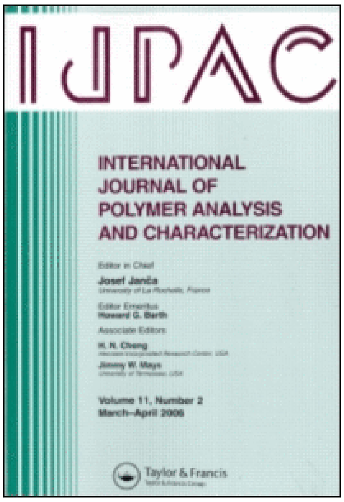

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S. Berdous[a]; N. Saidi-Amroun[a]; M. Saidi[a]; M. Bendaoud[a]
[a] Physics Faculty, Material Physics Laboratory, University of Science and Technology (USTHB), Algiers, Algeria

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CORRELATION BETWEEN MORPHOLOGY AND ELECTRICAL RESPONSE IN PEN POLYESTER FILM

S. Berdous, N. Saidi-Amroun, M. Saidi, and M. Bendaoud
Physics Faculty, Material Physics Laboratory, University of Science and Technology (USTHB), Algiers, Algeria

Thermoplastic polyesters have been widely used in electrical and electronic equipment and a large number of articles on these materials have been published in the past several decades. Poly (ethyethylene-2,6-naphthalene dicarboxylate) (PEN) is a new aromatic polyester that presents high-performance physical and chemical properties. It is well known that the study of the physicallchemical structure and its effect on the behavior of charge carriers is necessary for the development of electrical materials and the further improvement of high-performance apparatus and cables. In this study we try to identify the structure of PEN by using Fourier transform-infrared spectroscopy (FT-IR) and electron microscopy in order to explain its electrical response. This latter is determined by the measurements of charging and discharging currents as a function of temperature and electrical field. The currents are considered to be due to dipolar reorientation associated with structural motions and space charge relaxations due to trapping of injected charge carriers in energetically distributed traps.

Keywords: Electrical response; FT-IR spectroscopy; Morphology; PEN film; Transient regime

INTRODUCTION

For several years polymers have held a place in industry, in particular, in the electrical engineering field. The insulating virtues of some polymers such as polyethylene (PE), polyethylene terephthalate (PET), and polypropylene (PP) led to their use in various fields either as an insulator, as in the case of cables for energy transport, or as dielectric. The manifestation of a new material in this field, poly(ethylene-2,6-naphthalene dicarboxylate) (PEN), has encouraged many studies during the past few years from the point of view of their electrical properties, making this material a serious competitor with PET and PP.[1]

PEN is a polymer obtained by dicarboxylic polycondensation of the acid naphthalene-2,6 and ethylene glycol.[2] The process used for the manufacture of films calls upon the dicarboxylic cross esterification of the dimethyl ester of the acid naphthalene-2,6 with the ethylene glycol.[3]

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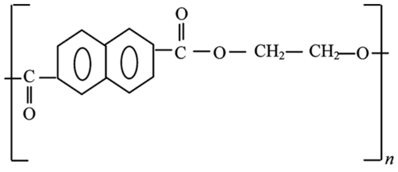
Du Pont de Nemours is thanked for providing us with PEN samples.

Correspondence: S. Berdous, Physics Faculty, Material Physics Laboratory, University of Science and Technology (USTHB), Bp N°32, El-Alia, Bab Ezzouar, Algiers 16111, Algeria. E-mail: gebesadz@yahoo.fr

The chemical structure is shown below.



