# Studies on PEO/PS Composite Thin Films

**Saranya, R**

**12PPH010**

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

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Signature of the Supervisor

# *ACKNOWLEDGEMENT*

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**Saranya, R**

# NOMENCLATURE

t = Thickness of the film (g)

W = Weight of the film

A = Area of the deposited film

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

# CONTENTS

# CONTENTS

## LIST OF TABLES



## LIST OF PLATES

# LIST OF FIGURES

# *INTRODUCTION*

# CHAPTER 1

# INTRODUCTION

## 1.1 INTRODUCTION

Modern thin film technology has evolved into a sophisticated set of techniques used to increase performance and aesthetic value of many products and make new functional systems and devices. The phenomenal rise in thin film researches is due to their extensive application in the diverse fields of Electronics, Optics, Space Science, aircrafts, defence and other industries. These investigations have led to numerous inventions in the form of active devices, passive components, piezo-electric devices, micro-miniaturization of power supply, rectification and amplification, sensor elements, storage of solar energy and its conversion to other films, interference filters, reflecting and antireflecting coatings. **[Grove]**

Thin film has received tremendous attention especially after the world war because of weapon systems, space science, solar energy utilization and also as optical and super conducting film materials, high memory computer elements, sensors and in microelectronic and hybrid circuits **[A.Goswami].** Some work is being done with ferromagnetic thin films as well for use as computer memory. Ceramic thin films are also in wide use. The relatively high hardness and inertness of ceramic materials make this type of thin film coatings prominent for protection of substrate materials against corrosion, oxidation and wear. In particular, the use of such coatings on cutting tools may extend their life by several orders of magnitude. **[Kamal Nain Chopra]**

Equipment manufacturers have made successful efforts to meet the requirements for improved and more economical deposition systems and for in situ process monitors and controls for measuring film parameters. The improved understanding of the physics and chemistry of films, surfaces, interfaces, and microstructures is made possible by the remarkable advances in analytical instrumentation during the past twenty years. A better fundamental understanding of materials leads to expanded applications and new designs of devices that incorporate these materials. A good example of the crucial importance of deposition technology is the fabrication of semiconductor devices, an industry that is totally dependent on the formation of thin solid films of a variety of materials by deposition from the gas, vapour, liquid, or solid phase. [**Dr. Veer Singh**]

## 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

The thickness of mechanical thin 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 micro structural size scale, the film is considered to be a micro structurally thin film. Most metallic thin films used in microelectronic devices and magnetic storage media are examples of microstructural thin film, where the film thickness is substantially greater than atomic or molecular dimensions.

Atomical 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]**

## 1.3 DEPOSITION TECHNOLOGIES

The act of applying a thin film to a surface or any technique for depositing a thin film of material onto a substrate is known as thin film deposition. There are many dozens of deposition technologies for material formation.

Modern thin film technology has evolved into a sophisticated set of techniques used to fabricate many products. Basically, thin film deposition methods are either purely

physical such as evaporation methods or purely chemical such as gas and liquid phase chemical process. Thus various deposition techniques fall into two main groups:

- Chemical method
- Physical method **[Krishna Seshan]**

## 1.3.1 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 require expensive equipment and is a scalable technique that can be employed for large area batch processing or continuous deposition. In 1933s, Bruckman deposited sulphide (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. **[R. Devi et al]**

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. Among various deposition techniques, chemical bath deposition yields stable, adherent, uniform and hard films with good reproducibility by a relatively simple process. The chemical bath deposition method is one of the simplest methods for preparing highly efficient thin films in a simple manner. The growth of thin films strongly depends on growth conditions, such as duration of deposition, composition and temperature of the solution, and topographical and chemical nature of the substrate.

The chemical bath deposition involves two steps, nucleation and particle growth, and is based on the formation of a solid phase from a solution. In the chemical bath deposition procedure, the substrate is immersed in an aqueous solution containing the precursors. Chemical bath deposition (CBD) is widely used in the laboratory and industry for the creation of thin films and nanostructures for semiconductors and photovoltaic.

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. Chemical Bath Deposition always yields stable, adherent, uniform and hard films with good reproducibility by a relatively simple process. Hence in this study deposition of PS/PEO thin films has been carried out by Chemical Bath Deposition method.

## 1.4 POLYMERS

The word polymer is derived from the classical Greek words *poly* meaning "many" and *meres* meaning "parts". A polymer is a long chain molecule that is composed of a large number of repeating units of identical structure **[Joel R.Fried].** Polymers are a large class of materials consisting of many small molecules called monomers, that can be linked together to form long chains. A typical polymer may include tens of thousands of monomers, because of their large size, polymers are classified as Macromolecules.

The real nature of polymer was established after 1930.These macromolecules have their molecular weight ranging between $10^3$-$10^5$ times molecular weight of the common organic crystalline compounds. They were found to have many physio-chemical properties different from those of common low molecular weight compounds **[M.S.Bhatnagar].**

The process of joining together small molecules or monomers by covalent bonds to form large molecules, with or without formation of other products is known as Polymerization. The use of polymeric material is increasing year by year and in many applications, they are replacing conventional materials such as metals, wood, and natural fibres such as cotton and wool. **[R.J.Young & P.A.Lovell]**

## 1.5 POLYMER THIN FILM

Macromolecular science has a major impact on the way we live. It is difficult to find an aspect of our lives that is not affected by polymers. Just 50 years ago, materials now taken for granted were not existent. With further advances in the understanding of polymer thin films and with new applications being researched, there is no reason to believe that the revolution will stop any time soon.

The technological advancements in the industrial sector across the world have intensified the demand for high performance polymer coatings during the past few

decades. The control of utilization of hazardous air pollutants, heavy metals as well as waste minimization has posed a significant challenge before the coating technologies and urged them to evolve eco-complaint high performance coating materials. Specialty polymeric coatings can be categorized as a special class of coatings that are engineered and designed to provide aesthetic sense and high performance surface protection under specific environmental conditions.

The characteristic features of these polymer coatings are

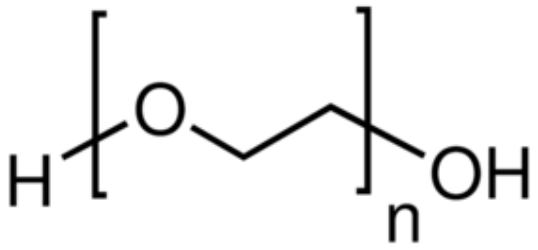
1. Their ability to withstand stress, strain exerted by different kinds of exposure in aggressive environments.
2. They can protect the metallic, concrete and other structures from corrosion and erosion.
3. Aesthetic retention. **[Faiz Mohammad]**

Although most of the development effort on thin film technology has been centered on inorganic materials, organic polymers offer highly desirable electrical and mechanical properties. The desirable properties of organic polymers, which are hard to obtain in inorganic materials and ceramics, include mechanical flexibility, which makes winding possible and the availability of such techniques as spraying, dipping or casting for making films in the range of 0.1 to 0.2 mils. In addition, these polymeric films show high dielectric strength and low dissipation factors. An organic polymer has been widely used for preparing insulating films also. **[Leon I. Maissel & Reinhard Glang]**

The main advantage of polymer thin film is that they can be prepared easily and cheaply. They are also stable and flexible and can be molded into any form. Due to their Piezoelectric and Pyroelectric effects in polarized materials, they have been used as electrets in microphones, air filters, electrophotogrhphy, radiation dosimeter, electrostatic voltage generators, and electro acoustic and electrochemical transducers. Thin polymer film can be made by various techniques such as glow discharge polymerization, electron beam irradiation, Ultraviolet irradiation, R.F sputtering, pyrolysis, electro less glow discharge and evaporation **[O.S. Heavens]**

## 1.6 POLYETHYLENE OXIDE

Polyethylene oxide is an oligomer or polymer of ethylene oxide**.** It is a non-ionic homopolymer**.** It has the following structure:




Where n represents the average number of oxy-ethylene group. The ethylene oxide monomer is nothing more than an epoxide ring. Two corners of the molecule consist of -CH2- linkages. The third corner is oxygen, -O-. In the presence of a catalyst the monomer forms a chain having the repeat unit $–CH_2-CH_2-O-$.

Polyethylene oxide or high-molecular polyethylene glycol is synthesized by suspension polymerization. It is necessary to hold the growing polymer chain in solution in the course of polycondensation process. The reaction is catalyzed by magnesium-, aluminium- or calcium- organoelement compounds. To prevent coagulation of polymer chains from solution, chelating additives such as dimethylglyoxime are used.

Poly (ethylene oxide) has a molecular mass greater than 20,000 g/m**.** It is a white to off-white powder obtainable in several grades, varying in viscosity profile in an aqueous isopropyl alcohol solution. It may contain a suitable antioxidant.

Polyethylene oxide has been used as a retention aid since the 1970's in various Canadian newsprint mills and other applications with high-yield pulps. In most of these cases the retention efficiency is maximized by first adding various proprietary phenolic compounds that appear to interact with the PEO and allow it to bridge between the adjacent solid materials in the furnace.

Recently, poly (ethylene oxide) is gaining the attention of research and development organizations and its application is extending into a wide range of drug delivery systems. Scientists are working on novel drug delivery systems like liposomes, niosomes, nano-technology, magnetic delivery systems, and sonophoresis, but in such type of dosage forms it is a challenge for research scientists and industrialists, to transfer pilot scale to production batches. So alternatively, controlled drug delivery systems, sustained delivery systems, orally disintegrating system, immediate release dosage forms etc., are used. The most important factor in all these release retarding dosage form is the use of polymers. One such polymer is poly (ethylene oxide).

## 1.6.1 PROPERTIES

**High binding efficiency**– PEO is water-soluble resins and has high-binding efficiency for pigments, fillers, and metal powders. These binders easily burn off at low temperatures with little or no tendency to char.

**Form novel complexes** - The strong hydrogen bonding affinity of PEO water-soluble resins accounts for the association of these polyether with various polar compounds, such as phenolic resins, mineral acids, halogens, urea, lignin sulfonic acids and poly (carboxylic acids).

