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An investigation on Transient Current characteristics in polymeric double layer Thin Films

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ABSTRACT

Transient current characteristics in charging and discharging modes for PVDF-PMMA double layer polymer samples replicated various mechanisms responsible for the transient conduction phenomenon. The isochronal are characterized by well-defined peaks, indicating thermal activation of discharging current over a particular temperature range. The time-dependent charging current characteristics in double-layered samples suggested space charge limited conduction (SCLC) mechanism and also that the polarization may be due to dipolar orientation and trapping of charge carries in the bulk.

Keywords: traps, space charge, isochronal, dipolar, tunneling

INTRODUCTION

Over the years, the involvement of polymers in electrical applications has been due to their electrical insulation properties. Polymers acquire persistent polarization due to the alignment of dipoles and migration of charge carriers over macroscopic distances. The electrical transport in polymeric materials^{1,2} has become an increasingly interesting area of research, partly because these materials possess great potential for solid state devices and partly because this field of study serves as a stepping stone towards the understanding of the general theory of polymer physics.³

Transient charging and discharging currents were investigated to understand time dependent polarization effect in organic polymers.⁴⁻⁶ The polarization of polymeric materials may be due to dipolar orientation and accumulation of charge carriers near the electrodes or trapping in the bulk.⁷ Under certain conditions additional charge carriers may be provided by injection from the electrodes, which also contribute space charge polarization.⁸⁻¹³ Hopping of charge carriers from one localized state to another¹⁴ and tunneling from electrode to those trap levels in the dielectric which are close to the Fermi level of the electrode material¹⁵ have also been proposed to account for the currents in the dielectrics. The current obtained immediately after the application of a step function voltage is known in most cases to decay with time until a

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steady state current is achieved.¹⁶ The discharging current flowing on removal of the voltage is usually the mirror image of the charging current except that a steady state current does not occur. The knowledge of currents is also necessary to discover the true conductivity of the material.¹⁷ Generally, it is expected that the transient and steady state conductivities should have similar characteristics or mechanisms. But it is not absolutely necessary and they may have different mechanisms in spite of having similar activation energies for both the conductivities. In the present study we shall try to reveal and interpret the changes produced in the transient current study in charging and discharging modes due to the formation of additional trapping sites for charge carriers as a result of the polymer-polymer interface in particularly PVDF-PMMA double layered samples and shall try to compare the results with that of the individual polymers i.e. PVDF and PMMA.

EXPERIMENTAL

PMMA and PVDF samples were prepared by using the solution grown technique.^{18,19} Thin films of the two polymers i.e. PMMA and PVDF were then stacked one over the other and compressed together under a compression moulding machine at a temperature of 65°C and pressure of 2500 lbin⁻² (17.25 MPa) to yield doublelayer samples with PMMA on one side and PVDF on the other. Thus PVDF, PMMA and PVDF-PMMA double-layer samples are prepared. They are kept inside separate vacuum desiccators; filled with silica gel in order to prevent them from moisture.

THEORY OF TRANSIENT CURRENTS

When an electric field is applied to a dielectric specimen placed between two electrodes, a charging current flow in the circuit which consists f a polarization component and a conduction component. After the specimen is fully charged, it is disconnected from the voltage source and is short-circuited. A discharge current equal to the depolarization current flows, which is of the almost same magnitude and opposite in polarity to that of the polarization current. Hence, depolarization current can yield information about the polarization process even when the corresponding polarization current is masked by the conduction current at charging. However, the origin of this dielectric charging and discharging currents is still the subject of much controversy in the literature and a number of mechanisms have been proposed. In view of the possible applicability of polymers in electronics and engineering, it becomes important to clarify the transient response of charging and discharging current in polymers. Assuming the transient current to be associated with a single relaxation time the transient current step response at a particular time (t) can be expressed as²⁰

$$I(t) = \frac{P_0}{\tau} \exp\left[\frac{-t}{\tau}\right]$$
(1)

where P_0 is the relaxed electric polarization and τ is the relaxation time. The electric polarization P_0 can be given as

