

# Optical and Thermo Electrical Properties of ZnO Nano Particle Filled Polystyrene

Mulayam S. Gaur,<sup>1</sup> Pramod Kumar Singh,<sup>1,2</sup> Ramvir S. Chauhan<sup>2</sup>

<sup>1</sup>Department of Physics, Hindustan College of Science & Technology, Farah, Mathura-Uttar Pradesh, India

<sup>2</sup>Department of Physics, R.B.S. College, Agra, Uttar Pradesh, India

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**ABSTRACT:** The study of optical and thermally stimulated electrical properties such as optical band gap, refractive index, X-ray spectra, SEM spectra, thermally stimulated discharge current (TSDC), differential scanning calorimetry (DSC) have been undertaken in ZnO nanoparticle filled polystyrene nanocomposite thin film of 30  $\mu\text{m}$  thickness. The appearance of single TSDC peak at temperature  $408 \pm 5$  K in nanocomposite samples shows the charge carriers injected from deeper trapping levels. It is due to the modification of surface and bulk properties of polystyrene by filling of ZnO nanoparticles. In other hand, the

strong interaction of nanoparticles with polymer matrix is the expected reason of improvement of crystallite size, optical energy band gap, refractive index, TSDC, glass transition temperature, and charge storage. It is confirmed from SEM images that the modifications of these properties are caused by creation of clusters in amorphous-crystalline boundaries of pristine polystyrene. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 2833–2840, 2010

**Key words:** activation energy; refractive index; crystallization; differential scanning calorimetry; glass transition

## INTRODUCTION

Recently, the introduction of inorganic nanoparticles in organic polymers have been paid much attention, because of organic-inorganic nanocomposite play a important role to improve the physical properties of conventional polymers such as mechanical, thermal, electrical, and optical. The ZnO nanofiller provide great attention to develop polymer-nanocomposites in recent years due to its significant properties such as high chemical stability, low dielectric constant, and high luminous transmittance, intensive ultraviolet and infrared absorption.<sup>1–3</sup>

From last few decades ZnO was known as transparent electro-conductive and piezoelectric material. But it has other interesting promising properties. Among them, the optical, ferroelectric, and magnetic properties are considered to be very important. UV lasing in polycrystalline ZnO films has been observed with optical gain about  $320 \text{ cm}^{-1}$ . It indicates that ZnO is the promising material for UV laser. Due to high excitonic binding energy, the lasing mechanism is not crashed at higher temperatures. At doping by noncentral ions the ZnO films display ferroelectric behavior. Thus ZnO is the

promising material for creating structures possessing very interesting ferroelectric, ferromagnetic, and magneto-optical properties.<sup>4</sup> Keeping these properties of ZnO in mind, we have chosen the ZnO nanoparticle as filler in present study.

The many methods have been applied for the formation of the nanocomposites such as situ polymerization, sol gel, melt compounding, etc. But in sol-gel technique, the solution mixing of each component in a cosolvent brings about a good molecular level of mixing overcoming the agglomeration tendency of the nanoparticle. It is applicable to the polymers that can be dissolved or swelled by the solvent.<sup>5–9</sup>

Many efforts are taken and many are under way to develop high-performance rapid manufacturing materials for engineering applications, including enhanced optical and thermo electrical, mechanical properties, transparency, and flammability by using polymer nanocomposite (PNC).<sup>10–12</sup> Polymer nanocomposite is suggested to be based on controlling the micro/nanostructure of materials by incorporating nanometer-size fillers as second-phase dispersions into polymer matrixes.<sup>13</sup> A lot of work has been reported in literature to cover the mechanical and optical properties by many group of researcher in last few decades,<sup>14</sup> but no more information is available to understand the thermally stimulated electrical properties of polymer nanocomposite and their suitable justification through other techniques.

The principle concern of thermally stimulated discharge current (TSDC) research is to study the

Correspondence to: M. S. Gaur (mulayamgaur@rediffmail.com).

charge decay and storage, relaxation processes by heating the charged polymer samples at constant heating rate. The decay process is thus investigated as a function of temperature instead of time. This has the obvious advantage of unraveling the nature of various decay and relaxation process very quickly, which makes TSDC an attractive proposition.<sup>15</sup>

UV-vis absorption spectra of pristine and nanocomposite materials investigate the promotion of electrons from lower state to the higher energy state in UV and visible region. UV-vis absorption and transmission spectra are helpful to investigate the change in bandgap and reflective index of polymeric material. Differential scanning calorimetry (DSC) is the technique that is used to study the thermal transition of polymer. Thermal transition is caused by heating of polymer at constant heating rate. DSC technique helps to separate the glassy and rubbery state by measuring the glass transition temperature ( $T_g$ ) and depend on the interaction of nanoparticles with polymer matrix. The X-ray diffraction (XRD) spectra ascribed the change in crystallite size and average intermolecular spacing after introducing the nanofiller in polymer matrix. These techniques are not only helpful to understand the change in shape confirmation due to motion of individual molecules of the polymer, but also elucidate the behavior of larger and more complex structural formation as well.