**Crosslink ability** - PEO water-soluble resins can be cross-linked to form gels that are highly water-retentive.

**Emollient** - When applied to the skin and hair, PEO water-soluble resins produce a soft and silky feel.

**Film former excipient** - PEO water-soluble resins can be formed into flexible films both by thermoplastic processing and casting techniques. Such films may be made of PEO water-soluble resins alone or blended with a wide variety of other polymers, such as polyethylene, polystyrene, polycaprolactone, ethylene vinyl acetate, nylon, etc.

**Low toxicity** - PEO, water-soluble resins, shows very low order of toxicity in animal studies by all routes of exposure. At the maximum practical oral dose to rates of about 2 g/kg of body weight neither death nor signs of toxicity are observed. Because of their

high molecular weights, the resins are poorly absorbed from the gastrointestinal tract and are completely and rapidly eliminated. These resins are neither skin irritants nor sensitizers, nor do they cause eye irritation as the dry powder or as aqueous solutions.

**Flocculant activity** - High molecular weight grades of PEO water-soluble resins effectively adsorb onto many colloidal materials and perform as efficient flocculating agents. They exhibit a high affinity for a variety of materials, including silica, clays, oxidized coal fines, lignins and paper fines.

**Lubricity** – PEO water-soluble resins impart a high degree of lubricity when in contact with water.

**Thermoplasticity** - As thermoplastics, PEO water-soluble resins are readily calendered, extruded, injection molded, or cast. Sheets and films of this material are heat-sealable and can be oriented to develop high strength. Films are inherently flexible, tough and resistant to most oils and greases. These resins are compatible with many natural and synthetic polymers. The combination of thermo plasticity and aqueous solubility and compatibility with "hydrophobic" polymers (e.g. polyethylene, polycaprolactone, ethylene vinyl acetate, nylon, etc.) makes PEO water-soluble resins a valuable asset for degradable plastics applications.

**Wet tack** – PEO water-soluble resins exhibit a high degree of wet tack and, thus, are useful as wet adhesives. The dried residue is non-tacky.

**Thickening power (aqueous)** – PEO water-soluble resins are nonionic and completely water-soluble at all temperatures up to the boiling point of water. Unlike most other high molecular weight, water-soluble resins, they do not exhibit an inverse solubility-temperature relationship, except near the boiling point. PEO water-soluble resins are extremely effective thickening agents in both fresh and salt water. Aqueous solutions are pseudoplastic (i.e. shear thinning).

**Drag reduction/Drift control** - Very low concentrations of the higher molecular weight PEO water-soluble resins can reduce the turbulent frictional drag of the water in which they are dissolved by as much as 80 percent.

**Viscoelasticity** - The flexibility of ether linkages combined with the extremely high molecular weight of PEO water-soluble resins produces solutions with elastic behaviour.

**Glass transition temperature -** The glass transition temperature (Tg) of the family of poly (ethylene oxide) products ranges from -50 to -57ºC. Molecular weight does not have a significant impact on the (Tg) within the family of products.

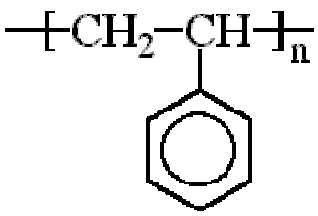
**Nontoxic and degradable -**PEO water-soluble resins are nontoxic and have received FDA approvals for a number of food and drug applications. Aqueous solutions of PEO resins are environmentally degradable due to oxidation and aerobic biodegradation.

## 1.6.2 APPLICATIONS

- Polyethylene oxides are used as a flocculating agent and coagulant in ore dressing processes, for sedimentation of dredges, paper pulp, and breeze. 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.
- PEO is used to decrease hydrodynamic resistance in industrial processes and is used in medicine for injections.
- It is also used as packing material for foods (water-soluble film).
- PEO acts as a binding agent and thickener for latex and paints.
- It is used as separator and also as electrolyte solvent in lithium polymer cells;
- Polyethylene oxide acts as polar stationary phase for gas chromatography.
- It is used in drug delivery system.
- In PEO copolymers of ethylene oxide and propylene oxide can be used as polyols in polyurethane systems, as block copolymers, and as functional fluids.

## 1.7 POLYSTYRENE

Polystyrene is also known as polyvinyl benzene. It has the following molecular structure:



Polystyrene is obtained by the polymerization of styrene solution in ethyl benzene in presence of benzoyl peroxide by solution polymerization method. Polymerisation of styrene had been accidentally observed as early as 1839. Styrene is produced from ethylene and benzene. When ethylene is passed into benzene in the presence of aluminium trichloride catalyst, ethyl benzene is produced, which on passing over a catalyst such as iron oxide or magnesim oxide at high temperatures gets converted into styrene and hydrogen. The reaction proceeds at a good rate at 600ºC.

The styrene thus obtained is further purified by distillation. Styrene can be polymerised by radical co-ordination cationic or anionic method. It is industrially produced by free - radical polymerisation, using the suspension or bulk technique. Commercially available polystyrene is usually amorphous in nature. Polystyrene generally consists of linear molecules and is chemically inert. Acids, Alkalis, Oxidising or reducing agents have little effect on it.

Copolymers of styrene with acrylonitrile, vinyl carbazole or diphenyl acrylamide are a few important ones. These co-monomer increases the heat and impact resistance of the polymer. Styrene-acrylonitrile (SAN) copolymer is a transparent plastic with very good impact strength and is used for moulding crockery items and machine components. Acrylonitrile-butadiene styrene (ABS) terpolymer is another commercially important plastic which possesses good strength and toughness. It is most suited for injection – moulded articles of household items and packaging containers. **[V.R. Gowariker]**

## 1.7.1 PROPERTIES

- Polystyrene is hard, clear plastic having good chemical resistance, good dimensional stability and moderate tensile strength.
- It is a good electrical insulator and shows no dielectric loss of mode rate frequencies. [**Dinesh Sharma]**
- Polystyrene is a transparent, highly stable and moisture resistant polymer.
- It is not affected by acids or other chemicals. It has low softening range (90-100ºC) and is brittle.
- Molecular weight of repeat unit is 104.1g/mol and it has a glass transition temperature of 100°C.
- Melting point of polystyrene is 240°C and its density is 1.047g/$cm^3$.
- Polystyrene softens slightly above 100°C and becomes a viscous fluid at temperatures around 185°C.
- Polystyrene is highly transparent and is able to transmit about 90% of visible light.
- Due to low cost and good modulability together with its transparency and colourability, this material is having widespread applications.
- Polystyrene can also be fabricated into a wide variety of extruded shapes, including films, monofilaments and oriented sheets [**Arora et al]**.

## 1.7.2 APPLICATIONS

- Polystyrene is used to make insulation and packaging materials as well as food containers such as beverage cups, egg cartons, and disposable plates and trays.
- Solid polystyrene products include injection-moulded eating utensils, videocassettes and audiocassettes, and cases for audiocassettes and compact discs.
- Many fresh foods are packed in clear vacuum-formed polystyrene trays, owing to the high gas permeability and good water-vapour transmission of the material.
- The clear windows in many postage envelopes are made of polystyrene film.
- The plastic recycling code number of polystyrene is #6. Recycled polystyrene products are commonly melted down and reused in foamed insulation.

- Polystyrene is widely used in the manufacture of articles such as moulded containers, lids, jars, bottles, radio and television cabinets, toys, formed plastics and many other household items **[V.R. Gowariker]**
- It is also used to make buttons, combs, refrigerator parts and in the manufacture of high frequency electronic insulators and lenses **[V.K.Ahluwalia].**
- Polystyrene has a fair light stability for all uses except those involving exposure to sunshine or ultraviolet light for a long time.

## 1.8 OBJECTIVES OF THE STUDY

The aim of the present work is

- To prepare PEO/PS thin film by Chemical bath deposition technique on Glass substrate.
- To measure the thickness of prepared film.
- To analyze structural and optical properties of the coated thin film using UV-Visible, PL and FTIR characterization methods.

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# *REVIEW OF LITERATURE*

# CHAPTER 2

# REVIEW OF LITERATURE

## 2.1 INTRODUCTION

Literature review refers to any collection of materials on a topic and it discusses published information in a particular subject area. 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. Several authors have studied the different properties of polymeric substances using various deposition and analytical techniques. This chapter discusses about studies on various polymers.

## 2.2 REVIEWS ON VARIOUS PROPERTIES OF POLYMER THIN FILM

**1. Gabriele Seguini et al., (2014)** assessed the phase behaviour in thin films of an asymmetric Polystyrene-*b*-Poly (methyl methacrylate) (PS-*b*-PMMA) block copolymer with a molecular weight of 39 kg $mol^{-1}$ at a wide range of temperature and time. Cylindrical PMMA structures featuring a diameter close to 10 nm and perpendicularly oriented with respect to the substrate were obtained at 180°C in relatively short annealing times ($t \leq 30$ min) by means of a simple thermal treatment performed in a standard rapid thermal processing machine.

**2. Amit Saxena et al., (2013)** studied the modification of the structural, optical and electrical properties of a polymer electrolyte film of polyethylene oxide complexed with sodium iodide (PEO: NaI) by doping with Si particles. Before forming the composite, the weight amount of polymer (PEO) and salt (NaI) are taken and films are prepared using a standard solution cast technique. Structural studies were carried out using Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) measurements. The surface morphology of the polymer electrolyte film doped with and without Si particles was studied using polarized optical microscopy (POM) under a cross polarizer. The interaction between the polymer and the semiconductor were confirmed from the Fourier-

transform infrared (FTIR) spectra, recorded in the attenuated total reflectance (ATR) mode using a Bruker Tensor 27 spectrometer.

