Chapter 2

Material Methods and Characterization Techniques

2.1. Introduction

The development of science and technology in recent years has replaced the traditional and arduous experimental techniques of analysis by sophisticated instrumental techniques of analysis, which give more precise and reproducible results. It is necessary to have a good insight into various experimental techniques for carrying out a fruitful research work [1, 2]. This chapter is devoted to the principles of the experimental methods and characterization techniques adopted in the present studies. Here, we discuss briefly preparation of cadmium oxide thin films by spray pyrolysis method. A brief description of the home built setup of the spray pyrolysis unit will be discussed. Next, the preparation of ZnO and CdSe nanoparticles respectively by aqueous route will be discussed. The X-ray diffractometry (XRD) and infrared spectroscopy (FT-IR) were employed for structural analysis. Elemental analysis was carried out by energy dispersive analysis of X-ray (EDAX). Surface morphology and particle size determination was done using scanning electron microscope (SEM) and transmission electron microscope (TEM). UV-Vis absorption and fluorescence spectroscopy studies were done for the as prepared samples. Current-voltage curves are recorded at room temperature using Keithley 617 programmable electrometer. Electrical conductivity measurements of CdO films deposited on glass were carried out using the four probe Hall effect measurements.

2.2. Cadmium Oxide Thin Film Preparation by Spray Pyrolysis Technique

Firstly, 0.04 M of cadmium acetate is dissolved in 30 ml methanol. CdO thin films are
prepared by spraying the above solution on properly cleaned glass slides at temperature 375 °C in open conditions. This is done using our home built spray setup. The specially designed atomizer made of teflon fitted in borosil conical flask was used. The advantage of this setup is, contamination of spray solution can be prevented.
that typically occurs in commercial setup which is usually made up of metal components. The substrate temperature is optimized to 375 \(^0\)C, since at higher temperature a significant decrease in transport of mass towards the substrate due to rapid evaporation of the solvent is observed. The deposition is done using the above shown home built setup. As the fine droplets of the spray solution reach the hot substrate, the pyrolytic decomposition of solution results in well adherent, uniform yellowish colored CdO films. To understand the effect of pH of spray solution on the electrical properties of CdO thin films, spray solution with different concentration of ammonia and HCl, respectively, were prepared.

2.3. Synthesis of Semiconductor Nanomaterials

2.3.1. Synthesis of ZnO Nanoparticles

Thiol-stabilized zinc oxide nanoparticles were synthesized via an aqueous route under moderate temperature to obtain better crystalline materials. The work has been focused to study the effect of molecular dipole moment of capping molecule on optoelectronic properties of these nanoparticles.

**Sample Preparation:** In a typical procedure, 0.5 M zinc nitrate hexahydrate (Zn\(\text{(NO}_3\text{)}_2\cdot6\text{H}_2\text{O}\)) is dissolved in distilled water and kept for heating at 80 °C. Finally 0.2 M of NaOH solution in distilled water is prepared separately in another flask and is added drop by drop to the above zinc nitrate solution. The solution is further heated for 3 hrs and the precipitate obtained is washed thoroughly with distilled water. The ZnO powder obtained is left to dry in open air. To synthesize thiol stabilized ZnO nanoparticles 0.2 M of each capping agent respectively is added to the 0.5 M zinc nitrate solution. Three different organic thiols such as 1-thioglycerol (C\(_3\)H\(_8\)O\(_2\)S), thioglycolic acid (C\(_2\)H\(_4\)O\(_2\)S) and 2-mercaptoethanol (C\(_2\)H\(_6\)SO) possessing different dipole moment were used as capping molecules. The white precipitates were
achieved by maintaining the temperature of bath at 80 °C and were then vigorously stirred at this temperature before filtering, rinsed with distilled water and finally centrifuged and dried for further measurements.

2.3.2. Synthesis of CdSe Quantum Dots

A single-step synthesis of stable, luminescent aqueous colloids of mercaptopropionic acid capped cadmium selenide nanoparticles of controllable size is established [3]. The reaction was carried out in open-atmosphere with one-pot by using selenium dioxide to replace selenium or its other hazardous, expensive and unstable precursors. Mercaptopropionic acid acts as both selenium reducer and capping molecule in the synthesis of these quantum dots. This new precursor of selenium has been successfully implemented to develop a new aqueous method to obtain fluorescent nanoparticles.

