $\alpha$ is the absorption coefficient. Similar considerations hold for reflectance measurements on metal films. Therefore, optical monitoring techniques are primarily used for dielectric films.

The sensing devices which allow measurements during the evaporation process are referred to as either thickness or rate monitors. They exploit different physical effects to determine the density of the evaporant stream, the mass of the deposit or a thickness-dependent film property.

[* This equation can also be written as $I = I_0 \exp (-\alpha d)$, where $\alpha = 4\pi k / \lambda$ and $k$ = extinction coefficient, $\lambda$ = wavelength of light used and $I_0 = (1 - R)$, which changes greatly with thickness $d$]
4.5.2 The measuring technique under SOM:

Thin films of Cadmium-Telluride (CdTe) of various thickness were deposited by thermal evaporation of CdTe (99.999%) onto suitably cleaned glass substrates held at room temperature under a vacuum (better than $1.33 \times 10^{-4}$ Pa) using HIND HIVAC coating unit (model 12 A 4). Films deposited under similar conditions were taken for observations. The thickness of the films were determined to an accuracy of 15 Å with the help of multiple beam interferometry method developed by Tolansky.

Various square wave like time varying signals are obtained while scanned at the edge of the thin films. Photographs 4.01 - 4.06 are some of the square waves developed while scanning the thin films of different thickness under SOM set-up. Three different points (upper part, middle part and lower part) at a distances of 3mm have been scanned for each film.

Figures 4.01 - 4.04 are the plot of voltage developed at different point for each thin film against the position of the scanned point. For the confirmation of the ability of SOM geometry in measuring thin film thickness, films of different thickness were scanned. Here only few numbers of graphical representations of thin film scanned under the designed SOM are shown. The graph for each film shows the voltage (ranging from 10 to 500 mV) developed when laser light passes through each point of the film i.e. the relation between the intensity of laser light penetrating through the thin film vs. the thickness of the film at the respective points.

Plotting voltage vs. thickness co-ordinates of respective thin film measured by Tolansky's method, a monogram of linear relation between the voltage formed at the photo-diode by transmitted laser light and the thickness of respective film within a certain limit of thickness is obtained. While plotting the monogram, only few number of points are considered, as the thickness of the used films are not uniform in all the points as observed through the voltage developed by each point.
SOME PHOTOGRAPHS OF THE FORMATION OF SQUARE WAVE WHILE SCANNING THE THIN FILMS UNDER SOM FOR THICKNESS MEASUREMENT.
FIGURE 4.01 - 4.04 SHOWS THE PLOTS OF VOLTAGE DEVELOPED AT DIFFERENT POINTS OF THIN FILMS AGAINST THE POSITION OF THE SCANNED POINT IN EACH FILM:

( The figures are also shows the inverse of the thickness pattern of each film, as the voltage developed by each point is inversely and exponentially proportional to thickness of the film as given in the equation 4.03.)
X-axis: Scan position
1. upper
2. lower

Y-axis: Voltage developed (in volt)
1. 0.44
2. 0.4

FIG. 4.01 (A)

X-axis: Scan position
1. upper
2. lower
3. middle

Y-axis: Voltage developed (in volt)
1. 0.14
2. 0.2
3. 0.06

FIG. 4.01 (B)

X-axis: Scan position
1. upper
2. middle
3. lower

Y-axis: Voltage developed (in volt)
1. 0.06
2. 0.07
3. 0.165

FIG. 4.01 (C)
X-axis: Scan position
Y-axis: Voltage developed
1. upper 0.38
2. middle 0.52
3. lower 0.4

FIG. 4.02 (A)

X-axis: Scan position
Y-axis: Voltage developed
1. upper 0.2
2. middle 0.52
3. lower 0.56

FIG. 4.02 (B)

X-axis: Scan position
Y-axis: Voltage developed
1. upper 0.08
2. middle 0.145
3. lower 0.09