$$P_0 = \frac{\varepsilon_0 \Delta \varepsilon_r SV}{d} \tag{2}$$

where $\epsilon_0\,$ is the absolute permittivity of free space, S the effective area of the sample, d the thickness, V the applied voltage and

$$\Delta \mathcal{E}_r = \mathcal{E}_{r_0} - \mathcal{E}_{r_\infty} \tag{3}$$

where ε_{r0} and $\varepsilon_{r\infty}$ are the low and high frequency limit of relative permittivity respectively. The complex relative permittivity ε_r^* for a single relaxation time can be given by the Debye equation as

$$\varepsilon_r^* = \varepsilon_{r\infty} + \frac{\Delta \varepsilon_r}{1 + j\omega\tau}$$
⁽⁴⁾

where ω is the angular frequency. However, for the polymers instead of a single relaxation time we have a distribution in relaxation time and the complex relative permittivity can be given by using the Cole-Cole equation as

$$\varepsilon_r^* = \varepsilon_{r\infty} + \frac{\Delta \varepsilon_r}{(1+j\omega \tau)^{1-\alpha}}$$
(5)

Eqn. (5) reduces to Eqn. (3) when $\alpha = 0$ where α is constant (0< α <1). Thus isothermal depolarization current measurement technique offers an alternative to the measurement of dielectric constant and loss tangent as a function of frequency and temperature. If the polarization process has not been completed during charging period, the magnitude of the depolarization current is not the same as that of the polarization current.

After the application or removal of a step voltage the transient current flowing through a dielectric decays following the Curievon Schweidler law i.e.

$$I(t) = A(T)t^{-n} \qquad (6)$$

where I is the current, A (T) is the time-dependent factor; t is the time after application or removal of the external voltage and n a constant which is generally close to unity. The current, in the time domain, for the short time region is characterized by the relation:

$$I(t) \propto t^{-n}; 0 < n < 1; t << \frac{1}{W_p}$$
 (7)

i.e. at frequencies which are larger than the loss peak frequency W_p , and for long time region with logarithmic slope steeper than unity.

$$I(t) \propto t^{1-p}; 0 < n < 1; t >> W_p$$
 (8)

The two power losses determine the time domain response of dipolar system. Similar behavior is observed in carrier dominated systems, however, low frequency dispersion below a frequency W_p , which corresponds to long time region, is described by the power laws with small value of n. Let n = 1-p, with p close to unity, for low frequency dispersion regime. The long time response of charge carrier system will then be denoted by

$$I(t) \propto t^{1-p}; p=1; \quad t \gg \frac{1}{W_p} \tag{9}$$

which corresponds to a very slowly time varying current. The complete representation of the universal dielectric response in the time domain, covering both dipolar loss peaks and strong low frequency dispersion associated with the charge carrier dominated system may be represented by

$$I(t) \propto t^{-n}; 0 < n < 2;$$
 (10)

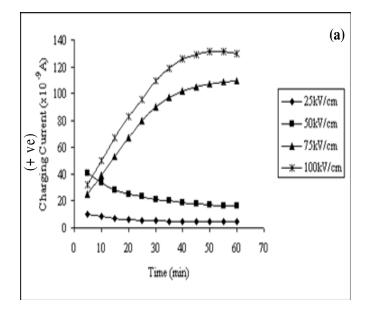
with the exponent n taking values in different ranges at long and short times, respectively²¹. In transient current measurements quantitative variation of activation energy on temperature i.e. A (T) is not seen. The application of a step voltage to a polymersample causes a flow of current which decays with time before reaching a steady state value. The total current²² consisting of the charging current (I_c) which decays with time and the static current (I_s) which remains constant, i.e.