**3. Roshani N. Bhagat et al., (2013)** prepared Poly (ethylene oxide) (PEO) complexed with Cadmium sulfide (Cds) by a solution cast technique. Several experimental techniques such as XRD, SEM, FTIR, DSC, PL and Raman spectroscopy are employed to characterize the sample. The powder X-ray diffraction (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. From UV spectra, it is found that the transmittance decreases with increase in filler concentration. As the filler concentration increases, band energy value decrease. In DSC, first peak of all the samples give the melting point of PEO and the second peaks are due to impurity of urea in Cds. The asymmetry in the Raman line shape of Raman spectra indicates the effect of phonon confinement.

**4. Dae Up Ahn et al., (2012)** systematically compared the morphological evolution of thin PS/PMMA films, with varying compositions. On native silicon oxide surfaces, the phase evolution in the films was dictated by the preferential substrate-wetting of PMMA. However, the resulting PS relief structures on the PMMA wetting layer varied with the blend composition, and transition from capillary-fluctuation-mediated breakup to random nucleation with increased PS concentration. In contrast, the morphological evolution of the PS/PMMA films on non-preferential surfaces was also dictated by the coarsening of PMMA domain, but preceded without the formation of a PMMA wetting layer. Both the PS and PMMA domains maintain direct contact with both the substrate and free surfaces throughout the evolution of the morphology.

**5. Julien Gaume et al., (2012)** described a novel 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 three polymers with different oxygen barrier (poly (methyl methacrylate) (PMMA), Polyethyleneterephtalate (PET) and Polyvinyl alcohol (PVA)), and the Oxygen permeability coefficients were obtained. The result was in good agreement with the literature also.

**6. Jing Wang et al., (2011)** synthesized Polystyrene-functionalized $C_{60}$ ($C_{60}$-PS) by atom-transfer radical polymerization. The structure of the hybrid was characterized by gel permeation chromatography and thermal gravimetric analysis. The self-assembly of Polystyrene-block-Poly (ethylene oxide) (PS-PEO)/$C_{60}$-PS film in annealing solvents was studied on a silicon wafer and at the air/water interface by transmission electron microscopy. The method is an easy route to produce arrays of ordered nanostructures. The addition of $C_{60}$-PS has a great effect on the self-assembly of PS-PEO. Treating the film under solvent vapor can modulate the orientation and ordering of PS-PEO micro domains. The $C_{60}$-PS enhanced the formation of lamellae microstructure, and the $C_{60}$-PS entered the PS phase, expanding the scale of PS domains. Nevertheless, it becomes more complex when it refers to the self-assembly at the air/water interface under solvent vapour for a long time. The selectivity of solvent to the polymer chains plays an important role as the annealing time increases.

**7. Xianke Gu et al., (2011)** used the scanning force microscope (SFM) to investigate morphology of poly (ethylene oxide) (PEO) and poly (acrylic acid) (PAA) blend. The morphology of the ultrathin films of the PEO and PEO–PAA blends spun on the silicon substrates was investigated. The crystallization of ultrathin PEO film exhibited smooth morphology using chloroform as a solvent, but recovered to crystallization image after dewetting. Crystallization of ultrathin PEO–PAA blend film disappeared in the presence of higher amount of PAA (>60%). However, the PEO film exhibited small hole in thin film and banded structure in thick film, when the amount of PAA was lower than 43%, PAA/PEO blend were fabricated via. Alternating spin-casting of PEO and PAA layers from aqueous solutions. All surfaces demonstrated PEO/PAA blends rather than a heterogeneous mixture.

**8. Ping Yang et al., (2010)** investigated the effect of solvent selectivity on the transition between crystallization and micro phase separation of the semi crystalline diblock copolymer polystyrene-b-poly (ethylene oxide) (PS-b-PEO) thin films. Square-shaped crystals formed due to lower barrier of crystalline nucleation in both poor and good solvent vapour for PEO. However, in poor solvent (cyclohexane) vapor for PEO, crystalline structure changed to micro phase separated structure in the square platelets due to the high mobility of PS blocks. While, in good solvent (water) vapour for PEO, competition between nucleation and dissolution of crystallization caused the formation of

imperfect crystals. Then imperfect crystals dissolved due to the high mobility of PEO blocks, and micro phase separation dominated the morphology of the film. The gain of far volume of soluble block and the low swelling of crystalline block are keys for micro phase separation and crystallization, respectively. X-ray photoelectron spectroscopy (XPS) spectra were measured with ESCALAB 250 (Thermo electron Co., U. K.) at room temperature by using an Al Kα X-ray source. The surface morphology of films was characterized by atomic force microscopy (AFM) and transmission electron microscopy (TEM).

**9. Marie F. Delcroix et al., (2010)** studied the water-induced surface reorganization of a thin film of a block copolymer [Polystyrene-b-Poly (ethylene oxide), PS-PEO], by cryogenic X-ray photoelectron spectroscopy (cryo-XPS). Experimental parameters were examined with a view to optimize the analysis. The absence of artifacts due to the low temperature analysis was checked, and the influence of the procedure used for sample hydration before analysis was investigated. Adequate timing of the different steps of the analysis and temperature program was also established. With this optimized protocol, an important reorganization of the block copolymer was detected, showing more pronounced exposure of the PEO block at the outermost surface in hydrated compared to dry environment. As this type of polymer surface is prone to be used for biomedical applications, an accurate knowledge of the chemical composition of the outermost surface in aqueous environment is crucial.

**10. Chunyan luo et al., (2010)** studied the solvent-induced crystallization and dewetting behaviours of Polystyrene-b-Poly (ethylene oxide)-b-Polystyrene (PS-b-PEO-b-PS) block copolymer films deposited on three different substrates, silicon, mica, and carbon-coated mica by atomic force microscope (AFM). When the common solvent dichloromethane was used for annealing, the films on all three of the substrates exhibited dewetting behavior; the dendritic crystallization patterns were found in the dewetted regions for the films on silicon and mica substrates, while for the films on carbon-coated mica, no crystallization pattern appeared. According to the observation of the crystallization patterns formed in the films with different initial thicknesses, it is shown that the width of the dendritic branches decreases with the increase of film thickness and solvent annealing time. When the PS-selective solvent toluene was used for annealing, the dewetting

process was absent, and the crystallization patterns were observed on the surface of the films on all the three kinds of substrates.

**11. Hsiang Lin et al., (2010)** have generated the submicron Polystyrene (PS)/Poly (methyl methacrylate) (PMMA) blends by the precipitation with a compressed antisolvent (PCA) technique. The generation of PS/PMMA blends was carried out by spraying a solution containing PS and PMMA into a precipitator. The blends without coalescence were observed to only be generated when both vapour and liquid $CO_2$ existed in the precipitator combined with appropriate total polymer concentration in solution, molecular weights (Mw) of PS and PMMA, mass ratio of PS to PMMA, flow rates of $CO_2$ and polymer solution, and liquid $CO_2$ level in the precipitator. Toluene with a solubility parameter smaller than that of tetrahydrofuran (THF) was found to be the more appropriate solvent for generating spherical PS/PMMA submicron blends. Individual and spherical PS and PMMA particles or spherical PS particles partially covered by PMMA films, however, were generated when the liquid $CO_2$ level was of 1/8 or lower in the precipitator.

**12. Nathalie Lefvre et al., (2010)** reported the self-assembling behavior in thin films of mixtures of Polystyrene-block-Poly (ethylene oxide) copolymers (PS-b-PEO), having PEO cylindrical micro domains, with poly (acrylic acid) homopolymers (PAA). The effect of adding PAA of different molecular weights, specifically interacting with the PEO block by hydrogen bonding, on the thin film characteristics such as morphology, micro domain orientation and spacing were studied. It is found that the addition of PAA induces an orientation of the cylindrical micro domains perpendicularly to the film surface. The lattice spacing increases with the amount of PAA added until a transition toward lamellar morphology is observed. This transition occurs at lower PAA content for PAA of small molecular weight. The experiments also reveal that the PAA homopolymer is localized in the center of the PEO micro domains. The trends observed in the experiments were validated by self-consistent field theory calculations using a new and specifically developed model.

**13. Wen Ping Hsu., (2010)** predicted by the mean field theory that PMMA is immiscible with poly (styrene-b-ethylene oxide) (PS-b-PEO) in the bulk state. The miscibility of PMMA with PS-b-PEO may be different in the two-dimensional state. The mixed monolayer behaviour of stereo regular (including isotactic, atactic and syndiotactic)

PMMA and PS-b-PEO was investigated on the basis of the measurement of surface pressure-area per molecule ($\pi$-$A$) isotherms at three different temperatures. The miscibility and nonideality of the mixed monolayer were examined by calculating the excess area as a function of composition. Mostly negative deviations from ideality were observed in the mixed monolayer at 25 °C and 32.5 °C. This is likely because of favorable interaction between PMMA and PEO. However, positive deviations occurred at 32.5 °C and 40 °C with atactic PMMA (or syndiotactic PMMA) mixed monolayer. With confinement in the two-dimensional state, the miscibility between PMMA and PS-b-PEO was greatly improved in comparison with the bulk state.

**14. Chiara Neto et al., (2009)** investigated the topography and surface composition of thin films of a polystyrene-b-poly- (ethylene oxide) (PS-PEO) block copolymer using a suite of complementary techniques, namely tapping mode atomic force microscopy (AFM), optical microscopy, X-ray photoelectron spectroscopy (XPS), neutron reflectometry, and wettability measurements. The copolymer films separate into lamellar structures oriented parallel to the silicon substrate, and bicontinuous and island/hole morphologies characteristic of this arrangement appear. Even though the crystalline topography of the film's surface and its wettability properties suggest the presence of PEO on the top surface, XPS and neutron reflectometry data point undoubtedly to the presence of a top layer of PS at the air/film interface. Tapping mode AFM images unequivocally demonstrate that in air only one block is present at the air/film interface. Neutron reflectometry data identify the nature of each phase-separated layer within the film. After many hours of thermal annealing, both PS and PEO blocks can be made to appear at the film/air interface, within isolated droplets formed upon film dewetting.