(1)

PEN is different from PET by the presence of an additional aromatic cycle, which confers on it better mechanical, thermal, and chemical properties. In general, the properties of PEN depend strongly on the morphology of the sample. Therefore, in order to tailor its properties to a given application, it is important to know the structure-property relationship. During the past two decades, several articles concerning PEN have been published that have dealt mainly with semicrystalline biaxially stretched and amorphous samples[4–6] as well as with structure and texture[7,8] and dielectric, photoluminescence, and electric properties.[9–11]

Several experimental techniques can meet the demand to detect the morphological structure and orientation in films. Among them, Fourier transform-infrared spectroscopy (FT-IR) is a promising technique and has a great advantage: it can probe directly to such subtle details as intermolecular interactions, localized molecular conformations, and orientations.[22] Many studies have been reported on infrared analysis of polyethylene terephthalate (PET),[26] but only a few studies have been reported on the structural analysis of PEN with different morphologies.[23–25]

Polyethylene naphthalate (PEN) is a common dielectric used in film capacitors (class-F insulator). One of its major advantages is the possibility to process it into ultrathin films (down to 10^{-6} m), which is important in today's trend of the miniaturization of electronic components. It is well known that the measurement of charging and discharging currents flowing through capacitors is often used to characterize their electrical properties.

It is generally observed that the current passing through a polymer film by application of a step voltage decays with time following approximately the power law[12,13]

$$I_{\mathrm{charge}}(t) = A(T)t^{-n} + I_{\infty} \tag{2}$$

This is the well-known Curie–Von Schweidler relationship where I_{charge} is the charging current observed at time t after the application of the field, $A(T)$ is a temperature-dependent parameter, n is a constant with a value near unity, but may be small as 0.6,[14,15] and I_{∞} is the steady-state component of the charging current corresponding to the intrinsic conductivity of the material. A reverse transient discharging current is also observed after the removal of the field and obeys the following relationship:

$$I_{\mathrm{discharge}}(t) = A'(T)t^{-n'} \tag{3}$$

where $A'(T)$ and n' are depolarization parameters similar to $A(T)$ and n, respectively. The origin of these currents is still the subject of much controversy in the literature.

Many articles have been published on the subject and a number of mechanisms have been proposed,[16–21] such as dipolar orientation, hopping of charge carriers from one localized state to another, interfacial bulk polarization (crystalline-amorphous boundaries, etc.), electrode polarization (hetero space charge), and injected homo space charge.

The process that actually occurs must be determined after a systematic study of the influence of parameters such as temperature, field strength, time, and so on.

The aim of this article is to present some morphologic and electrical characteristics obtained for PEN film, in order to try to understand the influence of the morphology on the conduction current behavior. An approach to the mechanisms governing transient conductivity is also proposed.

EXPERIMENTAL SECTION

Sample

PEN sample was obtained from Du Pont de Nemours (Luxembourg) as biaxially stretched film with thickness of 25×10^{-6} m. The semicrystalline PEN was prepared with aluminium evaporated electrodes, 1 cm in diameter on both sides.

Infrared Spectroscopy

The FT-IR spectra in the region of 4000–500 cm^{-1} were recorded using a Perkin–Elmer spectrometer (Spectrum One) in the absorption mode using 2 cm^{-1} resolution and 32 scans.

Electrical Measurement

A DC voltage was applied to a sample at a certain temperature, and the charging current was measured for a period of 10^4 s. The voltage was then removed and the discharging current of the short-circuited sample was also recorded for 10^4 s. The experimental apparatus used in our work is described elsewhere.[27,28] The semicrystalline PEN was examined over the temperature range 100°–180°C in an electrical field from 24×10^6 to 72×10^6 V/m.

