On the linear and nonlinear optical properties of thermally stable, ZnO/PS-PMMA nanocomposite films

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The linear and nonlinear optical properties of highly transparent and thermally stable zinc oxide (ZnO)/poly (styrene) - poly (methyl methacrylate) (PS-PMMA) nanocomposite films have been investigated and the details are given in this chapter. ZnO nanoparticles of size around 10 nm, as confirmed from Transmission electron spectroscopy (TEM) studies, are used to synthesize the ZnO/PS-PMMA nanocomposite samples. The nanocomposite films, deposited by spin coating technique, are characterized by XRD, FT-IR, AFM, UV-Vis-NIR spectroscopy, TGA, Photoluminescence (PL) spectroscopy, and Z-scan technique. In present work, the host matrix used is the blend of the two polymers, PS and PMMA. Both of these polymers are highly transparent and environmentally stable and proven to be excellent for forming nanocomposites with ZnO. Hence, in the present work, the nanocomposite samples of ZnO with PS-PMMA blend have been synthesized and their linear and nonlinear optical properties, investigated. Excellent optical limiting and efficient UV shielding are found to be the significant advantageous characteristics of these composite samples.
6.1 Introduction

Recently, nanocomposite materials have received a lot of attention and close scrutiny of scientists and researchers. The driving force behind the fabrication of novel composites is to achieve functional properties for promising applications in many fields of technology such as optoelectronics, electrochemistry, coating technology and catalysis [1]. Nanocomposite materials are multiphase solids where one of the phases has a dimension less than 100 nm. These materials typically consist of an inorganic (host) solid containing an organic component or vice versa, or they may also consist of two or more inorganic/organic phases in some combinational form with the constraint that at least one of the phases is nanosized. The development of polymer-based nanocomposites, which exhibit interesting optical properties, such as tailored absorption/emission or strong optical nonlinearities has attracted great interest because of their potential optoelectronic applications [2].

Nanostructures of ZnO have attracted significant attention due to their proposed applications in the low voltage and short-wavelength electro-optical devices, transparent ultraviolet protection films, gas sensors, optical limiters and spintronic devices [3-5]. A great deal of research has been focused on the development of ZnO/polymer nanocomposite materials using different polymer systems. Polystyrene (PS) and polymethyl methacrylate (PMMA) are transparent thermoplastic materials, with lots of prospects for making composite materials with nanostructured ZnO. Introduction of ZnO filler into polymeric matrices can modify the optical, electrical, and mechanical properties [6-8]. Composite materials consisting of zinc oxide and
polymers exhibit the merits of blending the advantageous properties of zinc oxide with the processability and flexibility of polymers.

In the present study, PS/PMMA blend is used for making ZnO-polymer nanocomposite. PS/PMMA blend is a well-known immiscible combination for which bulk and surface phase separation has been observed [9, 10]. However, due to the low entropy of mixing, polymeric blends are mostly incompatible and exist as phase separated under appropriate conditions. In incompatible polymer blends, the thin film of the blend may undergo phase separation even during preparation. This behavior has been observed in a variety of incompatible blend systems [11, 12]. In the present work, highly transparent thin films of ZnO/PS-PMMA composite are prepared by spin-coating technique. Although there are a few reports on the synthesis and various properties of this sort of nanocomposite materials [13-17], there are no detailed studies on the nonlinear optical characterization of ZnO/PS-PMMA nanocomposite films. The work presented in this chapter is an attempt to investigate in detail the linear and nonlinear optical properties of the spin-coated ZnO/PS-PMMA nanocomposite films and see how the choice of the polymer blend in the nanocomposite is influencing the optical characteristics. The results obtained are compared with those of the ZnO/PS and ZnO/PMMA composite films.

6.2 Synthesis details

ZnO nanoparticles were synthesized using the method described in chapter 4, and were found to be of average size ~10 nm by XRD analysis. The ZnO/PS-PMMA nanocomposite solution was prepared by adding 10 wt% of ZnO powder sample into the polymer blend solution.
(10% w/v) of PS and PMMA (PS wt% = 50, Mw-175,000 g/mol; PMMA wt% = 50, Mw-140,000 g/mol) in toluene and stirring the mixture for two hours and then sonicating for ten minutes to ensure that the ZnO nanoparticles could be dispersed well. This solution was used to deposit thin films by spin coating on ultrasonically cleaned and optically flat glass substrates (Spin 150). The thin film samples were dried in an oven at 60°C. The nanocomposite films of ZnO/PS and ZnO/PMMA were also prepared using the method described in chapter 4.

