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Studies on thin Films based on XRD Diffractograms Method

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Abstract—*Thin films technology has great effect on the growth of electrical, electronic industry especially into integrated chip area. The properties of thin films mainly depend upon purity of the material and the process of preparation. Although several techniques are available solution grown technique of preparation of thin films is most promising to get optimal thin films characteristics. The structure of thin film can range from completely disorder to highly ordered state. Therefore it is challenging for investigators to find structural integrity, physical, dielectric and conduction properties. All these properties will provide as selection of thin film for a specific application. In this context, studies are conducted on two types of thin films namely Polysterene (PS) and Polymethyle Mythcrylate (PMMA). The samples are being prepared on solution grown technique with a standard procedure. The samples then studied for physical properties and the structural data's using Bragg X-ray techniques. X-ray diffractometer furnished the required spectra for the thin films under the subject of study. The results have been discussed on the basis of diffractograms of each type of thin films. The results showed that, the spectra's uniqueness is important characteristic of thin films which will guide us to insight into micro aspects of thin films. These points and other important discussions have been presented in this research paper.*

1. INTRODUCTION

Today the very -large scale integration technology is used industrially, permitting the manufacture of ICs with more than 10^5 pixels per squire millimeter. This trend to miniaturization is continuing, leading to a demand for submicron device geometries. The dielectric properties of pure and copper phthalocyanine (CuPe) –doped polystyrene (PS) films were studied by A.P Srivatsava etal[4] in audio frequency range by varying the temperature from 30^0C to 120^0C. Narula et al [3] studied PVDF/PMMA poly blends over the entire composition range using DSC, IR, SEM and dielectric techniques. A few studies have been carried out on the optical, dielectric and electrical conduction properties of PMMA and PS by various researchers and scientists [1, 2, 3,]

However, not much work has been done on the optical, structural and electrical conduction properties on the blended PS and PMMA thin films. Hence the present work is focused on the preparation of both pure and blended PS and PMMA films and on the study of their physical properties, structure and optical, dielectric and electrical conduction. Thin films are prepared using a standard technique of solution growth applying relevant chemicals, the sample preparation, experimental method and other details are furnished in the following paragraphs.

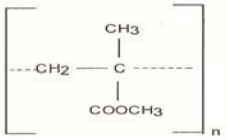
2. EXPERIMENTAL WORK

The details of samples, preparation, structural investigations are provided in this section.

2.1 Preparation of The samples of thin films

Polymethyle methacrylate (PMMA) films were formed from a solution of 2g of low molecular weight PMMA, dissolved in 100ml of benzene.

The structure of PMMA is



This solution of particular concentration was prepared in a glass beaker and it was continuously stirred for three hours by means of a Teflon coated pellet using a magnetic stirrer cum heater. Pre cleaned glass substrates were immersed vertically into the solution for a period of about 10 to 60 minutes so as to get 0.7μm film thickness.

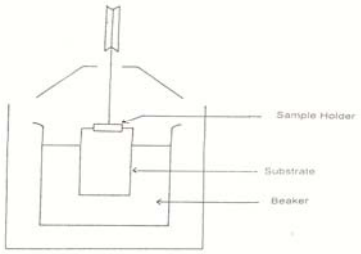
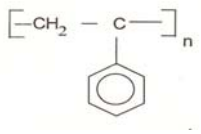


Fig. 1: Schematic Representation of Solution Growth Technique Setup

The substrates were withdrawn from the solution and then dried in an oven at 333K for one hour in order to eliminate the traces of solvent. Two substrates were immersed into the solution at a time and hence a set of films of same thickness was obtained. The procedure was repeated many times to prepare the required number of PMMA film samples.

