Experimental Details
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2.1 Introduction:

In this chapter the experimental details of magnesium oxide, aluminium oxide and their co-deposited mixed thin films preparations and characterization are elaborated. Vacuum evaporation technique has been used for the thin film deposition. The cost effective Vapour Chopping Techniques (VCT) is used to improve the quality of thin film. The equipments used for the thin film deposition and characterization of above mentioned thin films have been given.

The optical properties of MgO, Al$_2$O$_3$ and their co-deposited mixed thin films were studied by using UV-visible spectrophotometer. The mechanical properties such as adhesion and intrinsic stress of thin films were studied by Direct Pull Off (DPO) method and Interferometric method respectively. The secondary electron emission coefficient was measured by secondary electron emission coefficient measurement system. The optical signal loss was measured by prism coupling method. The effects of ambient air exposure on the thin films were also studied.

2.2 Substrate cleaning:

During the production of various devices by using sophisticated thin film technique; the proper substrate cleaning is required for homogeneous and uniform thin film deposition with higher adherence.

The first step of substrate cleaning is to pre-clean the substrate in an ultrasonic cleaner filled with distilled water for 5-10 minutes. Then slides are dipped in
the dilute chromic acid for 10 min. The substrates were wiped by clean cotton. During this whole cleaning process the handling of slides should be done with care. For handling the substrate, metal tweezers was used. Then, it was cleaned again in the ultrasonic bath. After this the slides were rinsed with glass wares cleaning detergent. It is important to use very dilute solution of cleaner, because it is difficult to remove. Again these slides were wiped by cotton. Finally these slides were rinsed by hot distilled water in ultrasonic cleaner for 40 °C. The purpose of this was to remove all traces of the detergent.

The substrates have been air dried in clean area, because cleaned substrates can pick up the dust from air by electrostatic attraction at a remarkable rate. These slides were ready for to use as substrates for the thin film depositions [1].

2.3 Method used for thin film deposition:

In this present work, vacuum evaporation technique was used to deposit magnesium and aluminium thin films. 99.98% pure magnesium turnings obtained from Alfa Aesar were used as source for Mg thin films whereas 99.99% pure aluminium wire obtained from Balzers Ltd. (Switzerland) used to deposit Al thin films. Deposition source material has been given in table 2.1. Co-deposited mixed thin films were prepared by using same source materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (gm/cc)</th>
<th>Melting point (°C)</th>
<th>Evaporation sources</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnesium</td>
<td>1.738</td>
<td>650</td>
<td>Filament: Mo, Ta, Chromel.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Boat/crucible: Mo, Ta, W</td>
</tr>
<tr>
<td>Aluminium</td>
<td>2.70</td>
<td>660</td>
<td>Filament: Mo, Ta, W, Nb, Chromel. Boat/crucible:</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Mo, Ta, W</td>
</tr>
</tbody>
</table>

Table 2.1 Source material description

The deposited Mg thin films were oxidized by thermal oxidation in air for various oxidation temperatures (673 K, 623K and 573 K) and durations (90 min
and 180 min). The magnesium thin films started to oxidize at 573 K, which was observed by the changing of colour of mirror like reflecting Mg thin films converted into colourless (transparent) films. The oxidation completed at 673 K and after the 673 K, MgO thin films starts to recrystallize. This was observed by the TGA-DSC report shown in fig. 2.1.

![TGA-DSC of MgO powder](image)

Figure 2.1: TGA-DSC of MgO powder.

The deposited aluminium thin films were oxidized by hot water oxidation. Thin films became transparent from colored (shiny) thin films. For Mg-Al co-deposited thin films were oxidized by steam oxidation with thermal heating

**2.4 Vacuum evaporation technique [2]:**

Vacuum evaporation technique was used in this work for the thin film deposition using Hind Hi-Vac (Model 12A40) vacuum system. In this work $\sim 10^{-6}$ Torr pressure was used.
The most important part of the vacuum system is the glass dome used as vacuum chamber of 2 cm thick and 15 cm in diameter. The chamber made up of steel material can also be used. The basic requirement is that, it should not degas under high vacuum. It is also known as the experimental chamber. The source evaporation heating arrangement was installed in this section. In resistive heating system, the evaporation is done by using the filament or boat. It is made up of the refractory materials like tungsten, molybdenum or tantalum etc. The use of the refractory material depends on the evaporating temperature range of the source material. This chamber also consists of substrate holder, which is fixed at a suitable distance from the evaporator. The chopper is also fixed inside the chamber.

Figure 2.2: Photograph of vacuum coating unit.

The bottom of this dome was covered with gasket of Neoprim rubber which was kept on a steel base plate. The vacuum grease was used to gasket. It forms a
thin gel layer in between dome and steel base plate and it helps to reduce the air leakage. The base plate had 12 port holes for electrical feed throughs. It consist a air admittance valve to leak the air while opening the dome.