**15. Jennifer Tata et al., (2009)** deposited Polystyrene-block-Poly (ethylene oxide) PS-b-PEO by spin coating method. In their study, atomic force microscopy (AFM),X-ray diffraction (XRD),Transmission electron microscopy(TEM), and Differential scanning calorimetry (DSC) was used for analysis. XRD measurements of polymer films were carried out with a conventional rotating anode source. FTIR was used to investigate the hydrogen bond interactions between block co-polymer and resorcinol. Thermal analysis was carried out by DCS. PEO resorcinol phase self-segregation in cylindrical domains with relatively good lateral order and perpendicular orientation were highlighted by TEM.

**16. Kamlesh Pandey et al., (2008)** prepared the solid polymer blend films based on polyethylene oxide and polyvinyl acetate (PVAC) in various concentrations by solution cast technique. XRD studies were used to analyse the structure of the materials. The study indicates that PEO can be effectively blended with PVAC to enhance its conductivity. The study of optical absorption gives information about band structure of solids. It was found that the energy gap and band edge values shifted to lower energies on blending with PVAC.

**17. Hans-Georg Braun and Evelyn Meyer., (2008)** have analyzed and found that the film formation of ultrathin polymers on micro heterogeneous surfaces is strongly influenced by molecular surface patterns which cause local wettability differences for liquid phases in contact with the surface. Surface coverage with polymers transferred by dip-coating from polymer solutions was controlled by surface heterogeneities prepared by soft lithography or by electron beam lithography of self-assembled monolayer. Crystallisable polymers were used for the formation of Poly (ethylene oxide) (PEO) ultrathin film. The competition in pattern formation resulting from the dewetting process and patterns which result from lamella crystallization of PEO in ultrathin films were investigated. Heterogeneous nucleation in these metastable films can be done by external stresses such as contact with an AFM tip. The nucleation on demand enabled the study of the diffusion controlled pattern formation that was observed during lamella crystallization and the growth process resulting in different morphological features that could be studied at elevated temperatures and in lateral confined areas which were realized by the film preparation on the microheterogenized surfaces.

**18. A.A.Khaydarov et al., (2007)** have prepared asymmetric Poly (styrene-*b*-methyl methacrylate) (PS-*b*-PMMA) diblock copolymers. Atomic force microscopy (AFM) was used to investigate the surface structure of thin films, prepared by spin-coating on a silicon substrate. It was shown that the nanostructure of depends on the molecular weight and volume fraction of the diblock copolymers. It was observed that a perpendicular lamellar structure exists for the high molar mass sample whereas hexagonal-packed cylindrical patterning for the lower molar mass. Small-angle X-ray scattering investigation of these samples without annealing did not reveal any ordered structure. Annealing of PS-*b*-PMMA samples at 160°C for 24 hour led to a change in surface structure.

**19. Juan Peng et al., (2007)** have systematically studied the thin film morphologies of asymmetric polystyrene-block-poly (ethylene oxide) (PS-b-PEO) diblock copolymer subjected to solvent vapours of varying selectivity for the constituent blocks. Upon a short treatment in neutral or PS-selective vapour, the film exhibited a highly ordered array of hexagonally packed, cylindrical micro domains. In the case of PEO selective vapour annealing, such ordered cylindrical micro domains were not obtained. Instead, fractal patterns on the micro scale were observed and their growth processes were investigated. Furthermore, hierarchical structures could be obtained if the fractal pattern was exposed to neutral or PS selective vapour.

**20. 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 ethyl benzene, 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 ethyl benzene 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 also discussed in detail.

**21**. **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. The formation of the blend morphology could be explained by the difference in the solubility of the two polymers in the solvent and the dewetting of PS-rich domains from the PMMA-rich phase. 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.

**22. Xue Li et al., (2004)** have investigated the surface morphology evolution of three thin Polystyrene (PS)/Poly (methyl methacrylate) (PMMA) blend films on silicon substrates. 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 ultrathin PS/PMMA blend film (16.3 nm), the discrete domains of the PS-rich phases upon the PMMA-rich phase layer were formed and the secondary phase separation occurred after a longer annealing time.

**23. Ton-That et al., (2001)** have examined the films of Polystyrene (PS) and Poly (methyl methacrylate) (PMMA) blends of two different thicknesses by X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM). Blends with different compositions were spin-cast onto a mica substrate with chloroform as the mutual solvent. XPS measurements revealed surface enrichment of PMMA in all compositions. The thicker (66 nm) films exhibit a higher degree of PMMA surface enrichment than the thinner (17 nm) films. AFM imaging allows distinctions to be drawn between blends with differing compositions. The blend films with less than 50% PMMA bulk concentration generally exhibit pitted surfaces; the pit size varies with film thickness and bulk composition. When the PMMA bulk concentration is greater than 50%, the film surface changes to show island-like phase-separated structure.

**24. Keiji Tanaka et al., (1996)** prepared PS/PMMA blend thin and ultrathin films by spin coating method at 293K.PS/PMMA the blend thick films were also prepared by a conventional solvent cast method. The surface phase state for the two dimensional PS/PMMA ultrathin films can be explained by the film thickness dependence of both the Flory- Huggins interaction parameter and the degree of entanglement along polymer chains. The surface morphology of the PS/PMMA blend films was investigated the on basis of AFM observation carried out in a repulsive force range. The chemical composition of PS/PMMA was evaluated on the basis of X-ray Photo electron Microscopy (XPS).

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# MATERIALS AND METHODS

# CHAPTER 3

# MATERIALS AND METHODS

## 3.1 INTRODUCTION

Thin films can be prepared from a variety of materials such as metals, semiconductors, insulators or dielectrics, polymers, etc, and for this purpose various preparative techniques have also been developed. Newer methods are also being evolved to improve the quality of the deposits with maximum reproducible properties and minimum variation in their compositions. Almost all film depositions are made on some solid substrates such as glass, quartz, ceramics, mica, minerals, metals, alloys, semiconductors, insulators, organic materials such as cellulose, plastics, polymers, rubbers etc. In this study glass was used substrate.

## 3.2 CHOICE OF SUBSTRATE

The substrate plays a major role in the nucleation and growth process. In addition, substrate surface also serves as the vehicle for imparting interfacial, structural, mechanical and physical properties of the film **[Milton ohring].** The microstructure of thin films strongly depend on the kind of substrate i.e. roughness, crystalline structure and the chemical state of the surface. Ideally, the surface should be extremely flat, smooth and free from crystalline defects. It should also be free of chemical impurities. The substrate must be stable to all types of solvent and to reagents, also to high temperatures. Mechanical strength and the possibility of repeated use are also desirable, combined with low cost. 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 film except for sufficient adhesion.

In practice, however substrates exert considerable influence on the thin film characteristics. There are a number of materials like glass, ceramics and quartz available for use as thin film substrates. Glass plates fulfil these varied requirements best; they can also rapidly coat by chemical bath deposition, and hence is preferred in this work. [**Maissel and Glang]**

The primary driving factor in the selection of a specific material as a substrate for a thin film coating is the wavelength region over which it must function. Transparency must be the major concern, as coatings can modify only the surface-related characteristics of the material, such as the reflectance; they cannot alter the internal transmittance or absorptance. The type of substrate upon which a film is deposited generally determines many of the characteristics of the film, such as durability, stability and cost. **[James. D.Rancourt].** There are three major categories of substrate:

- Glasses and similar crystalline materials.
- Organic materials and plastics.
- Metals.

Surface coating of glass with different types of film is one of the technologies that occupy a key position in product development **[Bach. Krause].**Glass substrate is used in this work**,** as they are economical and easy to coat thin films.

## 3.3 SUBSTRATE CLEANING

The process of substrate cleaning requires that the bonds are broken between contaminant molecules as well as between the contaminant and the substrate. This may be accomplished by chemical means or by supplying sufficient energy to vapourize the impurity by heating. The cleaning process followed is given below.

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. **[James. D.Rancourt]**

Initially all the glass wares were washed with ordinary distilled water. Then they were rinsed with Extran and washed completely with distilled water. The blue star glass plates of dimension (75mmx25mmx1.35mm) were washed with distilled water and immersed in $HNO_3$ for half an hour and washed with distilled water. The substrates were dipped in HCL for the same period. After that the slides were rinsed using soap solution

and distilled water. Finally the substrates were allowed to dry in a hot air oven for about an hour at a temperature of 60°C. Thus the substrates required for film coating in this study have been prepared.

## 3.4 EXPERIMENTAL DETAILS

### 3.4.1 CHEMICALS AND GLASSWARES USED

Polystyrene (99% purity), Poly (ethylene oxide) (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 Rankleen solution were used to clean the microscopic glass slides. Blue star glass slides of dimension 75mm×25mm×1.35mm was used for film coating. Beakers, conical flasks and measuring jar are other glass wares used.

### 3.4.2 EXPERIMENTAL PROCEDURE FOR PREPARATION OF POLYMER THIN FILM

Thin films of pristine poly (ethylene oxide) (PEO) and poly (ethylene oxide) (PEO)/ polystyrene (PS) blends were deposited on highly clean glass substrates. Required amount of PS and PEO was weighed using digital balance. The solution was prepared in conical flasks by dissolving known quantities of PEO and PS in Toluene solution at room temperature to yield different molar solutions. Then the solution was stirred at 40ºC to ensure homogeneous mixing. The solution was filtered by using 0.5µm Whatman filter paper to remove any undissolved impurities and dust particles. Thin films were prepared using constant temperature water bath with digital proportional temperature controller. Similarly, a homogeneous solution of pristine PEO is prepared. The glass slides were dipped in a beaker containing PEO and PEO/PS solutions kept in water bath. The rate of growth and thickness of the film depend on the nature of the substrate, the concentration of the solution, and the temperature maintained. Different molar solutions have been used to deposit polymer film, but only the following best coated films characteristics have been analysed. After the film deposition, coated substrates were taken out and put into hot air oven at 50ºC for 2 hour to remove all the traces of solvent.