The structure of Polystyrene (PS) is



Polystyrene (PS) films were formed from solution of 2gm of low molecular weight PS dissolved in 100ml of benzene. The solution of a particular concentration was prepared in a glass beaker and it is continuously stirred for one hour by means of a Teflon coated pellet using magnetic stirrer cum heater. Pre cleaned glass substrates were immersed vertically into the solution for a period of about 10 to 60 minutes so as to get 0.7μm film thickness required. The substrates were withdrawn from the solution and then dried in an oven at 333K to eliminate traces of solvent. Two substrates were immersed into the solution at a time and hence a set of films of same thickness was obtained. The procedure was repeated many times to prepare the required number of PS film samples on to the plain micro slides as well as over the Al electrodes.

Using the same procedure the PS and PMMA blend films of the ratio of 75:25, 50:50, and 25:75 are prepared. The solution growth technique is an important feature because sample preparation depends on it [4, 5] and enough care is exercised in the application of this technique for thin films.

2.2 Structural Investigations

Material characterization using X-ray diffraction plays a vital role in all aspects of thin film technology from fundamental research to manufacturing. The microstructure of the film is directly influenced by the type of substrate used and by the deposition rate, substrate surface etc. several analytical techniques are available for the analysis of the structure.

Among them XRD is a very simple and non destructive technique requiring only very small sample with advancement in technology and computer control over the higher power X-ray sources, diffractometers and counters, this method of analysis has become very popular and is very widely used. Hence in the present study the structural characterization is carried out by employing the X-ray diffraction technique.

2.3 X-ray diffractometer (XRD)

The X-ray diffractometer consists of three parts, a basic diffraction unit, a counter goniometer and an electronic circuit panel with the automatic recorder. The diffraction angles and the intensity of lines can be measured with great accuracy.

The basic principle in XRD is the Bragg's law, which describes the condition for constructive interference of X-rays scattered from atomic plane of a crystal. The condition for constructive interference is $2d\sin\theta = n\lambda$, where λ is the wavelength of X-rays, d is the lattice spacing, n is the order of diffraction and θ is the glancing angle of X-rays. The factor'd' is related to the (hkl) indices of planes and the dimensions of the unit cells. It is therefore seen that the diffraction direction is solely determined by the structure and size of the unit cell.

In the present investigation, the structure of the polymer films is analyzed using Shimadzu X-ray diffractometer (XRD - 6000) with the Nickel filtered CuK_α radiation ($\lambda = 0.15418$nm) at 40kV and 20mA in the 2θ range 10^0 to 80^0.

3. RESULTS AND ANALYSIS

In the present investigation, the structure of the polymer films are analyzed using Shimadzu diffractometer (XRD–6000) with the nickel filtered CuKα radiation in (λ =0.15418 nm). The operating conditions for all the samples were 40kV, 30 mA. Films for analysis were mounted on a suitable holder to eliminate substrate thickness effects on the position of the diffraction plane. Thin film samples coated on glass substrates of size approximately 1cm X 1cm were used for XRD studies.

Fig. 3.1–3.3 show the X–ray diffractograms of pure Polysterene (PS), Polymethyle Mythcrylate (PMMA) and blend (PMMA 50) PS and PMMA films of thickness, about 0.7 mm. above polymer film samples exhibit similar patterns. The variation of intensity with Bragg angle 2θ is shown in the fig. 2, 3 and 4. The spectra (Diffractogram) for blend thin film are entirely different as can be seen from the fig. 4.

