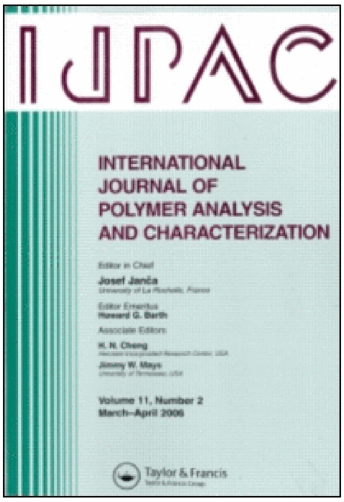

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Dielectric Characterization of Poly Ethylene Naphtalene 2,6 Dicarboxylate (PEN) by Thermally Stimulated Currents Technique (TSC)

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Abstract: The thermally stimulated currents technique has been used to study the relaxations at temperatures between 0° and 160°C in polyethylene naphthalate (PEN). Different material structures were obtained by different treatments. Consequently, for each sample, we have obtained one peak located around 130°C, which is attributed to the dielectric manifestation of the glass transition (α relaxation). The thermal windowing method permitted us to evaluate the activation enthalpy and the relaxation times for each sample. Compensation phenomenon was observed at a temperature T_c, which increases with the crystallinity rate χ_c. The area under each elementary peak permits calculating the relative dielectric dispersion. The knowledge of this latter and of the relaxation time $\tau(T)$ allows us to deduce experimentally the real and the imaginary parts of the dielectric permittivity ε^* for each temperature T and at an equivalent frequency in TSC ($f = 10^{-3}$ Hz). The imaginary part of ε^* decreases with χ_c. The Cole and Cole diagrams were then deduced.

Keywords: Annealing; Crystallinity; D56; PEN; TSC

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INTRODUCTION

Poly(ethylene naphtalene 2,6 dicarboxylate) (PEN) belongs to the polyester family. It results from the polycondensation of 2,6-naphtalene dicarboxylic acid and ethylene glycol.[1,2] The presence of the double naphthalene group in PEN's formula provides a higher stiffness to the macromolecular chain of the material. For this reason, PEN presents better thermomechanical properties than poly (ethylene terephtalate) (PET). The increased aromaticity properties in PEN favor its use as film materials in electronic and packaging[3–6] (miniaturization of capacitors, base film of long-playing videotapes, etc.).

The applications of this material in electronics require a wide knowledge of its dielectric behavior. That is why the combination of thermally stimulated current (TSC) measurements with differential scanning calorimetry (DSC) presents a powerful experimental tool to study the influence of morphological changes of the dielectric properties of PEN.

The thermally stimulated currents method is equivalent to dielectric loss measurements at very low frequency (10^{-2} to 10^{-4} Hz).[7] It has been widely used to study fundamental characteristics in various materials including films, powders, and crystalline and amorphous insulators. With regard to its high resolution power, the TSC technique permits identifying the origin of molecular relaxations and allows one to detect the transitions that other techniques cannot detect.[8–10]

The TSC method[11–14] consists of applying a DC voltage to a material in order to orient dipoles that are sensitive to electrical field. After cooling down, the sample is short-circuited and the depolarization current is measured at a constant heating rate. The resulting thermally stimulated current peaks represent typical relaxation processes similar to those of loss in dielectric measurements. Temperature can be scanned from −180° to 200°C. To study the relaxation mode in more detail, the peak cleaning method (thermal windowing method)[15] was used by resolving the complex peak into an elementary spectrum with thermal windows of 5 K.

Amorphous PEN is characterized by three characteristic temperatures[6]: glass transition temperature at about 124°C, melting temperature around 268°C, and crystallization temperature around 200°C. In this study, particular interest was directed to the glass transition phenomenon appearing around 124°C by using the TSC technique. Thermally stimulated currents in amorphous and semicrystalline PEN films have been investigated in the temperature range from 0° to 160°C. Thermal windowing technique[15] was applied to study individual modes of relaxation using Debye's model.[16] The area under each elementary peak permits us to calculate the relative dielectric dispersion. The knowledge of this latter and the relaxation time $\tau(T)$ allows 45 to deduce experimentally the real and the imaginary parts of dielectric permittivity for each temperature T and at an equivalent frequency in TSC ($f = 10^{-3}$ Hz). The Cole and Cole diagrams were then deduced.