**TABLE 3.1**

**THICKNESS OF PEO/PS BLEND AND PRISTINE PEO THIN FILMS**

| SAMPLE | MOLARITY | FILM THICKNESS (μm) |
|---|---|---|
| 1 | 1.5 mole of PEO + 1.5 mole of PS | 11.012 |
| 2 | 1 mole of PEO + 1.5 mole of PS | 17.23 |
| 3 | 1 mole of PEO + 2.5 mole of PS | 15.297 |
| 4 | 1.5 mole of PEO | 31.135 |

## 3.5 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 thickness with high accuracy. Microelectronic applications generally require the maintenance of precise and reproducible film metrology i.e., thickness as well as lateral dimensions. Even more stringent thickness requirements must be adhered to in multilayer optical coating applications **[Milton Ohing].** In this study gravimetric technique has been adopted to measure coated thin film thickness.

Gravimetric technique is one of the earliest and convenient techniques used for determining film thickness. It is a quantitative method that is based on the mass of a pure compound to which the analyte is chemically related. Since weight can be measured with greater accuracy than almost any other fundamental property, gravimetric analysis is

potentially one of the most accurate classes of analytical methods available. This method is one of the oldest analytical techniques, though they may be lengthy and tedious. In Gravimetric technique 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 is used for measuring the weight of the deposited film during this study.

The best technique for a specific application or process depends upon the film type, the thickness of the film, the accuracy desired, and the use of the film. These criteria include such properties as film thickness, film transparency, film hardness, thickness uniformity, substrate smoothness, substrate optical properties, and substrate size. Since thin film thicknesses are generally of the order of wavelength of light, various types of optical interference phenomena have been found to be most useful for the measurement of film thicknesses. There are optical techniques like ellipsometry and absorption spectroscopy which can be used to measure thickness. In addition to the optical techniques, there are mechanical, electrical, and magnetic techniques, which have been used for film-thickness measurements.

**PLATE 1**

**CHEMICAL BATH SETUP FOR THIN FILM COATING**

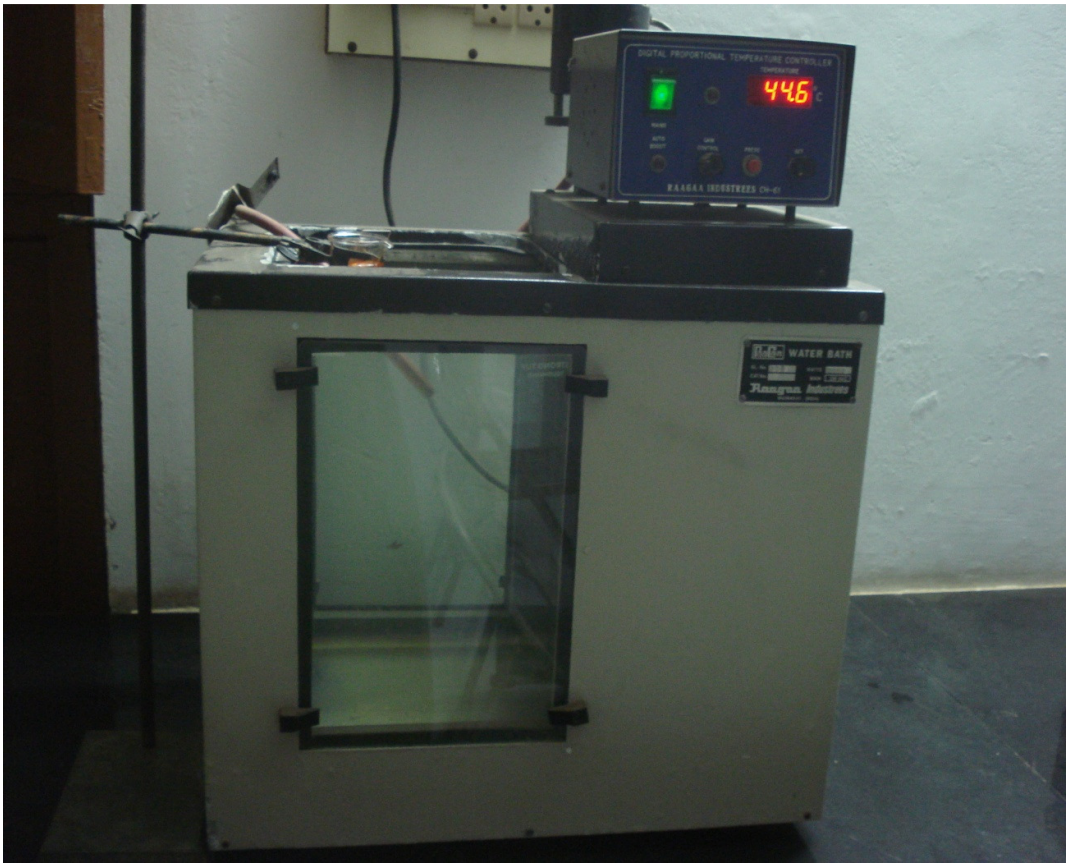

**PLATE 2**

**DIGITAL MICROBALANCE USED FOR THICKNESS MEASUREMENT**

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# RESULTS

# AND DISCUSSION

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 INTRODUCTION

Thin film characterization provides new challenges as they generally consist of a small amount of material, and thus pose problems from an analytical point of view. The measurements of thin film properties are indispensable for the study of thin film materials and devices **[Milton Ohing].** To make sure that coatings which were produced by a given process satisfy the specified technological demands a wide field 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. With the increasing interdisciplinary nature of application, demand for effective film characterization and property measurement in both individual films and multilayer coatings has recently been developed. There are a number of major considerations that determine the choice of an instrumental method to solve a specific problem in the surface analysis of thin films. The results obtained from UV-VIS spectrometry, Photoluminescence, and FTIR of the solution grown polymer thin film are discussed in this chapter.

## 4.2 ULTRAVIOLET AND VISIBLE SPECTROSCOPY

UV-Visible Spectroscopy is defined as the absorption and emission of radiation associated with changes in spatial distribution of electrons in atoms 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. **[H.K.Moudgil]**

The ultraviolet region corresponds to 200-400nm and visible region to 400 - 800nm. 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].**

Molecules containing π-electrons or non-bonding electrons (n-electrons) can absorb the energy in the form of ultraviolet and visible light to excite these electrons to higher antibonding molecular orbitals. The more easily excited the electrons, the longer the wavelength of the light it can absorb. It is mostly used for identifying conjugated systems which tend to have stronger absorptions. Absorbance is directly proportional to the path length of the absorbing species. This means that an absorption spectrum can show a number of absorption bands corresponding to structural groups within the molecule. **[Ghenadil Korotcenkov]**

## 4.2.1 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 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. 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].**

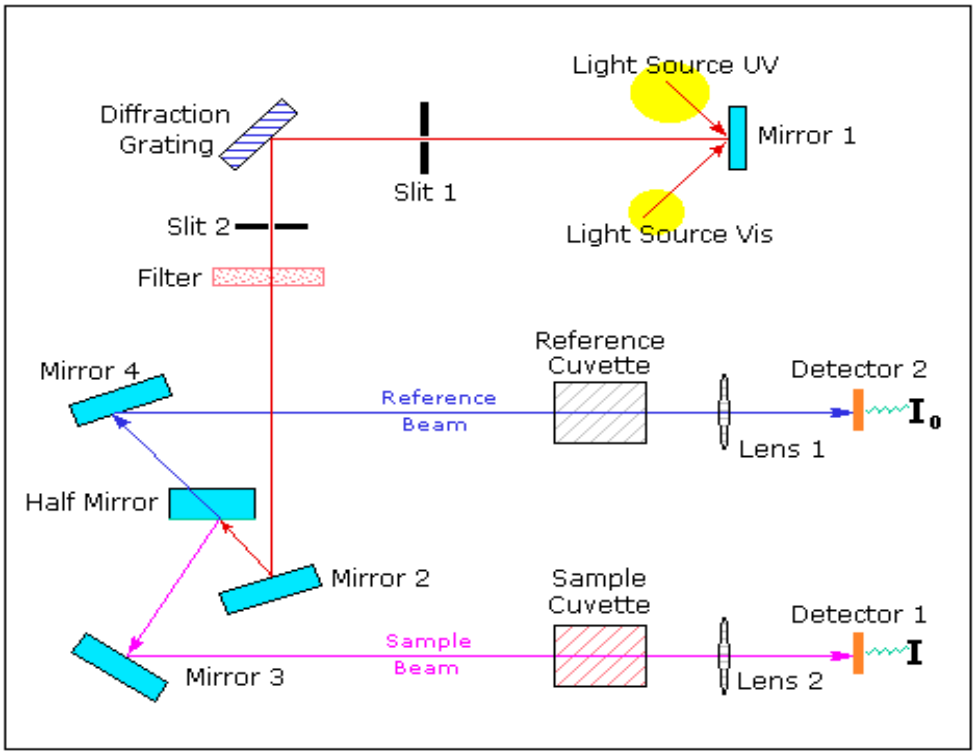



**Figure 4.1 Schematic diagram of UV-Visible Spectrophotometer**

## 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 (20-400$cm^{-1}$) as the sources are weak and the detectors insensitive. FTIR has made this energy region more accessible. It has made the middle infrared (400-4000$cm^{-1}$) also more useful **[G.Aruldhas].**

Fourier Transform Infra-Red (FTIR) spectroscopy is a tool for qualitative and quantitative analyses of the various chemical groups present in the material instead of recording the amount of energy absorbed when the frequency of the infrared (IR) light is varied, the light is guided through an interferometer. Molecular bonds vibrate at various frequencies depending on the elements and the type of bonds. For any given bond, there are several specific frequencies at which it can vibrate. According to quantum mechanics, these frequencies correspond to the ground state (lowest frequency) and several excited states (higher frequencies). One way to cause the frequency of a molecular vibration to increase is to excite the bond by having it absorb light energy. For any given transition between two states the light energy (determined by the wavelength) must exactly equal the difference in the energy between the two states [usually ground state ($E_0$) and the first excited state ($E_1$)].