**Sample Preparation:** CdCl₂.H₂O is dissolved in distilled water to which 250 mM 3-mercaptopropionic acid (3-MPA) is added. The solution is not completely clear and hence ammonia is added drop by drop till a clear solution was obtained. In a separate flask, 50 mM SeO₂ is dissolved in water, SeO₂ is readily soluble in water. This mixture is slowly added to the above solution. The pH value of the reaction mixture plays a very important role in the aqueous synthesis of semiconductor quantum dots, ensuring their stability and providing control to their growth. The optimum pH value for the QD synthesis depends strongly on the nature of the stabilizer. In the present synthesis protocol, the initial pH of cadmium precursor solution after the addition 3-mercaptopropionic acid was ~ 4.5. The pH of the solution was adjusted to 11 by dropwise addition of ammonia to get a clear solution. In the absence of ammonia and keeping the other synthetic parameters fixed, a relatively fast and uncontrolled growth of QDs was observed. Similarly, in case of selenium dioxide (SeO₂), for the formation
of selenium ions the presence of ammonia is a crucial parameter. The precursors are converted to CdSe QDs by refluxing the reaction mixture at 100 °C under open air conditions with condenser attached. Temperatures for all the samples were maintained at 100 °C and the solutions were refluxed for different time to obtain different sizes of CdSe QDs. Initially the solution is clear, just after 15 min of heating the sample turns light turbid. For S1, the sample is heated for ~30 min till it turns white in color; for S2 the same solution is heated for one hour till it turns yellow in color and for S3 the solution is heated for one and half hours till it turns orange in color. Later, all the colloids are allowed to cool under open conditions. Reactions were also carried out for a longer duration that gives red color precipitate that appears to be more close to the bulk.

2.4. Characterization Techniques

2.4.1. X-Ray Diffraction

The crystalline structure of thin films prepared in this work was examined using X-ray diffraction (XRD). XRD is a powerful non-destructive technique for characterization of crystalline materials. It gives information about chemical composition and structure of the material. A real three-dimensional crystal consists of many sets of atomic planes. These atomic layers are spaced at a distance \( d \) and are named as \( d \)-spacings. A large number of atomic planes having same \( d \) are assigned by its Miller indices \((hkl)\) and the \( d \)-spaces between \((hkl)\) planes denoted by \( d_{hkl} \). When X-ray beams irradiate the crystal surface at certain angles of incidence, these planes of atoms cause incident X-rays to be scattered. There is a constructive interference from specular reflectance of X-rays of particular values of \( d \)-spacing at a particular scattering angle \( \theta_{hkl} \) onto the \((hkl)\) planes, resulting in a diffraction peak. The condition for the constructive interference is given by the famous Bragg law,
where the integer \( n \) is known as the order of the reflection, \( \lambda \) is the X-ray wavelength and \( \theta \) is the scattering angle. The diffracted angle, \( 2\theta \), is defined between the incident

\[
2d_{hkl}\sin\theta_{hkl} = n\lambda
\]  

\text{(2.1)}

Figure 2.2: Schematic diagram of X-ray diffraction.
beam and the detector angle. The incident angle $\theta$ is always half of the detector angle $\Theta$. X-rays are incident on the samples over a range of angles $\theta$, while reflections are detected over the range of angles $\Theta$. This XRD scan mode is commonly known as the $0 - 2\theta$ scans. For single crystal samples, there is only one orientation for each $(hkl)$ plane where the Bragg diffraction law is satisfied [4, 5].

The entire X-ray diffraction setup is schematically shown in figure 2.2(b). X-ray diffractometer consist of three basic elements: an X-ray tube, a sample holder and an X-ray detector. X-rays are generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being $K_{\alpha}$ and $K_{\beta}$. $K_{\alpha}$ consists, in part, of $K_{\alpha 1}$ and $K_{\alpha 2}$. $K_{\alpha 1}$ has a slightly shorter wavelength and twice the intensity as $K_{\alpha 2}$. The specific wavelengths are characteristic of the target material (Cu, Fe, Mo, and Cr). Filtering, by foils or crystal monochrometers, is required to produce monochromatic X-rays needed for diffraction. $K_{\alpha 1}$ and $K_{\alpha 2}$ are sufficiently close in wavelength such that a weighted average of the two is used. Copper is the most common target material for single-crystal diffraction, with CuK$_{\alpha}$ radiation = 1.54056 Å. These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor. The geometry of an X-ray diffractometer is such that the sample rotates in
the path of the collimated X-ray beam at an angle \( \theta \) while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 20. The instrument used to maintain the angle and rotate the sample is termed as a goniometer. As the sample is rotated, the count rates will reflect the flux of diffracted X-ray photons for that orientation. When the geometry of the incident X-rays impinging the sample satisfies the Bragg equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor [4, 5].

