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Transient Current characteristics in PVDF-PSF blend samples

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ABSTRACT

The detailed studies of time dependent absorption and desorption currents at various polarizing fields and temperatures were carried out in PVDF-PSF polyblend samples of different ratio. The systematic analysis of various results from such studies has indicated that the time dependent desorption current in polymer blend samples may be due combined effect of dipole reorientation, space charge dissipation and hopping of charge carriers. Thermal activation of isochronal current in a specific temperature range followed by a peak centered at about 120-160°C, have indicated the close correlation of transient currents with the TSDC's.

Keywords: polyblend, transient current, space charge, absorption current, desorption current

INTRODUCTION

A significant progress has been made in many areas of science and technology of polymer blends and composite to understand state-of-the-art, and predict future trends in applications and new directions for these novel materials. The conduction processes could be studied using various methods which include ac conduction studies, charge particle bombardment, surface voltage decay, trapped charge decay, radiation induced conduction and photo conduction. However, there are uncertainties in each of the above methods. Thus, a systematic study of absorption and desorption currents can reveal the carrier injection trapping and polarization process which may be operative in a dielectric.

The absorption and desorption current data can be converted from time to frequency domain. It has proved experimentally¹ that the current obtained immediately after the application of a step function voltage is known in most cases to decay with time until a steady state current is achieved. Generally it is expected that the absorption-desorption 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. Thus, absorption and desorption current study is very sensitive tool to study the transport mechanism of charge carriers in polymeric dielectrics.² When the traps have a homogeneous energy distribution and the desorption current is not either too

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short or too long, the desorption current should decrease linearly with 1/t. therefore plot of log I versus log t gives a straight line with a negative slope equals to 1.

Hence, an attempt has been made for the first time to characterize the PVDF: PSF polymer blend electrets and this may give information about the molecular interaction and the extent of mixing between the two individual components and can also reveal the carrier injection, trapping and polarization processes.

EXPERIMENTAL

The films were prepared using the solution grown technique.³ The commercial PVDF (Solef 1015 PVDF Powder) and PSF (UDEL P1700 PSF Pellets) used were procured from Solvay Corp. Ltd., Belgium and supplied by Redox (India). The solution of particular concentration was prepared in a common solvent i.e. N, N, DMFat 50°C for 1 hr then kept at room temperature for 2 hr to become homogeneous. The cleaned optically plane glass plate at a constant temperature of 30°C were immersed in the solution for about 90 min. The plate was then slowly taken 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 hr to remove any residual solvent. Polymer film is then gently peeled off from the glass plate. Polyblend films of different compositions were prepared with PVDF: PSF weight percentage ratios of 80:20, 70:30 and 60:40. The circular shaped samples were prepared having diameter 5cm 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. The samples were out gassed in air for 24 h, and this was followed by room-temperature outgassing at a pressure of about 10^{-5} torr for a further period of 24 h. The samples were thermally polarized with fields of 25-100 kV/cm at various temperatures ranging from 30-120°C for 60 min during which the transient current in absorption mode was observed 5 min after the application of the field. The current was also observed in desorption mode 5 min after the removal of the field for the same period of time. To achieve the required degree of precision every consideration of perturbing parameter was taken into account, while designing the electrode assembly. Also, while carrying out measurements proper care was taken to avoid stray currents that may affect the results. The polarization was carried out by connecting a dc power supply (Scientific Equipments, Roorkee, EHT-11) in series with an electrometer (Scientific Equipments, Roorkee, DPM-111) which was carefully shielded and grounded to avoid ground loops and extraneous electrical noise.

Scanning Electron Microscopy (SEM) was performed on LEO-435-VP, variable pressure scanning electron microscope. Samples were sputtered coated with gold prior to testing.