Schematic representation of FTIR Spectrometer is shown in the figure 4.2. An FT-IR spectrometer works on the basis of Michelson Interferometer. The interferometer consists of a beam splitter, a fixed mirror, and a mirror that translates back and forth, very precisely. Radiation from the source strikes the beam splitter and separate into two beams. One beam is transmitted through the beam splitter to the fixed mirror and the second is reflected off the beam splitter to the moving mirror. The fixed and moving mirrors reflect the radiation back to the beam splitter. Again, half of this reflected radiation is transmitted and reflected at the beam splitter, resulting in one beam passing to the detector and the second back to the source.

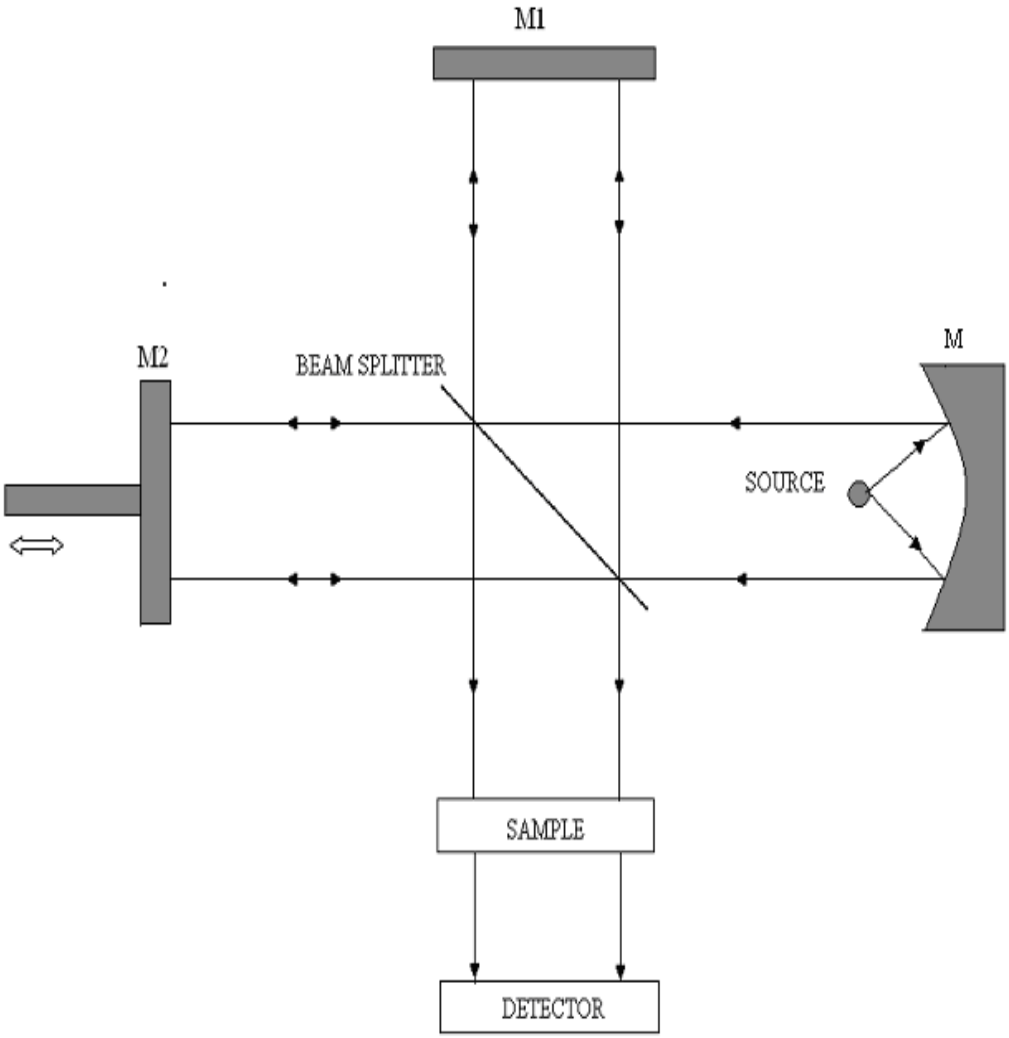



**Figure 4.2 Schematic diagram of FT-IR Spectrophotometer**

## 4.4 PHOTOLUMINESCENCE SPECTOSCOPY

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. Photoluminescence spectroscopy is a versatile technique enabling the study of light dynamics in matter, and it is an important approach for exploring the optical interactions in semiconductors and optical devices with the goal of gaining insight into material properties **[Leah Bergman et al].** This is one of many forms of luminescence (light emission) and is distinguished by photoexcitation (excitation by photons). Photoluminescence spectroscopy can be defined as the radiation emitted from a molecule or a solid which is excited by absorbing photons (Photon-excitation). [**Michel Che et al].**

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.

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 diagram of photoluminescence spectrometer 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. The efficiency of Photoluminescence depends on the lifetimes in the excited state with respect to the radiative and non-radiative relaxation mechanisms **[M.Balkanski et al].**

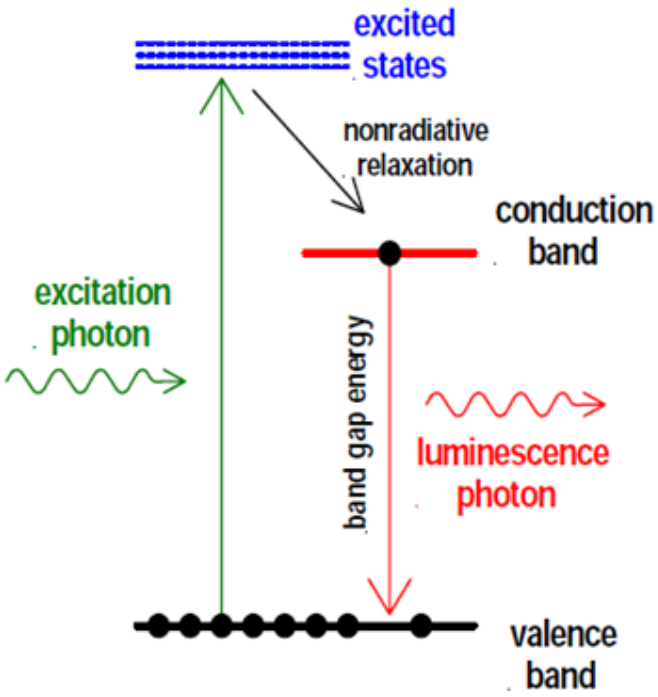



**Figure.4.3 Basic principle of photoluminescence**

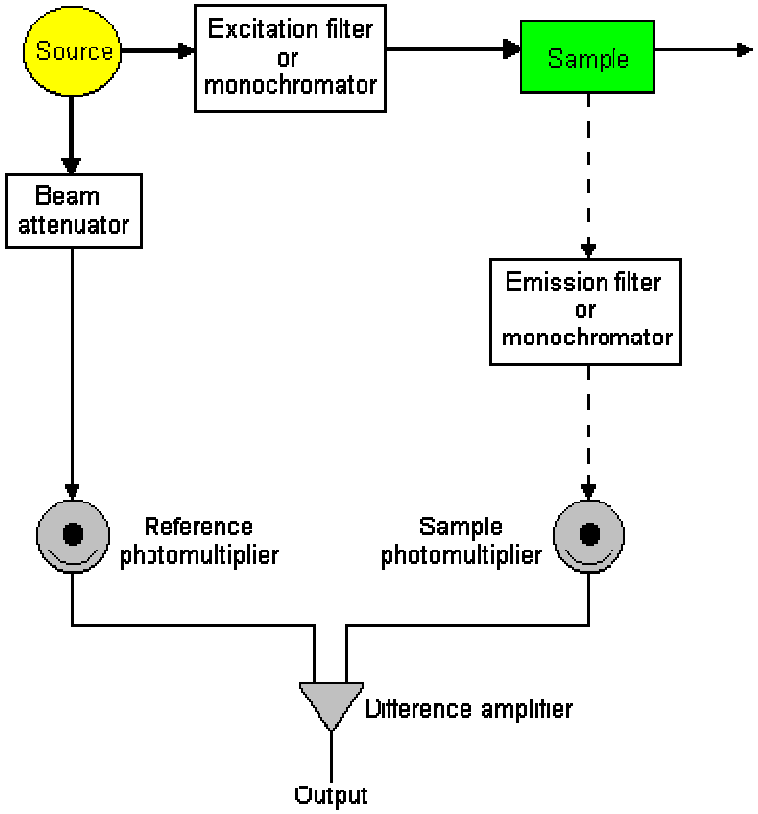



**Figure.4.4 Schematic diagram of Photoluminescence spectrometer**

## 4.5 DISCUSSION OF RESULTS OBTAINED

### 4.5.1 RESULTS OBTAINED FROM UV-VISIBLE SPECTROSCOPY

In UV-Visible spectrum, absorbance or transmittances are plotted against wavelength. The optical properties of the compound can be determined from the corresponding peak shape and wavelength. All the UV-Visible spectrum of polymer thin films is recorded by using UV-Visible-NIR (Jasco V-670 spectrophotometer). UV-Visible radiation is illuminated on the entire area of PEO/ PS thin films. The UV-Visible spectrum of pristine PEO film (sample 4) is shown in the fig 4.5. It is noted that the cut off appears at 294nm.The transparency window is present for a range of wavelength from 380nm to 1180nm.