6.3 Structural characterization

6.3.1 XRD analysis

X-ray diffraction spectra (XRD) of the samples were taken on a Rigaku X-ray Diffractometer with Cu - Kα (1.5418 Å) radiation operating at 30 kV and 20 mA. Scanning was carried out in the 2θ range from 10 to 70° at a scan speed of 5° per minute (2° per minute for the films).

The XRD patterns of ZnO nanoparticles, PS-PMMA blend and ZnO/PS-PMMA nanocomposite films are shown in figure 6.1. The average ZnO particle size is determined from the X-ray line broadening using the Scherrer equation, \[ \beta = \frac{k \lambda}{d \cos \theta} \], where \( \beta \) is the full width at half maximum (FWHM) in radians of the diffraction peak, \( \lambda \) is the X-ray wavelength, \( k \) is a constant (0.89), \( \theta \) is the Bragg angle of the peak and \( d \) is the average particle size. The particle size is found to be ~10 nm. The XRD pattern of the composite film consists of a broad non-crystalline peak of the polymer blend and sharp diffraction peaks of ZnO. The XRD pattern of PS-PMMA blend does not show any crystalline peak. The
presence of ZnO in the composite produces neither new peaks nor any peak shifts with respect to the polymer blend, showing that nano ZnO filled PS-PMMA composite consists of phase separated structures.

![Figure 6.1: XRD patterns of a) ZnO particles b) ZnO/PS-PMMA and c) PS-PMMA blend](image)

6.3.2 TEM and AFM analysis

The morphology and size of the synthesized ZnO particles were studied by transmission electron microscopy (TEM). The micrographs were taken using JEOL 3010 instrument with a UHR pole piece. This gives a lattice resolution of 0.14 nm and a point to point resolution of 0.12 nm. The TEM and HRTEM images of ZnO nanoparticles are shown in figure 6.2 (a & b). The morphology of ZnO is hexagonal. The average particle size of ZnO determined from the XRD peaks using the Debye-Scherrer formula is found to be close to the one estimated from TEM analysis.
The AFM pictures of the ZnO/PS-PMMA nanocomposite films are shown in figure 6.3. AFM images of the composite films were obtained with Park XE-100 Atomic Force Microscope. The uniform dispersion of the nanoparticles in the polymer blend matrix (Figure 6.3a) and the smooth surface morphology of the films (Figure 6.3b) are clear from the AFM images. The rms roughness estimated is around 10 nm. The morphology of PS-PMMA blend has already been reported by Saziye Ugur et al. [18]. According to them the morphology of PS-PMMA corresponds to that of an interpenetrating network through the whole volume of the sample. The ZnO particles are evenly dispersed in both PS and PMMA which is clear from the AFM image. The AFM pictures confirm the formation of a bi-continuous phase structure where both components form phases that partly or fully form a continuous phase.
6.3.3 FT IR analysis

The chemical structure of the synthesized ZnO particles and the composite film was studied using Fourier transform infrared (FT-IR) spectroscopy. The spectra of the samples were obtained with AVTAR 370 DTGS FT-IR spectrophotometer in the wave number range 400–4000 cm\(^{-1}\).

Figure 6.4 shows the FT-IR spectra of ZnO, PS-PMMA and ZnO/PS-PMMA nanocomposite. FT-IR analysis was used to characterize the functional groups of the nano-ZnO and ZnO/PS-PMMA nanocomposite. The spectrum of the nanocomposite exhibits the characteristic vibrations corresponding to polymeric groups and ZnO. In the spectrum of ZnO, the bands observed at 424, 453, and 527 cm\(^{-1}\) are assigned to Zn-O vibrations [19-21] out of which the bands at 453 and 527 cm\(^{-1}\) are present in the ZnO/PS-PMMA composite spectrum confirming the presence of ZnO in the composite.

