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Structural and Thermal properties of ion beam irradiated polystyrene/ZnO nanocomposite films

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Polystyrene/ZnO nanocomposite (PS/ZnO) thin films were prepared by the solution mixing method and irradiated with 55MeV carbon ion beam at various ions fluences ranging from 3×10^{11} to 3×10^{13} ions/cm^2. The structural and thermal properties of swift heavy ions (SHI) beam on irradiated films were studied by several characterization techniques such as Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), Differential Scanning Calorimetry (DSC) and the dielectric setup with LCR meter. The SEM measurement showed the uniform dispersion of ZnO nanoparticles in Polystyrene solution. The XRD pattern indicated the presence of ZnO nanoparticles in nanocomposite thin films. The increasing ions fluences significant loss of average crystallite size, percentage of crystallinity, glass transition temperature (T_g) and thermal stability were observed for many applications such as unique optical, mechanical and electrical properties. The dielectric loss, A. C. conductivity and dielectric constant were also increased with increasing values ions fluences.

KEYWORDS: PS (Polystyrene), Nanocomposite, XRD (X-Ray Diffraction), DSC (Differential Scanning Calorimetry) and T_g (Glass Transition Temperature).

INTRODUCTION

Swift heavy ions beam irradiation is the most effective method to modify the chemical, electrical, optical, mechanical etc. properties of polymer nanocomposite materials [1-4]. Now a days organic polymers and inorganic nanoparticles polymer matrix drawn great attention due to their many fold applications in electronics, microelectronics, cables, capacitors, power tools handles, safety jackets, electronic packing materials and sensor devices [5-7]. In recent decades considerable interests in polymer-nanocomposite films arose due to their joint combination of electrical, optical, mechanical and physical properties. The joint properties of polymer-nanocomposite are greatly dependent on the properties like miscibility and phase behavior. The effects of ions beam or others source of irradiations in polycarbonate or polystyrene or polymeric thin films have been investigated by many researchers however effect of ions beam, electrons, protons, gamma rays and laser irradiation on polymer-nanocomposite films has rarely reported [8-10].

SAMPLES PREPARATION

PS pellets obtained from Redox and ZnO nanoparticles of size less than 100 nm were obtained from Sigma–Aldrich (India). Benzene was supplied by Merck Pvt. Ltd. (India). PS/ZnO nanocomposite films were prepared by solution mixing method. The detailed method for nanocomposite films preparation and ions beam irradiation were reported in our earlier publications [11-19].

ELECTRODE PREPARATION FOR DIELECTRIC MEASUREMENT

The samples having diameter 5 cm. and thickness 25 ěm were prepared for good ohmic contact. Both the surfaces of nanocomposite films were vacuum aluminized using Vacuum Equipment Delhi. Vacuum coating unit with Penning and Pirani pressure gauges over central circular area of diameter 3 cm. on both sides vacuum aluminized samples have been used for electrical conductivity measurements.

The electrical measurements eg. dielectric constant, dielectric loss and A. C. conductivity of PS/ZnO nanocomposite films were determined by measuring their capacitance. Simultaneously, the loss factor was also measured. Capacitance and dielectric loss measurements were carried out using a parallel plate configuration of electrodes on both sides of PS/ZnO nanocomposite films by the LCR meter in the frequency range of 1– 4.5 MHz at room temperature. The measured values of capacitance then have been converted into dielectric constant and A. C. conductivity.

SCANNING ELECTRON MICROSCOPY

SEM images of PS and PS/ZnO nanocomposite films were shown in Fig. 1(a) & 1(b) respectively. The surface morphology and dispersion of nanoparticles in irradiated nanocomposite films were carried out by SEM. ZnO nanoparticles were found homogeneously and uniformly dispersed in nanocomposite films. The ZnO nanoparticles were represented by white spots in Fig. 1(b) and PS was shown by gray part in Fig. 1 (a) & 1(b).