**Pumping system** evacuates the gas molecules from the vacuum chamber and starts to reduce the pressure. The vacuum evaporation system used in this work consists of two pumps mechanical (rotary) and diffusion pump. Direct drive Oil sealed van type rotary mechanical pump (Model-ED-15) was used. It can evacuate the chamber pressure up to $10^{-3}$ to $10^{-4}$ Torr, depending on the evacuation speed.

To obtain the pressure up to $10^{-6}$ Torr, the diffusion pump was used. It works on the principle of evacuation of the molecule by momentum transfer from the streaming to diffusing molecules. The work fluid inside the chamber is heated in a boiler/ heater and their hot vapor rises to a chimney. The direction of the flow is reversed at the jet cap so that the vapour comes out from the nozzle directed away from the high vacuum side or in a downward direction. The vapour stream come out from the higher pressure to lower that’s why expands and acquire maximum velocity than the gas molecules at thermal equilibrium. The gas molecule thus diffuses in the stream of vapour and receives the velocity components and directed towards the fore vacuum side. Thus the zone of more reduced molecules generates at the point of nozzle and maximum number of gas molecules diffuses in that region and acquire the velocity. Thus the pressure in the lower region increases and it can be evacuated by using a backing pump such as rotary pump.

The pressure inside the chamber is measured by using **pressure gauges**. In our vacuum system two types of gauges i. e. Pirani and Penning were used depending on the range of pressure measurement. Pirani gauge was used for $10^{-4}$ Torr pressure range and above this range penning gauge was used up to $10^{-7}$ Torr.

Vacuum chamber, diffusion pump and rotary pump are separated by using different types of **valves**. High vacuum valve is used for the separation of vacuum
chamber and diffusion pump. Roughing valve is used to separate the vacuum chamber from the rotary pump. It is used for the roughly evacuate the vacuum chamber by using only rotary pump therefore nominated as roughing valve. Backing valve is used to separate the diffusion pump and rotary pump. The diffusion pump is not a self evacuating pump so it requires backing pump for evacuation. Rotary pump is used as backing pump there for this valve is known as backing valve.

2.5 Vapour chopping technique: [3-5]

Vapour chopping technique (VCT) is a cost effective technique. It modifies the various properties of thin films. VCT consists of a chopper which is a circular aluminium metal sheet of 10 cm diameter having a V-cut (155°) shape as shown in fig. 2.3. This thin circular vane was fixed to a light aluminium rod attached to a motor. The rate of chopping in this study was 6 rot/sec. As the chopper rotates, the filaments are exposed to the substrates at intervals. The vane was at the height of 10.5 cm from the source. The distance between source and substrate was 12.5 cm.

Figure 2.3: Photograph of the vapour chopping technique.
2.6 Oxidation of metal thin films:

In this work magnesium and aluminium metal thin films were prepared by using the appropriate deposition material sources. Co-deposited mixed Mg-Al thin films were deposited by using two different boats by resistive heating method. The vapour chopped thin films were prepared by using vapour chopping technique. These films were converted into oxides by using different oxidation methods. For magnesium oxide, thermal heating in ambient air was used. The thermal oxidation was carried out by using heater. The chromel-alumel thermocouple was used as temperature measurement sensor. This setup was put into the fume hood having continuous flow of air. The hot water oxidation was suitable for aluminium oxide. The aluminium metal thin films were boiled in hot water. Because of their different oxidation potential, the magnesium oxide and aluminium oxide doesn’t have a good oxidation performance in hot water and air respectively. We can prepare aluminium oxide in air but for this the substrate temperature required was above 450 °C, and at this temperature the glass substrate starts softening. Hence the steam oxidation method was used for the oxidation of co-deposited mixed Mg-Al thin films.

2.7 Measurements / Characterizations Techniques:

The prepared oxide thin films were studied using different characterization technique. The x-ray diffractometer (XRD), scanning electron microscope (SEM), UV-VIS spectrophotometer technique were used to identify crystal structural, surface morphology and optical studies respectively. In this section, different analytical instrumental techniques used to characterize the thin films are described with relevant principles of their operation and working.

2.7.1 Crystal Structure Determination:

In nanotechnology, XRD is a useful tool, since x-rays are in the nano-scale. It is used to identify the composition, phase and crystallite orientations of the material. X-ray diffraction is almost non-destructive and non-contact in regards to the sample being analyzed. It works on the basic principle of ‘Bragg’s law’.
\[ n\lambda = 2dsin\theta \]

Where,

- \( n \) = an integer (we use 1 for most calculations)
- \( \lambda \) = the wavelength of the x-ray (this is 1.54 Å if copper is the target metal)
- \( d \) = the distance between atoms in angstroms (Å)
- \( \theta \) = the diffraction angle in degrees

By knowing the distance and angles between the atoms of a substance, the crystal structure could be determined.

The instrument used for the crystal structure determination has the specification as follows:

**Instrument specifications:**

- **Model:** PW 3710/ PW1710 PHILIPS, Holland
- **Angle (2\( \theta \)):** 10 to 100 degree
- **Target:** Cu or Cr
- **Normal mode:** PW3710/PW 1710 APD Controller.
- **Specimen:** Fine Powder (quantity approximately 1 cm\(^3\))

Applications: Crystallographic studies, Quantitative analysis of organic, inorganic minerals, metals and alloys.

