Thermally Stimulated Depolarization Study in Polyvinylidenefluoride–Polysulfone Polyblend Films

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ABSTRACT: Thermally stimulated depolarization currents (TSDCs) in short- and open-circuit modes in polyvinylidenefluoride (PVDF)–polysulfone (PSF) polyblend have been recorded. The TSDC thermograms of PVDF and PSF in short-circuit mode show two peaks, whereas the polyblend of the two polymers shows a single peak. With the increase in PSF weight percentage in the polyblend, the magnitude of TSDC peak current increased and the peak current position shifted toward the lower temperature side. The single peak in polyblend appears at 165°C $\pm$ 10°C, which is at higher temperature than the temperature of low-temperature peak for individual polymers. This suggests that this peak may be due to dipolar polarization. Subsequently, shifting of peak toward higher temperature side with increase in polarizing temperature indicates the space charge peak. This contradiction has been explained on the basis of induced dipole theory. The behavior of short circuit TSDC could be explained in terms of the heterocharge caused by dipole orientation and ionic homocharge drift, together with the injection of charge carriers from electrodes and their subsequent localization in surface and bulk traps. However, two oppositely directed TSDC peaks observed in open-circuit mode in all the polyblend samples could be considered as the result of superposition of two overlapped and oppositely directed peaks, one caused by relaxation of dipole polarization and the other by the space charge. Thus, we have compared TSDC measured in open- and short-circuit modes to distinguish between these two relaxation processes and separate them. There is only one broad peak observed in the short-circuit mode of the polyblend, which entirely corresponds to the relaxation of dipole polarization. Insertion of a dielectric gap in the open-circuit mode does not affect the dipole current, but the space charge component flowing in the opposite direction is added to the former. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 118: 3715–3722, 2010

Key words: amorphous; interfaces; activation energy; polarization; induced dipoles

INTRODUCTION

There are many effects that ascribed to polarization, but many of these are not suitable because they reveal the results of different processes that take place at the same time and provide less information. In comparison to this, TSD current measurement yields several more or less distinct polarization bands,[1,2] which are in general due to individual depolarization mechanism. TSDC measurement is one of the important methods[3] for identifying and characterizing relaxation processes in electrified dielectrics and charged polymer electrets.[4–6] It also helps to understand the phenomenon of charge storage and charge decay process in electrets. The principle of TSDC is to orientate polar molecules or polar groups of macromolecules by applying a high electric field at an elevated temperature, and then the material is cooled to a room temperature under a constant field, which causes the molecular motion to cease. Subsequent heating causes the oriented dipoles to relax. This relaxation motion generates a depolarization current that relates directly to the molecular mobility and explores the internal physical and morphological structure of the material and therefore yields valuable information about the molecular interactions and the extent of mixing in polyblend. This technique can also reveal the information about the electrical behavior and structure of polymers, semicrystalline polymers, copolymers, and blends because it is a more sensitive alternative than other thermal analysis techniques for detecting the transitions that depend on changes in mobility of molecular-scale structural units.[7]

Several reports on TSC behavior of individual polymer thermoelectrets are available.[8–10] However, the nature of the various polarization processes and their relative contribution to the electret state of the polymer are not yet fully understood. This is particularly true for the space charge relaxation mechanism and the details of trap structure over the volume of the polymer. Such information can be

obtained by a combined study of short-circuit and open-circuit TSDCs. Most polymer materials possess several trapping levels for electrons and holes. The short-circuit TSDC cannot reveal them, but it is limited to yield information on bulk traps. More knowledge is gained by the combination of this method with the open-circuit TSDC. Short and open circuits are the two most widely used arrangements for the TSDC technique. In the former case, surface traps or near surface traps may not be detected because of the interface effect between electrode and sample surface. The electronic carriers in the electrode materials may be injected into the sample and produce TSDC peaks. In the latter case, there is no interfacial effect or extra carrier injection from the electrode and therefore the open-circuit technique is mostly used for dielectrics with real charge storage.

