

X-Ray Diffraction and UV-Visible Studies of PMMA Thin Films

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ABSTRACT:

Thin films of Poly(methyl methacrylate) were deposited on glass micro slides using Chemical Bath Deposition Technique. The bath compositions include double distilled Toluene, double distilled Benzene which served as a complexing agent. The films were deposited at room temperature. The Structural characterizations of PMMA were carried out by XRD, which shows an amorphous nature of the material. The absorbance and transmittance of the films was measured using UV-Visible Spectroscopy which reveals that PMMA has higher transmission with very little absorption in the UV-Visible region. These films because of their high transmittance in the UV-Visible region are suitable for various optical applications, and also in non-linear optics.

Keywords: Amorphous, PMMA, UV-Visible XRD.

1.0 INTRODUCTION:

The interest in polymer material has grown continuously in the last two decades. Various methods for preparation of thin polymer films have emerged. Polymeric substances make up the most important class of organic materials, technically and economically. The familiar plastics, fibers, elastomers and biological materials that surround us attest to this importance. Such substances which are composed of great many identical groups or repeating units are known as (high polymers). Polymers composed of more than one kind of repeating units are termed copolymers. PMMA is widely used sometimes with plasticizer and or small amount of other ester as copolymers. The resins are so wide spread under the trade name of plexiglas in United States and Perspex in Europe that these words have become synonymous to transport plastic panels, windows and wind shields. The resistance of these resins to shattering and to weather has led to their large scale application in aircraft. PMMA has drawn tremendous interest due to its optical properties and its possible use in non-linear optics.

New techniques for polymer particles of controlled composition and size have been developed. Polymer thin films play an increasingly important role in technological application ranging from coatings, adhesives and lithography to organic light emitting diodes and various organic material based devices, including sensors. PMMA is one of the earliest Polymers. It is extensively used in glazing applications as a replacement for glass in various applications with good weather resistance. This polymer unlike ionic and metallic crystals and low molecular weight substances consists of long chain molecules arranged in aggregates, which assume complex shapes and structures [1]. The organic films based on poly-methyl-methacrylate (PMMA) polymer have been obtained by pulsed laser deposition (PLD) on silicon substrates [2]. The homogeneous thin films have been obtained by UV-excimer pulsed layer deposition from PMMA bulk material. The structure of films is at variance with the structure of the bulk polymer.

The microstructure of the film is found to have profound influence on the optical, electrical and mechanical properties of the film. The physical properties of thin films are known to differ widely from those of bulk materials. The techniques based on X-ray probe have dominated the field mainly because of their simplicity, more reliability, quantitative and non-destructive nature. Of these techniques, X-ray diffraction has played a leading role, as a fundamental for material characterization [3]. XRD is a powerful tool in the field of scientific research. The application of X-rays in material analysis makes it possible to determine detailed information on the state of order and disorder of the system.

In this paper, we propose a method to prepare PMMA thin films by Chemical Bath Deposition. It is a non vacuum electroless technique has many advantages such as simplicity, no requirement for sophisticated instruments, minimum material waste, economical way of large area deposition and no need of handling poisonous gases like H₂Se or Se vapour and possibility of room temperature deposition. The transmittance, absorbance and structural properties of PMMA thin films are also reported.

2.0 MATERIALS AND METHODS:

2.1 Materials:

PMMA obtained from Alfa Aesar with average molecular weight (3, 50,000), Toluene, Benzene supplied by Merck were used in this study. Benzene and Toluene solutions are distilled before mixing with PMMA to produce thin films.

2.2 Synthesis of PMMA thin film:

The Chemical Bath Deposition procedure to prepare PMMA thin films was followed which involves placing 1 to 2g of PMMA in 50ml distilled Toluene, 50 ml distilled Benzene and 100ml distilled Toluene continuous and very slow stirring for about 60 minutes by means of magnetic stirrer is carried out while adding polymer to the reacting mixture, to yield a

homogeneous mixture. Initially, Blue star special super deluxe glass micro slides with dimension 75mm x 25mm x1mm were rinsed using Extran and washed thoroughly with distilled water. Then, the glass slides were soaked in a beaker containing nitric acid for ½ an hour and washed with de-ionized water and again soaked in hydrochloric acid for the same period. Finally the substrates are dried in a hot air oven for about an hour at a temperature of 100°C. Thus, the substrate for the film deposition has been prepared. Care must be taken so that there should not be any traces of water in the slide before deposition. The deposition time range was optimized as 20 to 35 minutes to obtain films of uniform thickness. After deposition the films were taken out and dried naturally.