THEORY OF TRANSIENT CURRENTS IN ABSORPTION AND DESORPTION MODES

In the absence of conductivity, the isothermal polarization current flowing through a dielectric after application of the constant voltage, also called the absorption current, is a pure displacement one depended on the applied polarizing field E_p is assumed to be constant and the so called response function $\varphi(t)$ characterizing the definite dielectric at a constant temperature. Thus, the polarization current density can be presented as

$$I_c(t) = E.\varphi(t) \tag{1}$$

The depolarization current density in the case of zero conductivity is

$$I_{d}(t) = E \left[\varphi(t+t_{p}) - \varphi(t) \right]$$
(2)

wheret_pbe the poling time.

If the poling time is long enough $(t_{p\to\infty})$, then $\varphi(t+t_p) \to 0$, so, as follows from Eqn. (1) and Eqn. (2), $I_c(t) = -I_d(t)$ i.e. $I_c(t)$ and $I_d(t)$ should be mirror images of each other. Therefore, the difference between. $I_c(t)$ and $I_d(t)$, if observed experimentally, can be caused by either a finite conductivity (g $\neq 0$), or by incomplete poling due to a relatively short poling time t_o . In both cases one would observe $I_c(t) \ge I_d(t)$.

To distinguish between influences of the two factors, one should analyze the shape of the polarization current at long time t. In the case of absence of the conductivity and long poling times, $I_c(t)$ and $I_d(t)$ coincide with both currents going to zero. If the sample has a definite conductivity, the through constant current will be observed at the $I_c(t)$ curve, while the $I_d(t)$ curve will go to zero. It follows from the above mentioned discussion that by comparing isothermal polarization and depolarization currents one can get information on the polarizing time required to obtain the sufficient polarization and on the value of the conductivity.

After the application or removal of a step voltage the transient current flowing through a dielectric decays following the Curievon Schweidler law as reported in literature.⁴

$$I(t) = A(T)t^{-n} \tag{3}$$

where I is the current, A (T) is the Temperature dependent factor; t is the time after application or removal of the external voltage and n is the decay constant which is generally close to unity.

The current, in the time domain, for the short time region is characterized by the relation:

$$I(t) \propto t^{-n}; 0 < n < 1; \quad t << \frac{1}{W_p}$$
 (4)

i.e. at frequencies which are larger than the loss peak frequency $W_{\rm p}$, and for long time region

$$I(t) \propto t^{-1+p}; \ p \approx 1, \quad t \gg \frac{1}{W_p}$$
(5)

with logarithmic slope steeper than unity. 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_c , which corresponds to long time region, is described by the power laws with small value of n. Let n= 1– p, with pclose to unity, for low frequency dispersion regime. The long time response of charge carrier system will then be denoted by

$$I(t) \propto t^{-1+p}; p \approx 1; \quad t \gg \frac{1}{W_p} \tag{6}$$

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:

(7)

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

Ι

with the exponent n taking values in different ranges at long and short times, respectively. In transient current measurements quantitative variation of A (T) is not seen. The application of a step voltage to a dielectric causes a flow of current which decays with time before reaching a steady state value. The total current as suggested⁵ consisting of the absorption current (I_c) which decays with time and the static current (I_s) which remains constant, i.e.

$$I = I_c + I_s \tag{8}$$

According to Aguilar⁶: for any given electric field, the time dependence of transient current is found to obey the law of dielectric responses. 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 absorption 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 bulk, etc. Khare⁷ et al highlighted that under certain conditions, additional charge carriers may be produced by injection from the electrodes which also contribute to space-charge polarization.

RESULT AND DISCUSSION

Transient current characteristics in absorption modes for PVDF:PSF blend samples (80:20) at polarizing temperature (T_p) of 30°C, 60°C, 90°C and 120°C are presented for different



Figure 1:Transient absorption current characteristics of PVDF: PSF (80:20) blend samples polarized at polarizing temperature T_p of (a) 30°C (b) 60°C (c) 90°C and(d) 120°C with different polarizing fields (E_p 's)

polarizing fields (E_p 's) of 20kV/cm, 40kV/cm, 70kV/cm and 100kV/cm are shown in Figure 1.