Previous reports of polyblend electrets have shown better charge storage than homopolymers. Polyblends are heterogeneous systems, and the possibility of charge storage at the trapping sites introduced by the grain boundaries of individual components is higher. Therefore, the attention has been paid to understand the modification in electrical properties thus brought about in molecular or morphological term. The two polymers differ remarkably in their glass transition temperature, that is, in their dynamical and structural properties. Polyblends of these two polymers are expected to yield strong and long-lived electrets useful for industrial applications. Keeping this in mind, detailed investigations have been carried out on the depolarization behavior of polyvinylidenefluoride (PVDF)–polysulfone (PSF) polyblend of different weight ratios as a function of the polarizing field, polarizing temperature, PSF weight ratio by the TSDC technique.

The polyblend samples were characterized by differential scanning calorimetric (DSC) technique, X-ray diffraction (XRD), and scanning electron microscopy (SEM) to study the formation of blend and microstructural properties of the materials.

THEORY OF TSDC

The thermally stimulated depolarization current (TSDC) technique has been widely used since its introduction in 1964 by Bucci and Fieschi.[11] This is also referred to as ionic thermal current technique and can be studied in the depolarization (TSDC) or in the polarization (TSPC) mode. In the TSDC mode, the sample with bound charge is polarized at a temperature T_p sufficiently high so that the relaxation time τ of the dipoles is short enough for the polarization associated with the dipoles to reach an equilibrium value P_o in a relatively short time. Then, the sample is cooled down to sufficiently low temperatures, and the external polarization field E_p is

switched off. Subsequent heating at a constant rate causes depolarization currents because of reorienting dipoles, which can be analyzed to yield the number of polarizable species, relaxation times, and their reorientation provided that the dipole moments are known.[12]

Bucci and Fieschi have developed a set of equations describing the depolarization current due to N_d reorienting noninteracting dipoles per unit volume, each with a dipole moment μ and reorientation energy E. The dipoles reorient with a unique characteristics relaxation time:

$$\tau(T) = \tau_0 \exp(E/kT_m), \qquad (1)$$

where $\tau(T)$ is the relaxation time at temperature T, τ_0 is the characteristic dipole relaxation time, E is the activation energy, k is the Boltzmann's constant, and T_m is the peak temperature.

If one has a partially oriented state at a temperature T, where the sample has a polarization P_0, and is heated at linear heating rate β in the absence of an electric field, then the depolarization current as a function of sample temperature, for a set of dipoles with a particular relaxation time τ, is given by[13–15]:

$$i(T) = P_0/\tau_0 \exp(-E/kT_m)$$
$$\times \exp\left[-1/\beta\tau_0 \int_{T0}^{T} \exp(-E/kT_m)dT' \right] \quad (2)$$

For the equilibrium polarization P_0 at the polarizing temperature T_p, the relation holds

$$P_0 = Nd\mu^2 E_p\alpha/kT_p \ \ \text{For } kT_p \gg E_p \qquad (3)$$

α is the geometrical factor and $0 < \alpha < 1$.

The discharge current versus temperature expression for the release of charge carriers from traps has the same form as Eq. (1) and differs only in the constant in front of the exponential.[15] So, the shape of the thermal current spectrum due to traps would be similar to that of dipoles. The low temperature tail of Eq. (1) is given by

$$\text{Ln } i(T) = \text{const} - E/kT_m. \qquad (4)$$

Thus, the slope of Ln i versus $1/T$ gives the activation of the discharge process. On differentiating Eq. (1) with respect to temperature and equating to zero (i.e., $di/dT = 0$), one obtains the temperature (T_m) where maximum current occurs. T_m is given by:

$$\tau_o = kT_m^2/\beta E \exp(E/kT_m) \qquad (5)$$

T_m was observed from I versus T plots, we can calculate τ_o and by putting the value of τ_o in Eq. (2), the relaxation time τ at T_m and at any temperature can be easily calculated.