2.3 Instrumentation:

The X-ray diffraction patterns were recorded with the diffractometer(X-PERT-PRO) equipped with a PW 3050/60 channel control goniometer and proportional counter. Radiation was generated from a copper anode tube ($_{Cu K\alpha}$ 1.54060 Å) using a X-ray generator operated at 40 kV and 30mA. UV-Visible Spectroscopy was carried out using JASCO-UV-VIS-NIR Spectrophotometer with bare glass slide as reference.

3.0 RESULTS AND DISCUSSION:

3.1 XRD studies:

Possible directions in which the film diffracted the beam of monochromatic x-ray are determined by Bragg condition,
 $n\lambda = 2d \sin \theta$

where n is the order of diffraction, λ is the wavelength of incident x-ray, d is the distance between planes parallel to the axis of the incident beam and θ is the angle of incidence relative to the planes in question. The XRD profiles for the sample PMMA thin films prepared by Chemical Bath Deposition are shown in figure. XRD pattern was obtained in the 2θ range between 0 and 90 degree, which are similar and without any sharp diffraction peaks thereby confirm the non-crystalline nature.

Poly -Methyl Methacrylate is known to be an amorphous polymer. The shape of the first most intense peak at 2θ values of 13° and 23.7° & d spacing around 3.745, 3.742 reflects the ordered packing of polymer chains while the second peak denotes the ordering inside the main chains with their intensity decreasing systematically. This explains the homogenous nature of PMMA thin films and similar results has been reported by [4]. Table 1 shows the measured values of XRD parameters and the calculated values of the crystalline size(P), interchain distance (r). Values of P,r have been calculated using the formula as follows

$$P = K \lambda / \beta \cos \theta \quad (1)$$

$$r = 5.8 \lambda / \sin \theta \quad (2)$$

Where λ is the wavelength of X-ray radiation, K the scherrer constant taken as 0.9, β is the half-height width

TABLE 1: XRD PARAMETERS FOR PMMA FILMS:

Sample	Position (θ)	β	d-spacing	P	r
	deg	deg	(Å)	(Å)	(Å)
PMMA	11.8689	4.0000	3.74526	2.032094	4.685227

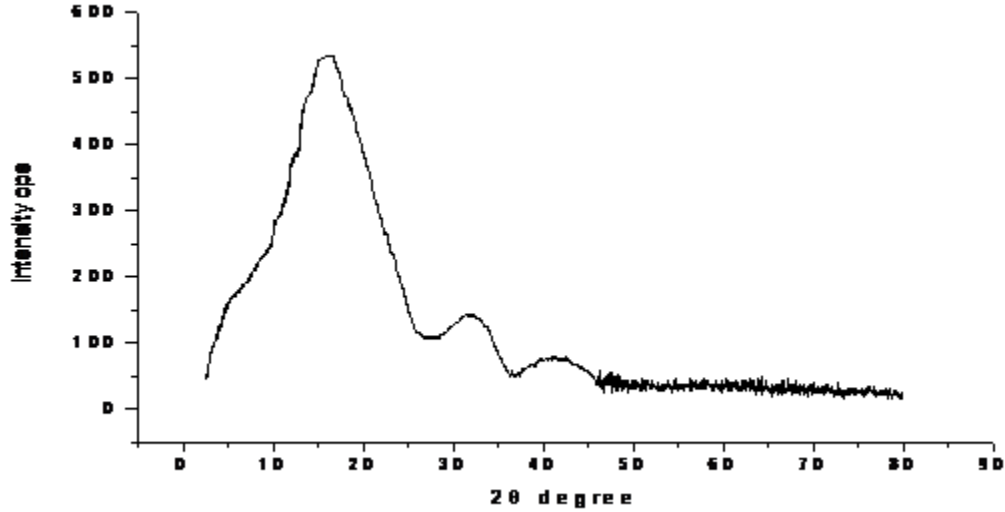


Fig. 1.XRD-Studies of PMMA

3.2UV-Visible studies:

The Transmittance and Absorbance measured for the sample is shown in figures .UV-VIS Spectra exhibited a transmittance of 50% and higher at or above 300 nm[5]. The Optical property of PMMA exhibits higher transmission through the visible wavelength range, very little UV absorption until 260 nm[6].