**2.4.2. Scanning Electron Microscope**

Scanning electron microscopes use a beam of highly energetic electrons to examine objects on a very fine scale. In a SEM, when an electron beam strikes a sample, a large number of signals are generated. This examination can yield information such as topography (the surface features of an object), composition (the elements and compounds that the object is composed of and the relative amounts of them) and crystallographic information (how the atoms are arranged in the object). The combination of high magnification, large depth of focus, good resolution, and the ease of observation makes the SEM one of the most widely used equipments.

A high-energy beam of electrons is used to scan across the surface of the sample. A schematic of a typical scanning electron microscope is shown in figure 2.3(a). The electrons are generated by thermionic emission from a metal filament (electron gun), and accelerated to \( \sim 25 \) kV (normally 10 kV to 30 kV used to make images). The electron beam is scanned, or ‘ rastered’ across the sample via magnetic scan coils. When the electron beam strikes a sample, it will scatter through the sample
Figure 2.3: (a) Scanning electron microscope working principle, (b) Electron beam interaction diagram
and the depth (below the surface) at which electron interaction occurs is called the penetration depth or the electron range. The volume of sample containing (most of) the scattered electrons is called the interaction volume (figure 2.3(b), and is usually represented as pear-shaped in cross-section since scattering causes the beam to spread laterally as the electrons continue to penetrate and slow down.

During the electron beam-specimen interactions, secondary products like secondary electrons, backscattered electrons, X-rays, heat and light will be formed. Image formation of surface structures using the SEM mainly depends on the production of secondary electrons. Also, by measuring the amount of energy present in the X rays being released by a specimen, it is possible to identify the elements in a given sample by quantitative analysis. For this work, the primary use of SEM was to generate high magnification images of the samples. Images are generated by variations in the scattering intensity of secondary electrons. Secondary electrons are low energy electrons and when produced deeper within the interaction volume, will be absorbed by the sample. Only secondary electrons close to the surface will be able to escape from the specimen. The low energy secondary electrons will be deflected by a positive pull exerted by the Faraday cage surrounding the secondary electron detector and therefore will contribute to the image formation. Different number of secondary electrons produced at different areas of the sample will provide image contrast according to the peak and valley on the sample surface. If at a certain spot on the sample more secondary electrons are produced, a bright spot will appear on the image. The current of electrons reflected from the surface is collected, amplified, and plotted as a two-dimensional ‘micrograph’ image of the signal intensity. To get the surface morphology of the sample, the sample surface must be electrically conducting and should be electrically connected with the instrument via sample holder, otherwise
the electron beam would charge up the surface leading to poor image quality. Therefore for non-conducting samples normally a thin layer (10-30 nm) of gold is deposited on top of the given sample by sputtering [6-8].

In conjunction with the scanning electron microscopy, energy dispersive x-ray spectroscopy (EDS or EDAX) is technique that utilizes X-rays emitted from the sample during the bombardment by the electron beam to characterize the elemental composition of material imaged in a SEM. When the sample is bombarded by the electron beam of the SEM, electrons are ejected from the atoms comprising the surface of sample. A resulting electron vacancy is filled by an electron from a higher shell, and a characteristic X-ray is emitted to balance the energy difference between the two electrons. The EDX X-ray detector measures the number of emitted X-ray versus their energy. The energy of the X-ray is characteristic of the element from which the X-ray was emitted. A spectrum of the energy versus relative counts of the detected X-ray is obtained and evaluated for qualitative and quantitative determination of the elements present in the sampled volume.