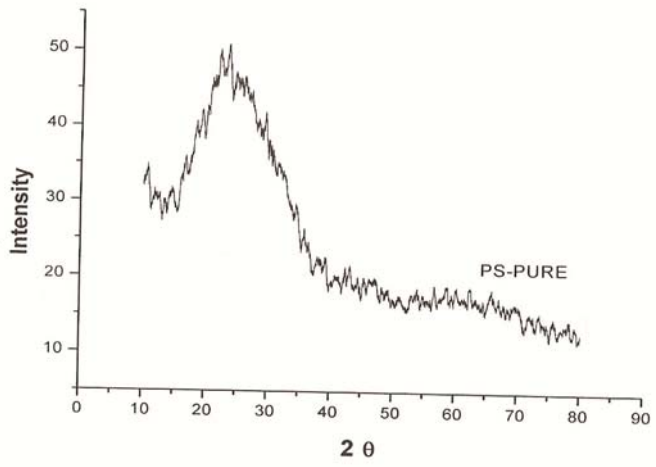


Fig. 2: The XRD Diffractogram for Pure Polysterene (PS) Thin Film

The diffractogram for PS and PMMA exhibited broad peak in each case. The absence of any characteristic peak indicates the amorphous structure of these films. However the presence of a very broad hump in the diffractogram reveals the mixed state of polycrystalline and amorphous structure of these films. Such a state could not be ruled out, because, polymer in general have homogeneous mixture of polycrystalline and amorphous regions [4, 6]. From these results it is clear that, blend thin film has showed mixed structure of crystallinity and amorphous. This result is in agreement with findings of earlier researchers [4, 6].

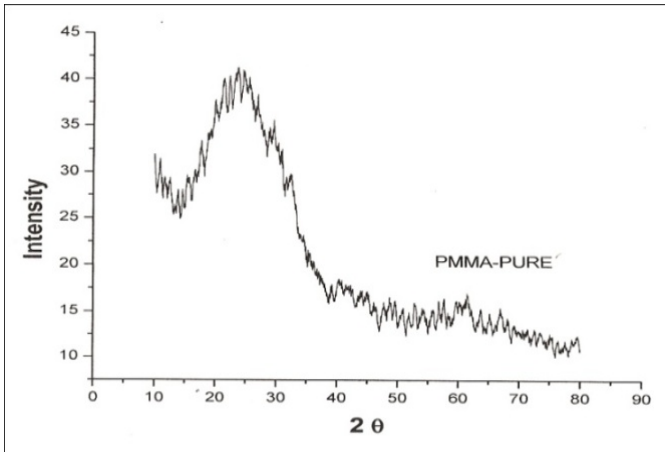


Fig. 3: The XRD Diffractogram for Pure Polymethyle Mythcrylate (PMMA) Thin Film

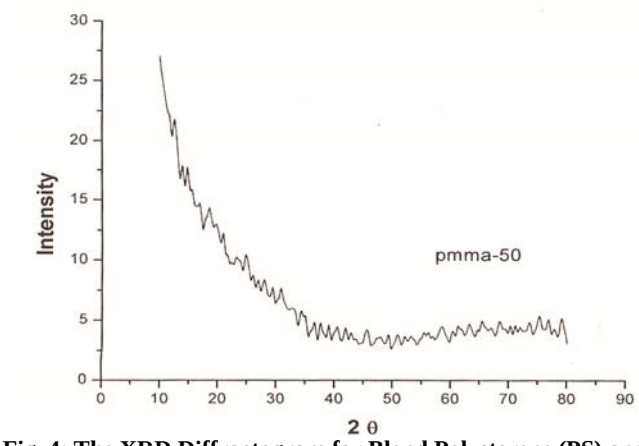


Fig. 4: The XRD Diffractogram for Blend Polysterene (PS) and Polymethyle Mythcrylate (PMMA) Thin Film

4. CONCLUSIONS

The structure, XRD studies, properties of solution grown pure polystyrene (PS) and polymethyle methacrylate (PMMA), blend films have been investigated in detail in the present study. The important results are summarized below.

1. From XRD technique to thin films indicated that the diffractogram for PS and PMMA have broad peak in intensity with 2θ spectra.

2. While blend thin films of PS and PMMA showed that, there is no such broad peak in diffractogram. This confirms the fact that structure is mixture of crystallinity and amorphous in the case of blend thin films (PS and PMMA).

The structure of both pure and blend PS and PMMA films have been found to be amorphous.

Thus the present investigation provides valuable data on XRD, optical and electrical conduction of solution grown pure and blend, PS and PMMA films.

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