The overall objective of this research is to investigate how the thermally stimulated electrical properties of nonpolar polymer nanocomposite such as PS-ZnO comprised of various parameters and their justification with the help of XRD, UV-vis, TSDC, DSC, and SEM due to reinforcement of nano-size particles in polymer matrix.

## EXPERIMENTAL

### Materials

Polystyrene used in the present study was supplied by Redox (Jabalpur, India) and manufactured by BDH and ZnO nano particle of size less than 100 nm was procured by Sigma Aldrich (New Delhi, India). All the samples of pristine polystyrene (PS) and ZnO filled PS nanocomposite samples were prepared by using the solution grown technique. The analytical grade benzene used as a solvent was procured from Merck India (New Delhi, India).

### Sample preparation method

The solution of PS in benzene was kept in magnetic stirrer for 7 h at 333 K to yield a homogeneous and transparent solution and then immersing an optically plane circular glass plate at a constant temperature of 303 K for about 1.5 h. The plate was drawn out from

solution. The circular samples of diameter 5 cm and thickness 30  $\mu\text{m}$  were peeled off from the glass plates.

To obtain the thin film samples of PS-ZnO nanocomposite with different weight ratio, the ZnO nano-particles of certain weight ratio are taken in 20 mL of benzene and then solution was kept at 333 K on magnetic stirrer for the period of 2 h. The milky solution was prepared. This milky solution was then added in PS solution drop by drop and stirred for the period of 7 h at temperature 333 K. Now the nanocomposite samples were cast by same method as stated above. All the samples were vacuum dried at 313 K to avoid the effect of solvent and other volatile for the period of 24 h.

### Experimental procedure

The detail of the TSDC measurement is reported in our earlier publications.<sup>16,17</sup> DSC was performed on a Perking-Elmer DSC 7 (Massachusetts) available in IUC Indore (India) under nitrogen purge at a heating and cooling rate of 10°C/min. Samples (10 mg) were heated from room temperature 303–423 K. Each test was repeated three times to observe the reproducibility, and glass transition temperature was evaluated using simple method reported in literature.<sup>12</sup> The SEM (LEO 435 VP) (Beaverton) available in IIT Roorkee (India) was used to observe the dispersion of nano particles in polymer matrix.

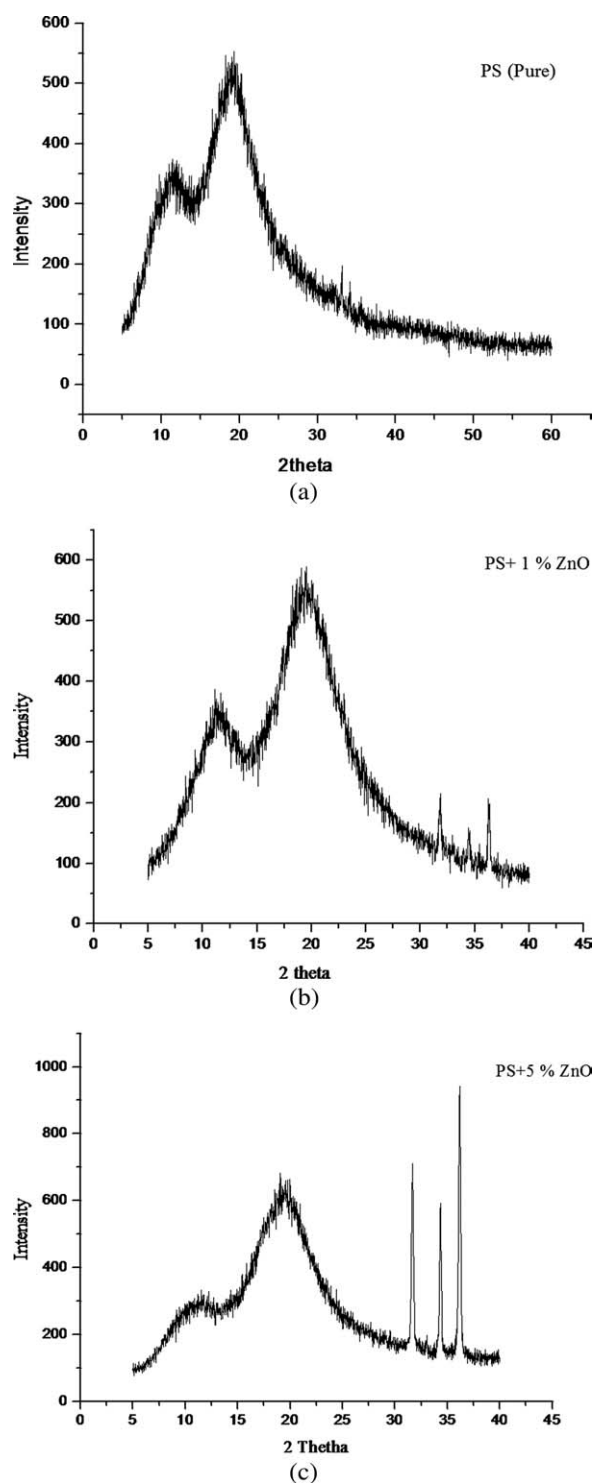
To study the crystalline structure, XRD patterns were recorded using the Cu K $\alpha$  ( $\lambda = 1.54 \text{ \AA}$ ) radiation in  $\theta$ - $\theta$  locked couple mode from the Bruker AXS D8 diffractometer (New Delhi, India) with scan speed of 1°/min. The diffraction angle ( $2\theta$ ) has been varied from 5° to 40° with a step size of 0.02. The measurements were done under ambient pressure conditions at room temperature. Each experiment was repeated at least twice and with both faces of the specimens alternatively exposed to the X-rays to check the reproducibility.