EXPERIMENTAL SECTION

Materials and Methods

The measurements were carried out on commercial PEN under reference LS 308063 supplied by Goodfellow (U.K.) and biaxially stretched (PEN_{biax}) in 125 μm thickness and 1.36 g/cm^3 density.

Amorphous PEN films (PEN_a) were prepared with a diameter of 13 mm and thickness of 125 μm by fusion and quenching in molds. PEN under amorphous and semicrystalline forms obtained by annealing amorphous films at 170°C for 15 min (PEN_{15}) and 45 min (PEN_{45}) were used.

The degrees of crystallinity of the samples were determined by a Q10 TA instrument DSC. The temperature of the instrument was calibrated with indium. The sample ($m = 10$ mg) was sealed in aluminum pans and the recording of the DSC thermogram was carried out at a heating rate of 10°C/min.

Depolarization current measurements (TSC) were carried out on samples in the form of square sheets. The electrodes were prepared on both sides of the sheet by vacuum evaporation with 100 nm thickness of gold in order to increase the ohmic contact with the electrodes. The sample placed between the plates of the condenser is put in a measurement cell maintained at a constant pressure of 400 torr under N_2 gas. Then, it is polarized by an electrical field E_p at the polarization temperature $T_p = 130°C$, for an interval of time $\Delta t_p = 2$ min, usually much longer than the polarization relaxation time, in order to reach saturation. The temperature is then decreased to $T_0 = 0°C$ with the polarizing field still on. The field was then switched off, the sample quenched to liquid nitrogen temperature, and the electrodes short-circuited in order to eliminate the parasite charge. The condenser was then connected to the input of an electrometer (Keithley 610C) and the sample heated at a constant rate (7°C/min). The temperature was measured by means of Pt100 probes located very near the sample.

The relaxation time decreases and a depolarization current was detected by the electrometer, which is connected to a microcomputer for the data acquisition.

Analysis of Elementary TSC Peaks

The analysis of TSC peaks is based on the classical Debye treatment with a single relaxation time, widely developed in the literature.[16] The expression of relaxation time $\tau(\mathrm{T})$ is given by the Arrhenius equation:

$$\tau(\mathrm{T}) = \tau_0 \exp \frac{\Delta \mathrm{H}}{\mathrm{kT}} \tag{1}$$

where $\Delta\mathrm{H}$ is the activation energy, τ_0 is the pre-exponential factor, and k is the Boltzmann constant.

The plot of $\ln(\tau)$ as a function of $1/\mathrm{T}$ is a straight line whose slope gives the values of $\Delta\mathrm{H}$. τ_0 is obtained by extrapolation at infinite temperature.

Compensation phenomenon is defined by the linear dependence of the activated state enthalpy and entropy.[17] It is considered that:

$$\tau_0 = \tau_c \exp \frac{\Delta \mathrm{H}}{\mathrm{kT_c}} \tag{2}$$

$\tau_c = \tau(\mathrm{T_c})$ is the compensation time and $\mathrm{T_c}$ the compensation temperature. At $\mathrm{T_c}$, all elementary processes would have the same relaxation time τ_c.

By assuming that each elementary process is described with Debye's model, the complex permittivity ε^* is given by the following equation[18]:

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + j\omega\tau} \tag{3}$$

where τ is relaxation time, ε_s and ε_∞ are respectively the static and optic dielectric constants, and $\omega = 2\pi\mathrm{f}$ where f is the equivalent frequency in TSC and is equal to $10^{-3}\,\mathrm{Hz}$.[19] Then, the real and imaginary parts of the dielectric constant are given by Equations (4) and (5):

$$\begin{cases} \Delta\varepsilon' = \varepsilon' - \varepsilon_\infty = \dfrac{\varepsilon_s - \varepsilon_\infty}{1 + (\omega\tau)^2} & (4) \\ \varepsilon'' = (\varepsilon_s - \varepsilon_\infty)\dfrac{\omega\tau}{1 + (\omega\tau)^2} & (5) \end{cases}$$

$\varepsilon_s - \varepsilon_\infty$ is calculated by using the following equation[20]:

$$\varepsilon_s - \varepsilon_\infty = \frac{\int_0^\infty \mathrm{J(t)dt}}{\varepsilon_0 \mathrm{E_p}} \tag{6}$$

where $\mathrm{E_p}$ is the intensity of the applied electrical field. The integral $\int_0^\infty \mathrm{J(t)dt}$ represents the area under each elementary peak.

