INVESTIGATION OF CHARGE CARRIER TRANSPORTATION PHENOMENON IN POLYMER COMPOSITES THIN FILMS

A

SUMMARY

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1.1 Summary

In recent times studies in polymer composites in the form of thin films have gained more attention, due to their fascinating properties, disordered structure, potential applications and growing need of solid state devices. Polymer composites thin films are being used in a number of exploratory electronic technologies. The polymer properties depend on average molecular weight and molecular weight distribution of the polymer. Any two polymer samples can have the chemical structure and almost similar molecular weight distributions, but may have different properties due to difference in their structure. All properties depend on inter-molecular and intra-molecular interactions of polymer chains in the aggregated state. Such interactions are caused by forces, such as dispersion force, induction force, dipole interaction and hydrogen bonding, acting within various parts of the same molecule as well as between the neighboring molecules.

The process of transport of carriers in macromolecular compounds involves a kind of intermediate state generally called ‘trap’. If a charge carrier falls into such a trap, it is captured and is no longer free for a certain time and its probability of recombination with a carrier of opposite sign is increased greatly. However, it may also escape from the trap after receiving a certain amount of energy, which is known as trap energy or activation energy. If the carrier is captured in deep traps, there are very few chances of detrapping while the chances of escape of carriers trapped in a shallow trap are higher. There is also a probability that the carriers once released from a shallow trap may be retrapped at deep levels. The net effect is that charge is effectively transported from one locality to another. Hence the knowledge of trap parameters like density, distribution and depth etc., required to understand the mechanism of charge and energy transfer in polymeric systems become important. In the present case different studies with PVK and ferric chloride doped PVK thin films were carried out.

Chapter-I contains the brief history, classification and glass transition temperature of polymers and polymer composites. In this chapter origin of traps in dielectric materials, preparation and classification of electrets are discussed. This chapter also describes the different techniques of polymer film formation like sputtering, pulse laser deposition, solution grown technique etc. In present study, solution casting technique is adapted for thin film formation on plane glass substrate and film thickness is controlled by selection of solution concentration. This chapter also focuses on charge carrier generation in polymer films and various applications of polymer thin films.
Chapter-II deals with theoretical background of studies. Basically, this chapter covered different conduction mechanisms, which are responsible for conduction in dielectric materials. In present case Poole-Frenkel mechanism seems to be mainly dominant for conduction in poly (9-vinylcarbazole) and ferric chloride doped poly (9-vinylcarbazole) samples. The chapter also deals with the theory behind thermally stimulated discharge current, and some formulae for determination of activation energy, total charge released and dipole relaxation time. Spontaneous current emission is also dealt in this chapter using VIJH model.

Chapter-III, describes the experimental details of the present work. Thin film formation methodology is explained which is used in present study. Chloroform is used as a solvent in present case, and mercury pool is used for uniformity in thickness of deposited film on glass substrate. Thickness of the film is monitored by selection of solution concentration. The Xrd of investigated thin films proved the amorphous nature of material, which is also reported in literature [1]. With the help of differential scanning calorimetry (DSC), the glass transition temperature of used material is found to be 155 to 165 C for pure and doped PVK which is less than reported value.

Chapter-IV, comprises of the dc-conductivity study of 2% ferric chloride doped PVK samples. LnI versus LnV plots show linearity at low field region whereas at high field the slopes reached 1.5 approximately for all temperatures. From slopes of linear part of Schottky plots the experimental value of $\beta$ is calculated. In the present case experimental value of $\beta$ is very close to theoretical value of $\beta$ suggesting Poole-Frenkel mechanism. The activation energy is determined by Arrhenius plots which is found in between 0.207 eV to 0.195 eV and may be due to typical electronic conduction, as the values less than 0.8 eV are normally considered to be due to electronic conduction mechanism, while the activation energies above 1 eV are attributed to ionic conduction.