The transient absorption current versus time characteristics for several polymers consisted of two slopes for each field. The initial slope of the curve so obtained decreases with the increase in applied field and temperature. The absorption current for PVDF and blends sample decays rapidly with time just after the application of step voltage until a steady state current is reached after 30 minutes whereas for PSF, the current decays for about 20 minutes and then become constant. The time-dependent absorption current characteristics for different ratios of blend (80:20, 70:30 and 60:40) shows that the rate of fall of the absorption current is low for lower fields than for higher fields, i.e. the time decreases with the increase in polarizing field. For polymer blend samples, the logarithmic plots observed in time dependent transient absorption currents, are linear and characterized by two slopes in most of the cases. The current decays at a faster rate for the first few seconds (short time region) and then decay rate slows down (long time region). The observed time dependence of the current transient may be represented by the Curie-Von Schweidler law. The index n has been found to be in the range: 0.029 to 0.56 at shorter times and 0.03 to 1.3 at longer times whereas for PVDF, the index n is in the range 0.22-0.49 at shorter time, however, at longer times it is in the range of 0.51-1.25 and for PSF, the different decay constant values lying between 0.31-0.59 at shorter times and 0.66-1.05 at longer times.It is also noticed that the rate of fall of the transient absorption 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 absorption currents decreases with time but increases with increase in polarizing fields. The characteristics also indicate that for all polymer blend samples, increase in current is more prominent in the higher temperature and field range. At higher polarizing fields for all polarizing temperatures, blend sample show a unique behavior of large burst of current which gradually rises with time and approaches a constant value. This behavior shows the thermally activated nature of blend samples.

The time dependent absorption current characteristic for blend samples suggests that the polarization in the sample may be due to a dipolar orientation and the trapping of charge carriers in the bulk. The ejection from the traps will increase with the increases in field and temperature. The sudden application of step-voltage causes a cloud of carriers, i.e. space-charge, to be injected from the contact into the sample. This free charge gives rise to a large out-burst of current. The free charge forced into the sample settles into the traps and the decay of current is observed, the rate being determined by the capture cross-section of traps for free carriers.

It can also be observed from SEM images of PVDF and their blends of weight ratio 80:20 in Fig. 2 and 3. It is seen in the figures that PVDF image has a rounded structure with porous regions in between. The polyblend image shows the filling of porous spaces of pure PVDF by the amorphous PSF, which leads to affect the size or the perfection 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.⁸



Figure 2: SEM image of pure PVDF



Figure 3: SEM image of PVDF: PSF (80:20) blend

The polarizing field dependent absorption current characteristics are represented for polymer blend samples at prescribed charging times (i.e. 600, 1200, 1800 and 2400 sec) for a fixed charging temperature (i.e. 30° C, 60° C, 90° C and 120° C) are shown in Figure 4.

In polarizing field dependent absorption currents for PVDF and PSF samples observed at various times for different polarizing temperature, the transient absorption and desorption currents set off gradually from their mirror image nature thus the process of electrode polarization is also unlikely to occur in this case. Apart



Figure 4: Field dependent transient absorption current characteristics of PVDF: PSF (80:20) blend samples at polarizing temperature T_p of (a) 30°C (b) 60°C (c) 90°C and (d) 120°C at different times

from the mirror image nature, the value of n should be 0.5 for shorter times and greater than $unity^{9-10}$ for longer times which is also not found true in the present case. This behavior has been found to be typical of all the amorphous polar polymers investigated, provided that the currents are measured at temperatures sufficiently lower than the glass transition temperature reported by¹¹⁻¹².

On the basis of experimentally observed facts for PVDF and PSF samples, tunneling can also be clearly ruled out as a possible mechanism since the currents obtained are strongly temperature dependent. So far as PVDF samples are concerned, it is suggested that a dipolar mechanism is responsible for the transient current in PVDF at shorter times for lower fields although a microscopic charge-carrier hopping mechanism may not be ruled out whereas in for PSF samples, mechanisms of dipolar origin are generally predominant at short time and low fields. This suggests that the dipolar mechanism is also not the predominant process for the entire time range. However, there seems a possibility of space charge relaxation at sufficiently longer time and that could be due to trapping of charge carriers in sufficiently deeper traps.