The bands centered at 1338 and 1412 cm\(^{-1}\) observed in the ZnO spectrum are attributed to the stretching vibrations of C=O and C=C
groups in acetate species. The characteristic vibration bands of aromatic
C = C of styrene units are observed at 1452, 1493, and 1601 cm\(^{-1}\) in the
spectra of PS-PMMA and ZnO/PS-PMMA but the relative intensity of
these peaks in the composite has slightly changed. In the spectrum of
the composite, the absorption band at 1735 cm\(^{-1}\) is characteristics of
C=O stretching vibration from PMMA. The peak at 1151 cm\(^{-1}\) is due to
the C-O-C stretching vibration of the polymer blend linkage [22, 23]. The
peaks due to the adsorbed acetate species cannot be clearly distinguished
in the ZnO/PS-PMMA spectra. This could be due to the presence of the
polymer blend matrix in which the ZnO nanoparticles are
homogeneously dispersed. The bands at 1382 and 1450 cm\(^{-1}\) correspond
to symmetrical bending vibration and asymmetrical bending vibration of
methyl group, respectively. The absorption band around 3424 cm\(^{-1}\) is
assigned to OH vibration on the ZnO surface. The bands centered at
2924 and 2850 cm\(^{-1}\) observed in PS-PMMA and ZnO/PS-PMMA spectra
are assigned to the asymmetric and symmetric stretching vibrations of
CH\(_2\), respectively.

The PS-PMMA matrix can bring about considerable surface
modification of the ZnO nanoparticles by eliminating most of the
adsorbed species on the surface. The changes in the relative intensities of
the bands in the region 1500 cm\(^{-1}\), in the composite can be due to the
minute presence of adsorbed species on the surface of the ZnO
nanoparticles.
The absorption bands corresponding to aromatic C-H stretching are observed in the range 2900-3200 cm\(^{-1}\). Because the characteristic bands of PS-PMMA and ZnO are both observed in the ZnO/PS-PMMA spectrum, it is clear that ZnO is well dispersed in the PS-PMMA matrix.

### 6.4 Thermal analysis

Thermo gravimetric analysis (TGA) of the samples was carried out on a Diamond TG/DTA instrument. Samples were heated to 1000\(^{\circ}\)C at a scan rate of 10\(^{\circ}\)C per minute in nitrogen atmosphere. The thermal degradation profiles of PS-PMMA and ZnO/PS-PMMA nanocomposite under nitrogen atmosphere are given in figure 6.5. The percentage weight loss of the samples at different temperatures is summarized in table 6.1. The presence of the nanosized ZnO increases the degradation temperature of the polymer composite. The degradation onset temperature (\(T_{\text{onset}}\)) of ZnO/PS-PMMA composite, measured as the
temperature required for percentage degradation is higher than that of PS-PMMA blend beyond 25% degradation. \( T_{0.1}, T_{0.2} \ldots T_{0.9} \) denotes the temperatures for 10%, 20% ... 90% degradation in weight, respectively (Table 6.1). The composite is more thermally stable compared to PS-PMMA blend. The dispersion of ZnO nanoparticles in the polymer blend enhances the thermal stability of the composite.

![TGA curves of PS-PMMA and ZnO/PS-PMMA nanocomposite.](image)

Table 6.1: TGA data of PS-PMMA and ZnO/PS-PMMA nanocomposite

<table>
<thead>
<tr>
<th></th>
<th>( T_{0.1} )</th>
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<th>( T_{0.3} )</th>
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6.5 Optical studies

6.5.1 UV-Visible absorption spectroscopy

UV-Visible absorption spectra of the samples were recorded on a JASCO-V 570 spectrophotometer in the wavelength range 200 to 800 nm.
The UV-Visible absorption spectra of the polymer blend and the ZnO/polymer nanocomposite films are shown in figure 6.6. Pure PS-PMMA blend films do not show any appreciable UV absorption, but an absorption window is found in the range 200-365 nm for the ZnO/PS-PMMA composite films. The ZnO/PS-PMMA films exhibit excellent UV absorption of about 94% spread over a uniform absorption window of about 165 nm width. Furthermore, the absorption peak wavelength of the composite films is substantially blue shifted relative to that of bulk ZnO (~373nm) due to the strong confinement effect [24].