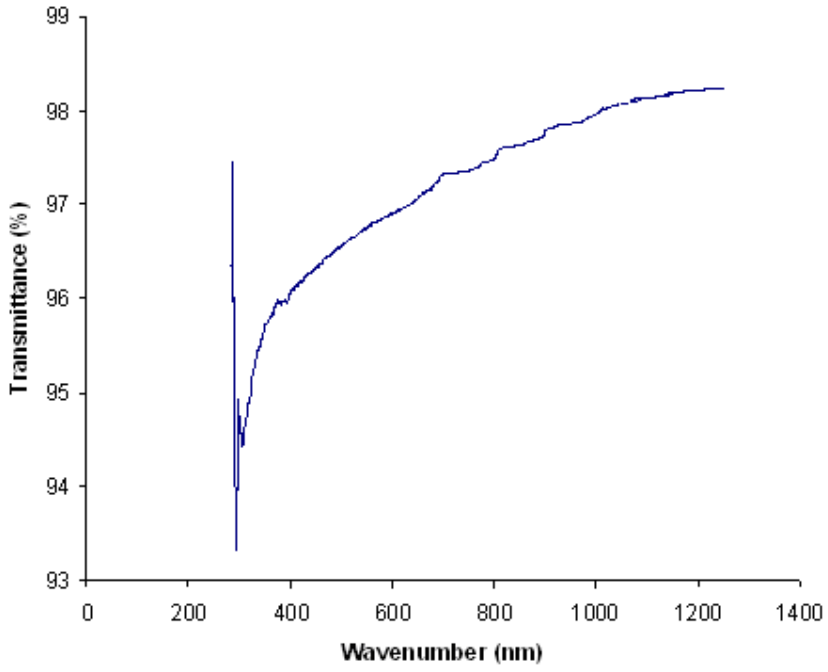



**Figure 4.5 UV–visible spectrum of (pristine PEO) sample 4**

The UV-Vis Spectrum of blend PEO/PS film of thickness 17.23 µm (sample 2) is shown in figure 4.6. It shows a cut off wavelength at 324nm and exhibits transparency from 650 nm to 1490 nm. The % of the transmittance is above 98 % from 939.5 nm to 1490 nm, which may be due to the transparent nature of polystyrene. Hence, the increase in transparency of blend film can be useful for Optical applications.

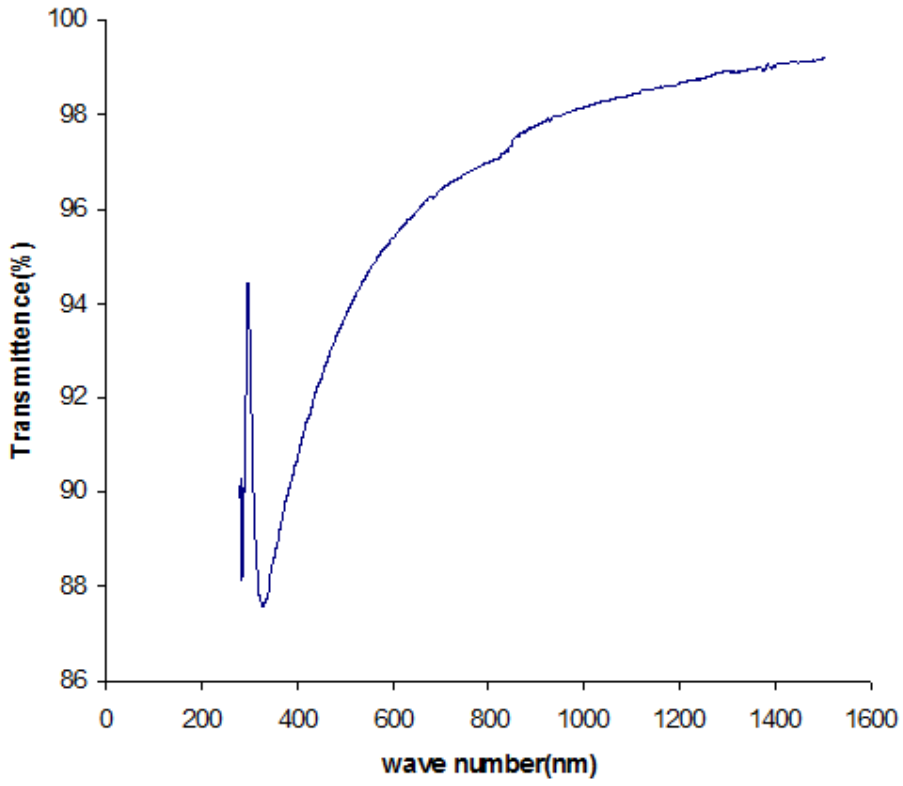



**Figure 4.6 UV-visible spectrum of (PEO/PS) sample 2**

## 4.5.2 RESULTS OBTAINED FROM FOURIER TRANSFORM INFRARED SPECTROSCOPY

FTIR spectra was recorded using SHIMADZU, FTIR spectrometer. The spectra for Sample 4 (pristine PEO) is shown in the fig 4.7. The peak obtained in the spectra correspond to 626$cm^{-1}$ represents the C-H stretching. The peak observed at 1028$cm^{-1}$ represents the C-O-C stretching, which confirms the presence of pristine PEO.

The FTIR spectrum of chemically deposited poly (ethylene oxide)/polystyrene blend is shown in figure 4.8. The peak corresponds to 957$cm^{-1}$ is due to $CH_2$ rocking. A sharp well defined peak due to C-O-C stretching is present at 1102$cm^{-1}$**. [V.M.mohan et al]** Because of C-H stretching mode three prominent peaks are observed at 2885$cm^{-1}$, 2921$cm^{-1}$ and 3025$cm^{-1}$. Thus from FTIR results, the presence of monomers ethylene oxide and styrene is confirmed**. [K.Kaniappan et al]**

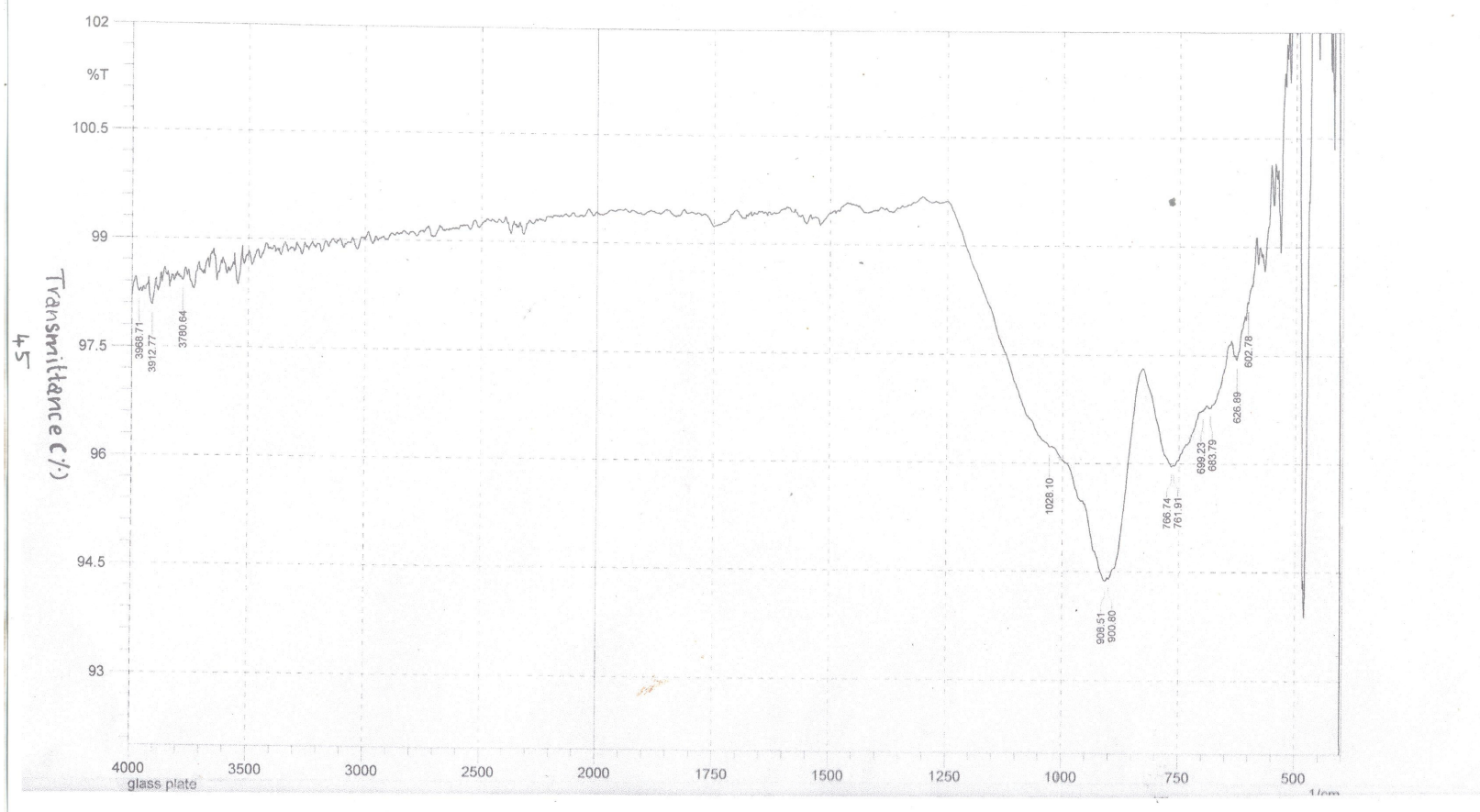
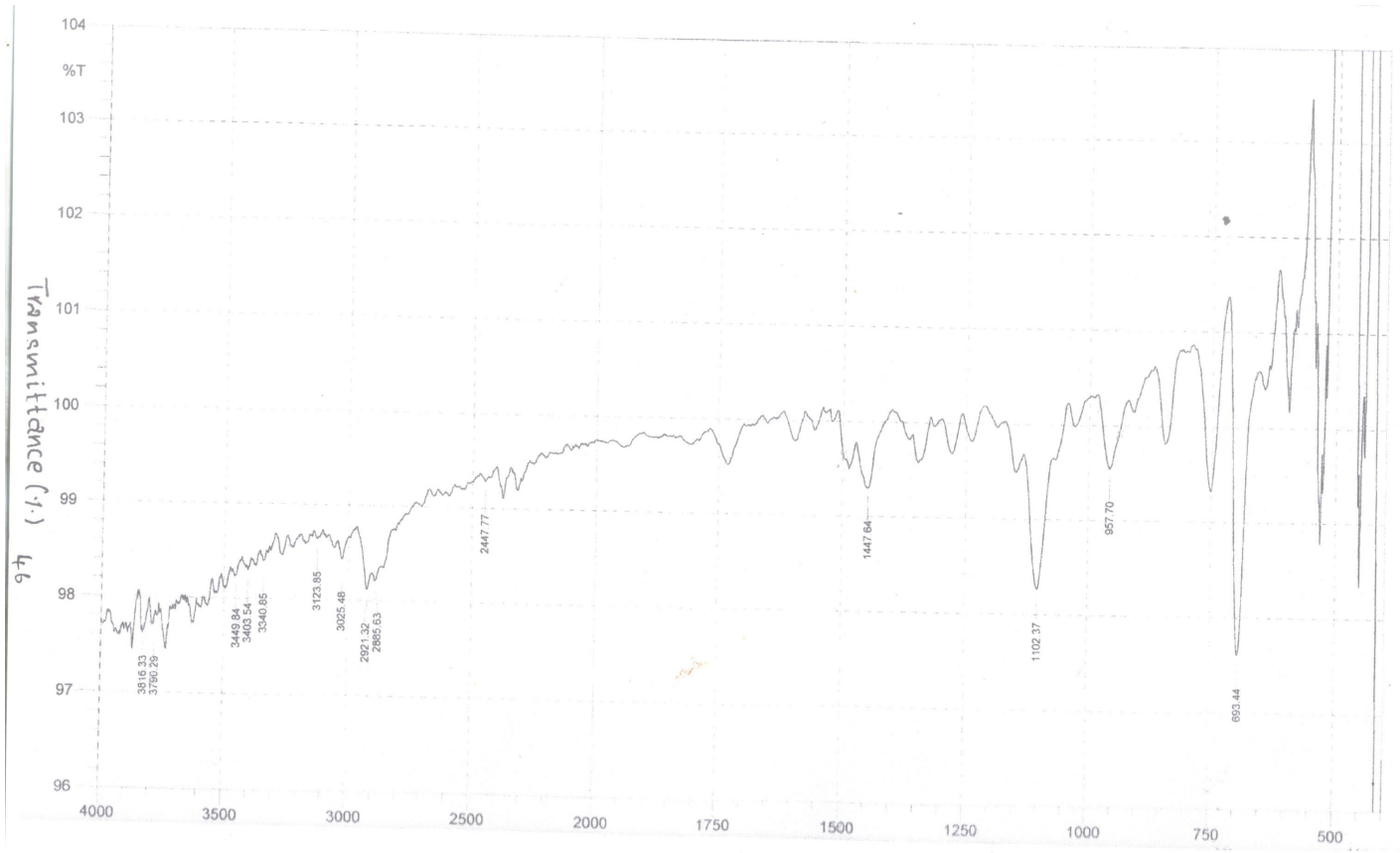