The field dependent transient absorption current characteristics of PVDF: PSF blend samples have been discussed in the light of the existing models: dipolar polarization,^{1, 12-13} hopping of charge carriers,¹⁴ interfacial bulk polarization,¹⁵ electrode polarization¹⁶ homo-space-charge.17 hetero-space-charge and injected Considering the variation of such currents with time, temperature, and blend composition. In the present case, absorption current at fixed times show thermal dependence and exhibits a complex dependence on electric field. Thus it seems that tunneling of empty traps is ruled out as a possible mechanism. In case of electrode polarization, the transient currents reported to be proportional to t^{-n} with the value of the decay constant, n = 0.5 at shorter times, and n > 1 at longer times. However, in the present case, at shorter times the value of 'n' is found to vary from 0.029 to 0.56 at shorter times and 0.03 to 1.30 at longer times; in case of absorption transients. These observations show that the process of electrode polarization is unlikely to be dominant in the present case.

Transient current characteristics in desorption modes for PVDF:PSF blend samples (80:20) at polarizing temperature (T_p) of 30°C, 60°C, 90°C and 120°C are represented in Fig.5 for different polarizing fields (E_p 's) of 20kV/cm, 40kV/cm, 70kV/cm and 100kV/cm.

For PVDF: PSF blend samples, the desorption currents versus time curves at different field with constant temperature are characterized by different slopes in short and long-time regions, having different decay constant values lying between 0.06 to 1.32 at shorter times and 0.16 to 1.94 at longer times ,which decays rapidly during the first 20 minutes with a tendency to a stabilization around zero value. The value of n was found to vary from 0.22-0.5 and 0.56-1.32 for PVDF and having decay constant values lying between 0.26-0.78 and 0.29-1.62 for PSF at lower and higher field. It is also marked that the rate of fall of the transient desorption 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



Figure 5: Transient desorption current characteristics of PVDF: PSF (80:20) blend samples polarized at polarizing temperature T_p of (a) 30°C (b) 60°C (c) 90°C and (d) 120°C with different polarizing fields (E_p 's)

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desorption currents decreases with time but increases with increase in polarizing fields. Researchers¹⁸⁻²¹ investigated that this current evolution may be due to a dipole reorientation or/and a charge detrapping mechanisms. The absorption current reduced by the steady state current is not the mirror image of the desorption current indicate a buildup of space charge in the sample.

Activation energy values obtained from activation plots of the discharge current at various prescribed times for various samples with different weight ratios of the two polymers are found to vary from 0.66-1.12 eV. The various facts, including the polar structure of the polymer, the power-law dependence of current on field, the observed value of thermal activation energy i.e. 0.20-0.83 eV for PVDF and 0.68-1.04 eV for PSF (Table 1) of current over a certain temperature range implies that a space charge due to accumulation of charge carriers near the electrodes and trapping in the bulk account for the observed current behavior. For samples with a fixed weight ratio of the two polymers, the activation energy value has been found to increase with increase in time of observation of the discharge current. Also, at any fixed time the activation energy value has also been found to increase with increase with increase with increase models.