The absorbance and transmittance spectra were recorded for pristine PS and ZnO filled PS at different concentration by means of UV-vis spectrophotometer (i.e., Hitachi U-2800 double beam spectrophotometer) (Tokyo, Japan) in the wavelength range from 190 to 1100 nm. All the observations were carried out with the accuracy of  $\pm 5\%$  experimental error, and results are reproducible. The optical band gap and refractive index have been calculated using UV-vis characteristics in absorbance as well as transmittance mode respectively for PS and PS-ZnO nanocomposite samples.

## RESULTS AND DISCUSSION

### XRD analysis

It has been observed that only the two peaks are corresponding to pristine PS; however, three more



**Figure 1** (a) X-ray diffraction spectra of pristine PS, (b) X-ray diffraction spectra of 1% ZnO nanoparticle filled PS, and (c) X-ray diffraction spectra of 5% ZnO nanoparticle filled PS.

sharp peaks are appearing in nanocomposites. These numbers of sharp peaks are showing the polycrystalline structure of PS-ZnO nanocomposite. The XRD spectra of pristine and ZnO filled 30  $\mu\text{m}$  thick polystyrene nanocomposite samples is shown in Fig-

ure 1(a–c). These figure shows a broad noncrystalline peak from 10–30° of polystyrene and sharp diffraction peaks for ZnO nanoparticle. The incorporation of ZnO nanoparticles produces neither a new peak nor a shift with respect to PS indicating that ZnO-filled PS nanocomposite consist polymer and nanoparticle phase. The XRD characteristics of pristine PS is characterized by one peak at 19.06° ( $d \approx 4.64 \text{ \AA}$ ,  $d = \lambda/2 \sin \theta$  is the lattice spacing or crystalline interplaner distance). However, the XRD peak of PS with 1% and 5% ZnO nanocomposite were found at 19.43 and 19.50, respectively. XRD pattern shows that the peak intensity and full-width at half maximum (FWHM) increases with concentration of ZnO in PS. The observed increase in peak intensity and FWHM is generally associated with decrease in crystallinity of the polymer nanocomposite. Crystal size and width of the peak have been related by Scherer formula, the average crystallite size  $L$ , have been calculated by Scherer formula

$$L = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where  $\beta$  is the FWHM of the peak (in radian) and  $k$  is the shape factor whose value is equal to 0.9. The  $k = 0.9$  in the above equation, the crystallite sizes of pristine PS and ZnO filled PS nanocomposite samples calculated and these results are shown in Table I. The crystallite size of polystyrene decreases after introducing the ZnO nanofiller with different concentration.