Chapter-V, describes the thermally stimulated discharge current studies for pure and ferric chloride doped PVK. Thermo-electrets are formed at poling temperatures 50 C, 100 C and 150 C for a set of poling fields 25 kV/cm, 50 kV/cm and 100 kV/cm. TSD current $I(T)$ is recorded as a function of temperature varying from 40 C to 170 C in steps of 5 C. TSDC thermograms show two peaks, first peak($\beta$) around 95 C to 105 C and second peak, $\alpha$ near transition temperature 150 C to 160 C for all poling temperature with a set of poling fields $E_p$ for pure, 1% and 2% ferric chloride doped PVK. Peak current linearly increases with poling field at fixed poling temperature $T_p$ and increasing trends are recorded with increase
Initial rise plots for different peaks of TSD thermogram corresponding to each poling temperature for a set of poling fields are drawn. The slopes of plots give the activation energy corresponding to peak for fixed poling temperature and poling field. Normally it is seen that activation energy decreases with increasing poling fields as well as polarizing temperatures. Due to increase in doping concentration activation energy is reduced with increasing fields and temperatures. When an electret is heated to obtain the TSD current, which generally appears near $T_s$, is either due to randomization of the oriented dipoles or due to release of charge carriers from traps. Generally polymers contain a small number of free charge carriers, i.e., ions, electrons or both [4, 5]. During electret formation, the carriers move comparatively free in the direction of the applied field over microscopic distance before they fall into deep traps from which, they are released only on receiving sufficient energy. Due to heat of samples at constant heating rate cause the release of charge from their trapping centers, giving a peak at the site of maximum release of charge [6]. The peaks are more pronounced at higher values of $E_p$. The depolarizing current is supposed to comprise of two components behaving differently as a function of temperature [7-9]. The first one is the deorientation of dipoles resulted the formation of a peak, whereas the second component is the conduction current derived from the motion of equilibrium charge carriers increasing continuously with temperature. The large area under the $\beta$ peaks than $\alpha$ may be due to the excess of dipoles in side chains than backbone chain. The linear field dependence of peak current suggests that the TSDC spectra may be either due to dipolar origin or migration of charge carries through microscopic distances with trapping [10]. Total charge released during each TSD cycle normally increases with inclusion of impurity as well as increasing poling fields and temperatures (shown in table 5.1 to 5.3). The linear dependence of charge released and the peak current on polarization field strength is a characteristic of dipolar origin whereas in the case of space charge polarization, the peak current and charge released will show a non-linear variation with polarizing field strengths.

Chapter-VI, displays the study of spontaneous current emission (SCE) of pure and doped poly (9-vinylcarbazole) (PVK) sample of thickness 55 $\mu$m. The samples were mounted on a electrode assembly and then the temperature is raised with a constant heating rate without any gradient (temperature, fields). Short circuit current is recorded for first as well as second heatings runs. Current versus temperature plots shows one peak around temperature 95 to 100 C due to liberations of moisture from polymeric side chain groups. The magnitude of current and peak height increase with increasing temperature as well as
impurity concentration. The effect of heating rates on SCE is recorded. It is seen that current increases on increasing heating rates upto 7 C/min and beyond that is decreases. On the basis of results it is proposed that SCE in poly (9-vinylcarbazole) pure and doped with ferric chloride may be water activated phenomenon below \( T_g \) and internal field controlled above \( T_g \). The decrease in magnitude of SCE peak may be due to plasticizer effect of moisture. Due to inclusion of ferric chloride in PVK matrix, increase in SCE may be due to charge species either by charge transfer complex formation or by creation of an excess charge cloud or by creation of defect states. Spontaneous current emission in PVK seems to be of electrochemical nature. SCE in pure and doped PVK samples is notably affected by variation in heating rates. Perhaps this may be due to the control of moisture release rate that affects the SCE.

Chapter-VII, carried out the summary and conclusions of present work as well as future aspects of this study.

1.2 Conclusions

On the basis of output comes from the results and discussion throughout the present work, following conclusions may be drawn.

1. The increase in conductivity is accounted for the creation of additional hopping sites for the charge carriers in doped samples.

2. In the lower field region (0.9 – 10.9 kV/cm) conduction through metallic electrode sandwich ferric chloride doped PVK thin films obeys the Ohm’s law.

3. At high field region (10.9 – 40.8 kV/cm) conduction process is bulk limited and electrode independent, governed by Poole-Frenkel mechanism.