2.4.3. Transmission Electron Microscope

The conventional electron microscopy is nowadays called TEM (transmission electron microscopy). The initial designs of the microscopes were able to magnify specimens’ up to seventeen times greater than that of a light microscope; modern designs have a resolution almost of 10,000 times greater. One of the major historical limitations of TEM was that electrons were largely unable to pass through thick specimens; until the diamond knife and ultra-microtome were designed in 1951; it was largely impossible to utilize this instrument to full capacity. While the theoretical upper limit of the transmission electron microscope is estimated to be as high as 10,000 times that of a light microscope, flaws in the equipment used lower the real
Figure 2.4: Transmission electron microscope working principle
limit. A schematic ray diagram of TEM is shown in figure 2.4. The ray of electrons is produced by thermionic emission by a pin-shaped cathode heated up by current. The electrons are accelerated by a high voltage at the anode. The acceleration voltage is between 50 and 150 kV. The higher it is, the shorter are the electron waves and the higher is the power of resolution. But this factor is hardly ever limiting. The power of resolution of electron microscopy is usually restrained by the quality of the lens-systems and especially by the technique with which the preparation has been achieved. Modern equipments have powers of resolution that range from 0.2 – 0.3 nm. The useful magnitude is therefore around 300,000. The accelerated beam of electrons passes a drill-hole at the bottom of the anode. Its following way is analogous to that of a ray of light in a light microscope. The lens-systems consist of electronic coils generating an electromagnetic field. The ray is first focused by a condenser. It then passes through the object, where it is partially deflected. The degree of deflection depends on the electron density of the object. The greater the mass of the atoms, the greater is the degree of deflection. Biological objects have only weak contrasts since they consist mainly of atoms with low atomic numbers (C, H, N, O). Consequently, it is necessary to treat the preparations with special contrast enhancing chemicals (heavy metals) to get at least some contrast. Additionally, they are not to be thicker than 100 nm, because the temperature rises due to electron absorption. It is generally impossible to examine living objects [9, 10].

After passing through the object, the scattered electrons are collected by an objective. Thereby an image is formed, that is subsequently enlarged by an additional lens-system (called projective with electron microscopes). Thus formed image is made visible on a fluorescent screen or it is documented on photographic material or digital data by high resolution CCD camera. Photos taken with electron microscopes
are always black and white. The degree of darkness corresponds to the electron density (= differences in atom masses) of the candled preparation.

2.4.4. Fourier Transform Infrared Spectrophotometer

Any absorption technique is based on the fact that, a chemical substance possesses the property of absorbing characteristics wavelengths, which they normally emit in the excited state. IR spectroscopy is the measurement of wavelength and intensity of the absorption of mid-infrared light by a sample through atomic vibration. Mid-infrared light (2.5-50 μm, 400-4000 cm⁻¹) is energetic enough to excite molecular vibrations to higher energy levels. The wavelength of IR absorption bands are characteristics of specific types of chemical bonds, and IR spectroscopy finds its greatest utility for identification of organic and organometalic molecules.

IR absorption spectroscopy is one of the most powerful techniques used for chemical identification, quantitative analysis and structural analysis of chemical substances. As compared to other absorption spectroscopy techniques such as UV spectroscopy and Raman spectroscopy, the IR spectrum provides a rich array of absorption bands, which provides a wealth of structural information about the molecules and their interaction. IR spectroscopy involves two kinds of fundamental vibrations for molecular – stretching and bending.

- **Stretching:** In this case, the distance between the two atoms increases or decreases, but the atoms remain in the same band axis. Stretching vibrations are found to occur in the order of band strength.

- **Bending:** In this case, the position of the atoms changes relative to the original band axis. Bending vibrations generally require less energy and occur at longer wavelengths. Band intensities in IR spectrum may be expressed either as transmittance (T) or as absorbance (A).
Transmittance: It is defined as the ratio of radiant powder transmitted by a sample to the radiant powder incident on the sample.