The crystallite size of the deposits is estimated from the full width at half maxima (FWHM) of the most intense diffraction lines by Scherrer’s formula as –

\[ D = \frac{(k\lambda)}{(\beta \cos\theta)} \]

Where \( D \) is crystallite size, \( \lambda \) is wavelength of X ray, \( \beta \) is FWHM of the peak in radians, \( \theta \) is the Bragg’s angle and \( k \) is constant. The value of \( k \) varies from 0.89 to 1.39, but for most cases it is closer to unity.

**2.7.2 Surface Morphological Study:**

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope. It has a higher magnification, larger depth
of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.

The Scanning Electron Microscopy (SEM) has been widely used for characterization of two-dimensional thin films surface morphology. All the SEMs’ have facilities for detection of secondary and back-scattered electrons, which uses incident electron beams with energy between 2 and 40 KeV. The scattering of electrons from atom results into secondary and backscattered electrons. The secondary electrons are orbital electrons knocked out of sample atoms by collisions with incident electron beam. The escape depth of secondary electron is low due to their lower energy (50 eV). Consequently, these electrons are generated at a specimen depth of few nanometers in metal and few tens of nanometers in insulator. The region from they originate, is a little larger than beam diameter. The typical resolution may be ~ 5 nm. [6]

The samples used for the scanning must be conducting to avoid charge build up caused by the impinging electrons and resulting in jumping of beam. Non conducting samples can be scanned by coating conducting layer of Pt-Au alloy.

For topographic examinations, the specimens are generally tilted some 20-40 degree towards the detector in order to get maximum number of secondary electrons. The detectors count the number of secondary electrons produced at its spot on the surface. Simultaneously, the spot of CRT is also scanned as the brightness of the spot is modulated by the amplified current from the detector. The overall effect is to produce a topographic image of the surface on the CRT screen. Magnification of the surface is simply attained by making the specimen raster much smaller than the CRT raster.

Backscattered electrons can also be used in SEM when higher energy electrons penetrate in to the bulk of the specimen, some undergo Rutherford back scattering with atoms and re-immerge as back scattered electrons. Thus, the back scattered electrons provides information about the bulk (~ 500 K). The resolution
is poor, due to spreading of electrons. This technique is very useful to study the microstructure, grain size, surface morphology etc.

Figure 2.4: The ray diagram of scanning electron microscope

The Scanning Electron Microscopy (SEM) has been widely used for characterization of two dimensional surface topography of thin film. The scanning microscope used in this work was JSM-6360, JEOL, Japan. fig. 2.4 shows the ray diagram of scanning electron microscope.

In Scanning Electron Microscope, a well-focused mono-energetic (~25KeV) beam incident on a solid surface and gives various signals as mentioned
above. Backscattered electrons and secondary electrons are particularly pertinent for SEM application, their intensity being dependent on the atomic number of the host atoms. Each may be collected, amplified and utilized to control the brightness of the spot on a cathode ray tube. To obtain signals from an area, the electron beam is scanned over the specimen surface by two pairs of electro-magnetic deflection coils and so is the C.R.T. beam in synchronization with this. The signals are transferred from point to point and signal map of the scanned area is displayed on a long persistent phosphor C.R.T. screen. Change in brightness represents change of a particular property within the scanned area of the specimen [7]. The ray diagram of scanning electron microscope is shown in fig. 2.4. Interaction of energetic electron beam with solid surface leads to several signals like elastically scattered electrons (i.e. change of direction without change of energy) from the coulomb field of the nucleus, whereas some other includes inelastically scattered electrons (with change of energy) from the electrons of the host atoms giving rise secondary electrons, Auger electrons and x-rays characteristics to host lattice. The secondary electrons (signal from approximately top 100 Å) are used to get contrast from surface morphology.

2.7.3 Atomic force microscope (AFM):

Any surface of thin film has irregularities on the surface. These irregularities depend on various parameters. Such irregularities can observe by using SEM or FESEM. But atomic force microscope (AFM) gives the proper view of those irregularities in the form of surface roughness. Because AFM works on simple principle, an atomically sharp tip is scanned over a surface with feedback mechanisms that enable the piezo-electric scanners to maintain the tip at a constant force (to obtain height information), or height (to obtain force information) above the sample surface. Tips are typically made from Si₃N₄ or Si, and extended down from the end of a cantilever. The nanoscope AFM head employs an optical detection system in which the tip is attached to the underside of a reflective cantilever. A diode laser is focused onto the back of a reflective cantilever. As the
tip scans the surface of the sample, moving up and down with the contour of the surface, the laser beam is deflected off the attached cantilever into a dual element photodiode. The photodetector measures the difference in light intensities between the upper and lower photodetectors, and then convert into voltage. Feedback from the photodiode difference signal, through software control from the computer, enables the tip to maintain either a constant force or constant height above the sample. In the constant force mode the piezo-electric transducer monitors real time height deviation. In the constant height mode the deflection force on the sample is recorded. The latter mode of operation requires calibration parameters of the scanning tip to be inserted in the sensitivity of the AFM head during force calibration of the microscope. The schematic diagram of AFM is given in fig. 2.5.

