Investigations on PEO/PMMA Composite Thin Films

Aishwarya, S

12PPH001

Thesis Submitted to

Avinashilingam Institute for Home Science and Higher Education for Women,

Coimbatore – 641 043.

In Partial Fulfilment of the Requirements for the

Degree of Master of Science in Physics

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Signature of the Head
of the Department

31.3.14

Signature of the
Supervisor

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Aishwarya.S

CONTENT

CONTENTS

NOMENCLATURE

t = Thickness of the film (g)

W = Weight of the film

A = Area of the deposited film

ρ = density of the material (gcm^{-3})

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

CHAPTER-I
INTRODUCTION

1.1 INTRODUCTION

A cursory consideration of the vast body of solid substances reveals what outwardly appears to be an endless multitude of external forms and structures possessing a bewildering variety of properties. The branch of study known as Material Science evolved in part to classify those features that are common among the structure and properties of different materials. **[Milton Ohring].**

Thin film technology is a branch of material science and simultaneously one of the oldest arts. Thin film science has received tremendous attention in the recent years especially after the world war because of numerous applications of films in diverse fields. Because of compactness, better performance and reliability coupled with low cost of production, low power consumption and low package weight, thin film devices and components are preferred over their bulk counterparts. The term thin film has often been loosely used in literatures to imply not only a layer of solid material but also of a liquid or gaseous phase. Solid films are oftenly prepared for industrial applications **[A.Gowsami]**

Thin film science and technology play a crucial role in the high-tech industries while the major exploitation of thin films has been in microelectronics. There are numerous and growing applications of thin films in communications, optical electronics, coating of all kinds, and in energy generation and conservation strategies. A great many sophisticated analytical instruments have already become indispensable in virtually every scientific endeavor irrespective to discipline **[Milton Ohring]**

A thin film may be arbitrarily defined as a solid layer having a thickness varying from a few Å to about 10μm.Since the thickness limitation is rather arbitrary, even somewhat thicker films may also come within the scope of the above definition. The ideal condition of the film formation involves the deposition of material, atom by atom (molecule by molecule) and layer by layer **[A.Goswami]**

Thin films both amorphous and crystalline are very important in modern technology. For instance, they may be used for protective coating on materials and they play a key role in the miniaturization of components on electronic devices. They also sometimes have special properties, which drive from their thinness and in particular, from their very large surface area to volume ratio. The structure and properties of the surface are often quite different from their interior **[Antony R.West]**

Thin film technologies have advantages over the crystallography techniques, because of the absence of defects such as dislocations, vacancies, stacking faults, grain boundaries and twins.

The material in thin film configuration can have structure distinct from those in bulk state and the structure of the thin film material can be controlled by the production method. A number of novel effects (such as Quantum Hall effect) are observed in thin films, often because the electrons are constrained to move in a plane.

1.2 CLASSIFICATION OF THIN FILM

Thin films are classified into three categories based on relative physical dimensions and make no reference to any length scale reflecting the underlying structure of the material. They are,

- Mechanical thin film
- Microstructural thin film
- Atomical thin film

In mechanical thin films, the thickness of the film is small compared to that of the substrate. Here the thin film thickness is much larger than all the characteristic micro structural length scales such as grain size, dislocation cell size etc. Such structures, typically tens or hundreds of micrometers in thickness, are deposited onto substrates by physical vapour deposition.

When the small dimension of the material structure is comparable to the characteristic microstructural size scale, the film is considered to be a micro structural thin film. Most metallic thin films used in micro electronic devices and magnetic storage media are examples of microstructural thin film, where the film thickness is substantially greater than atomic or molecular dimensions.

Atomically thin films constitute layers whose thicknesses are comparable to one or a few atomic layers .An adsorbed monolayer of gas or impurity atoms on a surface is an example of an atomically thin layer. Here the mechanical response of the thin layer is likely to be more influenced by interatomic potentials and surface energy than by macroscopic mechanical properties **[L.B.Freund & S.Sureh].**

Thin film growth has been phenomenal especially due to their tremendous applications in diverse fields such as, Electronic industries, military weapon systems, space science, solar energy utilization and as optical and superconducting film materials, high memory computer elements, sensors and in microelectronic and hybrid circuits and in others **[A.Goswami].**

1.3 POLYMERS

Polymers are large molecules made up of simple repeating units. The name is derived from the Greek *poly*, meaning “many”, and *mer* meaning “part”. Macromolecule is a term synonymous with polymer. Polymers are high molecular weight compounds whose structures are made up of a large number of simple repeating units. The repeating units are usually obtained from low molecular weight simple compounds referred to as monomers .The reaction by which monomers are converted into polymers is polymerization **[Arun Bahl & B.S.Bahl].**The structural unit enclosed by brackets or parentheses is referred to as the repeating unit or monomeric unit. The smallest possible repeating unit is referred to as the base unit.

Polymer synthesized from a single monomer is called homopolymer. If two or more monomers are employed, the product is a copolymer. Degree of polymerization refers to the total number of structural units, including the end

group and hence is related to both chain length and molecular weight **[Malcolm P.Stevens].**

The process of polymerization was divided into two groups known as condensation and addition polymerization or, in more precise terminology, step-reaction and chain-reaction polymerization.

Condensation or step-reaction polymerization is entirely analogous to condensation in low-molecular-weight compounds. In polymer formation the condensation takes place between two polyfunctional molecules to produce one larger polyfunctional molecule, with the possible elimination of a small-molecule such as water.

Addition or chain-reaction polymerization involves chain reactions in which the chain carrier may be a ion or a reactive substance with one unpaired electron called a free radical. A free radical is usually formed by the decomposition of a relatively unstable material called initiator **[Fred W.Billmeyer, JR].**

In the modern world, polymer thin films are utilized for a lot of technological applications in diverse fields such as surface coatings, micro and opto-electronics, holography, biosensors, etc. In thin films, substrate interaction, interfacial effects and confinement alter the kinetics of phase segregation and phase equilibrium. The presence of surface and an interface make them exhibit properties not generally detected in bulk materials. The theoretical understanding of the thermodynamic properties of these thin films can be improved by accurate analysis of the topographical and morphological features manifested by them and this, in turn, can lead to new outlooks and applications **[Kailas et al.,]**.

1.4 POLYMER THIN FILM

Polymer thin films are a novel class of materials that have found their way into a wide range of industrial and biomedical applications and are now an integral part of our everyday lives. Their universal presence in paints, coating, packaging,

adhesives, dielectrics and other common entities have firmly established their versatility, integrity and dependability. At the same time, their continuing expansion into new technologies and applications has inevitably revealed potential shortcomings and subtleness in their properties and behaviour.

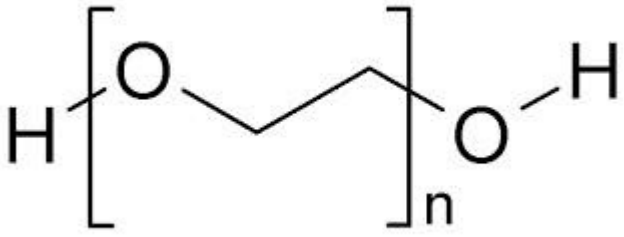
The characteristic features of the polymer coatings are

1. Their ability to withstand stress, strain exerted by different kinds of exposure in aggressive environments.
2. They can protect the metallic, concrete and other structures from corrosion and erosion.
3. Aesthetic retention **[Faiz Mohammad]**

Organic thin film material has arisen due to their extensive applications in the field of mechanics, flexible electronics and optics. Polymer organic thin films are employed typically in micro electronics and have been used in a wide variety of applications such as perm selective membranes and protective coatings as well as electrical, optical and biomedical films.

1.5 PEO Poly (ethylene oxide)

Poly (ethylene oxide) is also known as Poly (ethylene glycol) or Polyoxyethylene depending upon its molecular weight. PEO has the following structure,



Linear polyethers known as poly (ethylene oxide) are conveniently made by controlled ring opening polymerization of ethylene oxide or through polycondensation of ethylene glycol. PEO of low molecular weight (1,000-20,000) are greasy to waxy in appearance.

The ethylene oxide monomer is nothing more than an epoxide ring. Two corners of the molecule consist of $-CH_2-$ linkages. The third corner is an oxygen, -O-. In the presence of a catalyst the monomer forms a chain having the repeat unit $-CH_2-CH_2-O-$. The PEO products found to be most effective in various papermaking applications are those having very high molecular mass, much in excess of one million grams per mole. Factors that make PEO wet-end chemistry puzzling include (a) the fact that it can be used as a retention aid even though it is nonionic, (b) the fact that its performance is highly dependent on its shear history, and (c) the fact that it needs the presence of lignin or certain phenolic "cofactor" additives to achieve its best effect as a flocculant and retention aid. Observations of PEO behaviour suggest that the molecular chains are initially tangled with each other and that this tangling is somehow essential for effective flocculation. PEO is usually received as dry granules. These need to be dispersed with care, using a dilution ratio of at least 100 and avoidance of excessive shear.

PROPERTIES

- Poly ethylene oxide has the mean molecular weight > 104g/mol
- It's a white powder with no special smell
- The relative density is 0.21g/cm^3 and the apparent density is 0.2 to 0.5 g/cm^3.
- The glass transition temperature of poly ethylene oxide is 63~67°C
- Poly (ethylene oxide) becomes brittle at -50°C
- It dissolves completely in water, aqueous solution or alkaline and in some organic solvents.
- Poly ethylene oxides of low concentration usually have high viscosity.
- With water temperature close to the boiling point of water, PEO will precipitate out.

- Should be preserved in tight, light-resistant containers.
- Melting point of poly ethylene oxide is 66-75°C.
- With good chemical stability, PEO is resistant to both acid and alkali and also corrosion resistant.

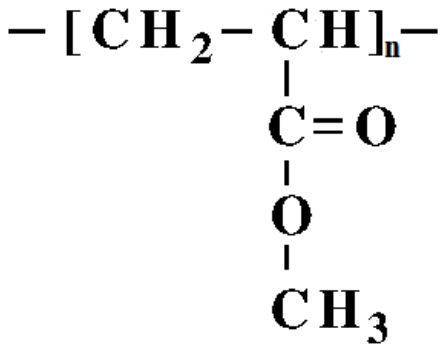
APPLICATIONS

- Poly (ethylene oxide) has wide application in pharmaceutical and biomedical arenas due to their excellent physical and biological properties, including solubility in water and some organic solvents and also it is lack of toxicity
- It is used to improve the therapeutic effects and compatibility of patients.
- It is used in drug delivery system
- PEO (polyethylene oxide) can serve as the separator and electrolyte solvent in lithium polymer cells. Its low diffusivity often requires high temperatures of operation, but its high viscosity–even near its melting point–allows very thin electrolyte layers to be created. While crystallization of the polymer can degrade performance, many of the salts used to carry charge can also serve as a kinetic barrier to the formation of crystals. Such batteries carry greater energy for their weight than other lithium ion battery technologies.
- It is used in biomedical research, tissue engineering scaffolds, surface functionalization.
- It is used as adhesives, particularly when formulated with association products such as lignin sulfonic acids, as a temporary binder in manufacture of acoustical tile and ceramics.
- PEO which has been radiation cross-linked and recovered in dry form, swells as it absorbs about 100 times its own weight of water. This cross-linked, water-swellable material is an excellent agar substitute as a culture medium offering advantages in assurance of its

aseptic quality. This material has been formulated as a base for medicated dressings.

1.6 PMMA Poly (methyl methacrylate)

Poly (methyl methacrylate) is a clear, colourless transparent plastic with higher softening point, better impact strength and better weatherability than polystyrene. It is available in molding and extrusion compositions, syrups and cast sheets, rods and tubes. It has the following structure



PMMA is sometimes called acrylic glass. Chemically, it is the synthetic polymer of methyl methacrylate. The material was developed in 1928 in various laboratories, and was first brought to market in 1933 by the Rohm and Haas Company, under the trademark Plexiglas. The methacrylate ester polymers are exceptionally clear, hard, glass plastics. They softens at temperature over 120°C, this permits fabrication of molding and extrusion techniques. PMMA is widely used sometimes with plastizer and or small amount of other esters as copolymers. The monomer methyl methacrylate is produced from acetone. PMMA is prepared by radical polymerization in bulk or suspension. It is often preferred because of its moderate properties, easy handling and processing, and low cost, but behaves in a

brittle manner when loaded, especially under an impact force, and is more prone to scratching than conventional inorganic glass.