### Energy band gap and refractive index

UV-Visible absorption spectra of pristine polystyrene and ZnO filled polystyrene nanocomposite spectra have been recorded and shown in the Figure 2. It is shown that the absorption is increased with increase of ZnO nanofiller weight percentage in PS, while transmittance will be decreased. The optical band gap has been determined at different ZnO nanofiller weight percentage from the fundamental absorption edge of UV-vis spectra. The increase of absorption in the infrared region of the spectrum is due to excitations of donor level electrons to the conduction band at these energies. The transmittance of the nanocomposite films is decreased with the increase of loading concentration of ZnO due to increase in surface roughness. A significant decrease in transmittance, observed for nanocomposite sample is due to Rayleigh scattering take place from the nanoparticles. High transmittance at UV-vis interface ( $\sim 400 \text{ nm}$ .) confirms that PS and ZnO are uniformly distributed in the films having a good optical homogeneity, because no substantial scattering effect from film exists. The band gap has been calculated from UV-vis absorption spectra using following relation<sup>18,19</sup>

TABLE I  
XRD Parameters

Samples	2θ (Degree)	β (Degree)	L (Å)	D (Å)	Intensity
PS (Pristine)	19.06	6.73	0.20	4.64	534.33
PS + 1% ZnO	19.43	7.81	0.18	4.56	566.23
1st Peak (ZnO)	31.82	0.31	4.64	2.80	214.34
2nd Peak (ZnO)	34.46	0.41	3.53	2.59	159.25
3rd Peak (ZnO)	36.26	0.41	3.55	2.47	205.09
PS + 5% ZnO	19.50	7.84	0.17	4.54	629.35
1st Peak (ZnO)	31.67	0.64	2.20	2.82	713.0
2nd Peak (ZnO)	34.31	0.55	2.63	2.61	597.09
3rd Peak (ZnO)	36.19	0.68	2.14	2.47	945.46

$$\alpha(\nu) = 2.303A/t \quad (2)$$

where  $\alpha$  is absorption coefficient,  $A$  is the optical absorbance, and  $t$  is thickness of the samples. The envelop method<sup>20,21</sup> was applied to calculate refractive index from UV-vis transmittance spectra using following relation

$$n = \left[ N + (N^2 + n_a^2)^{1/2} \right]^{1/2} \quad (3)$$

where

$$N = 2n_a \frac{(T_M - T_m)}{T_M T_m} + \frac{(n_a^2 + 1)}{2} \quad (4)$$

$T_{\max}$  ( $T_M$ ) and  $T_{\min}$  ( $T_m$ ) is the transmission spectra and  $n_a$  is refractive index in air.

It is found that bandgap of PS + ZnO nanocomposite decreases slightly and refractive index increases with concentration of nanoparticles in PS matrix as shown in Table II.

### Thermally stimulated discharge current

The effect of high dc field was observed in pristine PS and PS nanocomposite samples using TSDC method. TSDC of PS is found to be function of

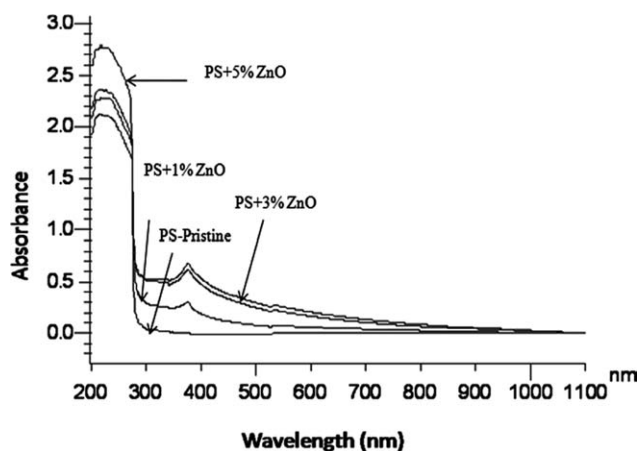


Figure 2 UV-Vis spectra of pristine and ZnO nanoparticle filled PS with different concentration.

polarizing field as reported earlier,<sup>22</sup> however, TSDC of nanocomposite is not yet been reported. The Figure 3 shows the depolarization current of PS, which is formed under the different polarizing field. The Peak observed at 368 K–373 K corresponding to glass transition temperature of PS. The observed value of activation energy is direct evidence that this peak is caused by dipolar and space charge relaxations jointly. The following relations are used to calculate the activation energy, relaxation time, and charge released, respectively, from TSDC peak as reported in literature.<sup>23,24</sup>

$$I = I_0 \exp \left[ \frac{A}{kT} \right] \quad (5)$$

where  $T$  is the absolute temperature and  $A$  is Activation energy.

$$\tau(T) = \tau_0 \exp(A/kT) \quad (6)$$

where  $\tau_0$  is the characteristic relaxation time.