The thickness of the nanocomposite films measured using the thickness profiler (Dekdak 6M Stylus Profiler) is found to lie in the range 1 to 5 µm for all the films investigated in the present work. In this thickness range, the spin coated films are also found to be homogeneous. In a recent work by Junlin Ge et al. [17] ZnO/PS-PMMA nanocomposite films are shown to exhibit UV-shielding efficiency in the wavelength range from 300 to 360 nm. Furthermore, the thickness of their films is very high, which is about 200 µm, and the technique of film deposition is solution casting where one cannot control the film thickness properly. In the present work, about 5 µm thick spin-coated film of ZnO/PS-PMMA nanocomposite is showing about 94% UV absorption in the wavelength range between 200 and 365 nm with 10% of ZnO loading in the composite. These films are highly transparent in the visible range and offer prospects of application as transparent UV radiation protectors in the wavelength range from 200 to 365 nm. The presence of ZnO nanoparticles, thus, enhances the UV absorption of the composite films and modifies the overall optical behaviour of polymer blend films.
UV-Vis absorption spectra of ZnO/PS and ZnO/PMMA nanocomposite films prepared under the same conditions are shown in figure 6.7 for comparison. An absorption window is found in the range 255-365 and 250-366 nm for the ZnO/PMMA and ZnO/PS composite
films respectively. Among these ZnO/polymer nanocomposites, ZnO/PS-PMMA shows the highest intensity of UV absorption with maximum width for the absorption window. The excellent optical characteristics observed in ZnO/PS-PMMA nanocomposite can be related to the formation of an interpenetrating network (IPN) of PS and PMMA in the polymer blend. The formation of IPN offers better possibilities for getting extended regions of pi conjugation which can influence the optical properties significantly.

6.5.2 Photoluminescence

The room temperature photoluminescence emission spectra of ZnO nanoparticles and the composite samples were obtained with Fluoromax-3 Spectrofluorimeter using Xe lamp as excitation source under an excitation at 325 nm. The photoluminescence emission spectra of ZnO and ZnO/PS-PMMA nanocomposite films are shown in figure 6.8. The PL spectrum of ZnO depicts an intense emission peak at 370 nm along with a broader but more intense emission peak in the longer wavelength side centred about 522 nm. A kink is also observed in the blue region around 460 nm. The broad PL band at around 522 nm has already been reported in bulk ZnO as well as in ZnO quantum dots by many researchers [25-27]. The composite films show intense luminescence emission centered around 375 nm in the UV region. The intensity of the broad luminescence observed around 522 nm relative to that of the UV luminescence decreases considerably and is almost quenched in the spectrum of the composite film. The kink observed in the blue region of the ZnO spectrum is less visible in the spectrum of the composite film.
The emission peak in the visible region observed for ZnO nanoparticles is due to the defect levels in ZnO. Since ZnO is nanostructured, there is the possibility of the formation of higher concentration of surface defects. This is the reason for the enhanced intensity of the emission peak in the visible region compared to the near band edge emission in the UV region. In the nanocomposite, due to the surface passivation effect of the polymer blend matrix, most of the dangling bonds will be compensated for. This in turn results in the observed quenching of the emission peak in the visible region in the ZnO/PS-PMMA nanocomposite. Subsequently, the emission gets completely confined to the near band edge emission in the UV region, in the nanocomposite. The polymer blend film alone does not show appreciable PL emission. The ZnO nanoparticles, in combination with PS-PMMA, can bring about functionalities suitable for the realization of efficient light emitting diodes, photo detectors, and other optoelectronic devices.

The photoluminescence emission spectra of ZnO/PS, ZnO/PMMA and ZnO/PS-PMMA nanocomposite films together are shown in figure 6.9 for comparison. Among the nanocomposite systems studied, the ZnO/PS-PMMA composite shows the highest intensity of photoluminescence emission.
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Figure 6.8: PL emission spectra of PS-PMMA and ZnO/PS-PMMA nanocomposite films (Inset shows PL emission spectrum of ZnO)

Figure 6.9: The PL spectra ZnO/PS, ZnO/PMMA and ZnO/PS-PMMA nanocomposite films
6.6 Nonlinear optical studies