PROPERTIES

- PMMA is a strong and lightweight material. It has a density of 1.17–1.20 g/cm^3, which is less than half that of glass.
- Molecular weight of repeat unit is 100.12g/mol and it has the glass transition temperature of 114°C.
- PMMA is hard and transparent polymer with good resistance to the effects of light and weathering.
- It is resistant to many chemicals but soluble in organic solvents such as ketones, chlorinated hydrocarbons and esters.
- PMMA transmits up to 92% of visible light and gives a reflection of about 4% from each of its surfaces on account of its refractive index (1.4914 at 587.6 nm).
- It also has good impact strength, higher than both glass and polystyrene
- Commercially produced PMMA is a thermoplastic that softens at 120°C.
- Melting point of polystyrene is 160°C and its density is 1.188g/cm^3.
- It is amorphous by nature, owing to the presence of bulky side groups in the molecules.
- Its environmental stability is superior to most other plastics such as polystyrene and polyethylene, and PMMA is therefore often the material of choice for outdoor applications.
- The mechanical and thermal properties of the polymer are good and the tensile strength ranges as high as 10,000 psi.
- Fabricability is quite good ,only slightly higher temperature is needed for molding PMMA than PS. **[Gowariker et al.,]**

APPLICATION

- PMMA has wide applications in injection-molded acrylic articles which include automotive lenses, reflective devices, instrument and appliance covers, optical equipment and home furnishings.
- It is an excellent substitute for glass. PMMA acrylic glass is commonly used for constructing residential and commercial aquariums.
- PMMA is used to make attractive sign boards and durable lenses for automobile lighting.
- Daylight redirection-Laser cut acrylic panels have been used to redirect sunlight into a light pipe or tubular skylight and, from there, to spread it into a room.
- Historically, PMMA was an important improvement in the design of aircraft windows.
- PMMA was used in Laser Disc optical media. (CDs and DVDs).
- PMMA has a good degree of compatibility with human tissue, and can be used as intraocular lenses in the human eye when the original lens has been removed in the treatment of cataracts.
- Poly (methyl methacrylate) has drawn tremendous interest due to its optical properties and its possible use in non-linear optics.
- Poly(methy1 methacrylate) (PMMA) polymer films can be used either as a chemically sensitive coating material in implementing biochemical sensors or as a wave guiding layer in guided shear-horizontal surface acoustic wave (SH-SAW) sensor platforms
- Plastic optical fiber used for short distance communication is made from PMMA, and perfluorinated PMMA, clad with fluorinated PMMA, in situations where its flexibility and cheaper installation costs outweigh its poor heat tolerance and higher attenuation over glass fiber.

1.7 OBJECTIVES OF THE STUDY

The aim of the present work is

- To prepare PEO/PMMA composite thin films on glass substrate by Chemical Bath Deposition
- To measure thickness of the prepared films.
- To analyze structural, optical and surface properties of the coated thin film using UV-Visible, Photoluminescence and FT-IR characterization methods.

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REVIEW OF LITERATURE

CHAPTER 2
REVIEW OF LITERATURE

2.1 INTRODUCTION

A literature review is a review of already existing and published articles on a given topic or area or certain subject with a view to accessing their relevance to a proposed project. A literature review can be just a simple summary of the sources, but it usually has an organizational pattern and combines both summary and synthesis. A summary is a recap of the important information of the source, but a synthesis is a re-organization, or a reshuffling, of that information. It might give a new interpretation of old material or combine new with old interpretations. Polymer materials have been widely used in various fields such as optical communications and electronic sensors. The continuing interest in the technological application of polymeric film is reflected in a large number of published papers dealing with various aspects. This chapter discusses about the studies on various properties of different polymers carried out by several researchers in different parts of the world**.**

2.2 REVIEWS ON VARIOUS PROPERTIES OF POLYMER THIN FILMS

1. Vijaya S.Sangawar et al., (2013) have prepared PEO complexed with CdS by solution cast technique. Several experimental techniques such as XRD, SEM, FTIR, DSC, PL and Raman spectroscopy are employed to characterize the sample. The XRD result indicates that the nanoparticles are crystallized in cubic phase. The UV-Visible absorption, differential scanning calorimetry, photoluminescence and Raman spectra of the sample are recorded. Transmission spectra of films were recorded using UV-Visible in the range of 400-800nm.It reveals that the transmittance decreases with the increase in filler concentration. In the photoluminescence spectra, the emission peak are shifted towards higher wavelength (upto 400nm).The red shift in the PL spectrum of all the blends is an

evidence for the formation of new bonds and miscibility of the polymers. The Raman spectra is recorded in the range 100-1000 cm^{-1} .The spectrum exhibited an intense, broad peak at 303.54 cm^{-1} corresponding to the LO phonon mode. Higher order phonon modes were also observed at 606.334 and 912 cm^{-1} in the spectra. From FT-IR spectra, pure PEO transmission is in the range 1250-950 cm^{-1} and the presence of the crystalline PEO phase is confirmed by the presence of triplet peak of the COC stretching vibration at 1148,1110 and 1062cm^{-1} with maximum at 1110 cm^{-1}.

2. Hans-George Braun et al., (2013) have studied on the direct contact of ultra thin polymer films with a solid substrate which result in thin film rupture caused by dewetting. With crystallisable polymers such as PEO, molecular self-assembly into partial ordered lamella structures is noted as an additional source of pattern formation. Morphological features in ultra thin PEO films (thickness < 10nm) result from an interplay between dewetting patterns and diffusion limited growth pattern of ordered lamella growing within the dwetting areas. Besides structure formation of hydrophilic PEO molecules n-alkyl terminated (hydrophobic) PEO oligomers are investigated with respect to self-organization in ultra thin films. Morphological features characteristic for pure PEO are not changed by the presence of the n-alkyl groups.

3. J.Shanthi and J.Priyanga (2013) have deposited solvent cast composite thin films of polystyrene and poly (methyl methacrylate) by spin coating on silica substrate. Thin films of few μm thickness obtained have been examined using UV-Visible, photoluminescence, XRD and SEM characterization methods. The band structure of polystyrene tailored by the addition of poly (methyl methacrylate), had 90% transmittance. The amorphous nature of the material was confirmed using XRD analysis. Homogeneous crack free surface of the coated film has been observed by SEM analysis**.**

4. Dae Up Ahn et al., (2012) compared the morphological evolution of thin PS/PMMA films, with varying compositions, during thermal annealing on preferential and non-preferential surfaces. On silicon oxide surfaces the phase evolution in the thin films was dictated by the preferential substrate –wetting of PMMA. In contrast the morphological evolution of the PS/PMMA films on non-preferential surfaces was also dictated by the coarsening of PMMA domain, but proceeds without the formation of a PMMA wetting layer. Both PS and PMMA domains maintain direct contact with both substrate and free surfaces throughout the evolution of morphology. A variety of dispersed and continuous phase separated structure can thus be obtained.

5. Julien Gaume et al., (2012) have studied on the experimental method for measuring the permeation of oxygen through thin barriers using the UV light-induced oxidation of a polymer as a sensor. This method is based on the photo-oxidation of poly (ethylene oxide) (PEO) encapsulated in a bag formed from the barrier. The method was applied to 3 polymers with different oxygen barrier properties [poly (methyl methacrylate) (PMMA), poly (ethyleneterephtalate) (PET), poly (vinylalcohol) (PVA)] and the results are compared with values reported in the literature. The results confirm that this method yields oxygen permeability co-efficient in good agreement with the literature data.

6. Chil-Chyuan Kuo et al., (2012) investigated surface roughness of thin films, which is an important issue in manufacturing engineering, because the performance of a coated film is significantly affected by the surface roughness of thin films. A fast and flexible optical measurement system to measure surface roughness of hard coatings deposited by cathodic arc evaporation. The objective is that to examine the repeatability and reproducibility (R&R) of an optical measurement system, percentage of equipment variation, appraiser variation and R&R is 7.25%, 1.42% and 7.39%.Thin film optical measurement system developed is acceptable according to measurement system analysis and the R&R technique.

7. Ryutaro Souda et al., (2012) have studied the surface mobility of polystyrene (PS), poly (methyl methacrylate) (PMMA), poly (ethylene oxide) (PEO) via interactions with an ionic liquid ad-species, 1-ethyl-3-methyl imidazolium bis [trifluromethanesulfonyl] imide, by using the secondary ion mass spectrometry as a function of temperature. The surfaces of PS and PMMA films become mobile below the glass transition temperature, as revealed from the uptake of ad-species. The enhanced mobility on the surface is ascribable to a precursor to the glass-liquid transition, from analogy of the glass transition behaviour of simple molecules. On semi crystalline PEO films, the phase transition of the ionic liquid (~210K) is quenched indicating that the PEO surface becomes mobile far below the melting point; the uptake temperature of ad-species depends on the sample preparation and polymer chain length. The result is explainable as liquid – crystalline co-existence or premelting.

8. Saibha sultana et al., (2012) prepared pure poly (ethylene oxide) (PEO) films and PEO-$FeCl_3$ films of various concentration and studied its thermal and optical properties. Various analytical techniques like XRD, EDX, SEM, TGA, and UV were used to examine the structural properties of PEO and PEO-$FeCl_3$ systems. X-ray diffraction (XRD) revealed the existence of some crystalline part in the PEO matrix. The concentration of $FeCl_3$ has a great effect on the crystallinity of PEO. Thermo gravimetric analysis (TGA) data established that accumulation of $FeCl_3$ to PEO films enhanced the thermal stability of PEO. The optical properties of these systems were also studied in the UV-Visible region as a function of $FeCl_3$ concentration at room temperature. The obtained data were analyzed in terms of absorption formula for non-crystalline materials. From the measured absorption data optical energy gaps (E*opt*) were determined. The optical energy gap was found to decrease with the increase in $FeCl_3$ concentration, which indicates that $FeCl_3$ concentration had a significant influence on optical energy band gap. The

absorption coefficient increased sharply at concentration of $FeCl_3$ wt (5%) as compared with the pristine PEO.

9. Sandip et al., (2011) have prepared Poly (3-methyl thiophene) thin films by chemical bath deposition technique on glass substrate; the prepared thin films were characterized for structural, morphological and optical properties. The variation in the oxidant concentration has an influence on the properties of the P3MeT thin films. The increase in the oxidant concentration leads to increase in the thickness of the film. The binding energy increases due to increase in oxidation concentration. The P3MeT thin films show smooth surface morphology with increase in oxidant concentration whereas the contact angle of the thin film decreases with increase in oxidant concentration. The optical absorbance of these thin films was found to increase with decrease in the optical band gap due to increase in oxidant concentration.

10. Prime Dominic et al., (2009) have analyzed the electrical and morphological properties of polystyrene layers in the nanometre thickness range for organic and polymer based electronic applications. This article aims in providing conduction data and information on trapped charges present in the polystyrene layer, as well as investigated how polystyrene properties change under differing annealing conditions. The maximum dielectric strength was found to be 4.0 MV cm^{-1}, while fixed trapped charge and mobile trapped charge average densities were calculated to be 9.9×10^{11} cm^{-2} and 2.6×10^{12} cm^{-2} respectively. Optimum electrical characteristics were obtained at an annealed temperature of 90°C, which is just below the glass transition temperature for polystyrene**.**

11. Lantz et al., (2009) have investigated the effects of various deposition techniques on the photoluminescence spectra of the conjugated polymer poly[2-methoxy-5--ethylhexyloxy)-1,4-(1-cyanovinylene) phenylene] (MEH-CN-PPV).

Photoluminescence spectroscopy provides insight into the internal morphology of organic thin films through the identification of interchain or intrachain recombination peaks. Thin films were deposited on glass substrates by drop casting, spin casting and resonant-infrared matrix-assisted pulsed laser evaporation (RIR-MAPLE) and were compared to the photoluminescence of the polymer in solution. The photoluminescence measurements reported in this article demonstrate that samples deposited by evaporative RIR-MAPLE have an internal morphology similar to that of MEH-CN-PPV in solution, leading to an enhancement of intrachain transitions in the conjugated polymer.

12. Yoshimura et al., (2009) have investigated the effect of ultraviolet (UV) light irradiation on polymer dry etching processes with the use of a low-energy mass-selected ion beam injection system. Etching yields of poly (methyl methacrylate) (PMMA) by Ar or CF_3 ion beam injections were evaluated from the loss of PMMA film weight measured by a quartz crystal microbalance (QCM) during the ion beam injection process with or without simultaneous UV light irradiation. Significant enhancement of the etching yield of PMMA was observed during the simultaneous irradiation with CF_3 ion beam and UV light over the sputtering yields obtained from separate ion-beam or UV-light injection processes. By contrast, no significant change of etching yields was observed when the UV light was superposed on the Ar ion beam.

13. Capan et al., (2009) have reported on the characterization of spin coated thin films of poly (methyl methacrylate) (PMMA) for their use in organic vapour sensing application. Thin film properties of PMMA were studied by UV–visible spectroscopy, atomic force microscopy and surface plasmon resonance (SPR) technique. Results obtained show that homogeneous thin films with thickness in the range between 6 nm and 15 nm have been successfully prepared when films were spun at speeds between 1000–5000 rpm. Using SPR technique, the sensing properties of the spun films were studied on exposures to several halo

hydrocarbons. The highest PMMA film sensitivity of 0.067 normalised responses per ppm was observed for chloroform vapour, for films spun at 1000 rpm sensitivity was ascribed mainly to its solubility parameter and molar volume values. Effect of film thickness on the vapour sensing properties is also discussed.