Table 1: Activation	energy values	(in eV) from	isochro	onal
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Poling field(40 kV/cm)	PVDF	PSF	PVDF:PSF (80:20)	PVDF:PSF (70:30)	PVDF:PSF (60:40)
(sec) 600	0.20	0.68	0.66	0.77	0.76
1200	0.49	0.86	0.86	0.86	0.85
1800	0.65	0.99	0.93	0.92	0.92
2400	0.83	1.04	0.97	0.93	1.12

The desorption current measured with PVDF, PSF and their blend samples for a number of polarizing temperature reveals that since the desorption current is due to the release of trapped charges, therefore, the characteristics of desorption current varies with the energy level, the distribution of energy level of the traps and the spatial distribution of traps in the bulk of polymer. The order of current in absorption and desorption mode was found to vary from 10^{-12} to 10^{-9} A for both PVDF and PSF but absorption currents has always been found to be higher than desorption currents. It seems to have large number of charge involved in the absorption period than desorption period. Since, 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 as reported earlier²²⁻²⁴. Whereas, in desorption period, the trapped carriers will be discharged towards both the electrodes showing a small current in the external circuit. The origin of transient absorption and desorption currents cannot be decided exactly, however, after a systematic analysis of the dependence of the current on various experimental parameters such as field strength, time and



Figure 6: Temperature dependent isochronal current characteristics of PVDF: PSF (80:20) blend samples with constant polarizing field (E_p) of (a) 20 kV/cm (b) 40 kV/cm (c) 70 kV/cm and (d) 100 kV/cm at different times

temperature, etc., the tentative discrimination can definitely be made between the various mechanisms²⁵ in pure and blend samples. The temperature dependence isochronal characteristics of PVDF, PSF and blends at different time with constant poling field of 20, 40, 70 and 100 kV/cm are shown in Fig. 6.

For PVDF, the isochronal are characterized by a peak located between 85-100°C for different values of field, which shifts towards lower temperature side with the increase in time and the peak appears at 110-130°C for PSF at different values of field. The peak observed in desorption current versus time curves of PVDF and PSF and the shifting of the peak time (t_m) towards lower time with increasing value of the absorption field depicts transient space charge limited conduction (SCLC)²⁶ for different organic molecular solids. The similarity in the appearance of the current transient and the experimental conditions appear to justify the carrier mobility calculation from the current-time plots on the basis of SCLC theory.

The PVDF: PSF blend samples polarized with different values of field are characterized by a broad peak located in between 120-160°C which shifts towards lower temperature side with the increase in PSF content in the blend as can be seen in Fig. 7. A careful observation reveals that, the isochronal current profile tends to shift towards lower temperature at shorter times. The transient currents thus exhibit the thermally activated behavior. The shifting of peak towards lower temperature side is similar to the behavior shown by blends in thermally stimulated discharge current study as reported in our previous study reported inour earlier work²⁷. The peak observed in blend samples is so broad that it probably contains several minor processes, one of which may be associated with the glass transition of the polymer blend and the second may be due to the thermal release of trapped carriers. The peak temperature is also found to decrease with increase in poling fields and time. This is a characteristic of the relaxation process. From these plots, relaxation time τ and activation energy E_a are evaluated using method reported in literature by Sessler²⁸ using the relation:

$$\tau(I) = \tau_0 \exp(E_a / \kappa I) \tag{9}$$

where τ (T) is the relaxation time and τ_0 is the relaxation constant. The activation energy calculated from the isochronal as shown in Table 1. In PVDF: PSF blend films, which contain segments with greater dipole moments than in individual polymer because under high field the reorientation process of dipoles is fast as compared to low field in the bled. It is expected that the blend film will experience morphological changes which create more traps to immobilize the charges and polymer chains becomes more facile at the higher values of temperature. The polymer blend is a heterogeneous system and as such it has a very large number of trapping sites. It appears that at shorter times and low charging temperatures, only shallow traps are involved which get implied earlier giving rise to higher value of n. At longer times and higher temperatures, more and more deep traps are filled. The trapping of charge carriers (i.e. electrons and holes) in deep traps may lead to induce dipole formation. The energy required for their disorientation and release of charge carriers from these trapping

sites will be high and as such the discharge current and value of n will be low. Thus, in conclusion it may be suggested that the observed transient-current curves are composite in nature and can be explained on the basis of dipolar and space charge effects. The space charge polarization sets up with a fast setting time,²⁵ while the dipolar polarization requires a longer time period because of inertia of dipoles and molecules. It appears that at shorter times, hopping charge and space charges contribute significantly resulting in a complex dependence on the charging field.