EXPERIMENTAL

The films used in this investigation were prepared using the solution grown technique. The commercial PVDF (Solef 1015 PVDF Powder) and PSF (UDEL P1700 PSF Pellets) used for this study were procured from Solvay Corp., Belgium and supplied by Redox (India). The solution of particular concentration was prepared by dissolving the two polymers in different weight ratios in a common solvent, that is, N,N-dimethylformamide at 50°C for 1 h and then kept at room temperature for 2 h to become homogeneous. The cleaned optically plane glass plate at a constant temperature of 30°C was immersed in the solution for about 90 min. The plate was then slowly drawn out of the solution, leaving a uniform polymer film on the plate. The dried samples were subjected to room temperature outgassing in air at 60°C at 10^{-5} Torr for a further period of 12 h to remove any residual solvent. Polymer film is then gently pealed off from the glass plate. Polyblend films of different PVDF–PSF weight ratios were prepared. The circular shaped samples were prepared having diameter 5 cm and 50 μm thickness. For good ohmic contact, the surfaces of the samples were vacuum aluminized using Hindhivac Vacuum coating unit with Penning and Pirani pressure gauges, ST-A6P3, over central circular area of diameter 3.5 cm. One-sided vacuum-aluminized samples (unilaterally) have been used for open-circuit TSDC measurements, whereas both-sided vacuum-aluminized samples (bilaterally) samples were used in short-circuit experiments.

The polymer samples were thermally polarized at different polarizing temperature and electric field. The sample holder forming metal-polymer-metal system was placed in a digital thermostatically controlled oven heated up to polarizing temperature and maintained constant for half an hour. The desired strength of electric field was applied for 1.5 h at polarizing temperature. The sample was then allowed to cool down at room temperature in the presence of applied field. Thus, total polarization time was taken to be 2.5 h for all samples. The polarized samples were kept shorted for an arbitrary time of 20 min so as to remove the frictional and stray charges. The short-circuit TSDC was then recorded by reheating the samples at a linear rate of 3°C/min. For measuring TSDC in open circuit, the polarized samples were mounted in an electrode assembly with the nonmetallized surface parallel to the sensing electrode at a distance of 2 mm, while the metallized surface rested on the other metal electrode. A high-voltage dc power supply (Scientific Equipments, Roorkee, EHT-11) provided stabilized DC voltages for polarization, while TSDC was measured using a sensitive digital electrometer (Scientific Equipments, Roorkee, DPM-111, India).

X-ray diffractometer having CuKα radiation (X-1.5418Å) by Rigaku, RU-200 Powder Diffractometer at room temperature, was used in structural characterization. The 2θ range for all the polymers was 2°–60°. The particle size (D) was calculated using Scherrer's formula[16] by using the full width at half-maximum (FWHM) intensity of the XRD pattern can be described by:

$$D = 0.89\lambda/\beta \cos\theta, \qquad (6)$$

where $\lambda = 1.5418$ Å (Cu Kα) and β is FWHM at the diffraction angle of θ.

SEM was performed on LEO-435-VP, variable pressure SEM. Samples were sputtered coated with gold before testing.

DSC was performed for all samples with Perkin Elmer Differential Scanning Calorimeter-Pyris 6 DSC in an inert atmosphere of argon flowing at the rate of 250 cc/min, which has an accuracy of ± 2°C. The rate of heat flow from 50 to 250°C was 3°C/min.

RESULTS AND DISCUSSION

The characteristics of short-circuit TSDC thermograms for PVDF and PSF polarized at various temperatures with polarizing field of 40 kV/cm are shown in Figures 1 and 2, respectively. PVDF is a
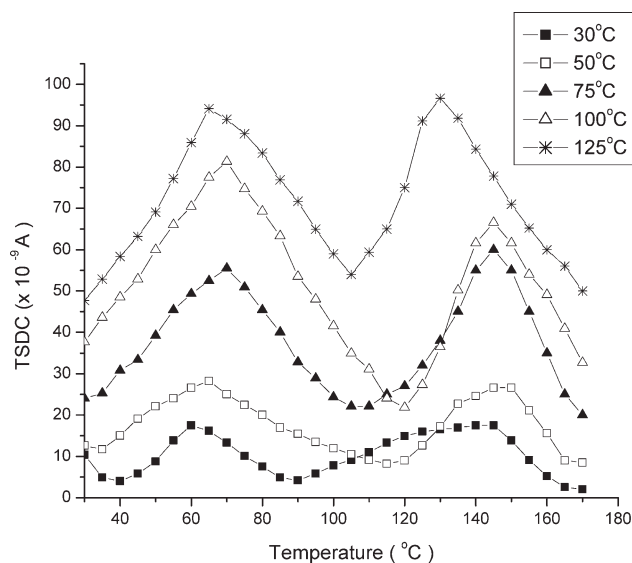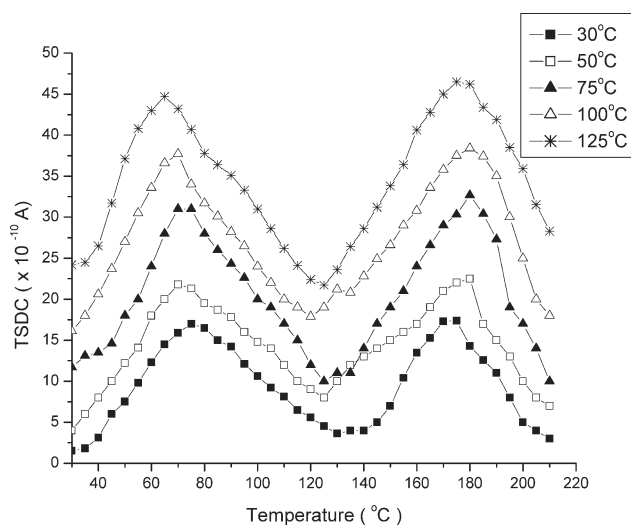