RESULTS AND DISCUSSION

The DSC heating scanning of PEN samples was undertaken to determine their degrees of crystallinity. The obtained thermograms are illustrated in Figure 1. The glass transition is clearly observed in the scan corresponding to the amorphous sample and lies at $T_g = 124°C$ approximately.

Crystallinity rates were deduced from the area of the exothermic, endothermic peaks, and the extrapolated heat of fusion for a pure crystal is ($\Delta H_f = 103.3\,J/g$).[21] The obtained rates are reported in Table I.

Figure 2 displays TSC spectra of PEN in amorphous and semicrystalline forms. Amorphous PEN shows a relaxation located at $T_M = 130°C$ in the temperature range from 0° to 160°C, which is attributed to the dielectric manifestation of the glass transition (α relaxation). It can be noted that there is a slight shift of the temperature of the peak maximum towards higher temperatures when the crystallinity rate χ_c increases. This behavior can be explained in terms of an increase of the amount of the interlamellar fraction with respect to the interspherulitic fraction. The same behavior was observed by Canadas et al.[22] Also, we notice a diminution of the maximal current intensity of the peak. This reduction is due to the diminution of the amorphous fraction of the material resulting from the appearance of a crystalline phase in this latter.[6,23] Then, some segments of the main chain are more ordered, they

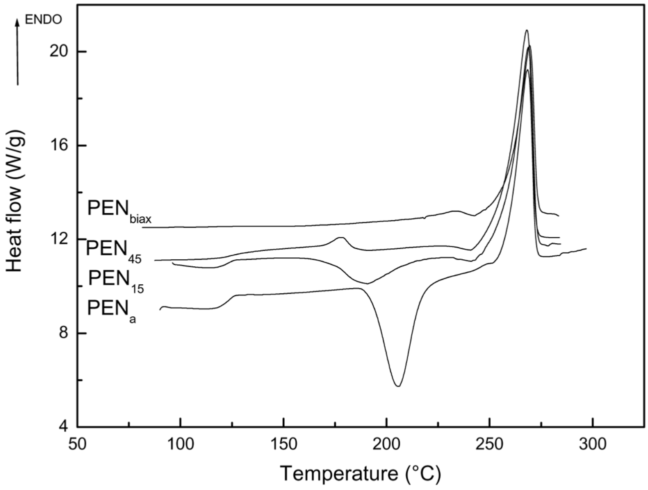


Figure 1. DSC curves obtained for each sample. The curves are normalized to 1 g of matter and shifted on the heat flow axis for legibility. The heating rate is 10°C/min.

Table I. Crystallinity rates of each sample deduced by DSC technique

Sample	PEN_a	PEN_{15}	PEN_{45}	PEN_{biax}
$\chi_c(\%)$	1	32.8	49.6	50.64

have less mobility, and the α peak decreases.[24] As concerns the PEN_{45} and the PEN_{biax}, we notice that although these samples have closer crystallinity rates, the maximal intensity of the TSC peak in PEN_{45} is more important than the TSC peak of PEN_{biax}: this behavior is probably due to the amorphous rigid fraction.[25] This latter is more important in PEN_{biax} than in the PEN_{45}. This difference could be due to the crystallization mode of each sample.

It is generally accepted that TSC spectra arise from dipole reorientation as well as from migration of the impurities and ionic conductors in the polymer. One way to differentiate between them is to examine the relationship between the current intensity and the electrical field. In the inset of Figure 2 is illustrated the evolution of the maximal intensity of PEN_a as a function of polarizing voltage V_p. A linear dependence of the α intensity peak value on V_p is observed, confirming the dipolar nature relaxations process. This increase is explained by the fact that the applied electrical field affects more dipoles as its intensity increases; the same result was found in PET,[23] in PVC,[26,27] and in other polymers.