$$I = I_c + I_s \tag{11}$$

For any given electric field, the time dependence of transient current is found to obey the law of dielectric responses.²³ Soon after the application of the poling field in a metal-insulator-metal system, the current rises rapidly and then approaches a constant value. This time-dependent part of the charging current is due to a dielectric polarization under the applied electric field which may be due to a dipolar orientation, accumulation of charge carriers near the electrodes, space-charge polarization of carriers injected from the electrodes or trapping in the bulk²⁴⁻²⁸ etc. Under certain conditions, additional charge carriers may be produced by injection from the electrodes which also contribute to spacecharge polarization.²⁹ The discharging current noticed after the removal of the step voltage decays with time in the direction opposite to that of the charging current. This will be due to a dipolar depolarization or redistribution of charge carriers due to the interfacial polarization or space-charge polarization.³⁰⁻³²

RESULTS AND DISCUSSION

Transient current characteristics in charging and discharging modes for PVDF-PMMA double-layered samples (Fig. 1 (a)-(d) and Fig. 2 (a)-(d)) at polarizing temperature (T_p) of 30°C, 50°C, 75°C and 100°C are represented for different polarizing fields (E_p's) of 25kV/cm, 50kV/cm, 75kV/cm and 100kV/cm. The order of charging and discharging currents for single and double-layered samples at different polarizing fields were found to vary from 10 ¹¹ to 10⁻⁹ A, but charging currents has always been found to be higher than discharging currents. It appears that charge involved in the charging period is greater than the discharging period. The possible reason for this difference may be that, initially transients occur with empty traps and current is as much as allowed by the injecting barriers. As soon as the traps become filled, the current reduces to the space charge limited current with traps. Whereas, in the discharging period, the trapped carriers will be discharged towards both the electrodes showing a small current in the external circuit. For double-layered samples, it is clearly observed that time dependent transient charging (Fig.1) and discharging (Fig.2) currents consisted of two logarithmic slopes for each field and satisfy Curie-Von empirical law but are not a mirror image of each other. The index n has been found to be in the range 0-0.5 at shorter times however, at longer times it has been observed to be in the range 0.5-1.0. It is also marked that the rate of fall of the transient charging currents is low for lower polarizing fields than for higher fields, i.e. the polarization time (t_m) decreases with the increase in polarizing field. It is evident that the magnitude of charging as well as discharging currents decreases with time but increases with increase in polarizing fields. The graphs also indicate that for all double-layered samples, increase in current is more prominent in the higher temperature and field range. At higher polarizing fields for all polarizing temperatures, doublelayered sample show a unique behavior of large out-burst of current which gradually rises with time and approaches a constant value. This behavior shows the thermally activated nature of double-layered samples.



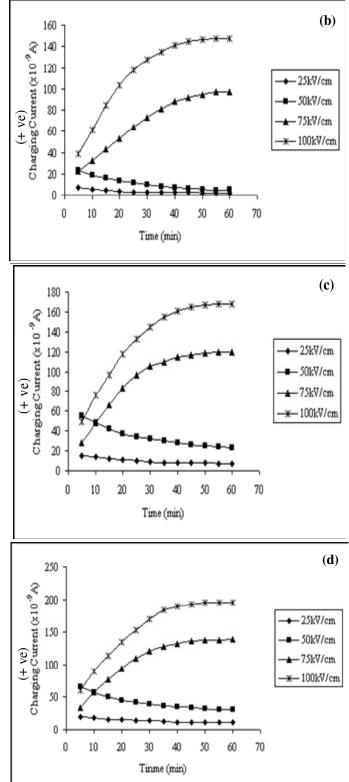


Figure 1: Transient current characteristics for PVDF-PMMA double-layered samples in charging mode for polarizing temperature (T_p) of (a) 30°C (b) 50°C (c) 75°C (d) 100°C and different polarizing fields (E_p 's)