FIG. 4.02 (C)
FIG. 4.03 (A)

X-axis: Scan position
1. upper 2. middle 3. lower
Y-axis: Voltage developed (in volt)
0.3 0.3 0.52

FIG. 4.03 (B)

X-axis: Scan position
1. upper 2. middle 3. lower
Y-axis: Voltage developed (in volt)
0.28 0.2 0.28

FIG. 4.03 (C)

X-axis: Scan position
1. upper 2. middle 3. lower
Y-axis: Voltage developed (in volt)
0.32 0.42 0.18
X-axis: Scan position
1. upper
2. middle
3. lower
Y-axis: Voltage developed (in volt)
- upper 0.36
- middle 0.52
- lower 0.4

FIG. 4.04 (A)

X-axis: Scan position
1. upper
2. middle
3. lower
Y-axis: Voltage developed (in volt)
- upper 0.24
- lower 0.013

FIG. 4.04 (B)

X-axis: Scan position
1. upper
2. middle
3. lower
Y-axis: Voltage developed (in volt)
- upper 0.185
- middle 0.15
- lower 0.19

FIG. 4.04 (C)
This also shows the ability of SOM in finding thickness pattern of a thin film at different point of the same film. Figure 4.05 is the plot of the above monogram. Reliability and repeatability was tested.

Following the same conditions and the similar set-up with same kind of sample, one can find out the thickness of a film, putting the value (voltage developed by the photodetector for a laser while penetrating through the particular point of the thin film where the thickness should be measured) over the monogram 24.

4.6 REVIEW OF METHODS FOR SURFACE STUDIES:

Figure 4.06 shows a diagram of different types of instruments and techniques that are suitable for measuring surface features in various range. The lateral dimensions or separations of the surface features, called surface spatial wavelengths or some time surface wavelengths, are plotted on logarithmic scale 22, 25, 26.

For smooth surfaces, optical and mechanical profilers produce line profiles or maps of an area and light scattering methods give information about surface statistical properties such as r.m.s. roughness or power spectrum but no surface topography. An optical profiler is non contact and cannot damage the surface. The highest sensitivity of the interferometer-based instruments are in the sub nanometer range. Optical profiler has some limitations. Their lateral resolutions are limited by the properties of the optical systems and by the wave length of the light beam illuminating the surface and the maximum step height that can be measured is less than half of the incident wavelength 27, 28.

Stylus instruments are most 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 ability to
FIG. 4.05: MONOGRAM OF THIN FILM THICKNESS MEASUREMENT.
Fig. 4.06: Techniques for measuring surface roughness in various wavelength regions.
measure in a production environment. The main drawback of optical methods is their difficulty to attain parameters.

The optical microscopes can give pictures of surfaces and sometimes quantitative information only. Scanning probe microscopes can be used to produce topographic maps of surfaces on an atomic scale. 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 scanning electron microscope requires steep surface slopes to produce an image with good contrast. The disadvantage of the above techniques are the need of vacuum chamber and difficult to use in production environment.

A differential interface contrast or Nomarsky (light) microscope is far superior to a scanning electron microscope for observing roughness 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.

Conventional SOM as designed by us offers capability of surface roughness measurement from around 4000 Å upwards. However, the chief advantage of SOM lies in thickness measurement of thin film (refer to page 30 – 32, chapter II).

Confocal microscopy is another alternative for producing topographic maps of surfaces whose heights are large compared with the focal range (depth of focus) of a microscope objective, i.e. more than about 1 micro meter.

4.7 TOPOGRAPHIC STUDY OF SOME SEMICONDUCTOR MATERIAL UNDER SOM SET-UP:

Topographic study i.e. the surface roughness, the unwanted speckles, microholes or cracks over a surface, their detection and measurement of sizes are the area of application of the SOM thus designed. Here we have discussed the observation of
various rough and smooth surfaces of semiconductor materials by application of the designed SOM.