![Schematic Diagram of AFM](image.png)

Figure 2.5: The schematic diagram of AFM.

An atomic force microscope Molecular Imaging (MI) USA make equipment is used in non-contact, acoustic AC mode to observed surface
topography and determines the roughness parameter of thin films. Its cantilever was NSC 12 (C) from MikroMasch with force constant 4.5 N/m and frequency 160 KHz.

2.7.4 Tolansky Interferometric method [8]:

Film thickness can be measured accurately from interference fringes using multiple beam interferometer and also from the fringes of equal chromatic order (FECO) techniques. In the former case, two reflecting surfaces are brought in close proximity such that a small wedge with a small air gap in between them is formed. If a monochromatic light is now incident on them at normal incidence, then an interference of light due to interactions of multiple reflected beams in air gap will take place resulting in a series of fringes (Fizeau) which can be observed in the back reflected light. The distance between fringes or lines depends on the air gap as well as the wavelength of the monochromatic light. This principle is adopted and suitably modified for the multiple beam interferometric method of the measurement of film thickness [8].

Figure 2.6: Tolansky method for thin film thickness measurement.
The film, the thickness of which is to be determined, is deposited on a flat surface so as to leave a sharp edge between the film and the uncoated region of the substrate. An optically flat surface is generally preferred as a substrate and often a good microscope glass slide is good enough for this purpose. The substrate with a half coated film is then deposited with a thin highly reflecting coating of a metal such as silver or aluminium so as to form a sharp step on the film edge. Another optically flat glass side or cover slip known as the reference plate, it is placed over the specimen with its film coated surface in contact with each other so as to leave a small air gap in the step. A monochromatic parallel beam of light passing through a beam splitter or a glass plate inclined at 45° is then incident on the two plate assembly and reflected light is then observed through a microscope or recorded on a photographic plate. A set of sharp fringes perpendicular to the step with equal displacements will be observed (as shown in fig. 2.6) and the thickness \(t\) can be determined using the relation.

\[
t = \frac{b\lambda}{2a}
\]

Where, ‘b’ is fringe shift at the step and ‘a’ is the distance between consecutive fringes and ‘\(\lambda\)’ is the wavelength of light used. These fringe displacements which are in the form of parallel lines occur at the film edge. The sharpness of fringes depends on the reflectivity of the film coating, the spread of the incident beam, air gap etc. This technique is capable to measure the thickness up to \(\lambda/2\) where, \(\lambda\) is the wavelength of light used.

2.7.5 UV–Visible Spectrophotometer:

Thin film optical properties study is most important for variety of applications. This deals primarily with reflectance (R), transmittance (T) and absorbance (A) properties and their relation to the optical constants of the film. The optical constant such as refractive index, optical band gap, and optical transmittance can be studied by UV-visible spectrophotometer. For crystallite materials, the transition of electrons from valence band to conduction band can be grouped in to direct and indirect process [6]. In direct inter band optical
transitions, the value of wave vector \( k \) for elements remains unchanged in E-k space and momentum also does not change, whereas in indirect inter band transition, the wave vector \( k \) for electrons changes in the E-k space. Thus, vertical transitions are important when the valence and conduction bands extreme are located at the same point in the Brillouin zone. For indirect transition, the band extreme differs in their positions in E-k space. It is possible to differentiate the nature of optical transition as direct allowed or direct forbidden by using classical relation –

\[
\alpha = \frac{\alpha_0(h\nu-E_g)^n}{(h\nu)}
\]

Where, \( E_g \) is the separation between bottom of the conduction band and top of the valence band, \( h\nu \) is the photon energy, \( n \) is constant. For allowed direct transition, \( n = 1/2 \) and allowed indirect transition, \( n = 2 \). Thus if the plot of \( (\alpha h\nu)^2 \) against \( h\nu \) is linear, the transition is direct allowed. Band gap energy \( E_g \) is determined by extrapolating the linear portion of the curve to the energy axis at \( \alpha = 0 \). Though the optical band gap, nature of transition, transmission etc are easily obtained from optical absorption, the reflectance \( R \) is assumed to be zero and the equation depends on only classical approach.

The optical transmittance and absorbance of the films deposited on to glass substrates were measured using a spectrophotometer (Hitachi Japan, Model No. 330). The light emitted from the source (deuterium or tungsten lamp) is passed through the two grating monochromators for obtaining monochromatic beam and then it is splitted in to two beams. After passing through the sample and reference compartment, the monochromatic beams are converged and then converted in to an electrical signal by a detector or photomultiplier tube. The detected signal is digitized by a 16 bit A/D converter. The wavelength range used was 350 nm to 850 nm. The internal block diagram is shown in the fig. 2.7.
Figure 2.7: Schematic of internal block diagram of spectrophotometer.