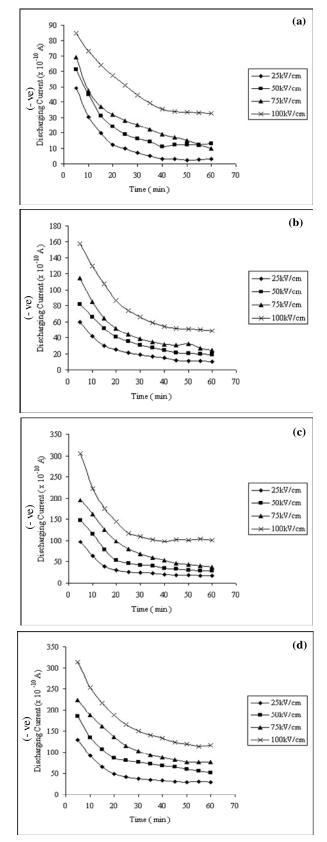


Figure 2: Transient current characteristics for PVDF-PMMA double-layered samples in discharging mode for polarizing temperature (T_p) of (a) 30°C (b) 50°C (c) 75°C (d) 100°C and different polarizing fields (E_p 's)

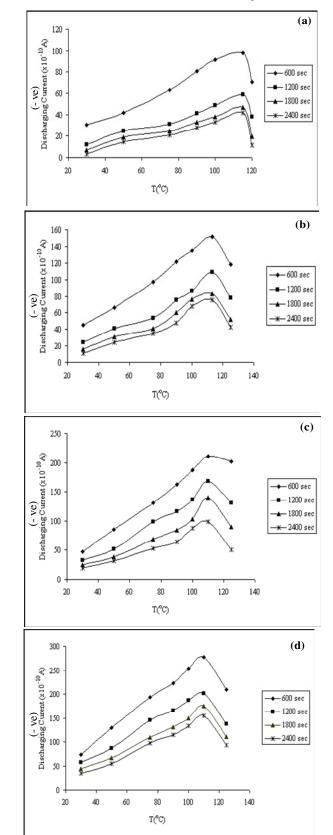


Figure 3: Temperature dependence of discharging currents (I_d) for PVDF-PMMA double-layered samples with polarizing field (E_p) of (a) 25 kV/cm (b) 50 kV/cm (c) 75 kV/cm (d) 100 kV/cm at different discharging times (sec)

For PMMA, PVDF and their double-layered samples, isochronals are characterized by a single peak. The peak for PMMA samples is located around 87-100°C and around 95-100°C for PVDF samples. For double-layered samples it lies in the temperature range 110-115°C shown in Fig. 3. In some cases, the peak was observed to shift towards lower temperatures for higher polarizing fields for single and double-layered samples, which is a characteristic of thermal relaxation process. Charges released from shallow and deeper trapscan be considered to contribute to these peaks. Thus, it may be obvious that the observed isochronal current peaks correspond to dipolar orientation due to molecular motions associated with the side chains. The contribution of space charge may not be completely expelled in the present study of single as well as double-layered samples.

The mechanisms of the time-dependent polarization effects are studied from an analysis of transient currents in charging and discharging modes with respect to polarizing fields and temperatures. The polarizing field dependent charging current characteristics are represented for double-layered samples (Fig. 4(a)-(d)) at prescribed charging times (i.e. 600, 1200, 1800 and 2400 sec) for a fixed charging temperature (i.e. 30°C, 50°C, 75°C and 100°C).

In double-layered samples, for low polarizing fields, with the application of a step voltage, the charging current flows, which gradually decay with time before reaching a steady state conduction level at very long times. The charging currents at low polarizing fields, reduces to zero when the sample is completely polarized. However, for high polarizing fields a steady-state level of conduction was reached in progressively shorter times as its value increased faster than that of the transient current and became strongly temperature dependent. The total current consisting of the absorption current (I_a) and the static current²⁵ (I_s), i.e. $I = I_a + I_s$. The time-dependent charging current characteristics shown in Fig. 1 (a)-(d) suggests that the polarization in the sample at low fields is attributed to dipolar orientation along with charge carrier hopping transport mechanism, accompanying molecular motions with a wide distribution of relaxation times. The trapping of space charge carries in the bulk; with their ejection from the traps is proposed to be the predominant mechanism of transient currents at high fields. It is observed that the sample takes a longer period of time to reach a steady-state situation at a temperature between T_g and room temperature³³. But at temperatures above T_g, the steadystate situation is attained quickly. This may be due to the increase in free volume at Tg which results in a decrease in viscosity and therefore yields sufficient room for free rotation of dipolar molecules. Also, at higher fields, changes in mobility of chain segments and charge carries may take place faster than at lower fields. Hence, the current may approach a stable value in a shorter period of time at higher fields and at temperatures above T_g.