Photograph 4.07 (A) is the surface of a vacuum deposited CdTe thin film observed by Scanning Electron Microscope (SEM) [Model: JEOL made, No. JSM–35 CF], which shows two speckles. The same specimen is scanned under the SOM in the reflected mode shown in photograph 4.08. The spikes are appeared as the speckles present over the thin film.

The line scan over a 200 μm area of a vacuum deposited CdTe thin film is seen in the photograph 4.09. The spikes are appeared as the uneven surface having some tracks formed over the thin film surface as observed from the photograph 4.07 (B) taken under a SEM.

Photograph 4.10 is the line scan of a semiconductor surface having some microholes over it, detected by the designed SOM. The spikes are appeared due to the microholes present over the surface.

To calibrate the designed SOM set-up in studying surface roughness, we have scanned three different known sizes (5, 10, and 20 micron dia.) of pinholes. The photograph 4.11, 4.12 and 4.13 shows the spikes formation while scanning the pinholes. The widths of the spikes at the base of the time scale for the three samples and the size of the known (standard) pin holes are plotted in the graph of figure 4.07. The plotted graph shows the linear relation between the pinhole size and the width of the spikes over the time base scale. From this monogram the size of an unknown pinhole can be found out by knowing the width of the spikes from the time based scale while scanning under the SOM.
Photograph 4.07: Taken by Scanning Electron Microscope (SEM).
(A) Speckles present over the thin film.
(B) Unwanted tracks formed over the thin film.

Photograph 4.08: Spikes (of speckles over thin film) are appeared, while observed under SOM in reflected mode.
Photograph 4.09: Spikes are appeared due to the uneven surface. (observed under SOM in reflected mode)

Photograph 4.10: Spikes are appeared due to the microhole present over a thin film (observed under SOM) in transmitted mode.
PHOTOGRAPHS OF THE SPIKES APPEARED, WHILE PIN-HOLES OF DIFFERENT DIAMETERS ARE SCANNED UNDER SOM SET-UP (IN TRANSMITTED MODE):

Photograph 4.11: Spike of a pinhole of 5 micron dia.

Photograph 4.12: Spike of a pinhole of 10 micron dia.

Photograph 4.13: Spike of a pinhole of 20 micron dia.
FIG. 4.07: PLOT OF PINHOLE SIZE VS. WIDTH OF THE SPIKE
4.8 CONCLUSIONS:

4.8.1 Advantages of SOM:

The SOM is an excellent system for measuring thin film thickness as well as for finding thickness pattern of a thin film at different point of the same film.

SOM geometry is also useful in studying surface morphology, formation of cracks, pinholes, speckles etc over a surface in micro level dimension.

Further, SOM is non-destructive in nature as the low power laser probing technique is used in measuring thin film thickness and reading surface profile. Hence the possibility of damaging the specimen is less in SOM system.

The system is found to be very fast. It requires only a fraction of a second to analyse the data as everything is done electronically. Also electronic recording of the measured data is possible in SOM system.

It requires no extra treatment of the sample while measuring or studying under SOM set-up.

While probing a specimen under SOM, it is exposed to atmosphere and no vacuum or special type of environment is needed around the specimen. Hence SOM can be used in studying the specimen itself during the growth of the specimen in an industrial atmosphere with a slight modification of the system.

In the photograph 4.11, it is observed that, the width of the spike of 5 micron pin-hole over the base line of the time scale is 7.30 milli-second. But the output device (i.e, CRO) of the present set-up of SOM is capable of measuring micro-second scale. Hence, it can be concluded that, the present set-up of SOM geometry is capable of measuring an object of size even smaller than 5 micron.
4.8.2 Disadvantages:

It requires another standard measuring technique to calibrate the SOM set-up.

Only a certain range of thickness and a particular type of thin film material can be measured for a particular setting under the SOM set-up.

The range of a particular material is very small. For CdTe thin film the range is between 1500 A° to 8000 A°. ³³,³⁶
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