$$Q = S/b \int_{T_0}^T I(T) dT \quad (7)$$

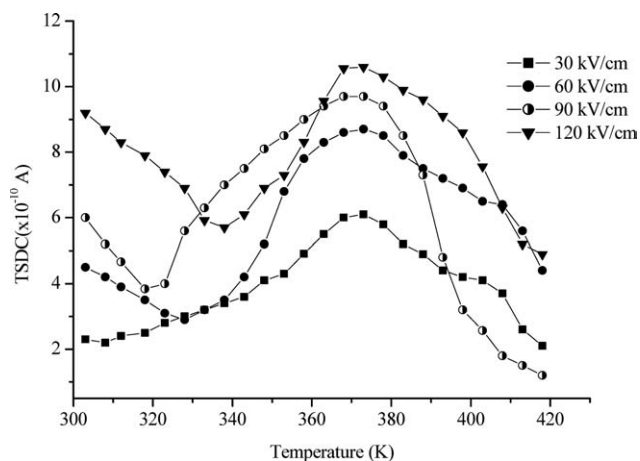
$S$  is the sample area and  $b$  is the heating rate.

The TSDC parameters are summarized in Table III. The TSDC of 1, 3, and 5% have been recorded, and they are showing the similar characteristics except change in magnitude of current. The TSDC of 5% ZnO filled nanocomposite is considered as a representative characteristic as shown in Figure 4. The

TABLE II  
The Variations of Energy Band Gap with Nanofiller in PS

S. No.	Samples	Bandgap (eV)	Refractive index
1	PS (Pristine)	4.44	1.52
2	PS + 1% ZnO	4.43	1.55
3	PS + 3% ZnO	4.39	1.60
4	PS + 5% ZnO	4.38	1.61





**Figure 3** TSDC spectra of PS sample polarized at room temperature with different polling fields.

TSDC spectra of pristine and nanocomposite samples were characterized with single peak. The important change has been observed in nanocomposite samples are (1) broadness of peak increases, (2) the magnitude of TSDC decreases with increase of polarizing field, and (3) the position of peak was observed quite above the glass transition temperature of PS. The TSDC characteristic of pristine PS is the increasing function of polarizing field. It is also observed that broadness of peak increases with increase of polarizing field. The observed results indicate that the TSDC peak of nanocomposites samples is having opposite trend than pristine PS (Fig. 4).

It is clear from SEM micrograph that particle dispersed in amorphous crystalline boundaries, these boundaries provide the conducting path for injection of charge carriers from crystalline to amorphous phase. ZnO nanoparticles filled these boundaries through solution mixing as well as these particles are accumulated and forming the small clusters. The change in surface morphology introduces a hindrance for conduction of charge carriers. This is one of the reason for decreasing function of current with field.

The opposite trends may be due to the decrement in crystallinity as discussed in XRD section. The similar trends of results are observed in low-density polyethylene/MgO nanocomposite samples.<sup>24</sup> Authors found that TSDC peak has been spared over  $10^{\circ}$ – $60^{\circ}$  as they increased the concentration of MgO nanofiller in low-density polyethylene, the height of current decreases. The similar behavior of TSDC peak of PS-ZnO nanocomposite samples has been recorded. This behavior is may be due to the charge carriers tend to be captured more strongly by the filling of ZnO nanoparticles. These results are evident to justify the fact that the amount of space charge is not much more in PS-ZnO nanocomposite samples.

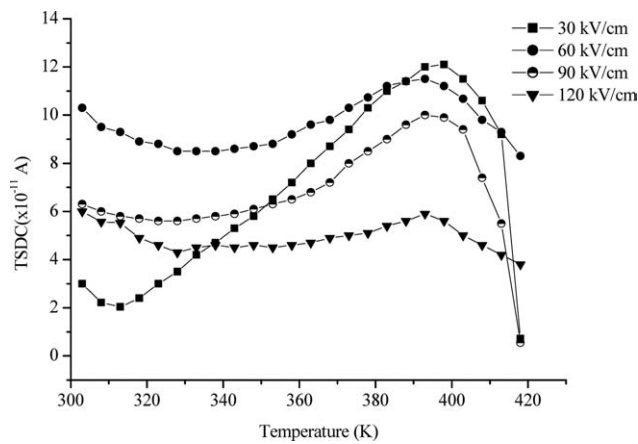
Generally, the charge released increases with the increase of applied electric field in polymeric insulating materials as discussed by several researchers.<sup>12,16,17</sup>

The results of pristine samples are in good agreement with the previous report; however, PS-ZnO nanocomposite shows the inverse [Fig. 5(a)] behavior of charge released. It may be due the blocking of charges in PS matrix after introducing the ZnO nanoparticles. This behavior of PS-ZnO nanocomposite samples shows the strong charge storage ability. Figure 5(b) shows the field dependent peak current of pristine and nanocomposite samples. It has been observed that the peak current decreases with increasing polarizing field for nanocomposite, while pristine PS shows the opposite trends, indicate the reduction of space charges in nanocomposites.