14. J-S Bae a et al .,(2009) prepared the free standing polymer film specimen by spin coating of PMMA solutions onto silicon substrates, removing the solvent and releasing the film safely by means of distilled water. Preliminary tensile tests was successfully done for the PMMA films made from 4 wt% concentration of solutions of two different molecular weights Mw=350000 and 996000 g/mol with thickness of 1.46 and 1.72 μm respectively. The stress-strain behaviour have been studied which resulted in the increment of young's modulus, yield strength and tensile strength as a function of logarithm of strain rate. The effect of molecular weight on the mechanical properties was discussed.

15. D.Shanmukaraj et al., (2008) have investigated the role of inorganic ceramic filler namely Al2O3 (15-25nm), TiO_2 (10-14nm) and ferroelectric filler $SrBi_4Ti_4O_{15}$ (0.5 μm) by citrate gel technique (CIT) on the ionic conductivity and electrochemical properties of polymer blend PMMA+PEO.EC/PC electrolytes were determined. The lithium-ion transport number and the electrochemical stability of the composite polymer electrolytes at ambient temperature were analyzed.

16. Halina Kaczmarek and Hanna Chaberska., (2008) have obtained thin poly(methyl methacrylate) films by casting polymer solution onto borosilicate glass and aluminum supports from organic solvents, such as chloroform, chlorobenzene, toluene, acetone and tetrahydrofuran. Atomic Force Microscopy has been chosen as a method for investigation of the influence of solvent residue and support type on the topography and roughness of the PMMA films before and after UV-irradiation

of 254 nm wavelength. It was found that, besides information on sample morphology, AFM provides valuable details on polymer–support interactions

17. Yadav et al., (2008) have reported the adhesion, structural and optical properties of spin coated PANI/PMMA composite thin films of different composition on glass substrate. The effect of post-deposition heating for 100 °C, 125 °C and 150 °C was also reported. The adhesion of the film was found to increase from $712 \pm 5 \times 10^4$ N/m^2 to $1602 \pm 3 \times 10^4$ N/m^2 and refractive index decreased from 1.852 ± 0.005 to 1.650 ± 0.004 with increase in concentration of PMMA. Due to post-deposition heating adhesion, optical band gap increased but refractive index decreased.

18. Jun zhu et al., (2008) investigated the crystallization patterns of ultra thin PEO/PMMA blend films crystallized at different under cooling by AFM. Dendrite pattern formed as a result of crystalline anisotropy at low under cooling and evolved into seaweed pattern with increasing under cooling. Although the configuration of macromolecules is far different from that of simple small molecules, the crystallization pattern transition in ultra thin polymer films can be interrupted by the classical morphology diagram developed on the basis of metals and simple molecules, indicating that polymer chains can be considered as simple dynamical units in a quasi-two-dimensional confined state.

19. Sarantopoulou et al., (2008), have reported that the Laser irradiation at 157 nm of poly (methyl methacrylate) (PMMA) thin films induces major variations of polymer film thicknesses from sorption (absorption/desorption) of methanol and ethanol analytes in the gas phase as much as 400%, in comparison to the film thickness variation of the non-irradiated areas. The structural changes of irradiated areas involve scission of polymeric chains, cross-linking and formation of new bonds. In addition, 157 nm induces surface and volume morphological changes in

the nano/micro domain, with different shapes, depending on the irradiation conditions. The reversibility of the sorption processes suggests that the polymer swelling has its origin as the tendency of the system to increase its volume during sorption.

20. F.-Z. Tighit et al., (2007) have deposited PMMA graft into the porous silicon by three different methods, like dip coating, spin coating and spreading method. FTIR, SEM, EDX has been carried out to evaluate the surface modification of the films. The EDX spectrum shows the presence of peaks attributed to the carbon and oxygen, which are the principle constituents of the PMMA polymer. FTIR spectroscopy detected the existence of specific interactions, especially a stronger hydrogen bonding which is suspected to occur between the hydroxyl groups present at the surface of the porous silicon substrate and the carboxyl groups of the polymeric compound.

21. Ivan Dmitriev et al., (2007) studied the effect of annealing on the structure and mechanical properties of melt extruded PVDF films by X-ray scattering, tensile testing, cycling loading, ultrasonic propagation and dynamic mechanical analysis. It has been found that the annealed films prepared at a melt draw ratio higher than 30 and annealed at the temperature close to the melting point exhibit hard elastic properties. Elastic recovery of the annealed PVDF films was about 60%.

22. Gulakari R.S. et al., (2007) studied the electrical conduction mechanism in polyvinyl chloride (PVC), poly (methyl methacrylate) (PMMA) blend films at various temperatures in the range 313K to 353K.The results were presented in the form of I-V Characteristics. Analysis have been made in the light of Poole-Frankel, Faoler Wordheim Schottky, log (J) Vs T plots and Arrehenius plots. It was observed that, Schottky-Richardson mechanism was primarily responsible for the observed condition.

23. Liang Cui et al., (2006) have discussed the effects of solvent nature on the surface topographies of polystyrene (PS)/poly (methyl methacrylate) (PMMA) blend films spin-coated onto the silicon wafer. Four different solvents, such as ethylbenzene, toluene, tetrahydrofuran and dichloromethane, were chosen. They are better solvents for PS than that for PMMA. When dichloromethane, tetrahydrofuran and toluene were used, PMMA-rich phase domains protruded from the background of PS. When ethylbenzene was used, PS-rich phase domains elevated on the average height of PMMA-rich phase domains. In addition, continuous pits, networks and isolated droplets consisted of PS formed on the blend film surfaces with the decrease of polymer concentrations. The mechanism of the surface morphology evolution was discussed in detail.

24. Yanxia Li et al., (2006) have investigated the surface and interface morphologies of polystyrene (PS)/poly (methyl methacrylate) (PMMA) thin-film blends and bilayers by means of atomic force microscopy (AFM) and X-ray photoelectron spectroscopy. Spin-coating a drop of a PS solution directly onto a PMMA bottom layer from a common solvent for both polymers yielded lateral domains that exhibited a well-defined topographical structure. Two common solvents were used in this study. The structure of the films changed progressively as the concentration of the PS solution was varied. Films of the PS/PMMA blend and bilayer were annealed at temperatures above their glass-transition temperatures for up to 70h. All samples investigated with AFM were covered with PS droplets of various size distributions. Moreover, they investigated the evolution of the annealed PS/PMMA thin-film blend and bilayer and a proper explanation was given for the formation of a relatively complicated interface inside a larger PS droplet.

25. Jianming et al., (2005) analyzed the change in the refractive index and thickness of spin-coated poly (methyl methacrylate) (PMMA) thin films upon irradiation by a conventional high-pressure mercury UV lamp. Significant increase

in refractive index and reduction in thickness were detected. The thickness reduction of an irradiated PMMA film is consistent with its weight loss .This is caused by the escape of the volatile molecules generated during the irradiation process. A slight increase in the refractive index was also found in the film, heat-treated above its glass transition temperature (T_g). They proposed three possible aliphatic structures that could be formed during the photochemical reaction and may exist in the main chain of irradiated PMMA after the irradiation. Their refractive indices in aggregate state are greater than that of PMMA based on an evaluation using the Lorentz-Lorenz equation. This is suggested to be an important reason for the refractive index increase in the UV-irradiated PMMA films. A UV-irradiated film, Heat-treated above its T_g, has a rough surface with many tiny holes as illustrated by atomic force microscopy. These holes are attributed to the evaporation of the small molecules generated during the irradiation process.

26. Xue Li et al., (2004) have investigated the surface morphology evolution of three thin polystyrene (PS)/poly (methyl methacrylate) (PMMA) blend films (<70 nm) on SiO_x substrates upon annealing by atomic force microscopy (AFM) and some interesting phenomena were observed. All the spin-coated PS/PMMA blend films were not in thermodynamic equilibrium. For the 67.1 nm and the 27.2 nm PS/PMMA blend films, owing to the low mobility of the PMMA-rich phase layer at substrate surfaces and interfacial stabilization caused by long-range Vander Waals forces of the substrates, the long-lived metastable surface morphologies (the foam-like and the bicontinuous morphologies) were first observed. For the two-dimensional ultra thin PS/PMMA blend film (16.3 nm), the discrete domains of the PS-rich phases upon the PMMA-rich phase layer formed and the secondary phase separation occurred after a longer annealing time.

27. Kenji Morota et al., (2004) have deposited the Nanostructures thin films by electrospray deposition (ESD) from poly (ethylene oxide) (PEO) aqueous solution. The surface morphologies of the deposited films were observed using scanning

electron microscopy (SEM). The SEM images revealed the correlations between the morphologies and the ESD conditions. By changing the applied voltage and solution properties such as viscosity, surface tension, conductivity, and molecular weight, PEO thin films with diverse nanostructures from nanospheres to nanofibers were fabricated. It was also revealed that the addition of alcohols to polymer solution, which enables simultaneously changing the viscosity, the surface tension, and the conductivity, enhanced the formation of the fibrous structure. These results indicate that the ESD method is potentially a useful option for producing nanoengineered polymer surface.

28. Christopher et al., (2003) have produced the films of atactic poly (methyl methacrylate) (PMMA) by spin-coating from toluene solutions, and their properties were compared to similar PMMA films produced by spin-coating from chloroform. Two-angle ellipsometry at λ= 6328 A˚ was used to probe the film thicknesses, refractive indices, and their overall quality and uniformity. Ellipsometry was also used with microspot optics at one angle to determine the film thickness uniformity. The films from toluene were approximately 4-fold thinner, but more uniform and of higher quality, than the films from chloroform, evidently because of the lower volatility and slower evaporation, of toluene. Films with thicknesses d_1=0.003 to 1 μm were produced for initial PMMA concentrations c=0.1–10 wt. %, and spinning speeds of 1000–3000 rpm. The thicknesses fit the equation d_1 (μm) =0.92 $c^{1.56}$ $\omega^{-0.51}$. The ω dependence agrees with predictions of simple theoretical models. The results may find use in production of high-quality polymer films for resists or other applications.

29. Higgins et al., (2002) investigated the dynamics of spinodal dewetting in liquid-liquid polymer systems. Dewetting of poly (methyl methacrylate) (PMMA) thin films on polystyrene (PS) “substrates” was followed in situ using neutron reflectivity. The development of roughness at the PS/PMMA interface and the PMMA surface characteristic growth times for the dewetting process. These

characteristic growth times are measured as a function of the molecular weight of the two polymers. By also carrying out experiments in the regime where the dynamics are independent of the PS molecular weight, they used dewetting to probe the scaling of the PMMA thin film viscosity with temperature and molecular weight. They also found out that the scaling reflects the bulk behaviour.

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MATERIALS AND METHODS

CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

The selection of the material depends on its properties and applications. Thin polymeric films are of growing importance in technical applications of microelectronics, optics, optoelectronics, etc, and hence in this research work polymeric materials have been analysed. Thin films can be deposited atom by atom on variety of substrates. There are many techniques involved in the coating of the material. Depending on the field of application the deposition technology has to match a broad spectrum of requirement in order to meet the desired functional parameters. Among the various available physical and chemical methods, the simple and economical chemical bath deposition method has been followed in this research work.

3.2 MATERIAL SELECTION

Selection of the best film for any particular use is a matter of matching film properties against the end use performance required. Poly (ethylene oxide), Poly (methyl methacrylate) have been chosen for thin film coating in this work. Poly (methyl methacrylate) is a clear transparent, thermoplastic with good optical, electrical, and mechanical properties. It is an excellent substitute for glass.

The two polymers, PMMA and PEO are blended with each other so as to yield a composite thin film with desired properties. Immiscible blends are known to have properties, which combine those of both the polymers, and to have segregated structures with domains predominantly formed from the individual homopolymers. PEO/PMMA blend is well-known immiscible combination for which bulk and surface phase separation has been observed. Among the commercially available polymers, poly (ethylene oxide), poly (methyl methacrylate) is a good material of choice. However due to the low entropy of mixing, polymeric blends are mostly incompatible and phase separate under appropriate conditions. Especially for thin

films, since the surface area-to- volume ratio of polymer blend film is relatively large, the presence of both air/polymer and substrate/ polymer interface play an important role in determining the morphology of polymer blend thin films. For ultra thin films the additional presence of a surface leads to behaviour, which is very different from bulk systems **[Krausch].**

3.3 CHOICE OF THE SUBSTRATE

A substrate is a solid substance or medium to which another substance is applied and to which that second substance adheres (If no substrate, it is called foil). The microstructure of thin films strongly depend on the kind of substrate i.e. roughness, crystalline structure and the chemical state of the surface. Unfortunately, an ideal substrate does not exist. Specific applications require different substrate materials which offer an acceptable compromise for the purpose on hand. Ideally the substrate should provide only mechanical support but not interact with the film except for sufficient adhesion. In practice, however the substrate exerts considerable influence on the thin film characteristics. The various substrate requirements are, atomically smooth surfaces, inertness to chemicals used in processing, high volume and surface resistivity, high thermal conductivity, coefficient of thermal expansion similar to film, high mechanical strength, high thermal shock resistance, zero porosity, good dimensional tolerance, excellent scribing, cutting, module- separation properties and low cost. There are a number of materials like glass, ceramics and quartz available for use as thin film substrates. **[Maissel and Glang]**.

