

Characterization of TiO₂ Doped Poly (methyl methacrylate) PMMA Thin Films Using XRD

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Abstract. In the present work PMMA thin films by using different concentration of TiO₂ as dopant were prepared. The films have been prepared by using solution cast method. The prepared films were characterized by X-ray diffraction (XRD) study. The XRD result shows that the film structure was changed from amorphous to crystalline nature by increasing the TiO₂ doping concentration. The calculated crystallinity index of the TiO₂ doped PMMA varied from 15.37% to 33.80% as the concentration is increased.

Keywords: Poly(Methyl Methacrylate) X-Ray Diffraction (XRD)

INTRODUCTION

Polymer thin films play an increasingly important role in the development of electronic devices, passivation coatings, chemical and electronic sensors, batteries, organic transistors, high-performance dielectrics, optical data storage and communications, and displays based on organic electroluminescent materials [1,2]. It is well known that the physical and chemical properties of a polymer can be controlled by doping the polymer by a suitable material, which has significant effect on their optical, thermal and electrical properties [3,4]. Among the optical polymers, Poly (Methyl Methacrylate) (PMMA) (CH₃CH₂COOCH₃) is most important commercial high polymer. It is extensively used in optical fibres, optical waveguides, optical sensors, LEDs, antireflective coatings, optical disks and lenses and diffractive optical elements [5,6]. Studies of transition metal oxide doped PMMA are important for determining and controlling the operational characteristics of different PMMA composites. Different properties of PMMA have been studied by using different dopants. Titania polymers i.e. TiO₂ doped PMMA thin films represent an important class of potential material for optoelectronic applications [7].

SAMPLE PREPARATION

Thin films of pure PMMA and doped PMMA with different concentration of TiO₂ were prepared by solution cast method. It is one of the simplest processes by which polymers can be shaped into a desired solid form. Most of the polymers are soluble in organic solvent like acetone (CH₃COCH₃), dichloromethane (CH₂Cl₂), Chloroform (CHCl₃) and Benzene (C₆H₆) etc. Substituting the value of cast surface area in the Mathematica program, the thin film of desired thickness can be obtained.

Pure PMMA Film

Two grams of PMMA granules were added to 100 ml of Benzene and left overnight to form homogeneous solution. The solution was poured over a smooth casting surface of a flat bottomed glass plate. In order to get the

uniformity in the film thickness the glass plate was kept on the surface which was balanced by the use of spirit level. The solvent was allowed to evaporate slowly over a period of 12-24 hours in a dry atmosphere and the film was peeled off the surface of glass plate. The film of thickness 70 microns was prepared.

Metal Oxide (TiO₂) Doped PMMA Film

The pure PMMA solution was prepared as described in the section 2.1.1. TiO₂ powder (dopant) weighing 0.2 mg was dissolved in 5 ml of Methanol and 5 ml of Xylene using sonicator. To prepare 0.1% TiO₂ doped PMMA film, 3180 µl volume of dopant solution was added to 31.80 ml volume of polymer solution. Similarly, to prepare 0.05% TiO₂ doped PMMA film, 1476 µl volume of dopant solution was added to 29.52 ml volume of polymer solution. The measurement of dopant solution was done by using the micro pipette. The measured solution was poured over the glass plate to obtain film of thickness of 70 microns. The characterization of the prepared undoped and TiO₂ doped PMMA films was done by using XRD and their structural properties have been studied.

X-Ray Diffraction of Samples

The X- Ray Diffraction Pattern has been recorded on prepared samples. The Miller indices, Crystallinity index has been calculated. The crystallinity index has been calculated using formula[8]:

$$\text{Crystallinity} = \frac{\text{Area of Crystalline Peaks}}{\text{Total Area}} \times 100\% \quad 1$$

RESULTS AND DISCUSSIONS

XRD Analysis

The pure and doped PMMA thin films are analysed by using X-ray diffractometer (D2 Phaser) with Cu-K_α radiation having a wavelength of 1.54056Å. The XRD patterns of pure PMMA and TiO₂ (0.05% and 0.1%) doped PMMA thin films are shown in Fig-. It shows the plot of counts per second against 2θ, where θ is the angle of incidence of X-ray beam. It can be seen that the pure PMMA sample shows an amorphous polymeric structure without any peaks. While the doped PMMA shows certain peaks which is due to crystalline structure of TiO₂. It can be seen from the figure that for 0.05% TiO₂ doped PMMA the peaks (111), (320) appears along 13.47° and 27.81° respectively and for 0.1% TiO₂ doped PMMA the peaks (111), (320), (322) appears along 13.57°, 27.49° and 31.73° respectively.