In UV-VIS spectroscopy, it allows light of given frequency to pass through a sample and detect the amount of transmitted light. The instrument compares the intensity of the transmitted and incident light and plot a graph of %T Vs confined varying wavelength. The percent transmitted is given by a formula:

\[
\%T = \frac{I_0}{I_n} \times 100
\]

where,

\(I_0\)=Intensity of transmitted light.

\(I_n\)=Intensity of incident light.

and the absorption is the measure of logarithm of reciprocal of transmittance.

\[
A = \log \left( \frac{1}{T} \right)
\]

Mathematically it is found that,

\[
\log (I_0) - \log (I_n) = kb
\]

where,

k- Proportionality constant

b- Path length of the sample.
The equation (3) is “Beer-Lamberts law” and defined as the intensity of transmittance decreases logarithmically as the path length increases arithmetically. But “Beer’s” found that increasing the concentration of absorber had the same effect as increasing the path length “b”. Thus the proportionality constant is in turn proportional to the concentration of “C” of the absorbing solute. Or k=aC

Thus the equation (2) becomes,

$$A= \log \left( \frac{1}{T} \right) = \log \left( \frac{I_0}{I_n} \right) = A B_c$$

---

where,

A - Molar absorptive factor.

Thus equation (4) is the fundamental equation, important in the spectrophotometric analysis. This is called as “Beer’s Lambert equation”.

2.7.5.1 Optical Absorption Studies

The essential feature of dielectric optical film material is their very low absorption ($\alpha<10^3$ cm$^{-1}$) in the relevant region of the spectrum. The equilibrium situation in semiconductor can be disturbed by generation of carriers due to optical photon absorption. Optical photon incident on any material may be reflected, transmitted or absorbed. The phenomena of radiation absorption in a material is altogether considered to be due to 1) inner shell electrons 2) valence band electrons 3) free carriers including holes as well as electrons and 4) electrons bound to localized impurity centers or defects of some type. In study of the fundamental properties of some semiconductors, the absorption by the second type of electrons is of great importance. In an ideal semiconductor, at absolute zero temperature, the valence band would be completely full of electrons so that electron can not be excited to a higher energy state from the valence band. Absorption of quanta of sufficient energy tends to transfer the electrons from valence band to conduction band. The optical absorption spectra of semiconductors generally exhibits a sharp rise at a certain value of the incident photon energy which can be attributed to the excitation of electrons from valence to conduction band (may also involve acceptor or donor impurity levels, traps
etc.). The conservation of energy and momentum must be satisfied in optical absorption process. Basically there are two types of optical transitions that can occur at the fundamental edge of the crystalline semiconductor, direct and indirect. Both involve the interaction of an electromagnetic wave with an electron in the valence band, which is rose across the fundamental gap in the conduction band. However, indirect transition also involves simultaneous interaction with lattice vibration. Thus the wave vector of the electron can change in the optical transition. The momentum change being taken or given up by phonon. The direct interband optical transition involves a vertical transition of electrons from the valence band to the conduction band such that there is no change in the momentum of the electrons and energy is conserved as shown in fig. 2.8 (a). Hence a wave vector $k$ for electron remains unchanged in E-K space. The optical transition is denoted by a vertical upward narrow. Absorption coefficient $\alpha$ for simple parabolic bands and for direct transition is given by the relation [9]

$$\alpha = \alpha_o \left( \frac{h \nu - E_g}{h \nu} \right)^n / h \nu$$

where $E_g$ is the separation between bottom of the conduction and top of the valence band, $h \nu$ is the photon energy, $n$ is constant and is equal to 1/2 or 3/2 depending on whether transition is allowed or forbidden and $\alpha_o$ is a constant depending upon the transition probability for direct transition. For allowed direct transitions $n = 1/2$ and for allowed indirect transition $n = 2$. Thus if the plot of $(\alpha h \nu)^2$ against $h \nu$ is linear then the transition is direct allowed. The band gap energy $E_g$ is determined by extrapolating the linear portion of the curve to the energy axis at $=0$.

When interband transition takes place between different k-states as shown in fig. 2.8 (b) since these must satisfy the momentum conservation laws, the only way such a transition can take place is through the emission or absorption of a phonon with wave vector $q$ as;

$$K' + q = k + K$$
The transition defined by equation (2.9) is termed indirect transition. For indirect transition

\[ a = a_o (hv - Eg)^n / hv \]

where \( Eg = Eg' \pm Ep \), \( Eg \) is indirect band gap energy and \( Ep \) is the phonon energy. For allowed transition \( n = 2 \) and for forbidden transition \( n = 3 \). The band gap energy is determined by extrapolating the linear portion of the plot \((hv)^n\) versus \( hu \) to the energy axis at \( \alpha = 0 \).

Figure 2.8: E-k diagrams showing (a) direct and (b) indirect inter-band transition.

### 2.7.6 Refractive index measurement:

Refractive index is one of the important optical constant in optically transparent thin film. In this present work, two methods have been used to measure the refractive index of thin spectrophotometric method.