3.4 SUBSTRATE CLEANING

Cleaning is one of the most difficult problems in the manufacture of low reflectance coatings, and it is a prime problem with other coating designs as well. One of the reasons that cleaning is so difficult is that it depends on all of the prior history of the part: at all stages of handling more dirt, grime and organics get added to the surface. If cleaning is not considered from the beginning of the fabrication a part, either on optical polishing machine. **[D James.Rancourt]**

Almost all depositions are made on some solid substrates such as glass, quartz, ceramics, metals, insulator, and organic materials like plastics, polymer, rubber etc the nature of which may differ widely from amorphous to polycrystalline, single crystal and fibre structure. The most important consideration in all these cases is that the deposit layers must be adherent to the substrates and should not peel off from the substrates under the normal conditions of stress and strain, mechanical or thermal to which the deposits are exposed during their uses **[A.Goswami].**

All the glass slides of dimension (75mm×25mm×1mm) were washed with ordinary distilled water. Then they were rinsed with Extran and washed completely with distilled water followed by de-ionized water. They were immersed in HNO_3 for half an hour and washed with distilled water. The substrates were dipped in HCL for the same period, and then the slides were rinsed using soap solution and distilled water. Finally the substrates were allowed to dry in a hot air oven for about 2 hour at a temperature of 60℃.This way the substrates required for film coating in this study have been cleaned thoroughly.

3.5 THIN FILM TECHNIQUES

The thin film deposition can be broadly classified as physical and chemical methods. Physical methods consists vaccum evaporation and sputtering; the material to be deposited has been transferred to a gaseous state either by evaporation or an impact process and then deposited on the substrate. Under chemical methods, we have the gas phase chemical processes such as conventional chemical vapour deposition (CVD), Laser CVD, Photo CVD, Metal organic chemical vapour deposition (MOCVD) and plasma enhanced CVD. The Liquid phase chemical techniques include spray pyrolysis, electrodeposition, chemical bath deposition, successive ionic layer adsorption, anodization and liquid phase epitaxy etc. The physical methods are plugged with certain drawbacks and difficulties. A careful and precise control of the boat temperature is an essential requirement for obtaining good stoichiometric films and to obtain the particular composition in alloy films. Chemical methods are relatively economical and easier ones as

compared to physical methods. Chemical methods are relatively economical and easier ones as compared to physical methods. On the other hand, there is no ideal method to prepare the compounds and alloys in thin film form, which will satisfy all the requirements.

3.6 TECHNIQUE ADOPTED IN THIS STUDY

3.6.1 CHEMICAL BATH DEPOSITION

The Chemical bath deposition (CBD) method is one of the cheapest methods to deposit thin films and nanomaterials, as it does not depend on expensive equipment and is a scalable technique that can be employed for large area batch processing or continous deposition. In 1933 Bruckman deposited Lead (II) sulfide (PbS) thin film by chemical bath deposition (CBD) or solution grown method. It is well known that the chemical bath deposition (CBD) is an economical growth technique that can deposit homogeneous stoichiometric films with high purity on non-planar substrates, which has been proved by growth of single- or poly-crystalline oxide thin films. Typically liquid solution containing precursors to the eventual film is prepared and a substrate is exposed to the solution. Over the course of seconds or minutes or hours (depending on the reagents and their concentrations), the precursors react to produce a solid material that grows, atom by atom, on all this surface exposed to the bath. This method of deposition, compared to other thin film deposition methods is often noted for its low cost and for the simple apparatus required to perform the deposition. It is, however, not always an option for thin film growth, as a suitable precursor solution does not exist for every material. But chemical bath deposition is mostly preferable to deposit liquids onto glass substrates.

The major advantage of CBD is that it requires only solution containers and substrate mounting devices. The one drawback of this method is the wastage of solution after every deposition. Chemical Bath Deposition always yields stable,

adherent, uniform and hard films with good reproducibility by a relatively simple process. The growth of thin films depends on growth condition, duration of deposition, composition, temperature of the solution, topographical and chemical nature of the substrate. Hence in this study deposition of PEO/PMMA thin films has been carried out by Chemical Bath Deposition. The method is shown in the Fig 3.1

3.7 EXPERIMENTAL DETAILS

3.7.1 CHEMICALS AND GLASS WARES USED

Poly (ethylene oxide) (99% purity), Poly (methyl methacrylate) (>99% purity), Toluene GR (>99% purity- Merck), were used to prepare polymer precursor solution. HCl GR (>40% purity- Merck), HNO_3 GR (>60% purity- Merck), Acetone GR (>99% purity- Merck), double distilled water and Extran were used to clean the microscopic glass slides. Glass Wares used for film coating are blue star glass slides with 75mmx 25mmx 1mm dimension, beakers, conical flasks and measuring jar.

3.7.2 EXPERIMENTAL PROCEDURE FOR CHEMICAL BATH DEPOSITION

Thin films of poly (ethylene oxide) and poly (methyl methacrylate) were deposited on highly clean glass substrates. The solution was prepared in a glass beaker by dissolving known quantities of PEO in different solvents at room temperature to yield different molar solutions. Then the solution was heated to 50˚C to ensure a homogeneous mixing of solvent. Similarly the blend of both PEO and PMMA solution were prepared by dissolving these two polymers in a common solvent and the solution was heated to 50˚C to ensure the complete mixing of these two polymers. Then the solution was filtered using 0.5µm Whatman filter papers to remove any undissolved impurities and dust before use. Thin films were prepared using Constant temperature water bath with digital proportional temperature controller. The beakers with PEO and PEO/PMMA solutions were kept in water

bath. The rate of growth and thickness of the film depend on the nature of the substrate, the concentration and temperature of the solution and also the time for which the substrate is left immersed in the solution. The chemical bath setup used in this work is shown in Fig 3.1.

3.8 THICKNESS MEASUREMENT TECHNIQUE

Thickness of a film is among the first quoted attributes of its nature. The reason is that thin film properties usually depend on thickness. Historically the use of films in optical applications spurred the development of techniques capable of measuring film thicknesses with high accuracy **[Milton Orhing]**. The desired film thickness is closely related to the deposition or formation rates, since economical considerations determine to a large degree, the selection of the most appropriate deposition technology. Thin films cover a thickness range from about one nanometer to several micrometers or from monolayers to thickness approaching bulk material characteristics. **[Krishna Seshan]**

Various types of optical interference phenomena have been found to be most useful for the measurement of film thickness, in addition to interference phenomenon, there are other optical techniques which can be used to measure thickness. Examples are ellipsometry and absorption spectroscopy. In addition to the optical techniques there are mechanical, electrical and magnetic techniques which have been used for film-thickness measurements. Electrical methods of measuring film thickness involve film resistance method, capacitance monitor and ionization method. Film thickness can also be measured mechanically by means of the following techniques such as profilometry, quartz crystal microbalance, microbalance gravimetric and ultrasonic multilayer film technology, etc. Among these the one that has found the widest acceptance is the microbalance gravimetric technique. **[Maissel and Glang]**

Instrument suitable for the Gravimetric determination of small quantities of mass are summarily referred to as microbalance technique. This is one of the oldest methods for film-thickness determination by weighing the substrate before and after deposition of the film. The basic requirement of Gravimetric technique is the weight

measurement should be done on the same substrate before and after deposition of the film. Another requirement is that the substrate should not be prone to chipping or other types of material loss and the deposition process should not result in the accumulation of material other than the desired film. The weighing should be very accurate since relatively small difference in large numbers is being sought. The weighing accuracy of ±1μg, which is attainable with digital balance, barely permits the detection of about micron film thickness. The film thickness (t) has been evaluated from the following relation

$$t = W/\rho A$$

Where,

Weight of the thin film on the glass substrate (W) = Weight after deposition-weight before deposition (g)

ρ = density of the material (gcm^{-3})

A = deposited area (cm^2)

DENVER microbalance with 4-digit accuracy used for measuring the weight of the deposited film during this study is shown in Fig 3.2.The thickness of Pristine PEO thin films and PEO/PMMA blend thin films deposited during this study is shown in table 3.1 and 3.2.

Thickness of Sample III and IV is less than 10 micron in comparison to Pristine PEO of thickness above 50 micron. Thus blending PEO with PMMA, better thin films were obtained in this study.

Table-3.1

Thickness of Pristine PEO Samples

Samples	Molar Ratio	Thickness (µm)
Sample I	1.5 mole of Pristine PEO	31.3
Sample II	2 mole of Pristine PEO	53.5

Table-3.2

Thickness of PEO/PMMA Blend Samples

Samples	Molar Ratio	Thickness (µm)
Sample III	1 mole of PEO & 2.5 mole of PMMA	7.69
Sample IV	1 mole of PEO & 2 mole of PMMA	8.92
Sample V	1.5 mole of PEO & 2 mole of PMMA	25.6

Figure 3.1 Chemical Bath Setup for Thin film Coating

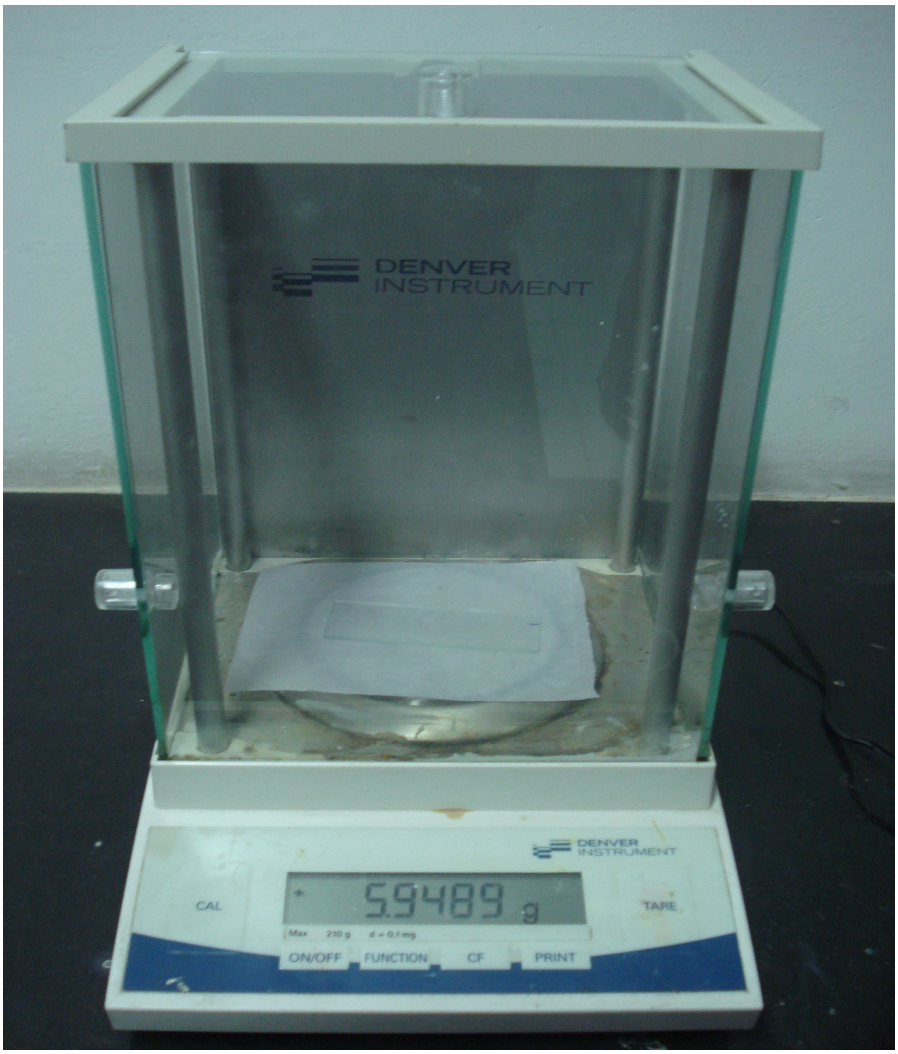

Figure 3.2 DENVER Microbalance Used for Thickness Measurement

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RESULTS AND DISCUSSION

CHAPTER 4

RESULTS AND DISCUSSION

4.1 INTRODUCTION

Scientific disciplines are identified and differentiated by the experimental equipment and measurement techniques they employ. The same is true for thin film science and technology. The role played by films was largely a utilization one necessitating measurement of film thickness and optical properties. With the increasing interdisciplinary nature of applications, new demands for film characterization and property measurement of both individual films and multilayer coatings have recently been explored. These are based on the interaction of radiation and electrons with the atoms of a solid, which result in the scattering of electrons and other associated phenomena. **[Milton Ohring].** To make sure that coatings which were produced by a given process satisfy the specified technological demands a wide variety of characterization, measurement and testing methods are available. The chemical composition, crystalline structure, optical properties, electrical properties and mechanical properties must be considered in thin film analysis. The results obtained from UV-VIS spectrometry, Photoluminescence, and FT-IR of the solution grown polymer thin films are discussed in this chapter.