**Figure 4.7 FTIR spectrum of (pristine PEO) sample 4**




Wave number ($cm^{-1}$)

**Figure 4.8 FTIR spectrum of (PEO/PS) sample 2**

### 4.5.3 RESULTS OBTAINED FROM PHOTOLUMINESCENCE (PL) SPECTROSCOPY

All the Photo-luminescence spectrum of polymer thin films is recorded by using FLUOROLOG HORIBA (Jobin YVON) Photoluminescence spectrometer. The photoluminescence spectrum of the pristine PEO (sample 4) is shown in the figure 4.9. The graph is plotted as wavelength versus intensity. The bandwidth value of the spectra is found to be 81 nm. The spectrum attenuates at 532 nm.

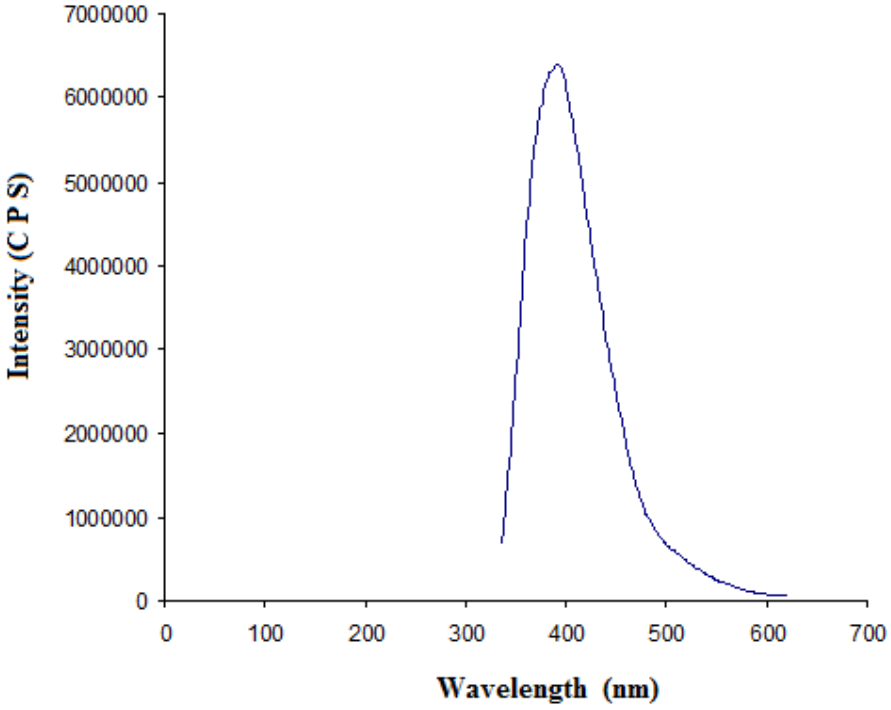



**Figure 4.9 Photoluminescence spectrum of (pristine PEO) sample 4**

The photoluminescence spectrum of the blend film PEO/PS (sample 2) is shown in the figure 4.10. The bandwidth value of the spectra is found to be 73 nm. The spectrum attenuates at 559 nm. A sharp peak is observed at 418nm which may be due to transition from conduction band to valence band **[B.Hari che et al].** The narrowing of bandwidth ensures the use of PEO/PS blend films as lenses, electronic insulators and filters for UV light

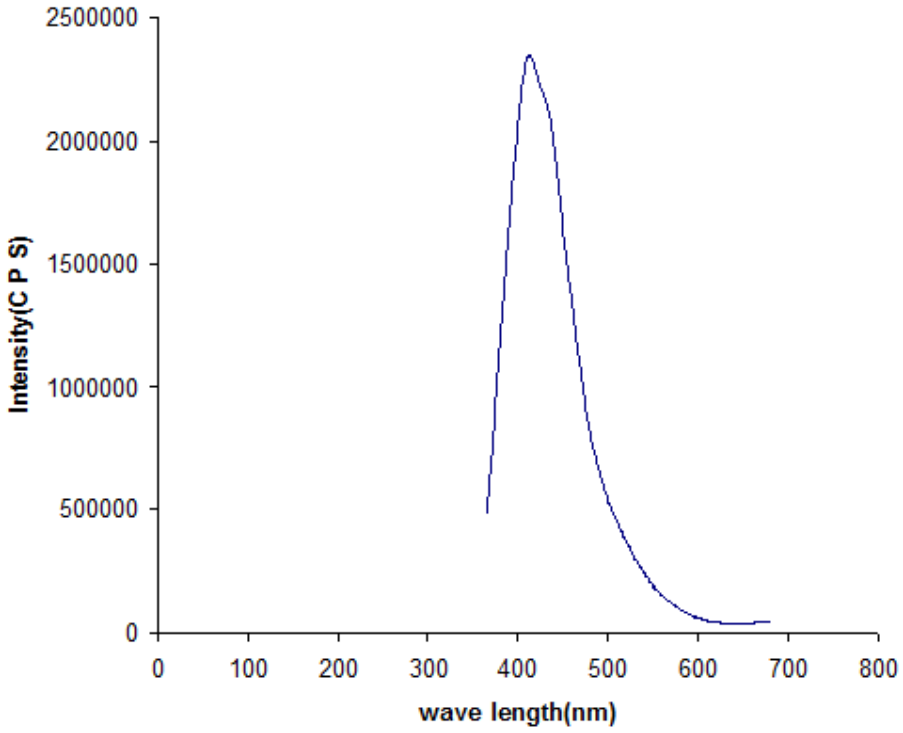



**Figure 4.10 Photoluminescence spectrum of (PEO/PS) sample 2**

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# SUMMARY AND CONCLUSION

# CHAPTER 5
# SUMMARY AND CONCLUSION

Polymer materials have been widely used in various fields such as industrial products, polymer optical fibers, optical waveguides and optical connectors due to their ease of processing, relatively low cost and mass production compared to silica based optical materials. Their universal presence in paints, coatings, packaging adhesives, dielectrics and other common entities has firmly established their versatility, integrity and dependability. The technical interest in the study of the properties of thin films has resulted in the invention of many novel devices.

The pristine Poly (ethylene oxide) (PEO) and Poly (ethylene oxide)/ Polystyrene (PEO/PS) blend thin films were successfully deposited by chemical bath deposition in this study. The thickness of the pristine PEO studied is 31.135μm and that of PEO/PS blend films lie in the range of 11 to 17.5 μm. The characterization techniques like UV-Visible Spectroscopy, Fourier Transform Infra-Red (FTIR) spectroscopy and photoluminescence (PL) Spectroscopy have been used to study the properties of the pristine PEO and PEO/PS blend films. In visible region the transmission of the PS/PEO films is found to be around 98%, because of transparent nature of polystyrene. The presences of functional groups of pristine PEO and PEO/PS blend films like C-H, C-O-C and CH2 in the films were confirmed, using Fourier Transform Infra-Red (FTIR) spectroscopy. The narrow bandwidth for PEO/PS blend films from PL spectrum validates its use as filters for UV light.