6.6.1 Open aperture Z-scan

Nonlinear optical properties of ZnO/PS-PMMA nanocomposite films were investigated by the Z-scan technique. Figure 6.10(a) shows the nonlinear absorption of ZnO/PS-PMMA nanocomposite films at typical laser energy of 25 µJ for an irradiation wavelength of 532 nm. The open-aperture curve exhibits a normalized transmittance valley, indicating the presence of induced absorption. The observed nonlinearity is found to be of the third order, as it fits to a two photon absorption process (TPA). The corresponding net transmission is given by

\[ T(z) = \frac{C}{q_0} \int_{-\infty}^{\infty} \ln\left(1 + q_0 L e^{-r^2}ight) \, dr \]

where \( q_0(z, r, t) = \beta I_0(t) L_{\text{eff}} \).

Here, \( L_{\text{eff}} = 1 - e^{-\alpha L} \), is the effective thickness with linear absorption coefficient \( \alpha \) and nonlinear absorption coefficient \( \beta \), \( L \) is the sample length and \( I_0 \) is the irradiance at focus \[28\]. The solid curve in Fig. 6.10(a) represents the theoretical fit to the experimental data. The experimentally obtained value of nonlinear absorption coefficient \( \beta \) at a pulsed energy of 25µJ is shown in table 6.2.

From the open aperture Z-scan curve it is found that, the ZnO/PS-PMMA nanocomposite films do exhibit large induced absorption behaviour. The observed dip in the open aperture curve shows the transmittance limiting efficiency of the nanocomposite films. Although similar investigations have been reported in ZnO/PMMA nanocomposite films \[15\], the transmittance minimum reported is only about 0.65. In
one of our earlier investigations the transmittance minimum obtained for ZnO/PS nanocomposite film is about 0.43 (chapter 5). In the present work, the curves of ZnO/PS-PMMA films show a better fit to the theoretical equations for TPA, and the transmittance minimum is about 0.25, which highlights the better optical limiting efficiency of the latter compared to the former ones. The calculated nonlinear parameters are given in table 6.2. The comparatively much lower transmittance value obtained for ZnO/PS-PMMA composite films (Figure 6.10a) shows that these films can be used as efficient optical limiters. Optical limiting (OL) devices protect light-sensitive sensors, such as eye or CCD cameras, from possible damage caused by intense light exposure. The observed improvements in the nonlinear optical properties of ZnO/PS-PMMA nanocomposite films compared to ZnO/PS and ZnO/PMMA nanocomposite films can be associated with the formation of an interpenetrating network (IPN) of PS and PMMA in the PS-PMMA polymer blend.

### 6.6.2 Closed aperture Z-scan

Figure 6.10b gives the closed aperture Z-scan trace of ZnO/PS-PMMA nanocomposite film for an irradiance wavelength of 532 nm from pulsed Nd- YAG laser of energy 25 µJ. The closed-aperture curve exhibits a peak to valley shape, indicating a negative value of the nonlinear refractive index $n_2$ [29]. For samples with appreciable refractive and absorptive nonlinearities, closed aperture measurements show contributions from both, i.e. the intensity-dependent changes in the transmission and the refractive index [28]. By dividing the normalized closed aperture transmittance data by the corresponding normalized
open-aperture data, one can eliminate the effect of nonlinear absorption and the resulting curves can be fitted with the theoretical equation for pure nonlinear refraction.

The value of the difference between the normalized peak and valley transmittance, $T_{p-v}$, can be obtained by the best theoretical fit from the results of divided $Z$-scan curve. The nonlinear refractive index $n_2$ is calculated from $T_{p-v}$ in closed aperture $Z$-scan using Eq. (6.2) and is given in table 6.2,

$$T_{p-v} = 0.406(1 - S)^{0.25} |\Delta \Phi_0|,$$

where $|\Delta \Phi_0| = (2\pi/\lambda) n_2 I_0 L_{eff}$, -----(6.2)

where $S$ is the linear transmittance of the far field aperture and $\lambda$ is the excitation wavelength. From the closed aperture $Z$-scan fit, $\Delta \Phi_0$ can be obtained.

The peak-valley trace in the closed aperture $Z$-scan curve shows that the samples have self-defocusing (negative, $n_2 < 0$) type optical nonlinearity, as reported for ZnO/PMMA nanocomposite [15].

