Study the Effect of Thermally stimulated discharge current on polar Polysulfone and Multiwall Carbon Nanotube Nanocomposite

Singh Rekha and Tiwari R. K.
Department of Physics, Jiwaji University-474011, Gwalior (M.P.), India

Abstract: The thermally stimulated discharge current (TSDC) has been observed in 50µm thickness of pure Polysulfone and Multiwall Carbon Nanotube nanocomposite. PSF/MWCNTs nanocomposite film were prepared by solution mixing of different weight ratio (0.25, 0.5 and 1). In TSDC spectra there were one relaxation peak observed and the magnitude of these peaks increases with increase in weight ratio of MWCNTs due to strong interaction of organic and inorganic component. The height of TSDC observed as a polarizing field. Height of TSDC peaks are increased in pure polar polysulfone while the peaks were more increasing with different weight ratio of MWCNTs due to amount of space charge is smaller in the MWCNTs nanocomposite rather than pure polar polysulfone. The surface morphology of pure PSF and PSF/MWCNTs nanocomposites at micro level were studied by SEM. The XRD characteristic confirms the formation of PSF/MWCNTs nanocomposites.

Keywords: TSDC; Nanofillers; MWCNTs and activation energy

I. Introduction

In recent years thermally stimulated discharge current is very well known and useful technique for investigation of various polymeric electret parameters such as activation energy, charge released, relaxation time and many more in the field of polymer nanocomposite. This technique has been reported during the past few years in the field of polymeric materials. Electret were first discovered by E. G. Sessler in 1919 and named thermo-electret. In year 1980 G.M. Sessler gave the electret definition and he defined the insulating or dielectric material exist a quasi-permanent electric charge. Several processes contribute to the discharge of electrets, but the driving force of them is the restoration of charge neutrality. The TSDC thermogram of PSF/MWCNTs nanocomposite film will consist of one peak, because the dipoles with low activation energy will disorient at low temperatures, while those with high activation energy will disorient at higher temperatures. If the differences in the various activation energies are not large, it is more appropriate to assume a continuous distribution of activation energies, for which all individual peaks overlap and merge into a broad peak. Such broad peaks are often seen as a result of disorientation of polar side groups in polymers at low temperatures i.e. α- peak. Another possible cause for the appearance of broad peaks is a difference in the rotational mass of dipoles. These differences occur in e.g., a polymer when heated to its softening temperature, where the dipoles are disoriented by the motion of main chain segments.

II. Experimental Method

The Polysulfone was procured by Redox (India) and Multiwall carbon nanotubes (i.e. length 50 µm, inner diameter 10 nm, surface area 200m²/gm) were supplied by Reinste Nano Ventures Pvt. Ltd, New Delhi. The dimethyl formamide (DMF) was used as received from Merck India Ltd. The pristine polysulfone and nanocomposite sample were prepared by solution mixing method. For the 0, 0.25, 0.5 and 1.5 wt. % MWCNTs concentration (weight fraction concentration for a total 5 g of PSF/MWCNTs, First, given amount of MWCNTs was dissolved in N-N, dimethylformamide by sonication for 25 min. in a sonicator. Then required amount of PSF dissolved in dimethyl formamide was added to MWCNTs dispersed solvent and the mixed solution was stirred and sonicated for 30 min. stirring and sonication were done in 5 min interval. Thin films of average thickness 50–60µm were prepared by casting the solution and evaporating the solvent at room temperature for overnight to remove solvent completely. For the thermally stimulated discharge current, samples were vacuum aluminized over the central circular area of 1.4cm diameter using high Vacuum Coating Unit (Vacuum Equipment Company Ltd, Noida, India).

III. Result and discussion

A. SEM analysis

Figure 1 shows that the effect of carbon nanotubes in PSF matrix were clearly observed. Figure 1B clearly shows that carbon nanotubes were present in PSF matrix and the dispersion of CNT was uniform. In SEM Figure of pure PSF the pores of PSF sample were easily seen. However, increasing of wt% of CNTs the size of pores of
PSF were reduced. It clearly shows in SEM image. In Fig 1(a and 1b) the structural density increased with increasing of wt% of CNTs.