The large outburst of charging currents at high fields as revealed in Fig. 1 (a)-(d) in double-layered samples can be explained on the basis of the theory of space charge limited conduction (SCLC) which is influenced by traps³⁴. The defects and impurities govern the charge transport mechanism act as trapping centers and get populated by the injected charge carriers from the electrodes. However, the exact nature of the traps present

in the double-layered polymeric heterogeneous system depends on their position with respect to the Fermi-level. In the present study, the large currents obtained just after the application of voltage subsided to much smaller steady values after a certain length of time. The possible explanation is that the sudden application of voltage causes a cloud of carriers, i.e. a space charge, to be injected from the contact into the sample. This free charge gives rise to a large burst of current. If the space charge remained untrapped, the value of the transient current would continue as a steady current. However, the effects of trap densities in the sample must also be taken into account. The free charge forced into the sample settles the rate being determined by the capture cross section of traps for free carriers³⁵. The attribution to SCL mechanism in double-layered samples is further supported by the following facts: (i) PMMA being an amorphous polar material ensures trapping of charge carriers in large number of trapping sites would result in the build-up of space charge, (ii) PVDF is a semi-crystalline polar polymer thus, charge trapping takes place at the molecular chain, the side chain and at the interface of the crystalline and amorphous regions of the polymer³⁶ (iii) polymerpolymer interface in double-layer samples give rise to additional molecular trapping sites for charge carriers³⁷. Blinov et al.³⁸ reported that during polarization, on application of high electric field in double-layered samples, the charge carriers are accumulated within the vicinity of the interface so that the local electric field in the interface is found to increase. This local electric field allows the movement of charge carriers through interface during application of external voltage.

The magnitudes of charging and discharging currents in double-layered samples have been found to be higher than that of the individual polymers. This can be explained in terms of Maxwell-Wagner Interfacial polarization. Maxwell-Wagner effect occurs in heterogeneous dielectric as a result of the build-up of space charges at interface between two media having different permittivity and conductivities when such a sample is heated and subjected to a field³⁹⁻⁴². The peak depicted by temperature dependent isochronal for discharging currents at constant times for double-layered samples is characteristic of the thermal relaxation process. Consequently, activation energy (E_a) evaluated from these plots using initial rise method was found in the range 0.60-0.94 eV. The field dependent charging currents at constant times for double-layered samples as shown in Fig 4 were observed to slightly depart from straight line. The steady state or isothermal characteristics reveal almost ohmic behavior initially in the low field region, which gradually becomes non-ohmic at higher fields. However, operating temperature seems to play a crucial role in determining nonlinear nature of these curves supporting the theory of charge injection forming trapped space charges. The increment in the current is approximately the same for whole range of temperatures. The nature of the thermo grams is non-linear but similar for all temperatures.

In the present study the double-layered system give rise to polymer-polymer interface which provides additional trapping sites for charge carriers. The polymer-polymer interface is supposed to create localized states of various depths which will lead to trapping sites distributed over a considerably wide energy range. Since there are many localized states, the release or excitation of the carriers in these states dominates the charge transport process. These localized states act as carrier trapping centers, and after trapping the injected charge from electrodes become charged and thereby expected to build up a space charge. This build-up of space charge playsthe key role in the determination of charge carrier generation and transport mechanism involved. The formation of polymer-polymer interface is considered to reduce the barrier between the trapping sites providing a conducting path through the polymer matrix and would result in the enhancement of charging current with increase in polarizing field and temperature. Thus, at high polarizing fields larger numbers of charge carriers including adventitious ions are generated probably due to impurities centers in the lattice or impurities induced as a cause of experimental limitations are ejected from the traps and contribute to the current flowing across the bulk of the sample.43-44