Figure 1 TSDC thermograms of PVDF samples polarized with polarizing field (E_p) of 40 kV/cm at different polarizing temperature (T_p) (a = 30°C, b = 50°C, c = 75°C, d = 100°C, and e = 125°C).

Figure 2 TSDC thermograms of PSF samples polarized with polarizing field (E_p) of 40 kV/cm at different polarizing temperature (T_p) ($a = 30°C$, $b = 50°C$, $c = 75°C$, $d = 100°C$, and $e = 125°C$).

polar and semicrystalline polymer. Its contribution to the polarization may be due to alignment of dipoles and formation of space charge/injection of charge carriers from metal electrode under the effect of the electric field. Two TSDC peaks are observed for PVDF.[17] The first peak (β-peak) at 65°C ± 10°C and second peak (α-peak) centered on 140°C ± 10°C are due to the release of charges trapped at crystalline/amorphous boundaries. The activation energy for all β-peaks varies for polarizing temperature-dependent TSDC thermograms. With increase in polarizing fields and temperatures, the magnitude of peak currents was found to be increase. The α-peak in PVDF shifts toward lower temperature side with increasing values of polarizing temperature, whereas it shifts toward higher temperature side for PSF.

In PSF, the two TSDC peaks appear below 185°C. The first peak (β-peak) at 80°C ± 10°C, which is associated with dipolar relaxation occurred almost at the same temperature with change in field, and second peak (α-peak) centered on 185°C ± 10°C that appears because of space charge relaxation and shifted to higher temperatures with increasing field strength. β-peak in polar polymers mainly arises from localized rotational fluctuations of the dipoles; it may be due to the movement of ions, side chain, and formation of dipoles referred to as dipolar relaxation.[18–20] The β-relaxation is associated to the dipole orientation of the polar side group (CH_2—CF_2) and sulfone present in PVDF and PSF, respectively. The occurrence of β-relaxation can be discussed from calculated value of activation energy associated with this peak and is very close to theoretically predicted value, that is, $\sim$ 0.30–0.50 eV in two polymers.[9] Further, the peak height increases almost linearly

with an increase in poling parameters. All these findings supported the dipolar orientation mechanism for β peak, whereas α-relaxation is related to combined dipolar and space charge mechanism.

The α-peak is contributed by large injection of charge carriers from the electrodes to the surface of the polymer, which are frozen-in during poling. The charge trapping in a polymeric material is the general behavior of polar polymers like PVDF and PSF. It takes place at the many possible regions of polymer structure such as at the molecular chain, side chain, and at the crystalline and amorphous interfaces.[21] The new trapping sites produce because of application of high DC field and temperature in a polymer matrix. Therefore, it is reasonable to understand that the peaks appearing in high-temperature region in both the polymers are originated because of space charge relaxation process, which occurs because of injection of charge carriers from the electrodes at the metal–polymer interface. The origin of α-relaxation process in PVDF is also confirmed by getting the activation energy[21] for this peak (i.e., 0.7–1.2 eV), which does not differ much for activation energy values reported for α-relaxation in many polymers.[22]