RESULTS AND DISCUSSION

Structure Characterization

Figure 1 shows the whole range FT-IR spectra of semicrystalline biaxially oriented PEN film. Chain conformations of PEN can be provided by the spectra, from the appearance of characteristic bands illustrated below 1800 cm^{-1}. Indeed, as summarized in Table I, the band at 768 cm^{-1} is insensitive to crystallinity and is due to deformation vibration of aromatic CH out-of-plane.[29,30] The band at 814, which is characteristic of the crystalline state, is assigned to *trans* conformation of CH_2 out-of-plane. The vibration of (O=C–O) out-of-plane in *trans* conformation of the amorphous state appears at 920 cm^{-1}. The bands near 1135 and 1096 cm^{-1} are assigned to naphthalene ring vibration and gauche conformation C-O symmetry stretching vibration, respectively.[31,32] The band at 1602 cm^{-1} is

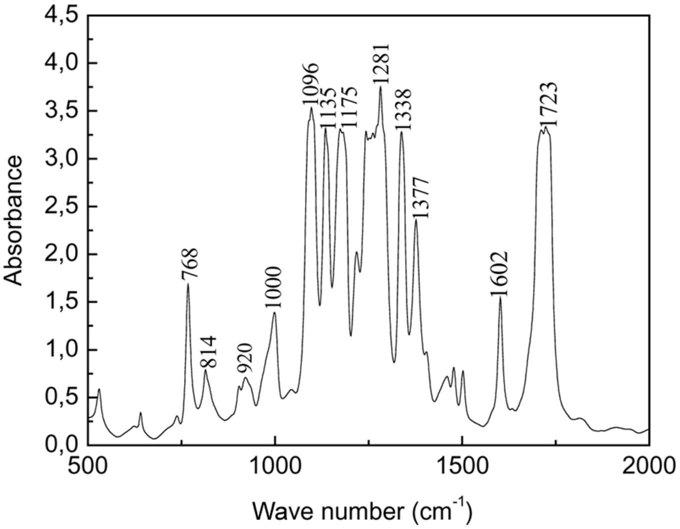


Figure 1. FT-IR spectra in the range 500–2000 cm^{-1} of semicrystalline PEN film.

assigned to naphthalene ring vibration,[30] and the bands at 1377 and 1338 cm^{-1} are attributed to gauche and *trans* conformation of CH_2 wagging vibration, respectively, in crystalline phase.[22,33] The band at 1723 cm^{-1} is assigned to C=O stretching mode.[24]

The crystallization in PEN has two polymorphs of crystals α and β according to the temperature of crystallization, 160°C and 240°C. The unit cell of the α-form was determined by Mencik[34] as triclinic ($a = 0.651 \times 10^{-9}$ m, $b = 0.575 \times$

Table I. Assignment of IR band of PEN

Wave number (cm^{-1})[a]	Band assignment[b]
768	δ (Ring CH out-of-plane)
814	Crystalline
920	(O=C–O)
1000	Crystalline
1096	v_s (C–O) gauche
1135	Naphthalene ring vibration
1175	Naphthalene ring vibration
1281	Naphthalene ring vibration
1338	ω (CH_2) trans
1377	ω (CH_2) gauche
1602	Naphthalene ring vibration
1723	v(C=O)

[a] The wave numbers in bracket represent the peak positions in absorbance IR spectra.

[b] v, stretching; ω, wagging; v_s, symmetry stretching; δ, deformation.

10^{-9} m, $c = 1.32 \times 10^{-9}$ m, $\alpha = 81°, 33$, $\beta = 144°$, $\gamma = 100°$). The other crystal modification, β-form, has been determined by Zachmann et al.,[35] it is also triclinic, with the following unit cell parameters: $a = 0.926 \times 10^{-9}$ m, $b = 1.559 \times 10^{-9}$ m, $c = 1.273 \times 10^{-9}$ m, $\alpha = 121.6°$, $\beta = 95.57°$, $\gamma = 122.52°$. Four macromolecular chains pass each cell, but they are not completely extended, which means that every naphthalene ring is twisted by 180°.[36]