Figure 7: Desorption current peak temperature versus increasing PSF weight % in the polyblend

In the present case of PVDF: PSF blend samples, several of the various concepts previously postulated to account for the transient conduction phenomenon can be ruled out on the basis of experimental results discussed above. Tunneling can be explicitly ruled out as a possible mechanism since the currents are strongly temperature dependent as suggested⁷ and the isochronal in the case of desorption current are characterized by peaks in our results, indicating thermal activation of desorption current over a particular temperature range. The transient absorption and desorption currents are not the mirror images of each other, which is also the essential condition for tunneling, dipole or electrode polarization process and supports the ruling out behavior of tunneling and electrode polarization for the individual polymers. In the present case, the large currents obtained just after the application of voltage subsided to much smaller steady values after a certain length of time as revealed at all poling temperatures can also be explained on the basis of the theory of space charge limited conduction (SCLC). It is reported²⁸ that this theory is based on the charge produced by the influence of traps. The defects and impurities can govern the charge transport mechanism and also act as trapping centers and get populated by the injected charge carriers from the electrodes. In the present study, the large currents obtained just after the application of field subsided too much smaller steady values after a certain length of time. The possible explanation is that the sudden application of field 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 remains untrapped, the value of the transient current would continue as a steady current as observed in our study. 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 carriers.²² These injected charge carriers easily passes through the interface between PVDF and PSF and move in to either of the PVDF or PSF region. This process enhances the conductivity of the blend samples. The properties of the amorphous regions dominate the conductivity behavior of such heterogeneous blend films reported in literature.²⁹ The polymer-polymer interface is formed in blend sample. The interface provides additional trapping sites for charge carriers which are distributed over a considerably wide energy range.³⁰⁻³² Since there are many localized states, the release or excitation of the carriers in these states dominates the conduction process. These localized states act as carrier trapping canters, and after trapping the injected charge from electrodes are expected to build up a space charge. This buildup of space charge plays the key role in the determination of conduction process. It is then concluded that measurements of time dependent absorption and desorption currents also clearly point out that current transients in PVDF, PSF and PVDF: PSF polymer blends originate due to dipolar orientation and trapped space charge undergoing limited hopping amongst localized states. Further absorption and desorption phenomenon depend strongly on the polymer structure suggested.²⁵ The magnitude of absorption and desorption currents in blend samples have been found to be higher than that of the individual polymers. This can be explained on the basis of Maxwell-Wagner effect studied³³⁻³⁶ occurs in heterogeneous dielectric as a result of the build-up of space charges at interface between two having different permittivity and conductivities when such a sample is heated and subjected to a field.

CONCLUSIONS

The systematic analysis of various results from such studies has indicated that the time dependent desorption current in polymer blend samples may be due combined effect of dipole reorientation, space charge dissipation and hopping of charge carriers. Thermal activation of isochronal current in a specific temperature range followed by a peak centered at about 120-160°C, have indicated the close correlation of transient currents with the TSDC's. Space charges have been found to make considerable contribution to TSDC's and also to time dependent desorption currents in the present case. As has been mentioned at several places, such charges may be attributed to the localization of bulk generated carriers and also to injected charge carriers into various traps indicating the existence of trapping levels of various origins which may be distributed both energetically and spatially. Trap depths of such levels obtained from desorption currents as well as TSDC's are of the same order indicating that in both cases the same group of traps is operative. However, absorption and desorption current study has shown these trap depths to be time dependent.

The present experimental evidences suggest that the transient currents observed at temperatures lower than the glass transition temperature as a result of the application of low to moderate voltage steps may be essentially explained in terms of a dipolar relaxation mechanism or a number of such overlapping processes. It directly follows that the position of the measuring temperature with respect to the temperature ranges of the various relaxation processes is the predominant factor determining the transient behaviour.

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