The less thermally stable TSDC peak can be attributed to less stable polarization, but the more thermally stable TSDC peak attributed to more stable polarization as reported for ferroelectric materials.<sup>25</sup> Since ZnO is a ferroelectric material, and it introduces the ferroelectric properties in polystyrene nanocomposite thin film. The comparatively large broadness of peak is observed in nanocomposite samples are due the ferroelectric behavior and saturation of polarization for long time. Therefore, existence of two components of polarization can be expected,

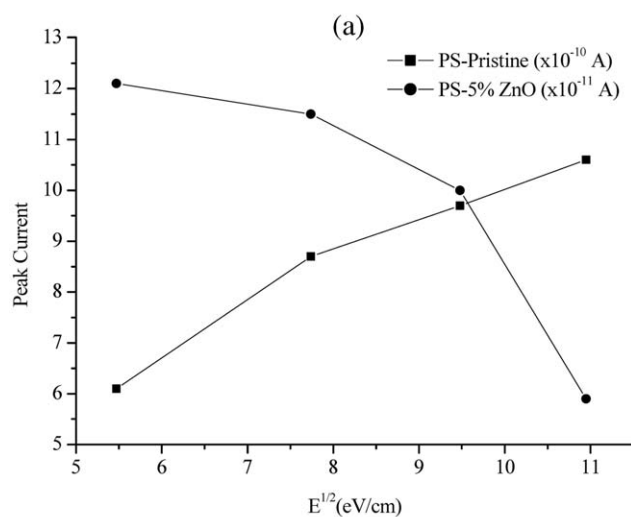
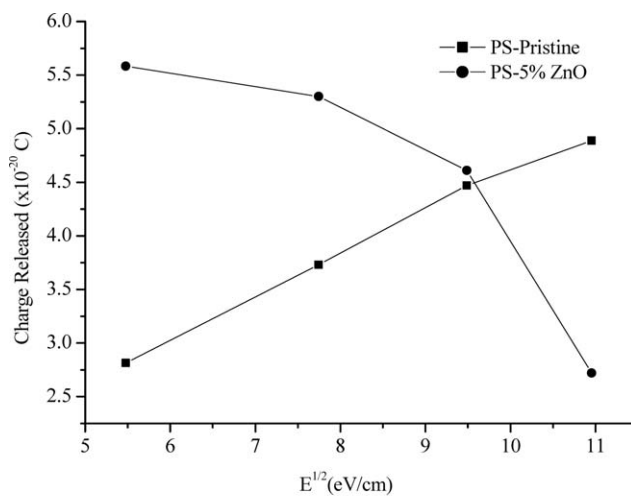
**TABLE III**  
TSDC Parameter of PS (Pristine) and PS-ZnO Filled Polymer Nanocomposite Samples

Samples	Poling fields (kV/cm)	Activation energy $A$ (eV)	Charge released $Q$ ( $\times 10^{-20}$ C)	Relaxation time $\tau$ ( $10^{-16}$ ) (s)
PS	30	0.08	2.81	1.00
	60	0.18	3.73	1.02
	90	0.07	4.47	1.00
	120	0.12	4.89	1.01
PS + 5 % ZnO	30	0.13	5.58	1.0127
	60	0.05	5.30	1.0055
	90	0.09	4.61	1.00934
	120	0.06	2.72	1.0060

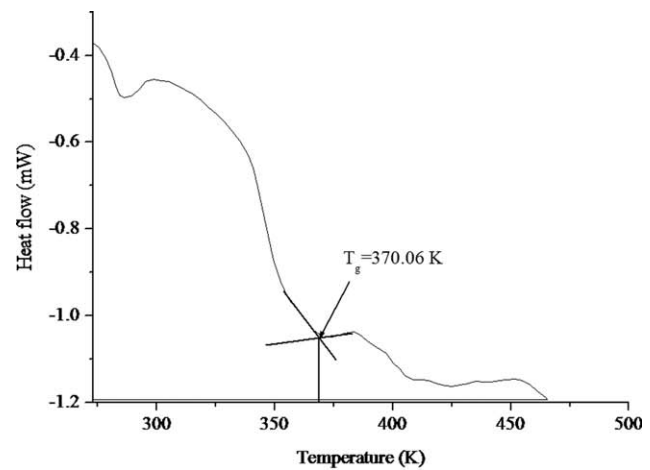


**Figure 4** TSDC spectra of 5% ZnO nanoparticle filled PS sample polarized at room temperature with different polling fields.