It can be concluded from the appearance of characteristics bands at 814, 1000, 1338, 1377, 1602, and 1723 cm^{-1} that the α-form crystalline structure is formed. In fact, Cakmak et al.[37] have shown that the α-structure is obtained when film is biaxially oriented, as our PEN sample is. Through X-ray observations they confirmed that the naphthalene planes are oriented preferentially parallel to the surface of the biaxially stretched films. Mencik,[34] in the structural study of PEN by means of X-rays, has shown that all the atoms of a chain are coplanar except for aliphatic hydrogen atoms, when the crystalline structure is α. This is an important difference with PET, of which the benzene ring makes a 12° angle with the carbonyl plane, which favors a piled up structure, i.e., a crystalline structure. This flatness is attributed to the resonance energy of naphthalene, which is higher than for benzene. With the C=O bonds of carbonyl groups having the character of a π bond and separated from the naphthalene by only one bond, the conjugation of the aromatic system may be extended to the carbonyl groups.[39]

Insulating polymeric materials such as PEN are generally semicrystalline. They are constituted by an intimate mixture of ordered crystals:

1. A crystalline phase (spherulite superstructure in the annealed amorphous PEN or dispersed lamellae in the biaxially drawn semicrystalline PEN).
2. A mobile amorphous phase, which is a more or less random chain conformation.
3. A restricted or rigid amorphous phase (RAP), whose existence is an opened question for PEN and which is a phase close to the crystallites.

From Figure 2, which presents a bulk view of PEN film under an electron microscope, we can deduce, according to several publications,[36,38,40] that it has a complex morphological organization. The bulk is constituted by a number of crystallites or lamellae classified in parallel layers separated by the amorphous, interlamellar regions.

Electrical Response: Transient Regime

Figure 3 shows typical time evolution of charging currents of PEN films with temperature parameter, for an applied field of 72×10^6 V/m.

It should be noted that the charging current shows a minimum (in other terms, maximum[41]) that becomes shorter with increasing temperature. For $T < 140$°C, the minimum shifts gradually towards the shorter times, but for $T > 140$°C this move turns out to be very quick. In addition, it is evident from Figure 3 that the initial slope of the charging current curves decreases with the rise of the temperature.

The main mechanisms that have been put forward in order to interpret the transient decay flowing through a dielectric after the application or removal of a step voltage were already recalled in the introduction. From the experiments discussed in this article it is not yet possible to unambiguously identify which of

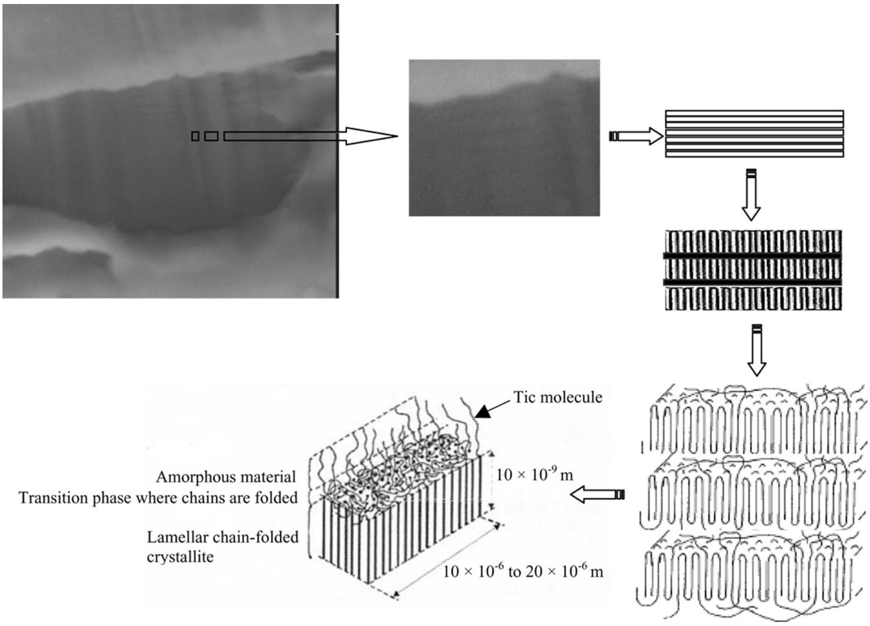


Figure 2. Lamellar structure of semicrystalline PEN film.

them is (are) the relevant one(s). Nevertheless, the analysis of our data and their comparison with the results obtained by other authors allowed us to present some experimental evidence leading us to take into account or to reject some of these models as representative of the current observed in polyester films of the PEN type.