X-RAY DIFFRACTION

The XRD patterns of PS and PS/ZnO nanocomposite films showed the amorphous nature of PS and crystalline ZnO nanoparticles in Fig.2. The XRD pattern of nanocomposites films were well reported in our previous research papers [20, 21]. The XRD patterns observed broad peaks of diffraction angles at 11.29^0 and 19.13^0 representing PS. Other three peaks represent ZnO at diffraction angles of 30.22^0, 32.87^0 and 34.73^0. It is also indicated the decrease in peak intensity. The full width at half-maximum increases with increasing of ions fluence. The decrease in the peak intensity and increase in the full width at half-maximum are generally associated with the decreasing in crystallinity of polymer. The average crystallite sizes were calculated by the Debye-Scherrer formula;

$$L = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

Where K is the Scherrer constant (0.9), л is wavelength of light source, в is the full width at half maximum and и is the Bragg diffraction angle. The percentage of crystallinity were calculated by

$$B = \frac{A}{A'} \times 100\% \qquad (2)$$

where A is the total area of the peaks and A' is the total area under the diffraction pattern.

It is evident from above formulae the crystallite sizes as well as percentage of crystallinity get decreased with increasing of ions fluences due to main chain scissoring.

DIFFERENTIAL SCANNING CALORIMETRY

DSC thermograms of PS and ions beam irradiated PS/ZnO nanocomposite films were shown in Fig.3. It was observed that the T_g decreased with ions fluences due to the formation of disordered polymeric material by ions beam irradiation. There was decrease in T_g and in molecular mobility as a result of scissoring of polymer chains. At extremely high ions fluence the cross-linking reaction had been arisen from the interaction between the scissioned species generated a highly crosslinked system. It did not exhibit any segmental mobility that was reflected in the form of absence of T_g.

DIELECTRIC CONSTANT

The results obtained for dielectric constant with respect to frequency dependence for PS and ions beam irradiated PS/ZnO nanocomposite films plotted graphically at room temperature as shown in Fig. 4 (a). It observed that the value of dielectric constant gets increased with increasing of ions fluence. It is also observed that the dielectric constant decreased with increasing the frequency. Due to increase in frequency, the charge carriers migrated through the dielectric material and get trapped against the defect site inducing an opposite charge in its vicinity.

DIELECTRIC LOSS

The variation of dielectric loss versus frequency at room temperature for PS and irradiated PS/ZnO nanocomposite films were shown in Fig. 4(b). The value of dielectric loss increases as ions fluence increases. The increase in dielectric loss with ions fluence may be due to chain scissoring of polymer, resulting in the formation of free radicals.

A. C. CONDUCTIVITY

A.C. conductivity versus frequency of PS and irradiated PS/ZnO nanocomposite films at room temperature were plotted in Fig. 4 (c). It was also observed from above graphs that A. C. conductivity gets increased on increasing the ions fluence and gets decreased at high frequencies. This is attributed to the formation of conjugated double bonds.

CONCLUSIONS

Swift heavy ions beam irradiation indicated significant enhancement in thermal, structural, optical, surface morphological and dielectric properties. It is also concluded that the crystallite size, percentage of crystallinity, glass transition temperatures and dielectric constant decreases, while dielectric loss and A. C. conductivity increases with

ion fluences. It could be attributed to formation of carbon nanoclusters, chain scissoring/crosslinking and a electronic energy loss. High-energy irradiation is proved a very effective tool for controlling the thermal and dielectric properties mainly due to bonds scissoring besides other structural modification.

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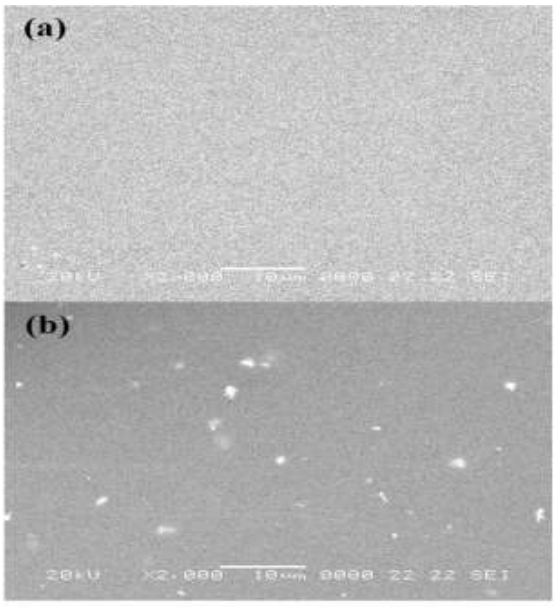