4. The activation energy for DC conduction is about 0.207-0.195 eV, which is suggestive of typical electronic conduction.

5. The presence of ferric chloride molecules in poly (9-vinylcarbazole) enhances conductivity by 4 to 6 times in the higher concentration of ferric chloride doping. The conductivity of pure poly (9-vinylcarbazole) (PVK) thin films is reported to be very weak.

6. The enhancement in conductivity due to formation of charge transfer complexes (C-T-C) between ferric chloride and poly (9-vinylcarbazole) has been attributed to the creation of excess charge cloud and formation of localized levels.

7. The dipolar relaxation process (\( \beta \)-relaxation) appears to be due to side chain of carbazole groups.
8. \( \alpha \)-peaks occur due to the joint motion/deorientation of the back bone chain and side chains of PVK.

9. Increase in poling fields helps in releasing and mobilizing the charges easily but the peak temperatures remain unaffected.

10. Dipolar relaxations have been found mainly responsible mechanism in case of TSDC.

11. Introduction of ferric chloride in PVK forms strong complexes, prevents the trapping of charge carriers, enhances the conductivity by increasing the mobility of the charge carriers.

12. SCE in poly (9-vinylcarbazole) pure and doped with ferric chloride may be water activated phenomenon below \( T_g \) and internal field controlled above \( T_g \).

13. The decrease in magnitude of SCE peak may be due to plasticizer effect of moisture.

14. The inclusion of ferric chloride in PVK matrix, increases SCE, which may be due to the production charge species either by charge transfer complex formation or by creating an excess charge cloud or by creating defect states.

15. Spontaneous current emission in PVK seems to be of electrochemical nature.

16. SCE in pure and doped PVK samples is notably affected by variation in heating rates.

1.3 Future of Present Work

The relaxation time of dipoles in the studied material (presented in the table 5.1 to 5.3) is higher so the charged electrets of such materials can be used for the purpose of charge storage devices for power backup in memory devices.

The resistivity of pure poly (9-vinylcarbazole) composite with ferric chloride is very high, so this material may be used for manufacturing the gate of MOSFET. The activation energy of 2% ferric chloride doped PVK has been found to be approximately 0.20 eV, so this material can also be used in light emitting diode and solar cell.
1.4 References

Investigation of Traps density in Poly(9-vinylcarbazole) using Thermally Stimulated Discharge Current Technique

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Abstract - In this paper the results of thermally stimulated depolarization current (TSDC) for pure poly(9-vinylcarbazole) (PVK) and PVK doped with ferrocene, thermoelectrets are presented. The measurements of TSDC has been carried out in temperature range 30 to 170°C with steps 5°C in function of different polarizing fields at constant polling temperature. The TSDC spectra comprises two maxima, namely β and α peak with their respective location around 100°C and 155°C. β peak was attributed to the dipolar nature and α peak was assigned to a space charge trapping mechanism in the investigated material. The Trap energy is evaluated from the Garlick- Gibson plot of initial rise method. Similarly other parameters such as relaxation time, total charge release are evaluated. It is observed in present study that the value of the activation energy reduce according to the increasing the concentration of ferrocene molecules as dopant and it is found at poling temperature 100°C and poling field 50 kV/cm for β peak of pure PVK, 0.73 eV and 2% ferrocene doped PVK, 0.24 eV.

Keywords: TSDC, PVK, activation energy, traps, total charge release, poling temperature, ferrocene

1 Introduction

The most well known study of molecular motion is the study of relaxation processes, take place inside the polymers. In these studies almost two techniques are used, the first is dielectric spectroscopy (DS) and the other is the thermally stimulated depolarization current (TSDC). Thermally stimulated depolarization current is a most popular technique for investigation of charge storage and transport processes in high resistive materials [1]. A lot of work has been published on TSDC in polymeric materials during the past few decades [2-4]. Thermally stimulated current (TSC) one gets information concerning the trap structure of dielectric materials [5]. There are several methods to determine the properties of trap centers in polymeric materials and semiconductors. Among them, thermally stimulated depolarization current measurements are distinguished by the ease with which the experiments are performed and the detailed information provide on trap states [6, 7]. Various dielectric relaxation processes in polymers can be accurately described by applying the thermally stimulated depolarization current (TSDC) technique [8]. The TSDC technique essentially consists of two steps, in the first step, a thermoelectret state is formed in the investigated polymers and then in the second step, the depolarization current spectra at a constant heating rate are obtained. The main advantage of this technique is its high sensitivity, which makes it possible to detect very low dipolar or carrier concentrations.