4.2 ULTRAVIOLET AND VISIBLE SPECTROSCOPY

Ultraviolet-visible spectroscopy (UV-VIS) refers to the absorption spectroscopy in the ultraviolet-visible spectral region. The absorption in the visible range directly affects the perceived colour of the chemicals involved. In this region of 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 defined as the absorption and emission of radiation associated with changes in spatial distribution of electrons in atom and molecules. In practice, the electrons involved are usually the valance or bonding electrons, which can be excited by absorption of UV or visible or near IR radiations. Excitation of bound electrons from the highest occupied molecular orbits increases the spatial extend of electron distribution, making the total electron density larger and often more polarizable. Ultra violet absorption spectra arise due to electronic excitation. The outer electron atoms or molecules absorb radiant energy and get excited. These electronic transitions are quantized and depend upon the electronic structure of absorber **[Patanian].**

Optical characterization in absorption mode was carried out on the samples by using the UV-Vis spectroscopy. When a beam of electromagnetic radiation strikes an object it can be absorbed, transmitted, scattered, reflected or it can excite fluorescence. The processes concerned in the absorption spectroscopy are: absorption and transmission. An optical spectrometer records the wavelength at which absorption occurs together with the degree of absorption at each wavelength. Ultra violet and visible spectroscopy is used for quantitative analysis of the samples. The absorption of radiation in a sample follows the Beer-Lambert law which states that the concentration of substance in a sample (thin film/solution) is directly proportional to the absorbance **[H.K.Moudgil].**

On passing electromagnetic radiation in the ultraviolet and visible regions through a compound with multiple bonds, a portion of the radiation is normally absorbed by the compound. The amount of absorption depends on the wavelength of the radiation and the structure of the compound. The absorption of radiation is due to the subtraction of energy from the radiation beam when electrons in orbital of lower energy are excited into orbital of higher energy. Since this is an electron excitation phenomenon, UV- visible spectroscopy is called electronic spectroscopy **[P.S.Kalsi].**

EXPERIMENT

The experimental arrangement of a typical double beam UV-Visible spectrometer is shown in Figure 4.1**.** A beam of light from a visible or UV light source (colored red) is separated into its component wavelengths by a prism or diffraction grating. Each monochromatic beam in turn is split into two equal intensity beams by a half-mirrored device. One beam is called as the sample beam and it passes through a small transparent container called the cuvette containing a solution of the compound being studied in a transparent solvent. The other beam called as the reference passes through an identical cuvette containing only the solvent. The intensities of these light beams are then measured by electronic detectors and then compared. The intensity of the reference beam which should have little or no light absorption is denoted as I_0. The intensity of the sample beam is I. The ultraviolet (UV) region scanned is normally from 200 to 400 nm and the visible portion is from 400 to 800 nm. Absorption may be presented as transmittance ($T = I/I_0$) or absorbance ($A = \log I_0/I$). If no absorption has occurred then T = 1.0 and A= 0. Different compounds may have different absorption maxima. **[Skoog et al.,]**

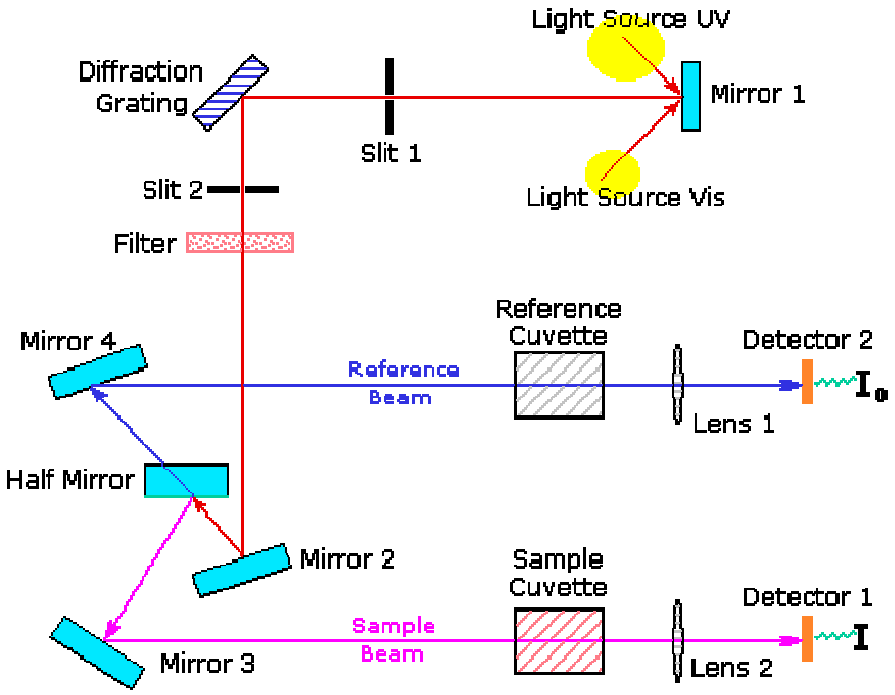

Figure.4.1 UV-Visible spectrometer

4.3 FOURIER TRANSFORM INFRARED SPECTROSCOPY

Infra-Red spectroscopy was first developed by astronomers in the early 1950s to study the infrared spectra of distant stars. It is a simple mathematical technique to resolve a complex wave into its frequency components. The conventional IR spectrometers are not of much use for the far IR region (20-400cm^{-1}) as the sources are weak and the detectors insensitive. FTIR has made this energy region more accessible. It has made the middle infrared (400-4000cm^{-1}) also more useful **[G.Aruldhas].**

Infrared (IR) radiation is an Electromagnetic radiation in the wavelength range that is adjacent to and less energy than visible radiation. The name of the region is derived from the fact that radiation in the region is less (infra) energetic than that of visible red radiation. IR radiation does not have sufficient energy to cause the excitation of electrons, however it causes atoms and groups of atoms of organic compounds to vibrate faster about the covalent bonds, which connects them. The vibrations are quantised and as they occur, the compound absorbs infrared energy in particular regions of the spectrum. The position of an IR absorption band is specified in frequency units by its wave number ῡ measured in reciprocal centimeters (cm^{-1}) or by its wavelength λ, measured in micrometers (μm). IR absorption occurs when the frequency of the alternating electric field that is associated with the incident radiation matches a possible change in a vibrational or rotational frequency of the absorbing molecule. **[Robert D.Braun].** A simple optical layout of a typical FTIR spectrometer is shown in Figure 4.2.

EXPERIMENT

An FT-IR spectrometer works on the basis of Michelson Interferometer. There are three basic components in an FTIR spectrometer: radiation source, interferometer and detector. The interferometer consists of three active components: a moving mirror, a fixed mirror, and a beam splitter. The two mirrors are perpendicular to each other. The beam splitter is a semi-reflecting device. The radiation from the broadband IR source is collimated and directed into the interferometer, which impinges on the beam splitter. At

the beam splitter, half the IR beam is transmitted to the fixed mirror and the remaining half is reflected to the moving mirror. After the beams are reflected from the two mirrors, they are recombined at the beam splitter. Due to changes in the relative position of the moving mirror to the fixed mirror, an interference pattern is generated. The resulting beam then passes through the sample and is eventually focused on the detector. The interferogram contains information over the entire IR region to which the detector responds**. [R.Wilfred Sugumar].**

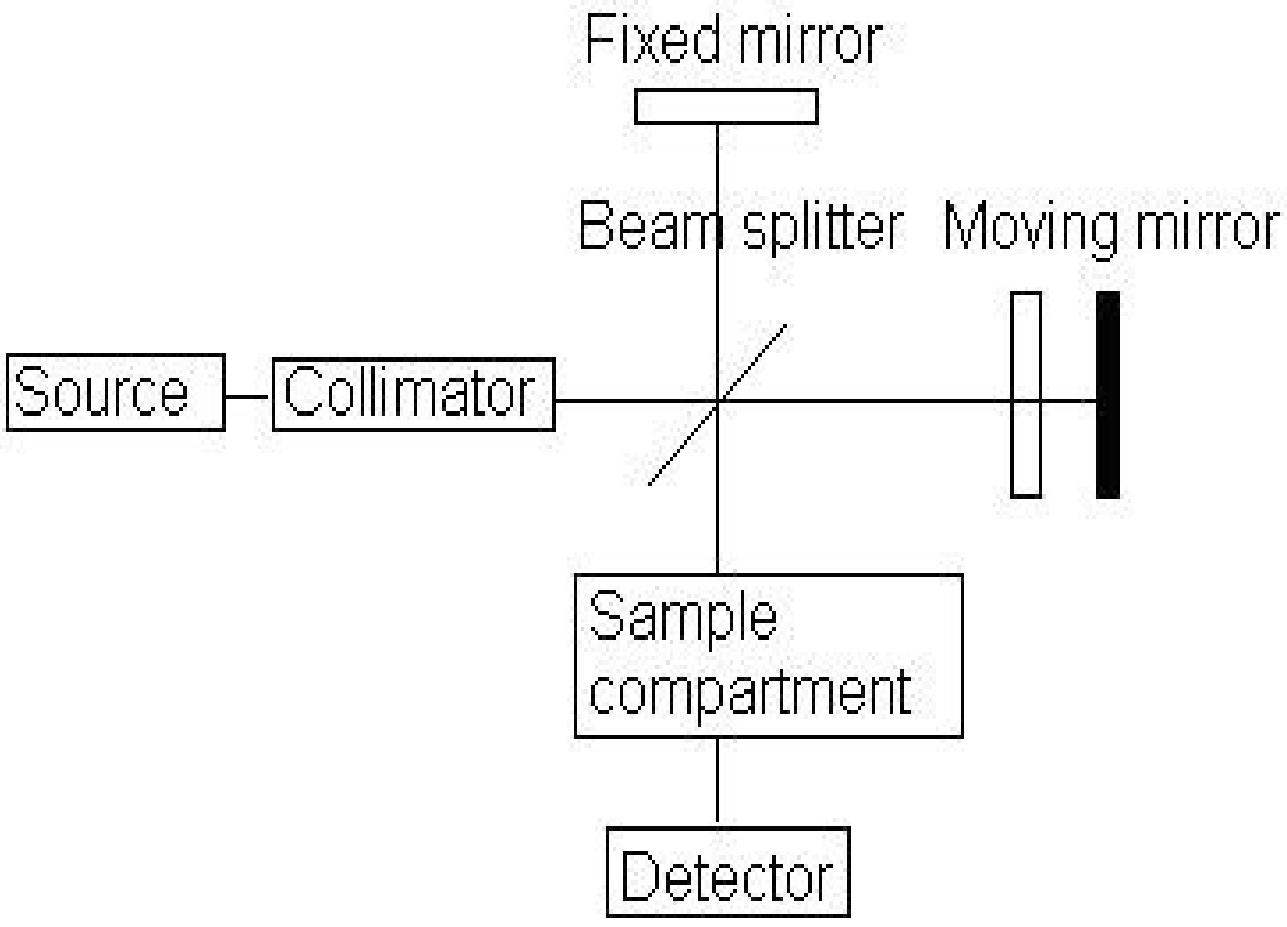

Figure.4.2 Schematic representation of FTIR spectrometer

4.4 PHOTOLUMINESCENCE SPECTROSCOPY

Photoluminescence describes the phenomenon of light emission from any form of matter after the absorption of photons (electromagnetic radiation). It is one of many forms of luminescence (light emission) and is initiated by photoexcitation (excitation by photons), hence the prefix photo. The excitation typically undergoes various relaxation processes and then photons are re-radiated. The period between absorption and emission can be extremely short: it ranges from the femtosecond-regime for the emission from, e.g., free-carrier plasma in inorganic semiconductors up to milliseconds for phosphorescent processes in molecular systems; however, it can also be extended into minutes or hours under special circumstances.