Short-circuit TSDC thermograms of different polyblend at different polarizing field and temperature are characterized by a single peak. The appearance of single peak at 165°C ± 10°C in polyblend could be explained on the basis of induced dipole theory, according to this theory during polarization, the charges originating from the bulk of the material get trapped in deeper traps and thus form induced dipoles. Later, these induced dipoles get aligned in the polarizing field.[23–25] It is, therefore, possible that the dipolar peak either gets covered by the space charge peak or its contribution to the total polarization is very small. The current magnitude is found to decrease for low field values but then increases with an increase in the polarizing field. The peak height increases linearly with an increase in field and suggests dipolar uniform polarization.[26] Thermal activation at a constant rate causes the release of charges from different trapping sites because of their mobilization giving a peak at the site of maximum release of charge. The dependence on the polyblend composition of the short-circuit TSDC is depicted in Figure 3. It can be seen from the figure that for polyblend containing a lower weight percentage of PSF, the depolarization current is high. However, as the PSF content is increased, the current is increased in magnitude. From the observed thermograms, it is also found that the current maxima move to low-temperature side with the increase in PSF in the polyblend composition. The activation energy, charge released, and relaxation time for the various peaks were evaluated with the initial rise method of
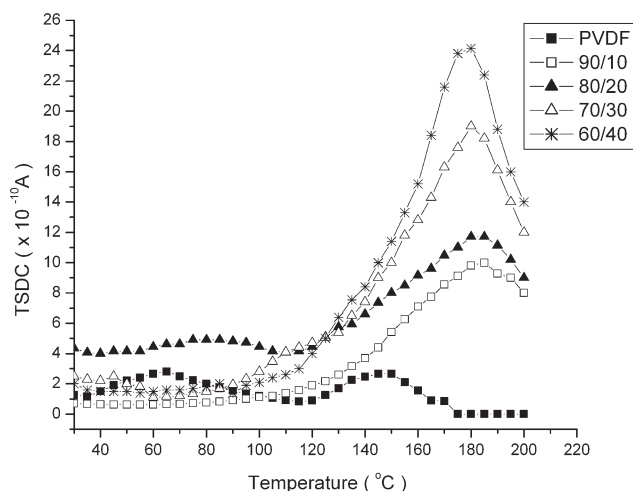
Figure 3 Short-circuit TSDC thermograms of PVDF–PSF polyblend polarized with polarizing field (E_p) of 40 kV/cm at polarizing temperature (T_p) of 50°C.

Garlick and Gibson.[27] The calculated activation energies of the order of 0.60 eV is an evidence that the peak is not only due to the dipolar polarization. There seems to be two possibilities for this sort of behavior, that is, (i) addition of PSF in PVDF produces heterogeneous structure and plasticization effect, which causes the injection of charge carriers from metal–polymer interface into the bulk and their deposition at the phase boundaries and (ii) plasticization effect enhances the mobility of charge carriers, which resulted the increase of peak current.

In polyblend samples, at least two phases (i.e., amorphous and crystalline) coexist and produce discontinuity of structure at amorphous and crystalline boundaries with increase in concentration of PSF. As carrier conductivity is different in different phases, therefore, the carriers get trapped at the interphase of the phase boundary. This trapping of charge carriers will originate induced dipoles, because positive and negative charge carriers are suppose to be trapped in deeper levels. Such trapped charge carriers may be generated in bulk because of ionization of the impurities or may get injected in the bulk from the impurities. Thus, they lead to induced dipole polarization in the polyblend. It is also possible that contribution of a permanent dipole of the host material is masked by the peak because of induced dipoles.

The dipoles of the polar components in the polyblend are so constrained under the field of polarization, thus the contribution of these dipoles into total polarization is very small when compared with the contribution of the induced dipoles. In fact the higher value of E_p is taken as an indication of induced dipole formation because in this process the energy required in disorienting the charge carriers and for their subsequent release from the trapping

sites is expected to be higher. As the PSF concentration in the polyblend is increased, the increase in current and the shift in the current maximum toward a low temperature can be ascribed to an increase in the carrier mobility and/or an increase in the mobile carrier densities due to an increase in shallow traps. The blending of PVDF with PSF thus brings about a definite change in the structural morphology of PVDF. A qualitative explanation of these effects can be given under the assumption that the amorphous regions of PSF act as trapping centers, whereas crystalline surface regions become deeper traps. It appears that in polyblends, with a sufficient number of available deep surface traps, the injected charge is located in the crystalline surface regions.