B. X-ray Diffraction
XRD results Fig.2 also reveal the structural features of the Pure PSF, Pure CNT and different wt% of PSF/CNT nanocomposite. The diffraction peaks in XRD pattern at 19.0, 25.0 and 45.0° indicating a good crystallinity of the supported nanoparticles. The broad peaks in the XRD pattern indicate that the PSF are small. The average crystallite size of PSF was calculated using the Debye–Scherrer formula

\[ d = \frac{k \lambda}{\beta \cos \theta} \]  

(1)

in which \( d \) is the crystallite size, \( \lambda \) the wavelength of the X-ray radiation, \( K \) is usually taken as 0.89, \( 2\theta \) is the Bragg angle of the PSF (111) peak, and \( \beta \) is the full width at half-maximum height. The diffraction peak at 2 \( \theta \) = 25° is related to the presence of carbon nanotubes with graphite structure. The diffraction peaks in XRD pattern indicating a good crystallinity of the supported nanoparticles.

C. Short Circuit Thermally Stimulated depolarization current study
Short-circuit TSDC characteristics are represented PSF /MWCNTs nanocomposite samples polarized at room temperature with different polarizing fields \( (E_p) \) s (a)2kV/cm,(b)4kV/cm and (c)10kV/cm respectively. It is observed from the various TSDC characteristics of nanocomposite samples are characterized by a single peak located at 145±10°C. For TSDC characteristic obtained polarizing at room temperatures, the depolarization current flows in the normal sense (i.e. the current is anomalous i.e. flowing in the same direction as the charging current and if changes sign and starts flows in a direction opposite to the charging current or is negative called normal current). For nanocomposite containing a lower weight percentage of MWCNTs, the depolarization current is small enough. However, as the MWCNTs content is increased, the current is increased in magnitude. From the observed characteristics, it is found that the shift in the temperature scale of the current maxima moves to low temperature side with the increase in nano-composition. Our TSDC result suggesting relaxation is the main mechanism of TSDC. For \( \alpha \)-relaxation processes, the activation energy more than 0.9eV. However, calculated activation energy for nano-composite samples is comparatively greater than pristine PSF sample. The electret behaviour of nano-composite samples is significantly affected by pure PSF and PSF/MWCNTs nano-composite because of change in structural morphology, which influences greatly the electrical behavior of the polymer. The PSF enhances the amorphous content in nano-composite and modified the trap structure due to which the large numbers of charge carriers are localized in shallow traps. With increasing MWCNTs content in the nano-composite, the modified trap structure where the large number of charge carriers get trapped, get detrapped and result in producing a discharge current. This phenomenon 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 blend and increase in mobility of charge carriers due to plasticization effects reported by Arthur (2003). The mixing between the polymers nanocomposite is such that it results in the increase of intermolecular interaction which reduces dipolar contribution towards the total polarization. The polarization in the nanocomposite is mainly due to induced dipoles. The numbers of peak in short-circuit TSDC and their various characteristics have indicated that traps are distributed over wide energy range.

IV. Conclusion
In this study, amorphous polysulfone (PSF) filled with multiwalled carbon nanotube (MWCNTs) nanocomposite of different wt% were prepared by solution grown technique. SEM image shows the morphology of uniform dispersion of PSF filled with different wt% of MWCNTs nanocomposite. In short circuit TSDC characteristics obtained from both sized metalized polymer nanocomposite electrets have led to the conclusion that the peak located at around 145±10°C can be attributed to dipolar relaxation. With increase in polarizing fields and temperatures, the magnitude of peak current was found to be increased. The \( \alpha \) peak occurred almost at the same temperature. The various other features of this peak further led to the conclusion that this peak is also contributed by space charge polarization produced due to macroscopic displacement of bulk generated charge carriers as well as due to injection of the charges into the bulk of the sample with subsequent trapping during polarization.

V. Acknowledgements
The authors Rekha Singh and R.K.Tiwari are gratefully acknowledge to Dr.M.S. Gaur for providing TSDC facility.
References


Fig. 1: SEM image of PSF /MWCNTs nanocomposite samples

Fig. 2: XRD spectra of MWCNTs pure, PSF pure and PSF /MWCNTs nanocomposite samples
Fig. 3: Short circuit TSDC characteristics of PSF/MWCNTs nanocomposite samples polarized at RT with different polarizing fields ($E_p$'s) (a) 2kV/cm, (b) 4kV/cm and (c) 10kV/cm.