Photoluminescence is a process in which a substance absorbs photons (electromagnetic radiation) and then re-radiates photons. Quantum mechanically, this can be described as an excitation to a higher energy state and then a return to a lower energy state accompanied by the emission of a photon. This is one of many forms of luminescence (light emission) and is distinguished by photoexcitation (excitation by photons). The period between absorption and emission is typically extremely short, in the order of 10 nanoseconds. It is a two step process (i) excitation of electrons from a lower energy state to higher state as a result of absorption of energy, and (ii) emission of light radiation when the electrons fall back to a lower energy state. Photoluminescence systems are excited by the absorption of photons and they re-emit energy in the form of radiation of the wavelength which is same or longer than the wavelength used for excitation. If the wavelength radiation absorbed and emitted is exactly the same the phenomenon is termed as the resonance radiation or resonance luminescence. The PL spectroscopy is suitable for the characterization of both organic and inorganic materials of virtually any size and the samples can be in solid, liquid or gaseous forms **[Supriya S. Mahajan]**. The basic principle of photoluminescence process is illustrated in figure 4.3

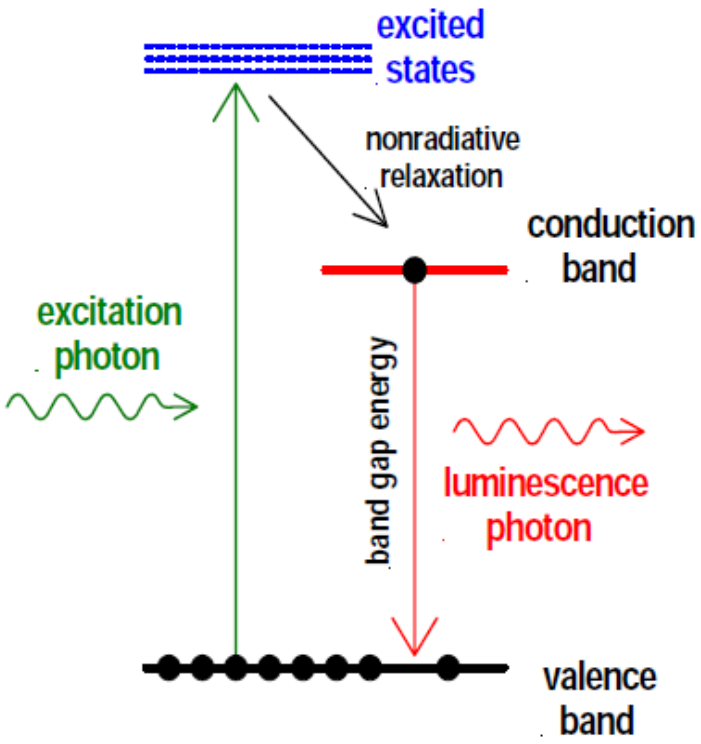

Figure.4.3 Basic principle of photoluminescence process

EXPERIMENT

Electromagnetic radiation in the UV and visible ranges is utilized in PL spectroscopy. The sample's PL emission properties were characterized by four parameters; intensity, emission wavelength, bandwidth of emission peak and the emission stability. The PL properties of a material can change in different ambient environments or in the presence of other molecules. Additionally, as the released photon corresponds to the energy difference between the states, PL spectroscopy can be utilized to study material properties such as band gap, recombination mechanisms and impurity levels. The schematic PL spectroscopy setup is shown in fig 4.4

Sample is placed in a quartz cuvette with a known path length. Double beam optics is generally employed. The first beam passes through an excitation filter or monochromator, then through the sample and onto a detector. This impinging light causes photoluminescence, which is emitted in all directions. A small portion of the emitted light arrives at the detector after passing through an optional emission filter or monochromator. A second reference beam is attenuated and compared with the beam from the sample, and then the PL spectrum is recorded in the signal processing unit. Solid samples can also be analysed, with the incident beam impinging on the material. Generally an emission spectrum is recorded, where the sample is irradiated with a single wavelength and the intensity of the luminescence emission is recorded as a function of wavelength. In converse, the emission peaks are identified and fixed to scan over an excitation spectrum of wavelength to identify whether the emission is associated with one or more excitation.

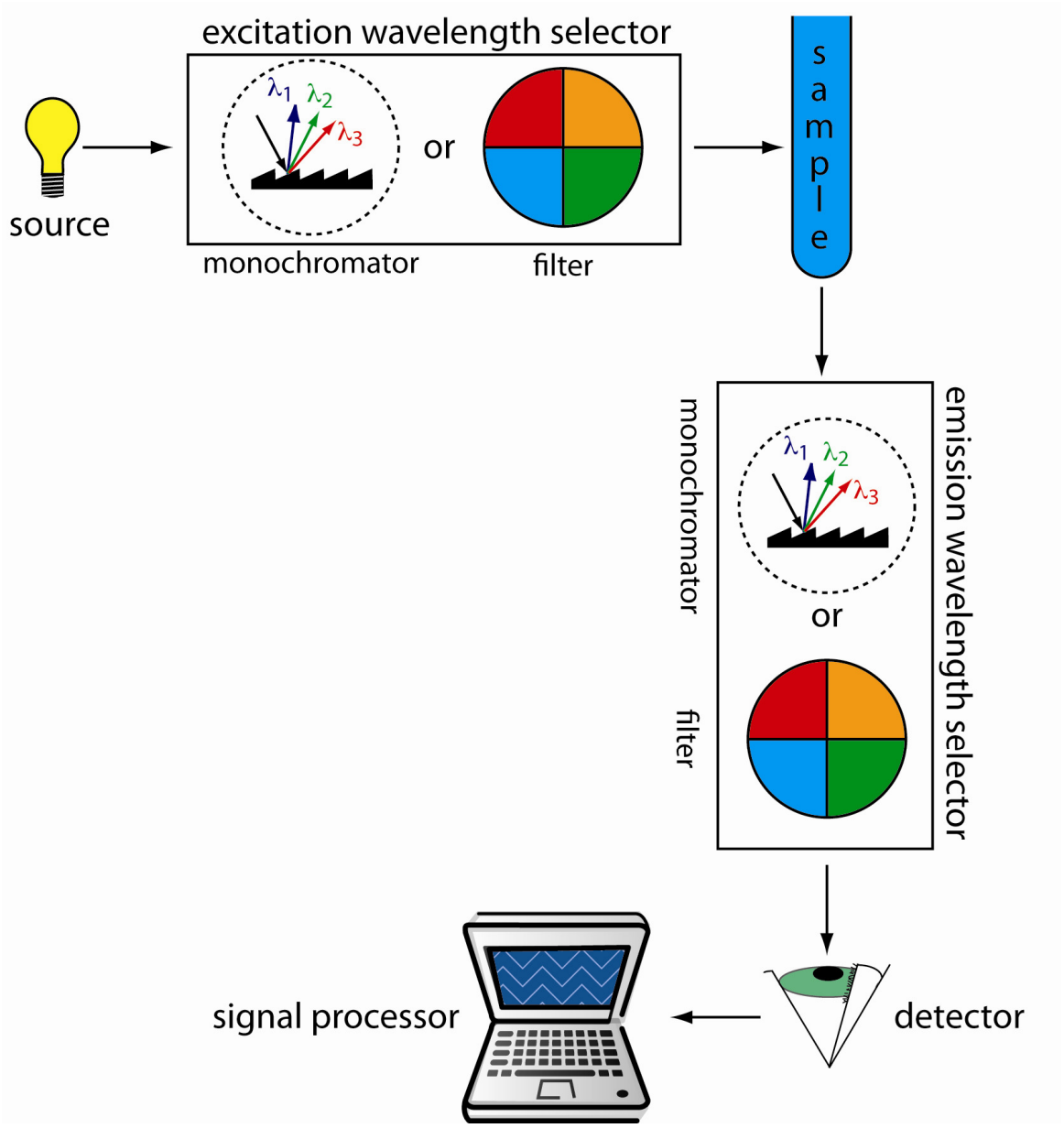

Figure 4.4 Photoluminescence spectrometer

4.5 ABBE'S REFRACTOMETER

A refractometer is a field device for the measurement of an index of refraction (refractometry). The Abbe instrument is the most convenient and widely used refractometer, Fig 4.5 shows a schematic diagram of its optical system. The sample is contained as a thin layer (~0.1mm) between two prisms. The upper prism is firmly mounted on a bearing that allows its rotation by means of the side arm shown in dotted lines. The lower prism is hinged to the upper to permit separation for cleaning and for introduction of the sample. The lower prism face is rough-ground: when light is reflected into the prism, this surface effectively becomes the source for an infinite number of rays that pass through the sample at all angels. The radiation is refracted at the interface of the sample and the smooth-ground face of the upper prism. After this it passes into the fixed telescope. The eyepiece of the telescope is provided with crosshairs: in making a measurement, the prism angle is changed until the light-dark interface just coincides with the crosshairs. The position of the prism is then established from the fixed scale (which is normally graduated in units of n_D).

The accuracy of the instrument is about ± 0.0002, its precision is half this figure. The most serious error in the Abbe instrument is caused by the fact that the nearly glazing rays are cut off by the arrangement of two prisms, the boundary is thus less sharp than is desirable. .

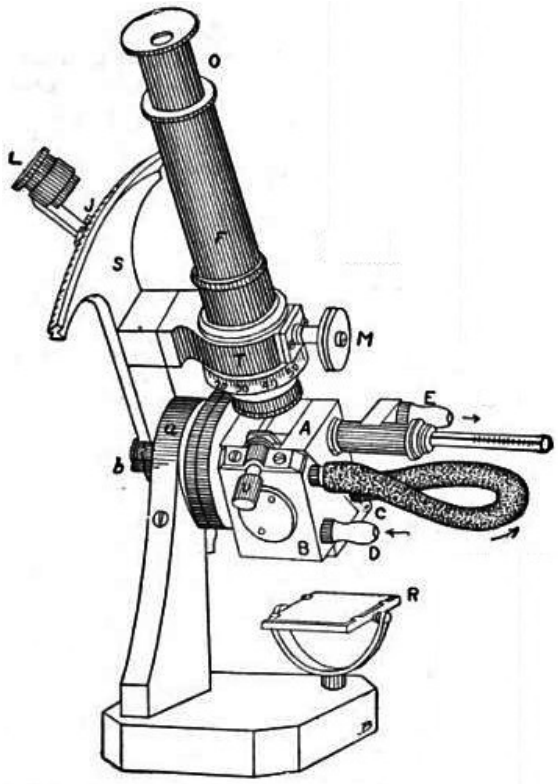

Figure 4.5 Abbe's Refractometer

4.6 DISCUSSION OF RESULTS OBTAINED

4.6.1 RESULTS OBTAINED FROM UV-VISIBLE SPECTROSCOPY

The UV-Visible spectrum for sample I and III is shown in the fig 4.6 and 4.7 respectively. From fig 4.6 (Pristine PEO), it is noted that the cut off appears at 294nm.It shows transparency for a range of wavelength from 380nm to 1180nm.

The UV-Visible spectrum for PEO/PMMA blend is shown in the fig.4.7.The cut off appears at 297nm.For this blend film transparency extends from 430nm to 1335nm and the percentage of transmission is above 99%.The addition of PMMA to PEO has extended the transparency region. This ensures that PEO/PMMA blend is a suitable material for many optoelectronic and optical devices.