Thermoelectrets effect was discovered in 1925, when Eguchi solidified a mixture of carnauba wax, resin and beeswax in presence of a high DC electric field [9]. The energy absorbed during the charging process resulted in space charge polarization by trapping of positive and negative charge at the interstitial sites [10]. However, the role of various polarization processes and their relative contribution to the electrets state of the polymers is
not yet fully understood. Particularly, the space charge relaxation mechanism and the details of trap structure (including the trap distribution in energy and also over the volume of the polymer) are still to be well understood.

The purpose of the present work is to obtained further information concerning the deep and shallow defects in undoped PVK and doped PVK using well-established technique of TSDC measurements.

2 Materials and Methods

The material used in the present study was poly(9-vinylcarbazole) (PVK) supplied by Aldrich Chemical Co. USA, having a glass transition temperature (T_g) ~ 200° C molecular weight average M_w ~ 1,100,000 product number 182605, CAS number 25067-59-8, refractive index 1.683 and density 1.2 g/ml at 25° C was obtained. The structure of macromolecule of PVK and ferrocene are given as below figure (2.1).

(a) Chemical structure of PVK monomer (b) Chemical structure of Ferrocene molecule

For the preparation of the thin film samples, polymer obtained was used as such without any further purification/recrystallization. Dopant ferrocene was used of Merk make. In present study, chloroform (AR-Grade) was used as a solvent for preparation of thin films. The thickness of the film varies was monitored as per selection of concentration of solution. Thin film samples obtained using solution casting technique was optically transparent.

For the preparation of polymer thermoelectrets in this study, thin film sample was sandwiched between circular aluminum electrodes of the required size and mounted on spring loaded sample holder. The whole assembly was kept in a digitally temperature controlled electric oven for thermal and electrical treatment. After achieving thermal equilibrium at a predetermined polarizing temperature T_p, a DC electric field i.e., polarizing field E_p, was applied for polarizing time t_p, 90 minute. The sample was the cooled down at room temperature under the presence of applied poling field. The field was then removed and sample short circuited for 15 minutes to remove stray charges. Now getting the TSDC spectra, according to experimental setup figure (2.2) connection plug K_3 was closed and sandwiched thermoelectrets is heated at constant heating rate β.

The depolarization current is recorded as a function of temperature using DPM-III digital picommeter with accuracy~10^{-12} Ampere.

In this study TSDC spectra were obtained on poly(9-vinylcarbazole) and 2% ferrocene doped PVK of 55 µm thickness with experimental parameters as follow: T_p = 100° C, E_p = 50 kV/cm, t_p = 90 min. T_f = 30° C and heating rate β = 3° C/min. The depolarization kinetic data i.e. activation energy, total charge release and relaxation time were calculated and listed in table 1 and 2. The activation energy was evaluated using the Arrhenius equation

\[ I(T) = A \exp\left(-\frac{E_{ac}}{kT}\right) \]

Where A is a constant, E_{ac} is activation energy, T is the absolute temperature and k is the Boltzmann constant. By plotting ln I as a function of (1/T), one gets a straight line with the slope \(-\frac{E_{ac}}{k}\). The relaxation time was determined using the formula

\[ \tau(T) = \frac{1}{\beta}\left(\frac{kT_m^2}{E_{ac}}\right) \]
where $T_m$ is the temperature corresponding to the TSDC peak point. The relaxation time $\tau(T)$ of dipole is affected by the parameters of heating rate $\beta$, peak temperature $T_m$ and activation energy $E_{ac}$.