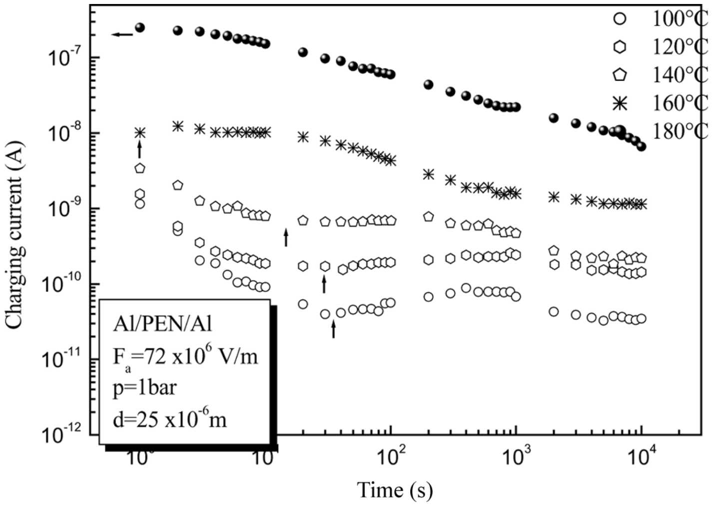


Figure 3. Time dependence of charging currents in PEN, at $F_a = 72 \times 10^6\,\text{V/m}$.

The charging current peak in Figure 3 is similar to the transient SCLC given by Many and Rakavy.[44] According to them, the transient space charge limited current (SCLC) in a trap-free insulator shows a peak at $t = t_m$:

$$t_m = 0.786\, d/\mu F_a \tag{4}$$

where F_a is the applied field, d the sample thickness, μ the carrier mobility, and ε the permittivity of the sample.

The above equation can be extended to the shallow trap case where carriers drift via shallow traps of depth E_t between the electrodes. Then the mobility μ is replaced by μ_e:

$$\mu_e = (n_f/n_t) \cdot \mu \approx \mu_0 \exp(-E_t/kT) \tag{5}$$

where n_f and n_t are the densities of free and trapped carriers and μ_0 is a constant.

The resulting value, $\mu = 10^{-16}\,\mathrm{m}^2 \cdot \mathrm{V}^{-1} \cdot \mathrm{s}^{-1}$, is comparable with other published data.[45,46] Such behavior of the mobility suggests[46,47] a hopping conduction between localized states.

Semicrystalline polymers, such as PEN, have lots of physical or chemical defects that provide localized states (trapping and hopping states) in the band gap, as presented in Figure 4. Typical physical defects include, for example, various disorders in chain folding, microcavities, density fluctuation, and defects in the amorphous/crystalline boundary. Another type of trap or hopping site may be due to chemical defects: chain branching, cross-linking, double bounds, impurities, residual catalysts, and so on.[42]

At temperatures much lower than the glass transition temperature ($T_g \approx 128$°C), several of the transient conduction phenomena in PEN were determined on the basis of the experimental facts. Previous works[44,45] on semicrystalline PEN sample have led to the identification of the dipole orientation