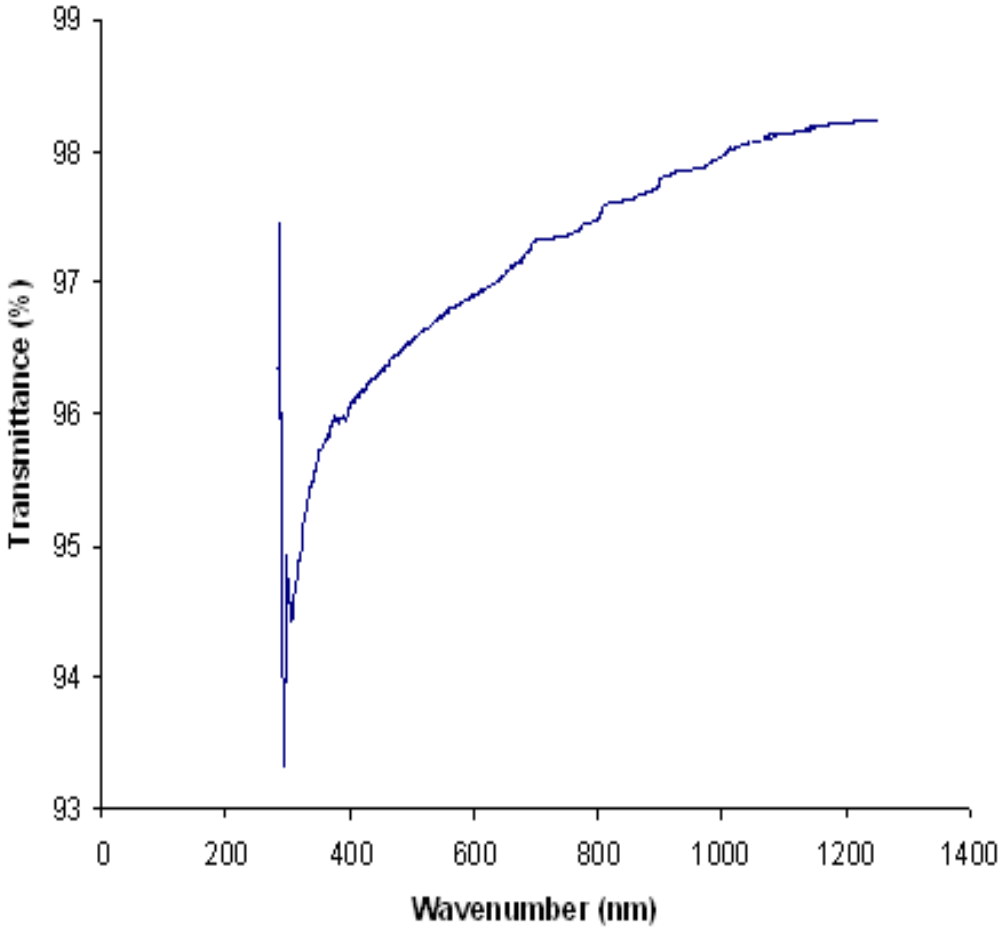

Figure 4.6 Transmittance spectrum of sample I (PEO) film

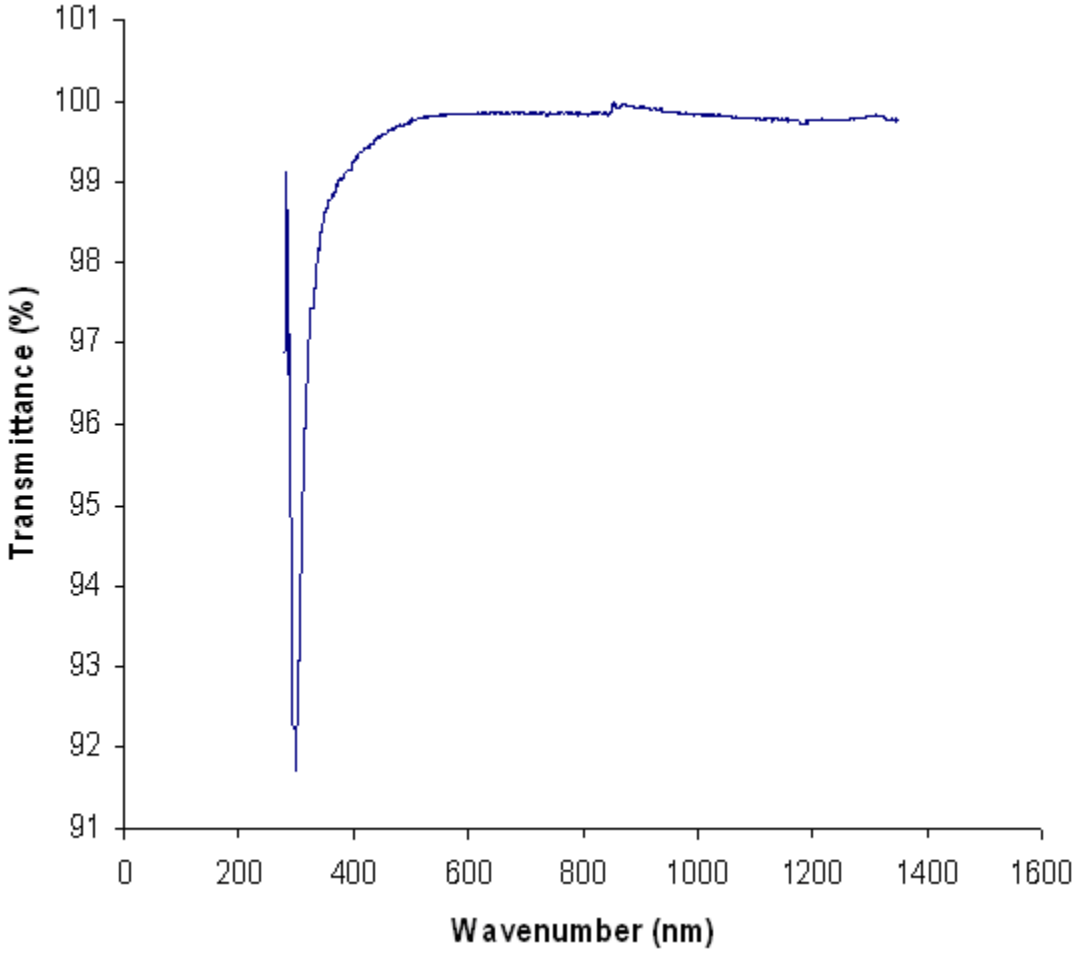

Figure 4.7 Transmittance spectrum of sample III (PEO/PMMA) film

4.6.2 RESULTS OBTAINED FROM FOURIER TRANSFORM INFRARED SPECTROSCOPY

FT-IR characterization study is carried out for sample I (Pristine PEO), which is shown in the fig.4.8. The peak obtained in the spectra corresponds to 626 cm^{-1} represents the C-H stretching. The peak observed at 1028 cm^{-1} represents the C-O-C stretching, which also confirms the presence of Pristine PEO in sample I.**[Vijaya S. Sangawar et al.,]**

The FT-IR spectra for sample III (PEO/PMMA blend) is shown in the fig 4.9. The functional groups are identified with respect to the various peaks present in the spectra. The peak at 1740 cm^{-1} is due to C=O stretching. The peaks at 2890 cm^{-1} and 2975 cm^{-1} represents the aliphatic C-H stretching mode. The peak at 3594 cm^{-1} is due to the absorption of –OH. Thus the presence of PMMA is confirmed. The peaks at 904cm^{-1} and 1031cm^{-1} represents the CH_2 rocking and C-O-C stretching, respectively due to the presence of PEO. The presence of both monomers ethylene oxide and methyl methacrylate is confirmed from FTIR spectra analysis**.[S.Ramesh et al.,]**

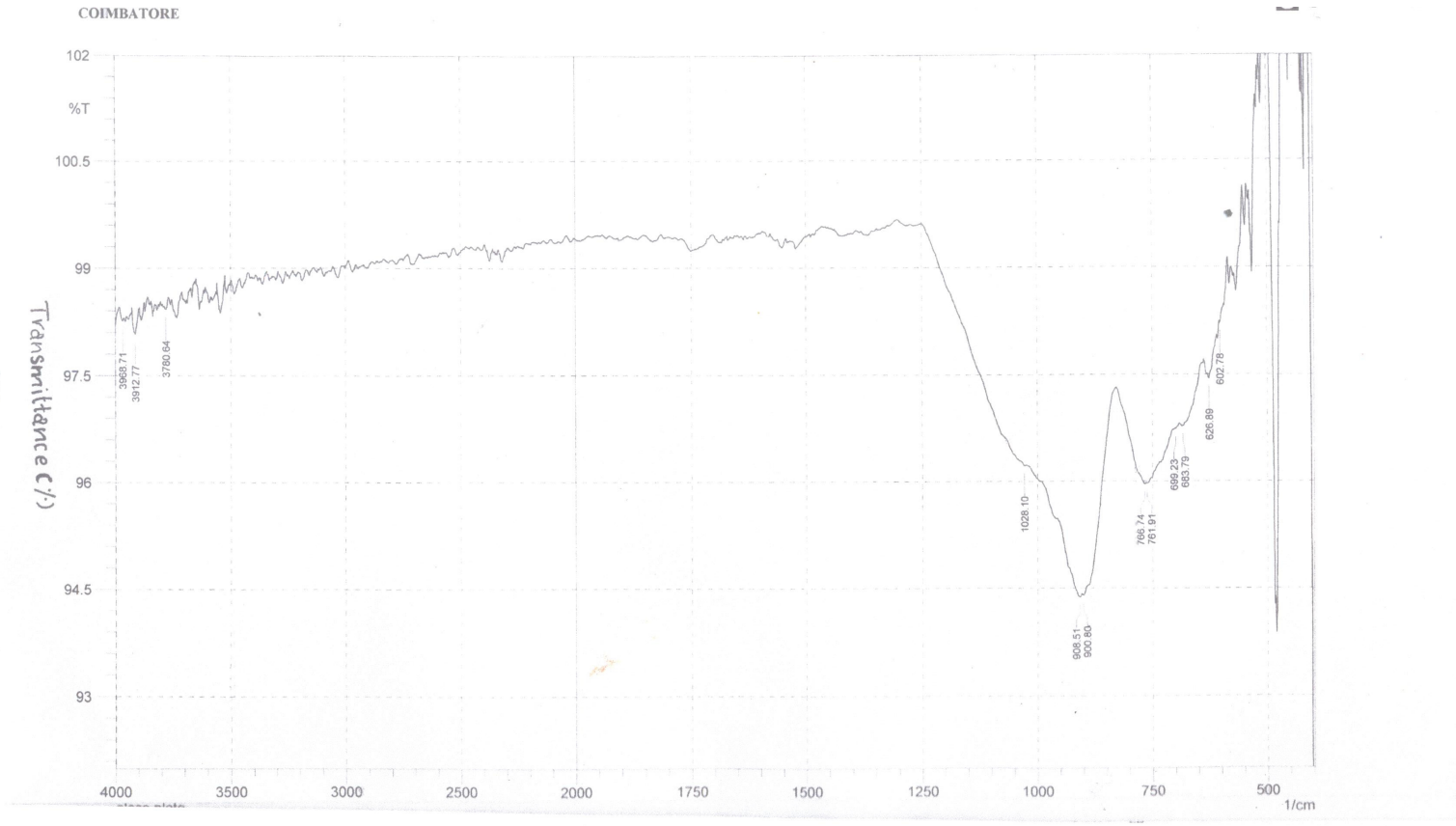

Figure 4.8 Fourier Transform infra-red spectrum of Sample I (PEO) film

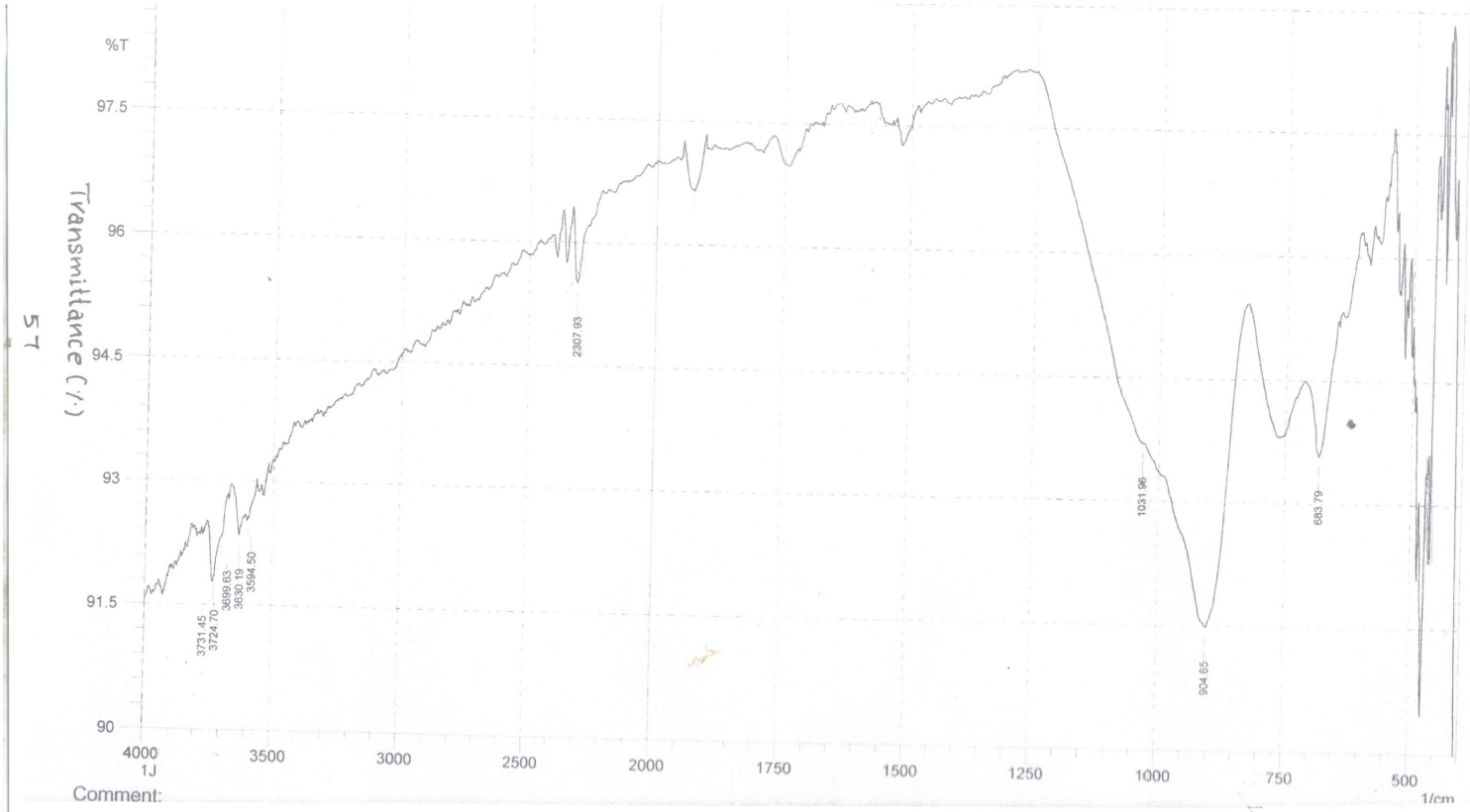

Figure 4.9 Fourier Transform infra-red spectrum of SampleII(PEO/PMMA) film

4.6.3 RESULTS OBTAINED FROM PHOTOLUMINESCENCE

The Photoluminescence spectrum of thin films is studied using FLUOROLOG HORIBA (Jobin YVON). The graph is plotted as wavelength versus intensity. For an excitation at 320nm, photoluminescence spectrum of sample I (pristine PEO) is shown in the fig.4.10. The peak is observed around 395nm and the band width is 81nm.