This method is based on the spectrometric results, by measuring the simultaneous absorption and transmittance of thin film at eye sensitive 621 nm wavelength, the reflectance has been calculated by using relation,

\[ \text{Transmittance} + \text{Absorption} + \text{Reflectance} = 1 \]

The refractive index \( n \) of thin films calculated by using the following formula [10].
\[ n = \left( \frac{n_s^2 T_f + n_s \left(1 + \sqrt{R_f}\right)^2}{T_f + n_s \left(1 - \sqrt{R_f}\right)^2} \right)^{1/2} \]

Where,

- \( n \) = refractive index of the film
- \( n_s \) = refractive index of the substrate
- \( T_f \) = transmittance of the film
- \( R_f \) = reflectance of the film

### 2.7.7 Prism coupling method for waveguide studies [11-12]:

The prism film coupling method provides a convenient and efficient method of launching a laser beam into a thin film structure.

In this method, the right angle prism is placed above the thin film guide and is separated from it by a small air gap having low refractive index. An incoming laser beam enters the film through prism. The coupler permits excitation of any one of the film mode by proper orientation of the direction of the incident beam.

The experimental arrangement shown in photograph (2.9)

![Figure 2.9: Experimental setup of prism coupling method.](image-url)
For efficient prism coupling, the refractive index of the prism should be higher than the refractive index of waveguiding medium (film) and the air gap between the prism and the top of the film surface should be in the order of wavelength of light used. The important condition is that the incident laser beam must have the proper angle of incidence so that the evanescent field in the gap travels with the same phase velocity to be excited in the film. Under this condition, when the laser beam is incident on the input prism, it will be launched into the thin film guide and can be taken out by a prism placed at the output end. The optical signal passing through thin film is showed in photograph (2.10).

![Figure 2.10: Photograph of the optical signal passing through thin film.](image)

In the present, we have used the two special right angle prism made of extra dense flint glass with a refractive index 1.79. These two identical prisms had a base area of 1 cm² and angle 90°-45°-45° (Supreme Optics, Pune). All sides of the prism were highly polished and a sharp edge was maintained at the angle of 90°. The top of the prism was cut up to the width of 2 mm for mounting purpose.

The prism was pressed from the top of the prism as well as from the back side of the thin film waveguide using the fine brass screws. Care was taken to press the prism slightly away from the sharp edge at the prism angle of 90°. Two rectangular brass fixtures were used to mount the input and output prisms. The
spacing of the air gap between the base of the prism and the top surface of the film was adjusted in the order of wavelength for effective coupling. The experimental arrangement is as shown in the photograph.

A streak of light observed in thin film waveguide is shown in the photograph fig. 2.10. The optical transmission loss is measured by measuring the output intensity of the output prism. The output intensity of 1.5 cm guiding length has been measured in the form of current using the silicon photo diode by keeping the input intensity constant (80 µm). After all these measurements, the transmission loss has been calculated for 1.5 cm guiding length in decibels using the formula

\[ a = 10 \log \left( \frac{\text{input intensity}}{\text{output intensity}} \right) \text{dB/cm} \]

2.7.8 Secondary electron emission yield measurement system:

Secondary electron emission yield was measured by secondary electron emission measurement system built at Samtel Display center of Indian Institute of Technology, Kanpur (IIT). The pictorial view of the secondary electron emission measurement set-up is shown in fig. 2.11.

In this technique, a discharge is produced in a cell (or pixel) containing pure noble gases or their mixtures and conditions similar to those existing in real PDPs. and Paschen curve (breakdown voltage versus p×d, where p is the pressure in the system and d is the separation between the two MgO thin films) are obtained and matched with the model calculations to yield the ion-induced secondary electron emission coefficient (\( \gamma \)) values. The firing voltage in such a set-up is uniquely related to the ion-induced \( \gamma \)-value without involvement of any fudge factor. The method has been described by Auday et al [17] and was later improved by Bachmann et al at [8]. The breakdown voltage for a particular gas discharge set-up depends only on the product p×d (Paschen law). Plasma firing voltages vs. the (p×d) were measured using the set-up depicted in fig. 2.12.
The dielectric layer (MgO or Si-doped MgO in the present case) to be tested is deposited onto 1"×1" cover slip/glass substrate. Two such layers are arranged to form an open glass cell with the coated inner surfaces facing each other at a well-defined distance (0.2 - 0.5 mm) by a spacer. Confining plasma in a small gap in a higher pressure condition is a challenging task. The plasma spreads to the metallic chamber walls suitable for sustaining it. Therefore, the cell area and electrodes are covered with a glass assembly as shown in fig. 2.12 (b). Copper electrodes of 10 mm diameter are used to connect the mounted cell to an AC high voltage source. The cell is then joined in series with a reference capacitor $C_{\text{ref}}$ having a known value (fig. 2.12 c). Charge transfer occurs due to firing of plasma between the cell and $C_{\text{ref}}$. The voltage ($V_r$) measured across $C_{\text{ref}}$ is displayed.
against the total voltage applied to the system (V_a) in the oscilloscope, as ‘Lissajous’ figure. In the absence of plasma discharge, Lissajous figure may attain any shape (i.e. elliptical, circular, straight line, etc.) depending on the frequency fig. 2.12 (d).