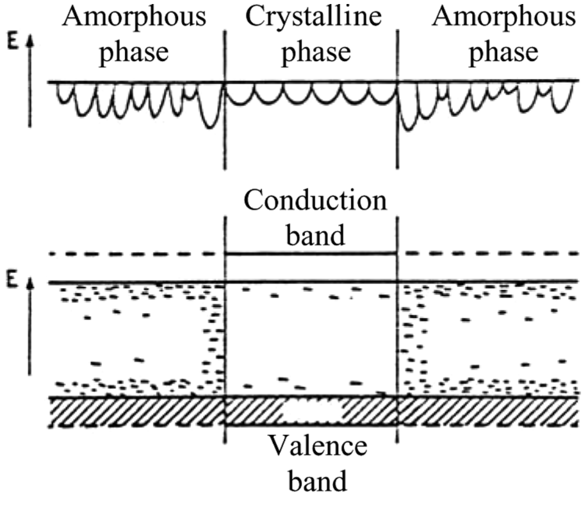


Figure 4. Band structure of semicrystalline polymers.

current. These transient currents are associated with the empirical law of Curie with a slope of $n = 1$. These currents are attributed to the progressively dipolar orientation. The dipoles will be oriented with the electrical field, and the amplitude of this orientation is related to the material structure and to the thermal activation. In addition, the more probable dielectric relaxation linked to the dipolar effect is the β^*, which is associated with the out-of-plane motions of the naphthalene ring and perhaps to the aggregates of naphthalene. This process is likely in a cooperative nature due to its high activation energy.[38]

Figure 5 shows charging and discharging current characteristics for an applied field of 72×10^6 V/m for temperatures of 100, 120, 130, 140, 160, and 180°C.

It can be seen that the discharging current plots are entirely different from those of charging currents. The difference between charging and discharging currents increases with the rise of temperature. It appears that the charge involved in the charging period is greater than in the discharge period. The possible reason for this difference may be that the initial transients occur with empty traps, and currents will be as large as allowed by the injecting barriers. As the traps become filled, the current reduces to the space charge limited current with traps (SCLC). In the discharging period, on the other hand, the trapped carriers will be discharged towards both electrodes, showing a smaller current in the external circuit. The transient charging and discharging currents are not the mirror images of each other, which is the essential condition for tunneling, dipole, or electrode polarization process. The value of n for the discharge was observed to be less than 1 and to decrease with the rise of temperature. This can be observed by the decreasing slope of the initial discharging current, i.e., at lower values of charge but for longer periods of time, the value of n becomes greater than 1.[43] The field dependence of the charging currents at 140°C is shown in Figure 6.

For any given electrical field, the time dependence of the transient current is found to obey the law of dielectric responses. Soon after the application of the

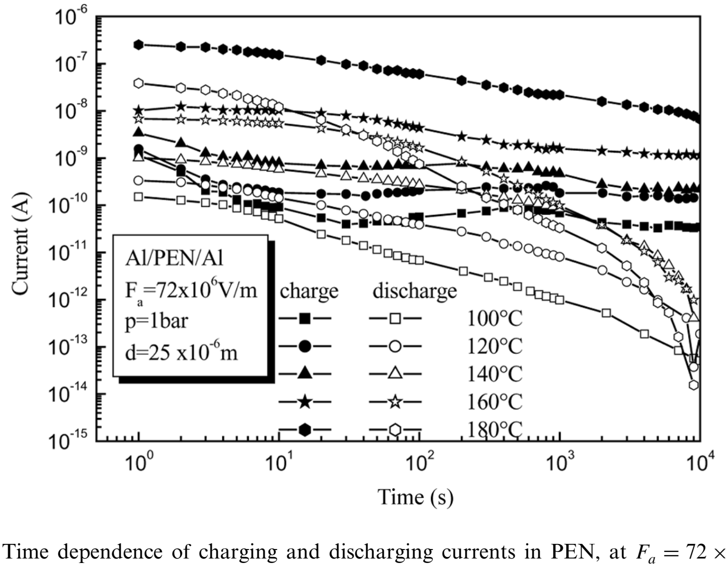


Figure 5. Time dependence of charging and discharging currents in PEN, at $F_a = 72 \times 10^6$ V/m.