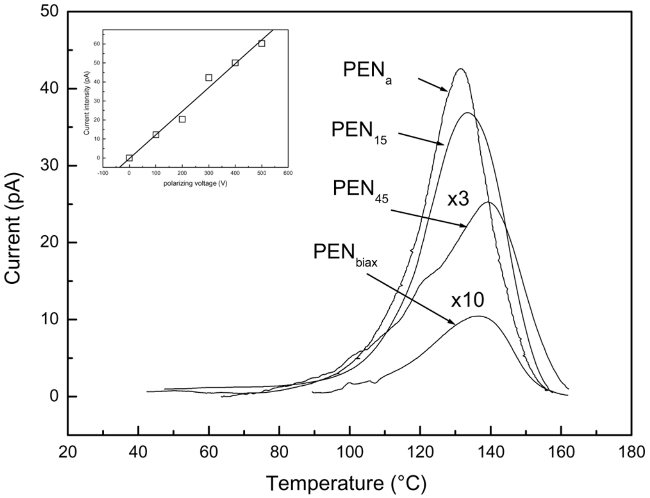


Figure 2. TSC scans of the α relaxation for samples with different crystallinity rates. (Inset: maximal current vs. polarization voltage in the PEN_a sample.)

In order to study this relaxation mode in more detail, the complex spectra have been experimentally resolved into a set of elementary peaks using the thermal windowing technique with a thermal window of 5°C. Figure 3 shows, as an example, the elementary spectra obtained on the PEN_a sample. Each elementary spectrum could be considered as characteristic of sequences relaxing with the same relaxation time and can be transformed into an Arrhenius representation and plotted as $\ln(\tau)$ versus $1/T$. The corresponding Arrhenius diagram is displayed in the inset of Figure 3.

The peak parameters characterizing each elementary spectrum were evaluated by using the Bucci-Fieschi-Guidi method[28] and are listed in Table II. As shown in the inset of Figure 3, the four first lines pass through a common point located at $T = T_c$, suggesting a compensation law. The coordinates of this point for each sample are reported in Table III.

Moreover, the linear evolution of $\ln(\tau)$ versus ΔH for each sample, shown in Figure 4, confirms the existence of such phenomenon characterizing the mobile amorphous phase. It is associated with the existence of an order in the amorphous phase of the material[23,29] and was observed in several materials such as PVC,[26,27] PET,[23] and PC.[30] We remark

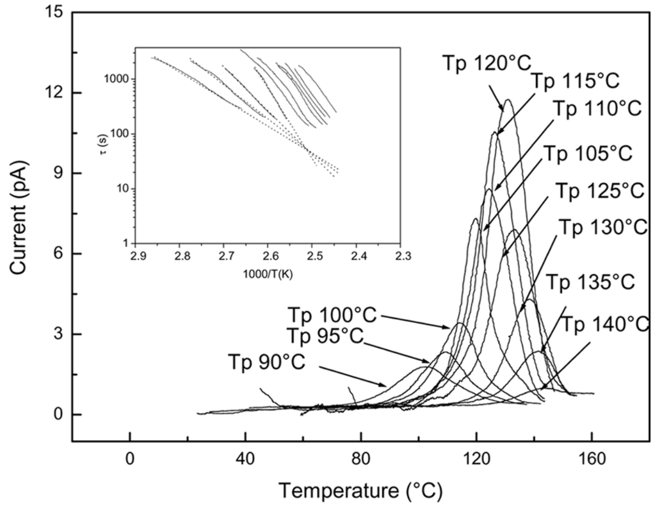


Figure 3. TSC peak profile for PEN_a sample obtained by thermal windowing technique, polarization window $\Delta T = 5°C$, T_p varies from 90° to 140°C, polarization field 160×10^4 V/m, heating rate 7°C/min. (Inset: Arrhenius plot of the relaxation times obtained from TSC measurement for α peak in PEN_a sample. The graph shows a compensation phenomenon around $T_c = 24.5°C$, $\tau_c = 53.14$ s.)