The amorphous region in polyblend increases with increase in PSF concentration. Therefore, carrier mobility is thus enhanced by the hopping centers in the amorphous region, and more charge is localized in the shallow traps. The detrapping of a large number of charges from these traps results in partial blocking of the electrode polymer interface and reduces the carrier mobility, giving TSDC. The peak temperature and the magnitude of the peak current depend on the weight percentage of polymers and hence morphology (or the crystallinity) of the polyblend.

To understand the role of surface trap in TSDC, the open-circuit TSDC is recorded. The open-circuit TSDC thermograms for polyblend samples are shown in Figure 4. Initially, the current is anomalous, that is, flowing in the same direction as the charging current at lower temperatures; however, change of sign and start to flow in a direction opposite to the charging current or is negative called normal current. The low temperature relaxation
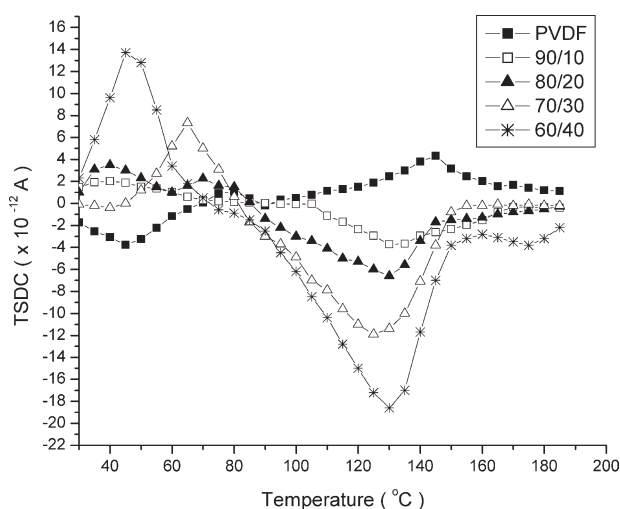

Figure 4 Open-circuit TSDC thermograms of PVDF–PSF blend samples polarized with polarizing field (E_p) of 40 kV/cm at polarizing temperature (T_p) of 50°C.
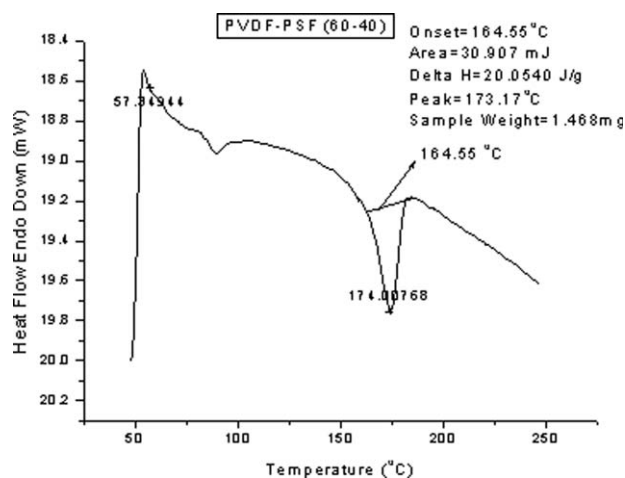
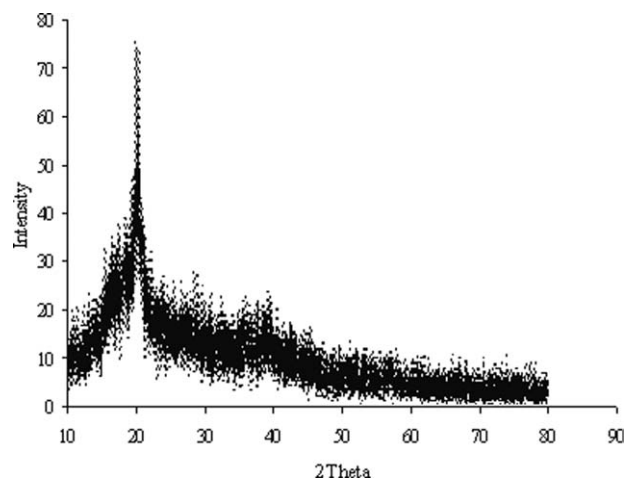**Figure 5** DSC curves of PVDF–PSF polyblend (60–40 wt %).