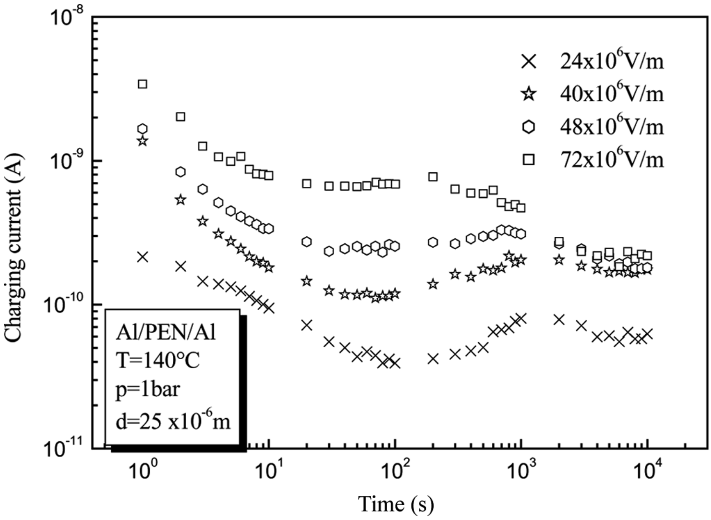


Figure 6. Time dependence of charging currents in PEN, at $T = 140°C$.

poling field in a metal-insulator-metal system, the current rises rapidly and then approaches a constant value. It can be seen that our results deviate from this behavior. From Figure 6, it is also observed that the rate of fall of the absorption current, $(I_{\text{charge}} - I_{\infty})$, is lower for lower electrical fields than for higher fields. It means that the polarization time decreases with the increase of electrical field, suggesting a dependence of the resistance of the film on the level and duration of

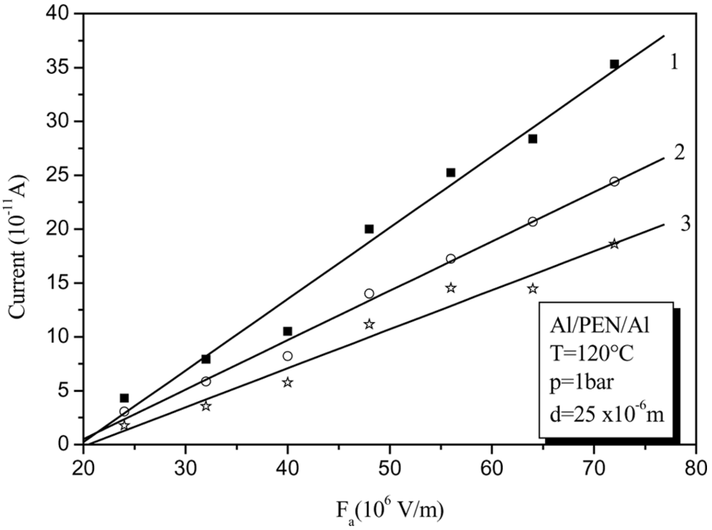


Figure 7. Field dependence of the isochronal transient currents, at $T = 120°C$, chosen after various observation times for PEN film: (1) $t = 3\,s$; (2) $t = 5\,s$; (3) $t = 10\,s$.