The photoluminescence spectrum of sample IV (PEO/PMMA blend) is shown in the fig.4.11where the excitation is at 320nm. The peak is observed around 396nm and the band width is 74nm.

For an excitation at 320nm, photoluminescence spectrum of sample V (PEO/PMMA blend) is shown in the fig.4.12. The peak is observed around 396nm and the bandwidth is 73nm**. [Vijaya S. Sangawar et al.,]**

The bandwidth of both samples IV and V (PEO/PMMA blend) decreases when compared with the sample I (pristine PEO). This proves that PEO/PMMA blend is a more suitable material for low pass filter.

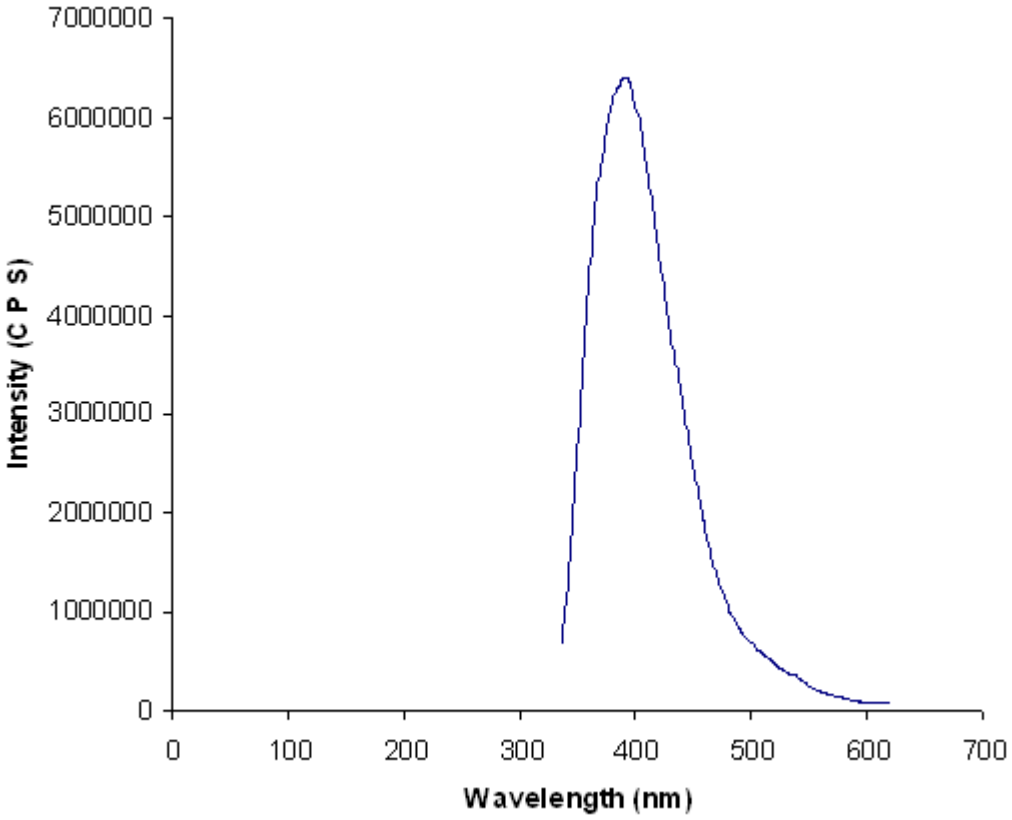

Figure 4.10 Photoluminescence spectrum of sample I (PEO) film

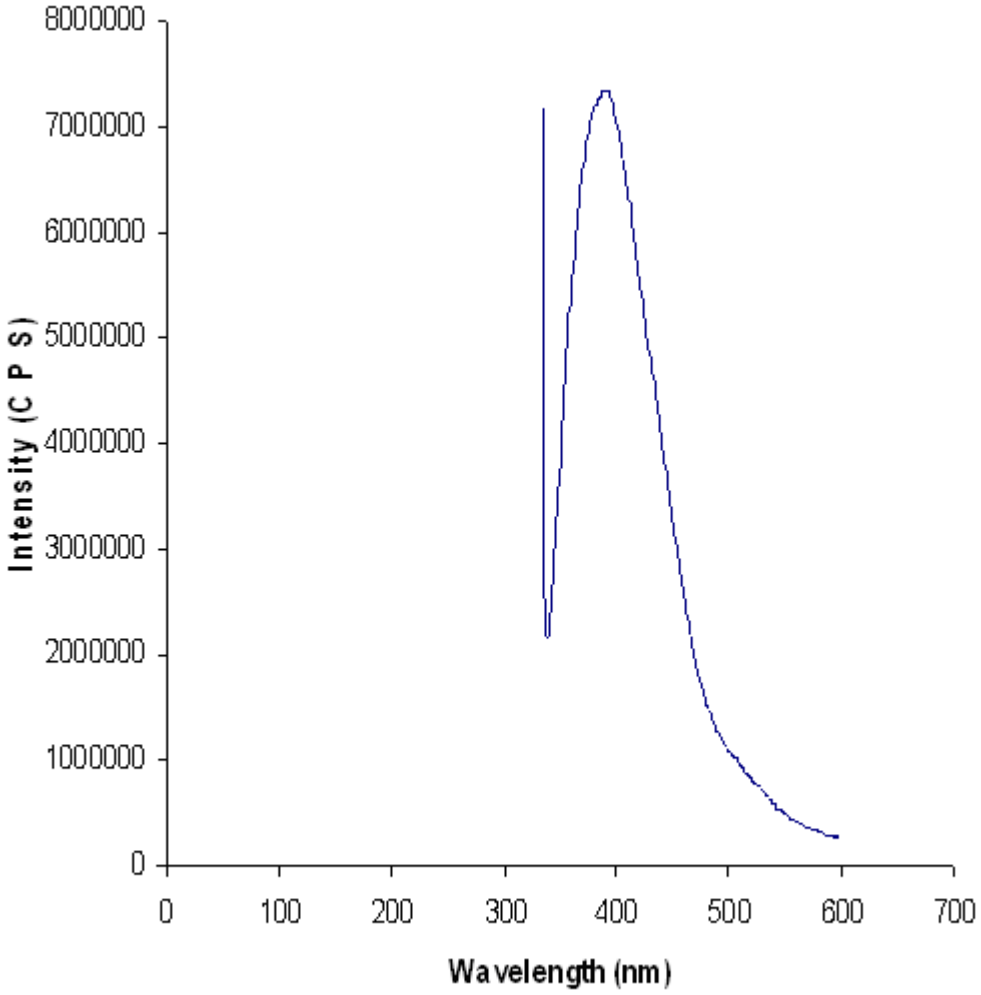

Figure 4.11 Photoluminescence spectrum of sample IV (PEO/PMMA) film

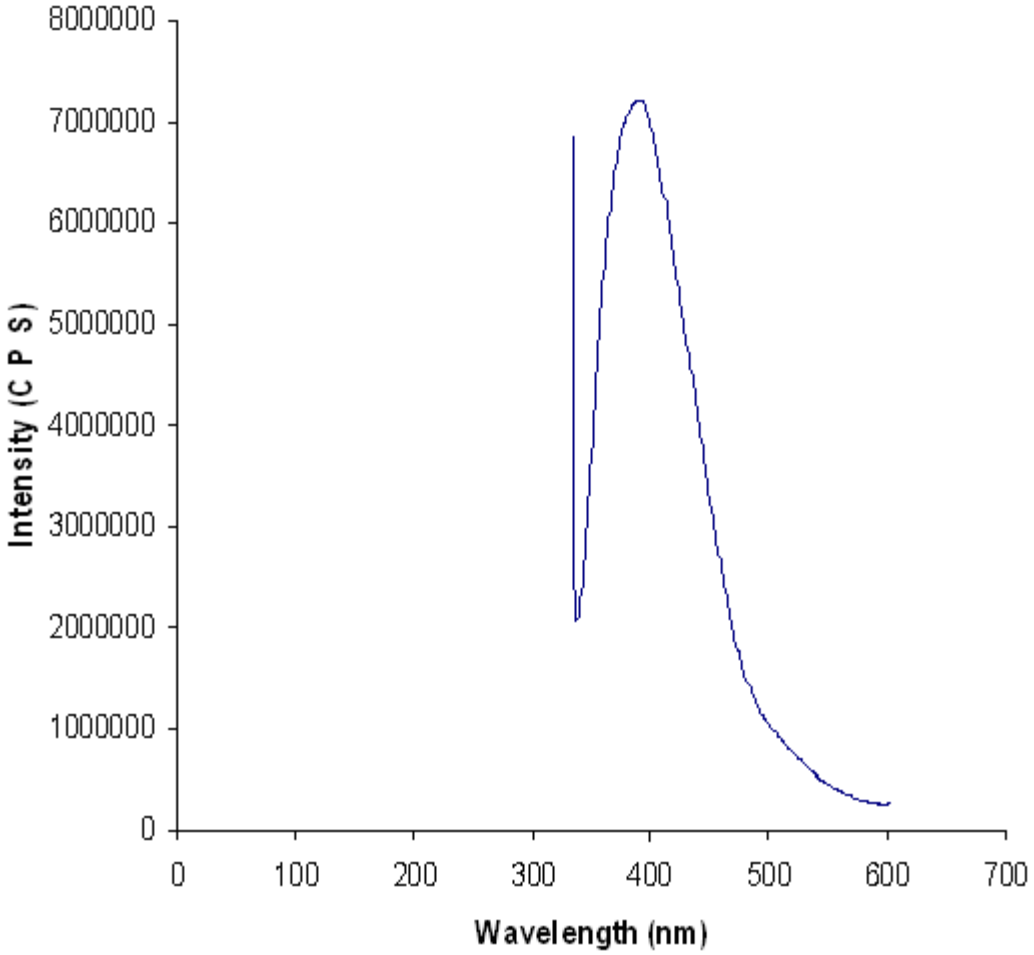

Figure 4.12 Photoluminescence spectrum of sample V (PEO/PMMA) film

4.6.4 RESULTS OBTAINED FROM REFRACTIVE INDEX

The refractive index is determined for sample III and IV using the Abbe refractometer. The results obtained are listed in the table 4.1. It is observed that the values of refractive index increases as the temperature of measurement increases. The refractive index for sample IV is greater than that for sample III at all three measured temperatures. This may be due to the reason that sample IV is thicker by 1.2μm than sample III. The values of refractive index lie ≃ close to 1.33 to 1.347 and hence these films can act as durable lenses and acrylic glass**. [Jinaming Yu et al.,]**

Table 4.1 Values of Refractive Index for PEO/PMMA Blend Samples

Temperature	Refractive index	
	Sample-III	Sample-IV
40°C	1.333	1.341
45°C	1.341	1.345
50°C	1.344	1.347

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SUMMARY AND CONCLUSION

CHAPTER 5

SUMMARY AND CONCLUSION

The increasing complexity of electronic systems has stimulated the development of thin film microelectronics. Different useful properties exhibited by many materials as thin film, lead to the development of thin film technology. Polymer thin films are a novel class of materials, have wide range of industrial and biomedical applications and are now an integral part of our every day use. Their universal presence in paints, coatings, packaging adhesives, dielectrics and other common entities has firmly established their versatility, integrity and dependability.

In this study, Poly (ethylene oxide) thin films and Poly (methyl methacrylate)/Poly (ethylene oxide) blend thin films were successfully deposited by chemical bath deposition. Thickness of the Pristine PEO films lie in the range of 31.1 to 53.5μm.The thickness of PEO/PMMA blend lie in the range 7.69 to 25.6μm.The transparency region is extended from 430nm to 1335nm for PEO/PMMA blend films which ensures that this blend is a suitable material for optical devices. Elucidation of functional groups was done with the help of Fourier Transform Infra-Red (FTIR) spectrometer. The refractive index values obtained ensures that the film can be used as durable lenses and acrylic glass. The Photoluminescence spectrum of the blend film reveals that the decrease in bandwidth validates its use as potential material for low pass filter.