Figure 2.12: Secondary electron emission measurements setup.

As soon as plasma ignites, the Lissajous figure between V_R and V_a begin to show up (fig. 2.12 d). The plasma firing voltage (V_i) was determined from the Lissajous figures using
\[ V_f = \left\{ \frac{0.5 \times C_{\text{diel}}}{(C_{\text{gap}} + 0.5 \times C_{\text{diel}})} \right\} \times V_s + \left\{ \frac{C_{\text{ref}}}{C_{\text{diel}}} \times V_r \right\} \]

where \( C_{\text{diel}}, C_{\text{ref}} \) and \( C_{\text{gap}} \) are capacitances across two adjacent dielectric layers, reference capacitor and inter electrode gap respectively as shown in fig. 2.11 (c). \( V_s \) and \( V_r \) are calculated from Lissajous figure that appears after plasma strikes inside the discharge cell, as shown in fig. 2.12 (d).

The firing voltage \( (V_f) \) also deduced theoretically using

\[ V_f = D^2 \cdot \frac{(p \times d)}{[\ln \left[ C \cdot (p \times d) / \ln \left\{ (1/\gamma_i)^{-1} + 1 \right\} \right]^2} \]

Where, \( C \) and \( D \) are constants having values for He, Ar, Ne and Xe in table 2.2

<table>
<thead>
<tr>
<th>Constants</th>
<th>He</th>
<th>Ne</th>
<th>Ar</th>
<th>Xe</th>
</tr>
</thead>
<tbody>
<tr>
<td>( C )</td>
<td>4.4</td>
<td>8.2</td>
<td>29.2</td>
<td>65.3</td>
</tr>
<tr>
<td>( D )</td>
<td>14</td>
<td>17</td>
<td>26.6</td>
<td>36.1</td>
</tr>
</tbody>
</table>

Table 2.2 The values of \( C \) and \( D \) constants [30].

Figure 2.13: Photograph of plasma obtained during SEE yield measurement.

The experimental \( V_f \) vs \((p \times d)\) plot was compared with the theoretical paschen curve to see best fit and in turn, determine the value of gamma. The plasma cleaning is required for to suck out all the adsorbed gas impurities by MgO thin film showed in fig. 2.13. For the SEE yield measurement and analysis high voltage amplifier, spectra RGA vacscan plus, function waveform generator,
oscilloscope, Neon gas cylinder, ultra high vacuum chamber and temperature controller equipment are required with SEE system.

**High voltage amplifier:**

An electronic amplifier is a device for increasing the power of a signal. It does this by taking energy from a power supply and controlling the output to match the input signal shape but with a larger amplitude. In this sense, an amplifier may be considered as modulating the output of the power supply. The Trec Model 609E-6 high voltage DC –stable power amplifier was use in this work. It is designed to provide precise control of bi-polar output voltages in the range of 0 to ± 4 kV DC or peak AC with an output current capability of ±20 mA DC or peak AC. It can be configured as a noninverting amplifier with a fixed gain of 1000 V/V, or as an inverting or differential amplifier. Features include an all-solid state designe, a slew rate grater than 150 V/µs, a small signal-bandwidth of grater than 35 kHz, and low noise operation.

The four –quadrant active output stage sinks or sources current into reactive or resistive loads throughout responses and high slew retes demanded by reactive loads. When the amplifier enters a current trip condition, the high –voltage output is disable, the Trip status indicator will illuminate and the trip minotor output will provide to TTL low.

**Waveform generator:**

A functional/arbitrary waveform generator (AWG) is a piece of electronic test equipment used to generate electrical waveforms. These waveforms can be either repetitive or single-shot (once only) in which case some kind of triggering source is required (internal or external). The resulting waveforms can be injected into a device under test and analyzed as they progress through the device, confirming the proper operation of the device. Agilent 33220A 20 MHz functional/arbitrary waveform generator was used in this work. Wave generator can generate any arbitrarily defined wave shapes such as sine, square, ramp, triangle, noise and
pulse as their output. The outputs may display a graph of the waveform on generator’s screen in graph mode.

**Oscilloscope:**

An oscilloscope is a type of electronic test instrument that allows signal voltages to be viewed, usually as a two-dimensional graph of one or more electrical potential differences (vertical axis) plotted as a function of time or of some other voltage (horizontal axis). Although an oscilloscope displays voltage on its vertical axis, any other quantity that can be converted to a voltage can be displayed as well. In most instances, oscilloscopes show events that repeat with either no change, or change slowly. The oscilloscope is one of the most versatile and widely-used electronic instruments. The HP-54600 B oscilloscope has been used to plot the Lissajous figures. Lissajous figures obtained in between applied voltage \(V_a\) and voltage across the reference capacitor \(C_{ref} (V_R)\). which helps to measure the sustain voltage \(V_s\) and \(V_R\).