Figure 1. SEM images of (a) PS and (b) PS/ZnO nanocomposite films

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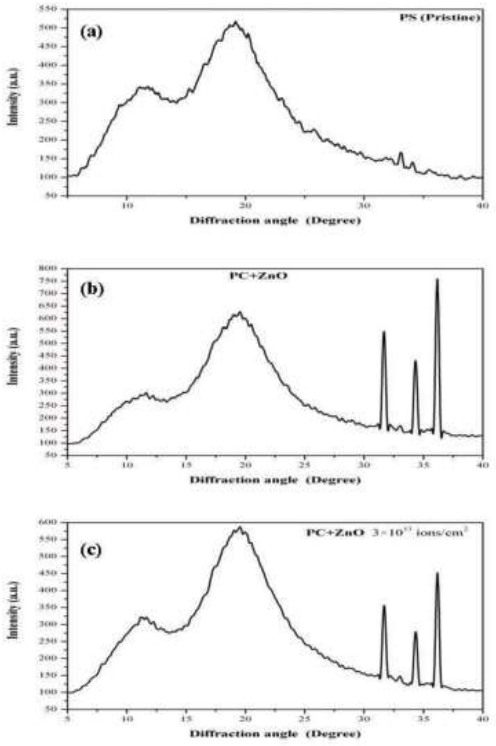

Figure 2. X-ray diffraction pattern of PS and PS/ZnO nanocomposite films

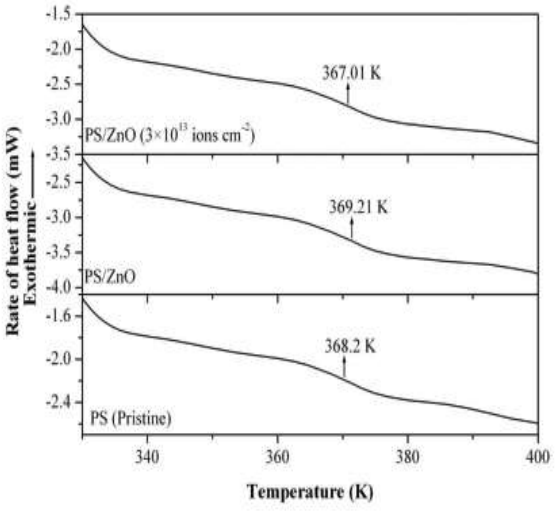

Figure 3. DSC thermograms of PS and PS/ZnO nanocomposite films

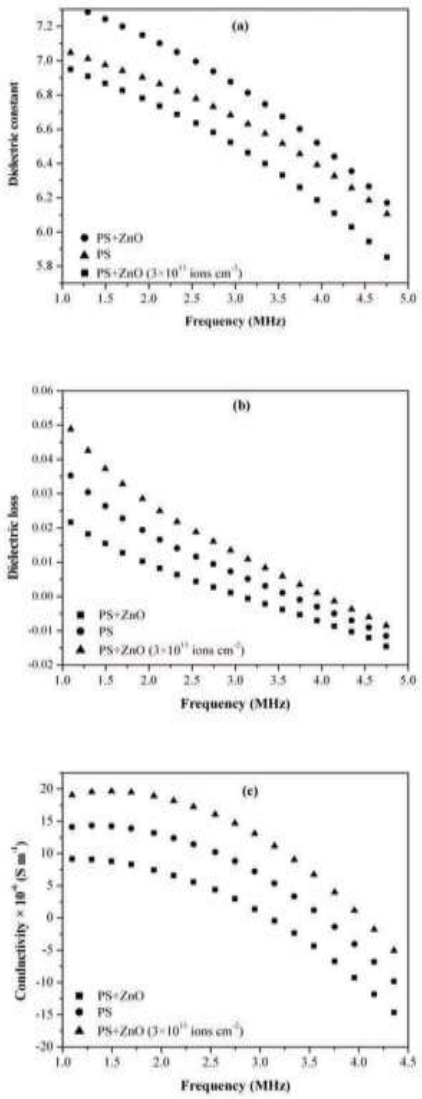

Figure 4. (a) Dielectric constant - frequency, (b) Dielectric loss - frequency and (c) A. C conductivity - frequency