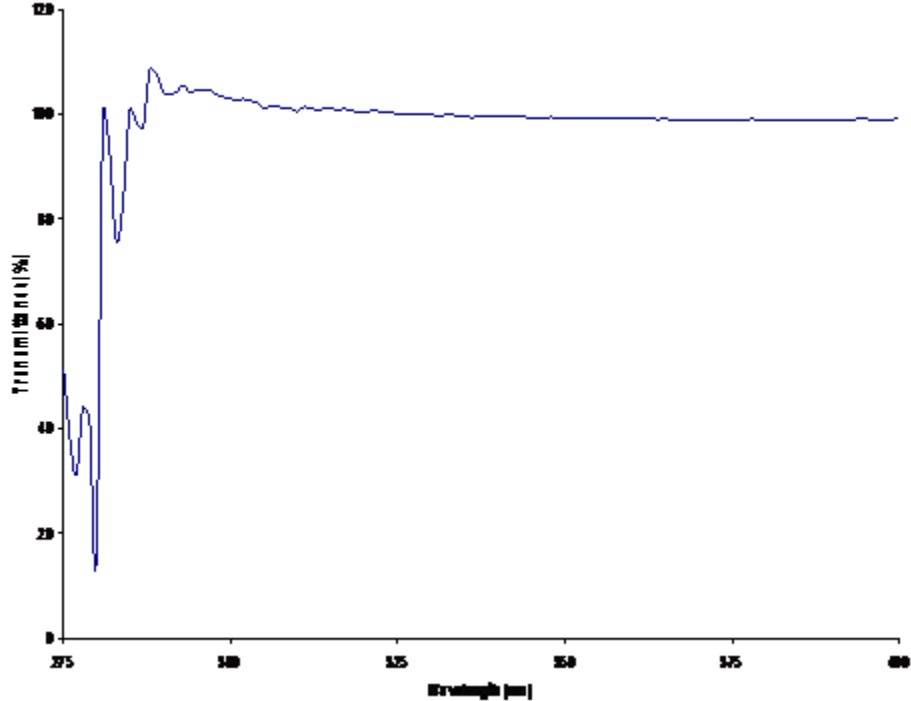


Fig. 2.Transmitivity of PMMA

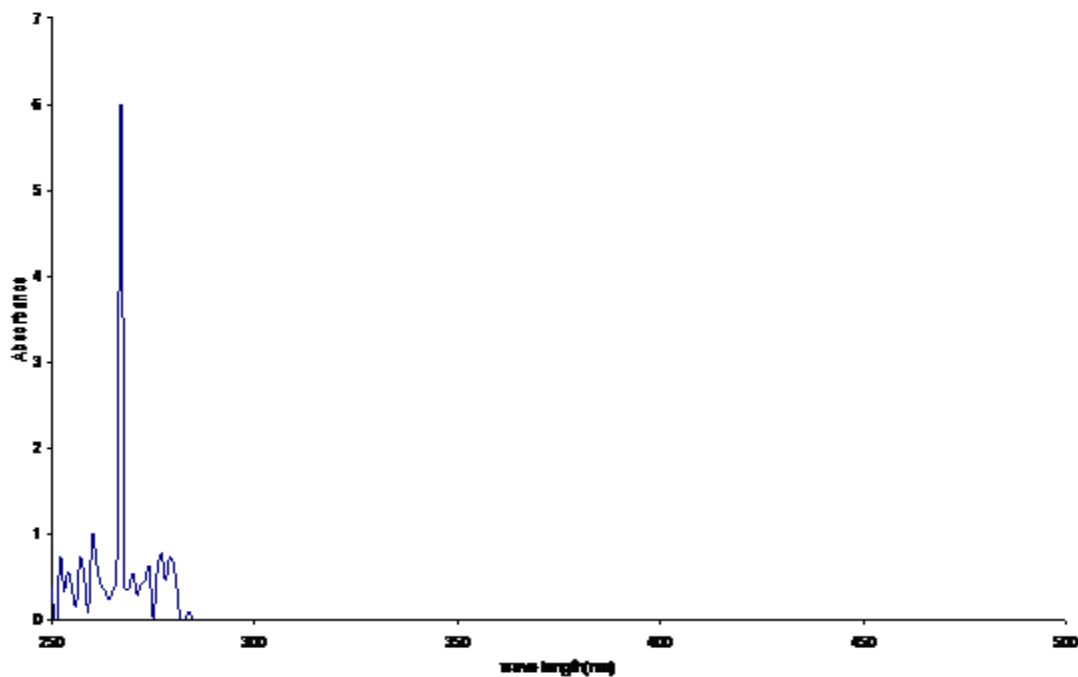


Fig. 3. Absorbance of PMMA

4.0 CONCLUSION:

Poly- Methyl Methacrylate (PMMA) thin films with thickness ranging from 0.53-0.58 microns were deposited successfully by Chemical Bath Deposition Technique. It has been attempted to understand the Structural nature of PMMA thin film samples. XRD technique indicates that the Chemical Bath Deposited film possesses the amorphous nature (i.e.) disorder in polymer chains. The behaviour of the films show higher transmission through the visible wavelength range with very little absorption.

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