Figure 6 XRD pattern of PVDF–PSF polyblend (60–40 wt %).

observed in the TSDC measurement of the polyblend can be attributed to dipole orientation taking place at the glass transition temperature of the homogeneously mixed phase. The dipoles become aligned in the direction of electric field and produce such effects in the lower temperature region; on the other hand, the high-temperature peak can be attributed either to drift of surface charge into the samples or due to neutralization by intrinsic charge carriers. Further, the temperature of polarity reversal increases with the increase in polarizing temperature. In open-circuit TSDC thermograms, increase in the magnitude of current and shifting of peak toward low temperature side with the increase in concentration of PSF in the polyblend is observed. The activation energy calculated for the first peak ranges from 0.25 to 0.67 eV, and for second peak it varies from 0.32 to 0.81 eV. The current of the second peak also increases with increasing E_p, but the position of this peak tends to shift toward lower temperature side. Two peaks in the open-circuit mode can be considered as the result of superposition of two overlapped and oppositely directed peaks, one caused by relaxation of dipoles and the another by the space charge. Thus, the two TSD currents in open- and short-circuit geometry have been found to be relevant enough to distinguish between these two relaxation processes. The single broad peak observed in the short-circuit mode of the polyblend samples. It is reasonable to presume that this peak entirely corresponds to relaxation of the dipole polarization[15,28] as discussed above. Insertion of a dielectric gap in the open-circuit mode does not affect the dipole current, but the space charge component flowing in the opposite direction is added to the former. Therefore, if $I_p(T)$ and $I_c(T)$ are two components of the total TSD current, then the resulting current in the short- and open-circuit modes can be presented as

$$I_{SC}(T) = I_P(T) \ \text{ and } \ I_{OC}(T) = I_P(T) - I_C(T) \quad (7)$$

From Eq. (7)

$$I_C(T) = I_{SC}(T) - I_{OC}(T). \quad (8)$$

It is observed that discharge current is the function of PSF content as it is evident from Figure 4 may be understood in terms of increased charge formation. Because of the in-built heterogeneous structure of the polyblend, charge carriers may pile up at the phase boundaries. The observed value of activation energy can also be considered as an indication of induced dipole formation because in this case the energy required to disorient the charge carriers and for their subsequent release from the trapping sites is expected to be higher.
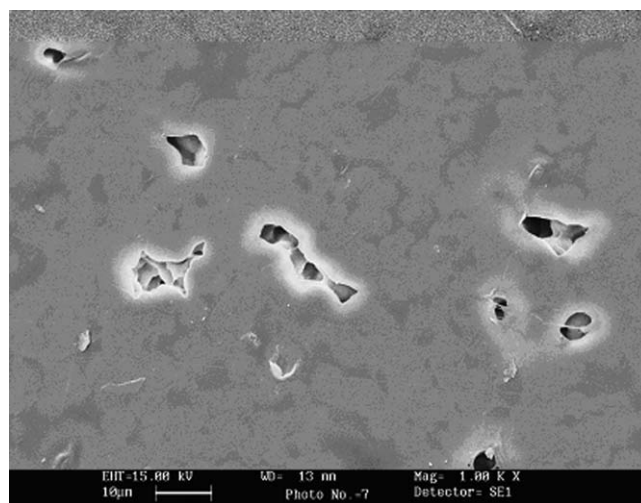