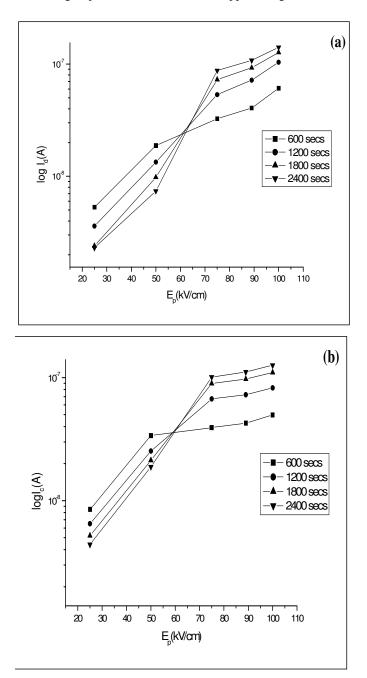
In conclusion, it would be reasonable to justify that charging currents has always been found to be higher in magnitude than the discharging currents; which replicates that charge involved in the charging period is greater than the discharging period. The possible explanation may be that, initially transients occur with empty traps and current is as much as allowed by the injecting barriers. As soon as the traps become filled, the current reduces to the space charge limited current with traps. Whereas, in the discharging period, the trapped carriers will be discharged towards both the electrodes showing a small current.

It is evident from both time dependent transient charging and discharging current graphs in double-layered samples, that in most of the cases; the magnitude of charging as well as discharging currents decreases with time and satisfy Curie-Von Schweidler law. However, for double-layered samples, the transient charging and discharging currents are not the mirror images of each other (in any respect and up to any extent) thus ruling out tunneling or electrode polarization processes. The time-dependent charging current characteristics suggests that the polarization in the doublelayered sample at low fields is attributed to dipolar orientation along with charge carrier hopping transport mechanism, accompanying molecular motions with a wide distribution of relaxation times. Trap-controlled hopping model describes the charge transport mechanism in the present study, which combines hopping with trap controlled transport. The trapping of space charge carries in the bulk; with their ejection from the traps is proposed to be the predominant mechanism of transient currents at high fields.

On the basis of effects brought about by different polarizing conditions on transient charging and discharging current characteristics and the activation energies calculated for doublelayered samples; we conclude that polarization may be due to dipolar orientation and also due to the trapping of charge carriers produced in the bulk and electrode-injected charges in deeper traps and their subsequent release due to the application of the step field. At higher polarizing fields double-layered samples show a unique behavior of large out-burst of charging current which gradually rises with time and approaches a constant value. This is due to the formation of space charge as a result of the

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Maxwell Wagner Interfacial polarization. The decaying trends of currents with time, the near linear nature of the charging currents against fields, the temperature dependence of the transient currents seems to be sufficient and reliable ground to assume that the possible controlling mechanism for the transient phenomenon is the charge injection and formation of trapped charges.



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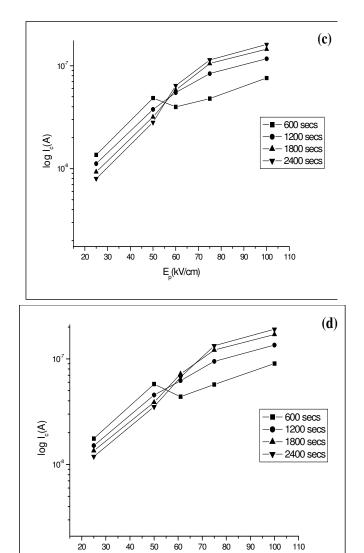


Figure 4: Polarizing field dependence of charging currents (I_c) for PVDF-PMMA double-layered samples at polarizing temperature (T_p) of (a) 30°C (b) 50°C (c) 75°C (d) 100°C at different charging times (sec)

E (kV/cm)

CONCLUSION

The transient charging/ discharging currents in double-layered samples have been considered to be due to dipolar and space charge processes. However, "trap-controlled hopping mechanism" has also been found to be dominant at high temperatures. The time temperature characteristics obtained from the discharging current observed at various fixed times at various temperatures has helped to correlate the observed discharging currents with thermally stimulated currents observed in the present case.

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