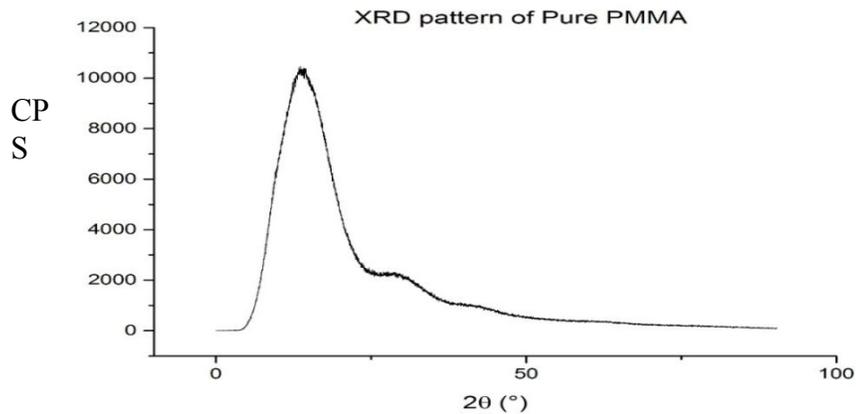


FIGURE 1. Counts per second vs 2θ for pure PMMA

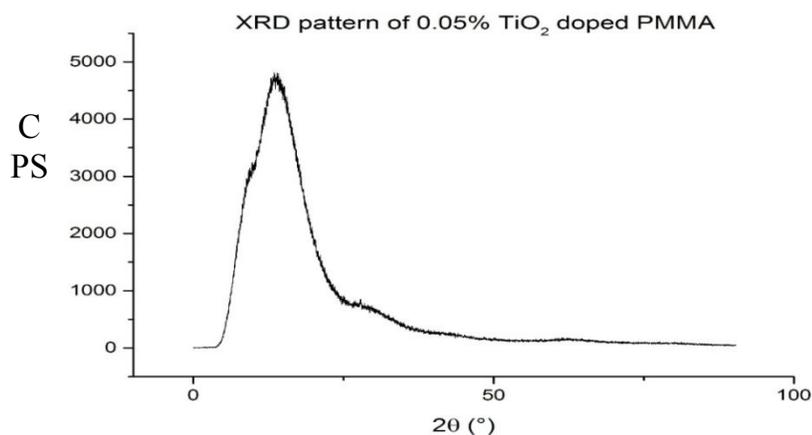


FIGURE 2. Counts per second vs 2θ for 0.05% TiO_2 doped PMMA

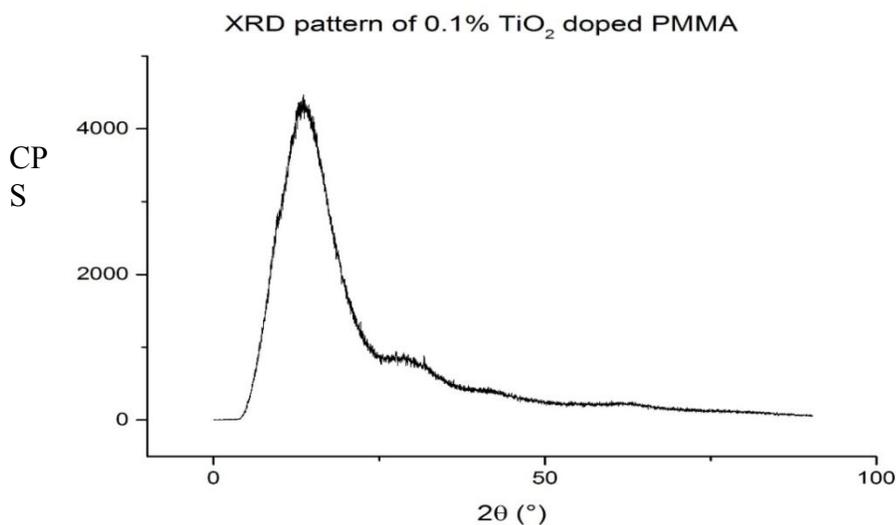


FIGURE 3. Counts per second vs 2θ for 0.1% TiO_2 doped PMMA

CONCLUSION

Transparent Polymer thin films of TiO_2 doped PMMA have been prepared by solution cast method. The effect of doping different concentrations of TiO_2 on the structural properties has been studied. It has been found from the XRD patterns of undoped, 0.05% and 0.1% TiO_2 doped PMMA thin films shows that the pure PMMA sample has an amorphous polymeric structure without any peaks. While the 0.05% and 0.1% TiO_2 doped PMMA shows certain major and minor reflection peaks which can be due to the crystalline structure of TiO_2 . The crystallinity Index (CrI %) for pure PMMA film, 0.05% and 0.1% TiO_2 doped PMMA film are found to be 15.37%, 29.45% and 33.80% respectively. Therefore we see that the CrI (%) increases with increase in the doping percentage.

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