**Ultra high vacuum chamber:**

Ultra high vacuum (UHV) is the vacuum regime characterized by pressures lower than about \(10^{-7}\) pascal or 100 nanopascals (10–9 mbar, ~10–9 torr). UHV was used for the purpose of extreme cleanliness to maintain the vacuum system, and baking of the entire system to remove water and other trace gases that adsorb on the surfaces of the chamber. At these low pressures the mean free path of a gas molecule increase, so gas molecules will collide with the chamber walls many times before colliding with each other. The required condition for plasma cleaning can only maintain under the ultra high vacuum. The glass assembly showed in fig. 2.10 b) put into this chamber.

**2.7.9 Direct pull off method for adhesion measurement:**

In the present work, the adhesion was measured by a simple and convenient direct pull off (DPO) method [13-16]. The adhesion testing apparatus for adhesion measurement used is shown in fig. 2.14.
This adhesion testing apparatus consists of two hard metal chucks aligned on the same axis with one movable and other fixed. The movable chuck was attached to a spring balance of maximum capacity of 50 Kg, with an accuracy of ± 0.5 Kg. The spring arm of balance was connected to a pulling mechanism through a metal cable. Aluminium studs of 0.5 cm diameter and 5 cm length attached to the film side and back side of the substrate with an adhesive ‘Araldite’. In order to align the studs during curing a special stud fixture was made which is shown in the fig. 2.13. After curing the adhesive, the adhesion was measured by monitoring the load when the failure occurred at the film substrate interface. The adhesion was calculated using the formula

\[ \sigma = \frac{(g \times F)}{A} \]

Where

\( g \) = gravitational force
\( F \) = load in kg used to occur failure at film substrate interface
\( A \) = area of stud (0.005 m)

![Diagram of adhesion testing apparatus](image)

Figure 2.14: Schematic diagram of (A) adhesion testing apparatus (B) Sample holder for stud alignment.
2.7.10 Interferometric method for measurement of intrinsic stress [15]:

In this method, the deflection of a circular plate after deposition determines the stress in the films. The deflection is related to the radius of curvature of circular plate which can be measured interferometrically by observing the changes in the optical fringes between the plate and optical flat forming the Newton’s ring. The departure of the circularity of the fringes offers the possibility of observing stress anisotropy. The radius of curvature of the plate is measured by determining the diameter of fringes on circular plate using an optical microscope. The experimental arrangement for stress measurement is shown in the fig. 2.15. The obtained fringes are given in figure.
The substrate used was soda lime cover slips of diameter 1.9 cm and 0.022 cm thickness. In order to obtain a measurable curvature due to film stress, the glass substrate must be thin. Majority of commercially available cover slips are not optically flat. However few are intrinsically concave in shape. Cover slips of measurable curvature are selected by placing them on to $\lambda/10$ optical flat and choosing the ones which formed circular or oval fringes (Newton’s rings) on being viewed in monochromatic light.

When the substrate is placed on the optical flat and is lightly pressed and released, the fringes patterns moves first in and then out if the substrate is lying with its concave side down and vice-versa if it is lying with its concave side up. This occurs because the distance between the substrate and optical flat at given point which is responsible for the interference fringes formation in accordance with the equations,

\[ 2d = n \lambda \quad \text{(Minima)} \]
\[ 2d = (n+1) \lambda/2 \quad \text{(Maxima)} \]

The fringe pattern obtained by placing the substrate (concave side down) on the optical flat and viewing it in monochromatic light is measured using traveling microscope. Light from monochromatic source is made incident normally on to the substrate placed on optical flat using a partially reflecting surface. Light reflected from the optical flat and from the substrate film interface interferes constructively and destructively and the resultant view pattern is viewed with the help of a traveling microscope focused from the top.

From the fringe pattern, the radii of the fringes are measured in the X and Y direction and plots of $d_n$ vs $r_n^2$ are obtained where $d_n = n (\lambda)^n/2$ is the distance between the curved film and the optical flat which is responsible for fringe of order ‘n’ and $r_n$ is the radius of the fringe of order ‘n’.

The slopes of $K_x$ and $K_y$ of the plots of $d_n$ vs $r_n^2$ are related to the value of the tension per unit area $T_x$ and $T_y$ in the X and Y directions.

\[ K_x = 3t \left( T_x - T_y \right) / (Yh^2) \]
and

\[ Ky = 3t \left( T_y - T_x \right) / (Yh^2) \]

These are used to calculate the stress “S” in the film,

\[ S = (Yh^2 (K_x - K_y)) / (6t (1 - \nu)) \]

Where

- \( Y = 2.5 \times 10^{10} \) …..Young’s modulus
- \( \nu = 0.22 \) ………Poisson’s ration
- \( t = \) film thickness
- \( h = 0.022 \) cm substrate thickness

since the uncoated glass cover slips are not totally flat but have a measurable curvature, the values of slopes obtained from the uncoated glass cover slips are denoted by \( K_x' \) and \( K_y' \). These values are subtracted from the values obtained for coated glass cover slips denoted by \( K_x'' \) and \( K_y'' \) so as to obtain correct \( K_x \) and \( K_y \) values due to stress “S” in the films.
References:

    (1996) 120.