![Figure 6.10: The open aperture (a) and closed aperture (b) Z scan curves of ZnO/PS-PMMA nanocomposite film](image)
6.6.3 Determination of nonlinear susceptibility

The nonlinear susceptibility is calculated by the analysis of the open aperture and closed aperture Z-scan data using the procedure described in chapter 2.

The nonlinear refractive index $n_2$ is related to the real part of nonlinear susceptibility, $\text{Re} (\chi^3)$ by the relation,

$$\text{Re} (\chi^3) = n_0 n_2 / 3\pi \quad (\text{esu}), \quad \text{-----------------------------}(6.3)$$

where $n_0$ is the linear refractive index.

The imaginary part of third order susceptibility, $\text{Im} (\chi^3)$ determines the strength of the nonlinear absorption. Equation (4) given below, relates the nonlinear absorption coefficient $\beta$ to $\text{Im} (\chi^3)$

$$\text{Im} (\chi^3) = \varepsilon_0 n_0^2 c^2 \beta / \omega \quad (\text{m}^2 \text{ V}^{-2})$$

$$= n_0^2 c^2 \beta / 240 \pi^2 \omega \quad (\text{esu}) \quad \text{-----------------------------}(6.4)$$

where, $\varepsilon_0$ is the permittivity of free space and $c$ the velocity of light in vacuum.

From the real and imaginary parts of $(\chi^3)$, the modulus of third order nonlinear susceptibility can be found out.

$$| (\chi^3) | = [\text{Re} (\chi^3)]^2 + [\text{Im} (\chi^3)]^2]^{1/2} \quad \text{-----------------------------}(6.5)$$

The magnitude of $(\chi^3)$ is significantly affected by the molecular orientation and it determines the strength of nonlinearity of the material.
The third order nonlinear susceptibility value of ZnO/PS-PMMA composite film at a pulsed energy of 25 µJ for a wavelength of 532 nm is given in table 6.2. The observed higher susceptibility value of ZnO/PS-PMMA films measured by the Z-scan technique, compared to both ZnO/PS and ZnO/PMMA films, establishes the fact that the ZnO/PS-PMMA nanocomposite films have better nonlinear optical response and can be chosen as ideal candidates with high prospects of applications in nonlinear optics [30,31].

<table>
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<th>Band gap (eV)</th>
<th>$\beta$ (cm/GW)</th>
<th>$n_2 (\chi^3)$ ($10^{-9}$ m²/W)</th>
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</table>

The open aperture and closed aperture Z-scan curves of ZnO/PMMA and ZnO/PS nanocomposite films are shown in figure 6.11. The transmittance minimum observed for the ZnO/PMMA and ZnO/PS nanocomposite films are 0.41 and 0.37 respectively (Figure 6.11a&c), whereas the value obtained for ZnO/PS-PMMA is much lower and is 0.25 (Figure 6.10a). The closed-aperture curves exhibit a peak to valley shape, indicating a negative value of the nonlinear refractive index $n_2$ for both the ZnO/PMMA and ZnO/PS nanocomposite films.
6.7 Conclusion

In the present work, ZnO nanoparticles and transparent ZnO/PS-PMMA nanocomposite films have been prepared using simple and reproducible methods. The ZnO/PS-PMMA composite films with ZnO content of 10 wt % exhibit strong UV absorption (around 94%), wide UV absorption window of width around 165 nm and high transmittance (around 93%) in the visible region. These nanocomposite films with strong UV absorption and high transmittance in the visible region offer prospects of applications as optical coating materials for UV protection and shielding. Furthermore, these composite films are found to be more
thermally stable compared to films of PS-PMMA blend. The composite films show a self-defocusing type (negative refractive index, $n_2$) optical nonlinearity and good nonlinear absorption behaviour. The linear and nonlinear optical characteristics of these films are compared to those of ZnO/PS and ZnO/PMMA films prepared under the same conditions. The much lower transmittance of about 0.25 observed for the ZnO/PS-PMMA nanocomposite films is ideal for the fabrication of efficient optical limiters in sensor protection applications. The main factor contributing to the excellent optical characteristics of the ZnO/PS-PMMA nanocomposite is assumed to be the formation of an interpenetrating network (IPN) of PS and PMMA in the polymer blend.

References


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