The total charge release is obtained using the following relation

$$Q = S \cdot \beta \int_{T_s}^{T_f} I(T) dT$$

Where $S$ is the sample area and $\beta$ is the heating rate. It gives the direct information about decay of stored charges. The charge released from these traps may be due to the thermal excitation and motion of molecular chain that causes the lowering of trap depth.

Experimentally value of total released charge ($Q$) is determined by Gaussian fit TSC peak and the total charge released in proportional to the area under the peak.

3. Results

The Thermally stimulated discharge current (TSDC) spectra of poly(9-vinylcarbazole) pure and doped with 2% ferrocene have been shown in figures 3.1(a) and 3.2(a) for different polarizing fields $E_p$ (25 kV, 50 kV, 100 kV) at fixed polarizing temperature $T_p = 100^\circ C$. Two peaks are observed first $\beta$ peak around temperature 90$^\circ$ C to 110$^\circ$ C and second $\alpha$ peak found between 150$^\circ$ C to 160$^\circ$ C. Figures 3.1(b) and 3.2(b) show the initial rise plot corresponding to TSD spectra of figures 3.1(a) and 3.2(a) for pure and doped PVK thin films (thickness 55 µm).

3.1. Effect of Polarizing Field $E_p$

1. Figure 3.1(a) shows the TSD thermograms for pure PVK at $T_p = 100^\circ$ C for three different fields (25 kV/cm, 50 kV/cm, 100 kV/cm). Two peaks ($\beta$ and $\alpha$) are found for each polarizing field. The magnitudes of both peaks corresponding to each poling field increase by increasing the strength of poling fields. For initial poling field (25 and 50 kV/cm), $\beta$ and $\alpha$ appear at nearly 105$^\circ$ C and 155$^\circ$ C respectively but at poling field 100 kV/cm the position of both peaks change and they appear at 100$^\circ$ C and 150$^\circ$ C respectively. Activation energies $E_{ac}$ associated with peaks of figure 3.2(a) are determined from the slopes LnI(T) versus 1000/T of corresponding to initial rise plots from figure 3.1(b). The others parameters like as relaxation time $\tau(T)$, total charge released $Q$ are placed in table 1 and 2.

2. Figure 3.2(a) represent the TSD thermograms for 2% ferrocene doped poly(9-vinylcarbazole) at poling temperature $T_p = 100^\circ$ C for polarizing fields (25 kV/cm, 50 kV/cm and 100 kV/cm) respectively. Plots of thermograms displayed similar nature but the magnitude and area under the peaks of $\beta$ and $\alpha$ increase with increase in poling temperatures as well as poling field.
Figure 3.2(b) shows the initial rise plots for TSD spectra of figure 3.2(a) for each β and α peaks corresponding to poling fields 25 kV/cm, 50 kV/cm and 100 kV/cm at polarization temperature $T_p = 100^\circ C$. The slopes of the curves determined the value of trap energy or activation energy, which are reducing almost with increasing poling fields.

Table 1.0, displays the values of evaluated depolarization parameters.

4.1 Affect of Poling Fields

Figures 3.1(a) and 3.2(a) represents the affect of poling fields on pure poly(9-vinylcarbazole) and 2% ferrocene doped PVK at different polarizing fields for fixed poling temperature, 100$^\circ C$. They show two relaxation peaks, first peak β appears at about temperature 70$^\circ C$ and second peak α arises at temperature 155$^\circ C$ i.e., around $T_g$ in all the cases and figures 3.1(b) and 3.2(b) show their initial rise plots. In each case, two peaks (β and α) are observed with their peak temperature ($T_m$) almost independent of $E_p$ but the peak current ($I_m$) increases with increase in poling fields. This increment in case of α peak is more pronounced than that in β peak.

One of the arguments advocated for distinguishing a dipolar polarization and space charge polarization is based on the field dependence of the TSDC properties, such as the peak current, peak temperature and charge released. The linear dependence of charge released and the peak current on polarization field strength is a characteristic of dipolar origin whereas in the case of space charge polarization, the peak current and charge released will show a non-linear variation with polarizing field strengths.