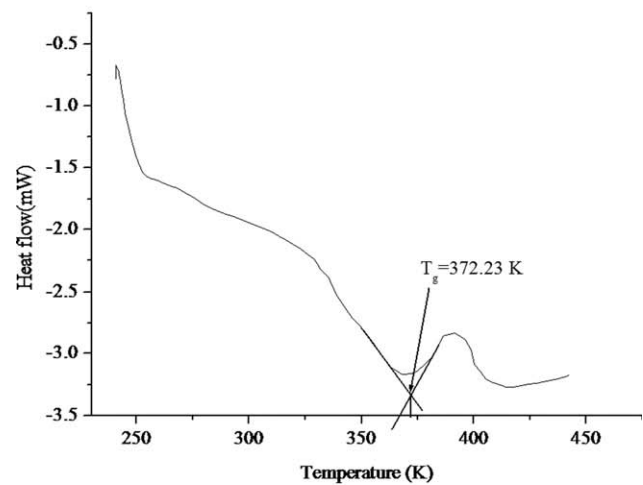
one related to ferroelectricity in crystalline phase and another to aligned dipoles in amorphous phase. These two components of polarization merge to-



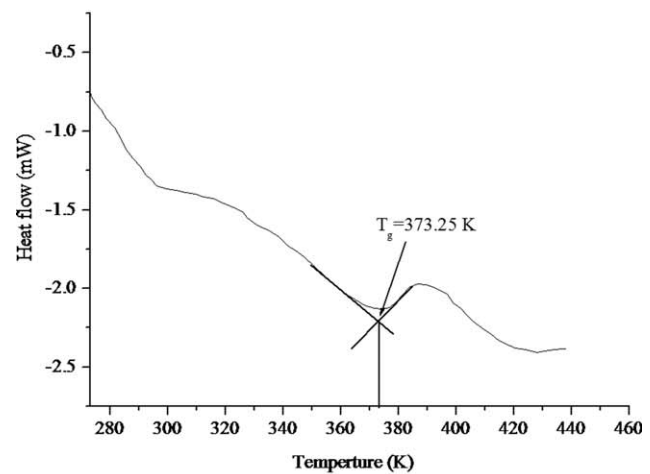
**Figure 5** (a) Charge released versus  $E^{1/2}$  for  $\alpha$  peak of TSC thermogramme. (b) Peak current versus  $E^{1/2}$  for  $\alpha$  peak of TSC thermogramme.



(a)



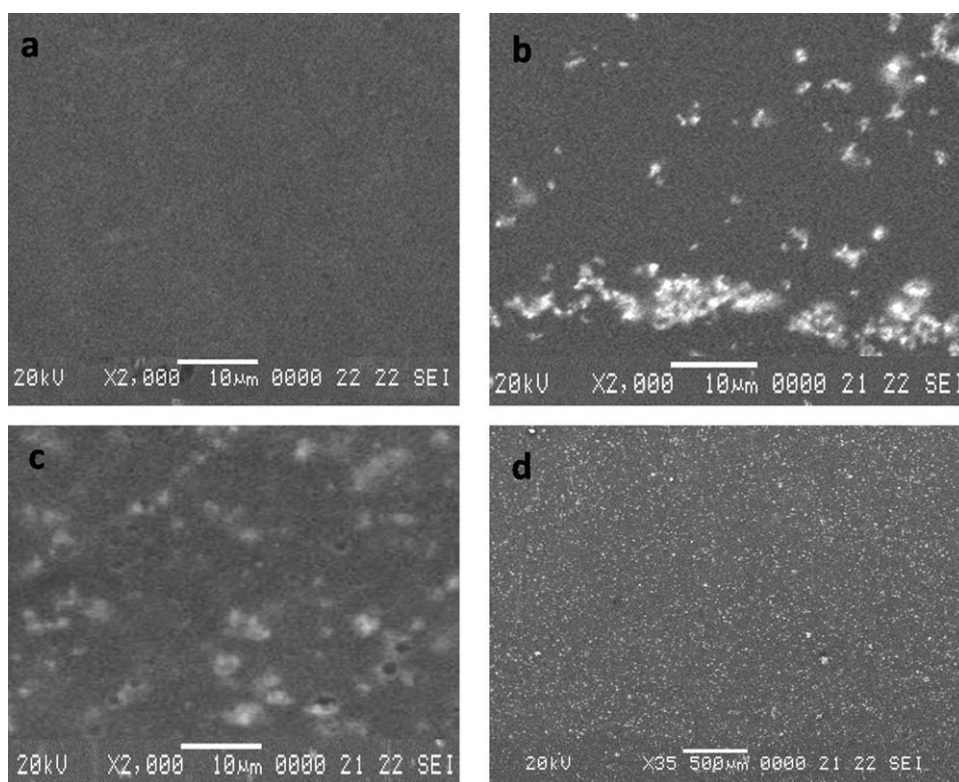
(b)