Absorbance: It is defined as the logarithm, to the base 10, of the reciprocal of the transmittance, i.e. \( A = \log_{10} \left( \frac{1}{T} \right) \)

The sample is prepared in the form of pellets, for which the sample powder was mixed with dry potassium bromide and by applying high pressure a pellet was prepared. The instrument was first set for 100% transmittance. The spectrum was scanned by placing the sample pellet in the sample beam in the range 400-4000 cm\(^{-1}\). A schematic diagram of commercial FTIR spectrophotometer is shown in figure 2.5.
The key components of Fourier transform (FT) system are the source, the interferometer and the detector. The interferometer provides a means for the spectrometer to measure all optical frequencies simultaneously. The interferometer modulates the intensity of individual frequencies of radiation before the detector picks up the signal. The product of an interferometer scan is called an interferogram, a plot of intensity versus mirror position (indicated by data point number). The interferogram is a summation of all the wavelengths (cosine waves) emitted by the sample; for all practical purposes it cannot be interpreted in its original form. Using a mathematical process called Fourier Transformation (FT); the system computer converts the interferogram into a spectrum. The spectrum shows the sample emission at all the frequencies measured and thus can be used to identify the sample and its characterization vibration bands. FT-IR spectrometers record the interaction of IR radiation with experimental samples, measuring the frequencies at which the sample absorbs the radiation and the intensities of the absorptions. Determining these frequencies allows identification of sample’s chemical makeup, since chemical functional groups are known to absorb light at specific frequencies. FT-IR spectroscopy is exceptionally suitable for obtaining spectra in energy limited situations (small quantities of samples, trace impurities in mixtures, weakly absorbing samples, etc.) and conditions under which conventional dispersive instruments fail to produce the desired spectra. The use of FTIR in research, analytical and quality control laboratories has brought new and extended capabilities to all users [11, 12].

2.4.5. UV- Vis Absorption Spectrophotometer

It refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means it uses light in the visible and adjacent (near-UV and near-infrared [NIR]) ranges. The absorption or reflectance in the visible range
directly affects the perceived color of the chemicals involved. In this region of the electromagnetic spectrum, molecules undergo electronic transitions. This technique is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state. UV-Visible spectroscopy is a non-destructive technique and relies on the interaction of light with matter. A light beam of a certain wavelength range interacts with the sample and the intensity of the transmitted or reflected signal is recorded as a function of the wavelength. Molecules containing π-electrons or non-bonding electrons (n-electrons) can absorb the energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbitals. The more easily excited the electrons (i.e. lower energy gap between the HOMO and the LUMO), the longer the wavelength of light it can absorb.

A diagram of the components of a typical UV-vis spectrometer is shown in the figure 2.6. The basic parts of a spectrophotometer are a light source, a holder for the sample, a diffraction grating in a monochromator or a prism to separate the different wavelengths of light, and a detector. The radiation source is often a tungsten filament (300-2500 nm), a deuterium arc lamp, which is continuous over the ultraviolet region (190-400 nm), Xenon arc lamp, which is continuous from 160-2000 nm; or more recently, light emitting diodes (LED) for the visible wavelengths. The detector is typically a photomultiplier tube, a photodiode, a photodiode array or a charge-coupled device (CCD). Single photodiode detectors and photomultiplier tubes are used with scanning monochromators, which filter the light so that only light of a single wavelength reaches the detector at one time. The scanning monochromator moves the diffraction grating to "step-through" each wavelength so that its intensity may be measured as a function of wavelength. Fixed monochromators are used with CCDs
and photodiode arrays. As both of these devices consist of many detectors grouped into one or two dimensional arrays, they are able to collect light of different wavelengths on different pixels or groups of pixels simultaneously. A spectrophotometer can be either single beam or double beam. In a single beam instrument, all of the light passes through the sample cell. I₀ must be measured by removing the sample. This was the earliest design and is still in common use in both teaching and industrial labs.

In a double-beam instrument, the light is split into two beams before it reaches the sample. One beam is used as the reference; the other beam passes through the sample. The reference beam intensity is taken as 100% Transmission (or 0 Absorbance), and the measurement displayed is the ratio of the two beam intensities. Some double-beam instruments have two detectors (photodiodes), and the sample and reference beam are measured at the same time. In other instruments, the two beams...
pass through a beam chopper, which blocks one beam at a time. The detector alternates between measuring the sample beam and the reference beam in synchronism with the chopper. There may also be one or more dark intervals in the chopper cycle. In this case, the measured beam intensities may be corrected by subtracting the intensity measured in the dark interval before the ratio is taken.