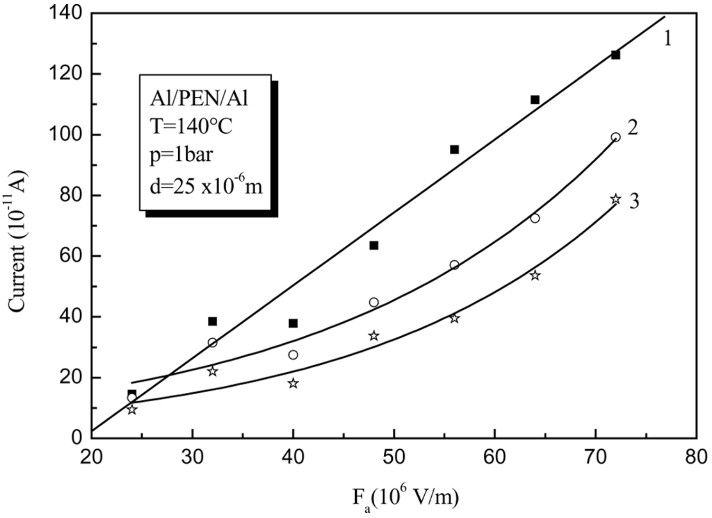


Figure 8. Field dependence of the isochronal transient currents, at $T = 140°C$, chosen after various observation times for PEN film: (1) $t = 3\,s$; (2) $t = 5\,s$; (3) $t = 10\,s$.

the applied field. The time-dependent charging current suggests that the polarization in a sample may be due to dipolar orientation and trapping of charge carriers in the bulk, whose injection from the traps will increase with increasing field and temperature. 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. However, one must take into account the effects

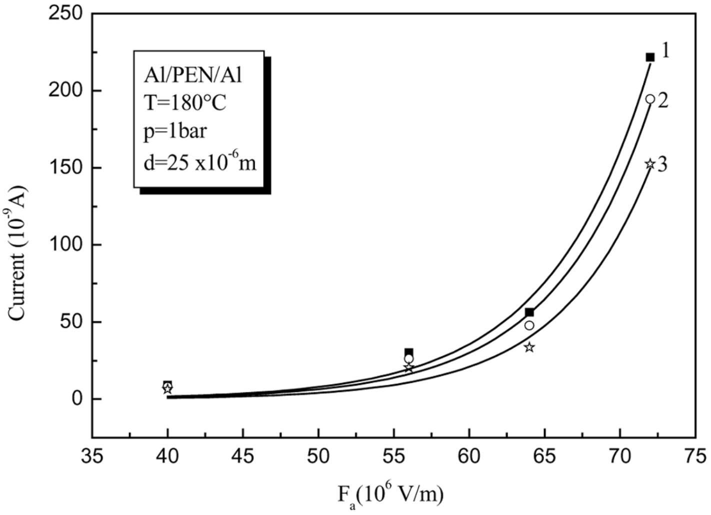


Figure 9. Field dependence of the isochronal transient currents, at $T = 180°C$, chosen after various observation times for PEN film: (1) $t = 3\,s$; (2) $t = 5\,s$; (3) $t = 10\,s$.

of trap densities in the sample. The free charge forced into the sample settles into the traps and one observes the decay of current, the rate being determined by the capture cross section of traps for free carriers. The shifting of the minimum is also due to the progressive superposition of the current growing exponentially.[38]

The field dependence of isochronal currents (i.e., the charging current at a particular time) recorded in the transient range at 120°C, $T < T_g$, is shown in Figure 7. It shows a good linearity in the whole range of field for times 3, 5, and 10 s, emphasizing the ohmic nature of the mechanism responsible of the transient decay.

On the other hand, at $T = 140°\text{C}$, temperature above T_g, the isochronal currents observed at 5 and 10 s are characterized by a pronounced supra linear dependence, deviating from the ohmic behavior (Figure 8). This behavior is also observed at 180°C for each isochronal time (Figure 9).

The supra linear behavior supports the theory of charge injection forming trapped space charges.[42,48]

CONCLUSION

In our work, the characterization of the structure of PEN polyester film allowed us to conclude that it has a very complicated morphology where the crystalline and amorphous parts are mixed. It indicates the presence of lots of defects (physical and chemical) that provide localized states or traps in the band gap of the sample.

The temperature and electrical field dependence of the charging currents in the transient regime, the difference observed between charging and discharging currents, and the supra linear nature of the isochronal current seem to be sufficient and reliable grounds to assume that the possible controlling mechanism for the transient phenomenon is the charge injection and formation of trapped charges.

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