(c)

**Figure 6** (a) DSC thermograph of PS polymer matrix, (b) DSC thermograph of PS nanocomposite with 1% ZnO, and (c) DSC thermograph of PS nanocomposite with 5% ZnO.

gether in nanocomposites is the reason for appearance of single peak at a temperature above  $T_g$  of polystyrene. It is due to uniform dispersion of ZnO nano particles in polymer matrix. Although there is



**Figure 7** SEM Micrograph of PS and PS-1% ZnO, PS-3% ZnO, and PS- 5% ZnO nanocomposite samples.

no other experimental evidence except injection of charge carriers has been shown to be important in ferroelectric behavior.<sup>26–28</sup> It is remarkable that the TSDC peak is positioned at higher temperature in nanocomposites, indicating that the trapped charges are more stable than the pristine PS.

#### Differential scanning calorimetry

Thermal analyses of polymer nanocomposite samples have been investigated by using DSC technique. The DSC characteristics of pristine and ZnO nanoparticle filled PS are showing in Figure 6(a–c). These result shows that glass transition temperature ( $T_g$ ) increases with increase in concentration of nanofiller upto 3.19 K as shown in Figure 6. The role of nanoparticle in polymer matrix is to act as a plasticizer and hindering agent of molecular motion or mobility of side chain. This behavior of nanoparticle is responsible for increase of glass transition temperature of polymeric material as observed in nanocomposite samples. The interaction of ZnO with polystyrene creates the region of low mobility in the vicinity of the polymer-nanofiller interface. This region of the polymer nanocomposite exhibits a dominant effect on the average glass transition temperature, which help to increase the average glass transition of the polymer nanocomposite samples

due to the strong interaction of the nanoparticle with polymer molecules. This behavior is similar to that observed in poly (methyl methacrylate) (PMMA), and poly (2-vinyl pyridine) (P2VP) nanocomposites containing 10- to 15-nm-diameter silica nanospheres or 47-nm-diameter alumina nanospheres.<sup>29</sup> Wei et al.<sup>30</sup> reported that density of composites is larger than that of a pristine polymer; it will help to improve the glass transition temperature of the polymer.

#### Scanning electron microscopy

SEM micrograph of pristine polystyrene and ZnO filled polystyrene nanocomposite spectra have been recorded as shown in the Figure 7 with different weight concentration of nanoparticle. After introducing the nanoparticle in polymer matrix, we get a remarkable change in surface properties and texture of the PS + ZnO nanocomposite. Figure 7(a) shows pristine PS and Figure 7(b), 7(c) showing a forming of small clusters, and 7(d) shows a good dispersion in PS matrix. The surface roughing and crystalline texture appears to be decreased, which help in formation of smooth texture of the surface on increasing the concentration of nanoparticles. These changes may be attributed to the mixing of nanoparticle in polymer matrix in the reduction of crystallinity of host polymer.

## CONCLUSIONS

The following conclusions have been drawn from the present investigation. (1) PS-ZnO nanocomposite were prepared by sol-gel method and dispersion of nanoparticles were confirmed by SEM. (2) XRD results shows the polycrystalline nature of nanocomposite with decrease in crystallite size. (3) The optical energy band gap decreases and refractive index increases with the increase in concentration of ZnO nanofiller due to modification of surface and bulk properties. (4) The reduction of space charge and charge released causes due to the formation of clusters and blocking of charge carriers in amorphous-crystalline boundaries. (5) Glass transition temperature increases due to strong interaction of nanoparticle with polymer matrix. Finally, it is concluded that the PS-ZnO nanocomposite greatly enhances the charge storage ability and reduces the effect of space charges. These modifications in electrical properties of PS-ZnO nanocomposite suggested its suitability for charge storage devices.

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