For a dipolar peak, the peak temperature will be independent of poling fields as well as poling temperature, whereas the peak temperature depends on the poling fields and temperature of the space charge polarization. The linear field dependence of peak current suggests that the TSDC spectra may be either due to dipolar origin or migration of charge carriers through microscopic distances with trapping. The increasing value of peak current with poling field and the observed
activation energy values indicate that the peak may be contributed significantly by dipolar and ionic polarization.

4.2 Affect of Impurity

Earlier it has been discussed that PVK is a hole donating and ferrocene is an electron donor, therefore charge transfer complexes (CTCs) may be formed between them. Due to impregnation of ferrocene in PVK chains, creates conductive pathways for charge carriers and prevent their trapping at particular sites in the molecular chains. Some workers are also having same opinion of weaker trapping of carriers [11]. Ferrocene also enhances the conductivity of the polymer.

PVK doped with ferrocene exhibit pronounced field affect as compared to pure PVK. In doped system, the magnitude of peak current increases with field. For $T_p = 100^\circ$ C and $E_p = 100$ kV/cm peak current for $\beta$ for pure PVK is found to be $5.9 \times 10^{-10}$ Ampere whereas it is $12.9 \times 10^{-10}$ Ampere for 2% ferrocene doped PVK. For $\alpha$ peak the corresponding values are $6.3 \times 10^{-10}$ (for pure PVK) and $13.0 \times 10^{-10}$ Ampere (for 2% ferrocene doped PVK). Increase in magnitude may be due to the increase in mobility of the carriers [12].

Inclusion of ferrocene in poly(9-vinylcarbazole) has developed, perhaps, a space charge cloud by filling all the traps present in the polymer matrix. At high temperature negative currents are found perhaps due to the contribution from net charges. It is also not improbable that up to these higher temperatures trapping sites would be destroyed and charges of only one sign give unidirectional current. High values of activation energies may be taken as standing proofs for this reason.

5.0 Conclusion

On the basis of preceding discussion, following conclusions may be derived.

1. The dipolar relaxation process ($\beta$-relaxation) appears due to side chain of carbazole groups.

2. $\alpha$-peaks occur due to the joint motion/deorientation of the back bone chain and side chains of PVK.

3. As increase in poling fields helps in releasing and mobilizing the charges easily but the peak temperatures remain unaffected.

4. Dipolar relaxations have been found mainly responsible mechanism in present case.

5. Introduction of ferrocene in PVK forms strong complexes, prevents the trapping of charge strong complexes, prevents the trapping of charge carriers, enhances the conductivity by increasing the mobility of the charge carriers.

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TABLE 1.0 : TSDC parameters for 2% ferrocene doped Poly(9-vinylcarbazole) samples at $T_p=100$ C, with different Poling field.

<table>
<thead>
<tr>
<th>Poling field</th>
<th>Peak</th>
<th>Peak current</th>
<th>Peak Temperature</th>
<th>Activation energy</th>
<th>Relaxation time</th>
<th>Charge released</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_p$ (kV/cm)</td>
<td>($\beta$)</td>
<td>107</td>
<td>105</td>
<td>0.26</td>
<td>$9.21 \times 10^2$</td>
<td>28.4</td>
</tr>
<tr>
<td>$n$(Coul.)</td>
<td>25</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\alpha$</td>
<td>115</td>
<td>155</td>
<td>0.60</td>
<td>$5.17 \times 10^2$</td>
<td>9.51</td>
<td></td>
</tr>
<tr>
<td></td>
<td>β</td>
<td>120</td>
<td>105</td>
<td>0.24</td>
<td>9.92 x 10^2</td>
<td>61.7</td>
</tr>
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<td>---</td>
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<tr>
<td>50</td>
<td>α</td>
<td>105</td>
<td>155</td>
<td>0.51</td>
<td>5.93 x 10^2</td>
<td>8.82</td>
</tr>
<tr>
<td></td>
<td>β</td>
<td>129</td>
<td>105</td>
<td>0.16</td>
<td>8.92 x 10^2</td>
<td>41.8</td>
</tr>
<tr>
<td>100</td>
<td>α</td>
<td>130</td>
<td>155</td>
<td>0.45</td>
<td>6.92 x 10^2</td>
<td>9.35</td>
</tr>
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References