Samples for UV-Vis spectrophotometry are most often liquids, although the absorbance of gases and even of solids can also be measured. Samples are typically placed in a transparent cell, known as a cuvette. Cuvettes are typically rectangular in shape, commonly with an internal width of 1 cm. (This width becomes the path length, L, in the Beer-Lambert law). Test tubes can also be used as cuvettes in some instruments. The type of sample container used must allow radiation to pass over the spectral region of interest. The most widely applicable cuvettes are made of high quality fused silica or quartz glass because these are transparent throughout the UV, visible and near infrared regions. Glass and plastic cuvettes are also common, although glass and most plastics absorb in the UV, which limits their usefulness to visible wavelengths [13, 14].

2.4.6. Photoluminescence (PL) Spectrophotometer

The electronic structure of the materials could be conveniently probed using photoluminescence (PL) spectroscopy. The process is contactless and non-destructive. During the study, a beam of light is directed onto a sample. The sample absorbs the light and imparts excess energy into the material and this phenomenon is known as photo-excitation. The sample can dissipate this excess energy either through the emission of light or luminescence. The luminescence induced by photoexcitation is referred to as photoluminescence. A measure of several material properties could be obtained from the intensity and spectral content of the photoluminescence. Electrons
move to permissible excited states after photo-excitation. Photoluminescence (PL) spectroscopy adopts a beam of light, usually ultraviolet, which excites the electrons causing them to emit light of lower energy, typically, but not necessarily, visible light. The excess energy is released when these excited electrons return to their equilibrium states. The energy dissipated in the form of emission is radiative in nature. If the process is nonradiative, it may not emit light. Fig. 2.7 depicts the block diagram of the experimental set-up used to record the luminescence spectrum of phosphor sample; the light emitted from the Xe-lamp enters the excitation monochromator. The light emerging from the excitation monochromator is split by the beam splitter and a fraction is directed to the monitor detector. All the driving component i.e., the wavelength drive motors and slit control motors are operated by signal sent from the

**Figure 2.7: Schematic representation of Photoluminescence instrument**

[Diagram of Photoluminescence instrument]

Light Source → Monochromator → Sample in cuvette

Light Source → Monochromator → Detector → A/D converter → Computer / Software

Sample in cuvette

Light Source

Monochromator

Detector

A/D converter

Computer / Software
computer. The output signal from the monitor detects and fluorescence detectors (photomultiplier) are processed by the computer via the A/D converter transmitted to the CRT or graphic plotter [13, 15].

2.4.7. Filmetrics

The filmetrics measures thin-films characteristics by either reflecting or transmitting light through the sample, and then analyzing this light over a range of wavelengths. Because of its wave-like properties, light reflected from top and bottom interfaces of a thin-film can be in-phase so that reflections add, or out-of-phase so that reflections subtract. Whether the reflections are in or out of phase (or somewhere in between) depends on the wavelength of the light, as well as the thickness and properties of the film (e.g., reflections are in-phase when \( \lambda = (2n \cdot d)/i \), where \( \lambda \) is the wavelength, \( n \) is the refractive index, \( d \) is the film thickness and \( i \) is an integer). The result is characteristic intensity oscillations in the reflectance spectrum. In general, the thicker the film, the more oscillations there are in a given wavelength range. Figure 2.8 depicts the filmetrics setup.

The amplitude of the oscillations is determined by the refractive index and extinction coefficient of the films and substrate. Therefore, by analyzing the period and amplitude of these oscillations, the filmetrics determine thickness and optical properties (\( n \) and \( k \)) of multiple thin-films.

Thickness measurement details

Optical thin–film thickness measurements require the successful completion of two tasks: acquisition and then analysis of an accurate reflectance spectrum. To determine the film thickness, FILMeasure calculates a reflectance spectrum that matches as closely as possible the measured spectrum. FILMeasure begins with an initial guess for what the reflectance spectrum should look like theoretically, based on the user’s
input of a film structure for the sample. Then FILMeasure varies the parameters it is solving for until the calculated reflectance spectrum matches the measured data. Mathematically, this procedure is complicated by the fact that as the thickness of the films in the calculation is varied there can be many near matches. Therefore, an approach that simply homes in on a solution by finding successively better approximations will not work unless the starting guess for optical thickness is within approximately 1000 Å of the actual thickness.
References