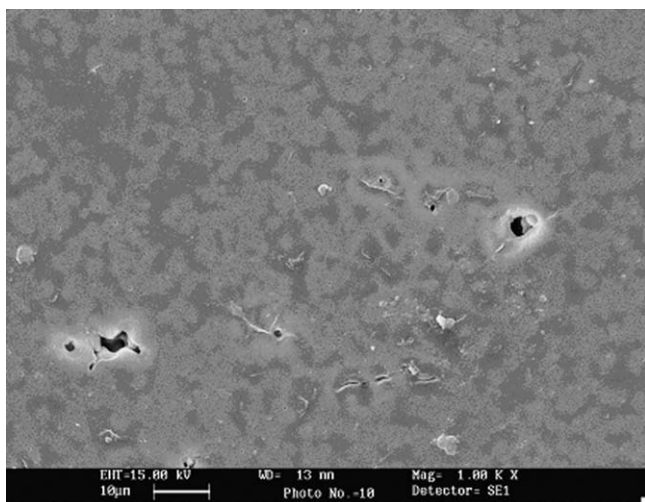**Figure 7** SEM micrograph of PVDF–PSF polyblend (80–20 wt %).

Figure 8 SEM micrograph of PVDF–PSF polyblend (60–40 wt %).

The direction of the open- and short-circuit currents in polyblend is same as that of the neutralizing current of homocharge up to a certain temperature region. The observed results of polyblend have indicated that the polarization phenomena in this case are due to the existence of heterocharge, dipole orientation, and ionic homocharge drift together with the injection of charge carriers from electrodes and their subsequent localization in surface and bulk traps. These results are found similar with the results obtained from short-circuit measurements except the polarity reversal observed in open-circuit TSDC measurements of polyblend.

The DSC curve was obtained by taking the onset of the heat capacity versus temperature curve measured at 3°C/min heating rate as shown in Figure 5. The presence of a single peak confirms the miscible behavior of the polyblend. It is also observed from the DSC curves that the temperature of the α-peak maximum, that is, high-temperature peak in TSDC curves of polyblend samples is usually observed at the polymer/blend's glass transition temperature, is very close to the glass transition temperature measured experimentally by DSC.

The blend samples were characterized by XRD to study the formation of polyblend. Only representative characteristics (i.e. 60 : 40 wt %) is shown in Figure 6. The important results of XRD characteristics are (i) β-phase PVDF is used in present study, (ii) the polyblend sample shows single sharp and other broad peeks of negligible height, (iii) when the PSF introduced in PVDF matrix the other peaks of PVDF is almost disappeared or of very small height and (iv) peak height gradually decrease with increase in PSF contents. These results confirm the decrease in crystallinity and particle size with increasing amorphous content in the polyblend.

It is also observed in XRD pattern that, with increase in PSF content in the polyblend, the intensity of peak is shifted toward left side that confirms the behavior of the polyblend and shows the increase in amorphous region. According to Lovinger,[29] PVDF presents Bragg peak positions at 2θ equal to 20°, 27°, and 40°. The XRD of polyblend shown in Figure 6 presents the characteristic diffraction pattern, indicating that the polymer blend was successfully formed. As we know that smaller crystals produce broader XRD peaks. Thus, crystallite size was calculated using Scherrer's formula[30] by FWHM and found to vary from 187 to 104 Å, and peak broadening with decreasing crystallite size confirms the reduction in particle size with the increase in PSF content in the blend.[31,32]

The SEM images of polyblend in Figures 7 and 8 of different weight ratios show that the amorphous phase of PSF is partially filling the original porous spaces of the pure PVDF, affecting the size or the imperfection of the polymer crystal. The presence of PSF to the PVDF matrix appears to modify the polymer grain formation process, altering the size and morphology of the pores.

CONCLUSIONS

The electret behavior of polyblend samples is significantly affected by blending of PSF with PVDF because of change in structural morphology. The PSF enhances the amorphous content in polyblend and modified the trap structure due to which the large numbers of charge carriers are localized in shallow traps. The detrapping of carriers in large numbers from such sites results in a discharge current with increasing PSF content has been explained on the basis of induced dipoles created because of the piling up of charge carriers at the phase boundary of heterogeneous structure of polyblend and increase in mobility of charge carriers due to plasticization effects.

The numbers of peak in short- and open-circuit TSDC and their various characteristics have indicated that traps are distributed over wide energy range. However, well-defined and nonoverlapping features of these peaks indicated that polyblend contains the trapping level of different depth. The comparative analysis of two types of TSDC has helped to determine the geometrical surface trap structure of the polyblend. It has been concluded from the observed activation energy that the surface traps are energetically shallower than the bulk traps.

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