Table II. Specific parameters for α relaxation process involved in the release of the current in the PEN_a, determined by the Bucci-Fieschi-Guidi method

Sample	T_p (°C)	T_M (°C)	ΔH (eV)	τ_0 (s)
PEN_a	90	102.4	0.97	3.06×10^{-11}
	95	109.4	1.30	1.88×10^{-15}
	100	114.4	1.64	8.61×10^{-20}
	105	119.8	2.61	4.38×10^{-32}
	110	124.2	1.90	1.57×10^{-22}
	115	126.3	2.07	1.27×10^{-24}
	120	130.9	2.28	5.95×10^{-27}
	125	131.1	2.05	4.75×10^{-24}
	130	138.7	2.21	6.43×10^{-26}
	135	141.8	2.21	8.33×10^{-26}
	140	144.5	2.06	1.24×10^{-23}

Table III. Coordinates of the compensation point for each sample

Sample	PEN_a	PEN_{15}	PEN_{45}	PEN_{biax}
χ_c (%)	1	32.8	49.6	50.64
T_c (°C)	124.5	150.7	169	163
τ_c (s)	53.14	9.25	0.65	2.29

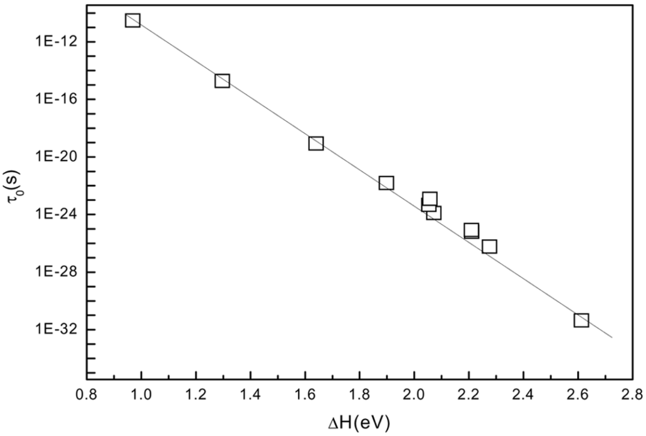


Figure 4. Illustration of the compensation phenomenon observed with the α peak in the PEN_a sample.

that the compensation temperature increases with the crystallinity rate. This evolution is in good agreement with the empirical law we established in our previous work.[26]

In the Debye hypothesis, and by using Equation (6), we have computed the dielectric dispersion for each elementary peak. The real and the imaginary parts of the dielectric permittivity ($\Delta\varepsilon'$ and ε'') were then deduced by using Equations (4) and (5).

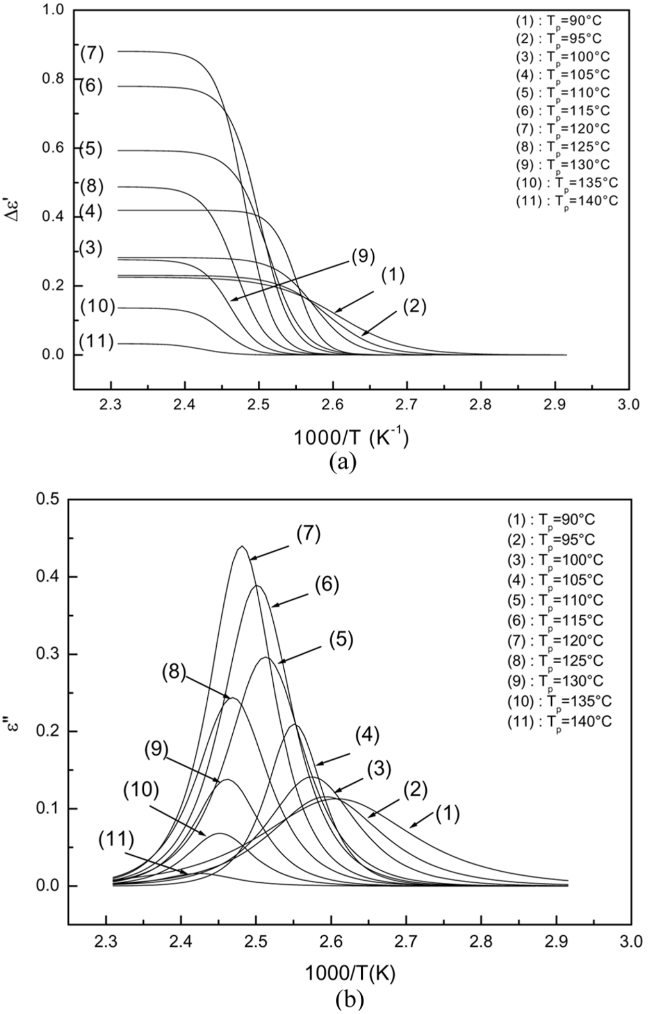
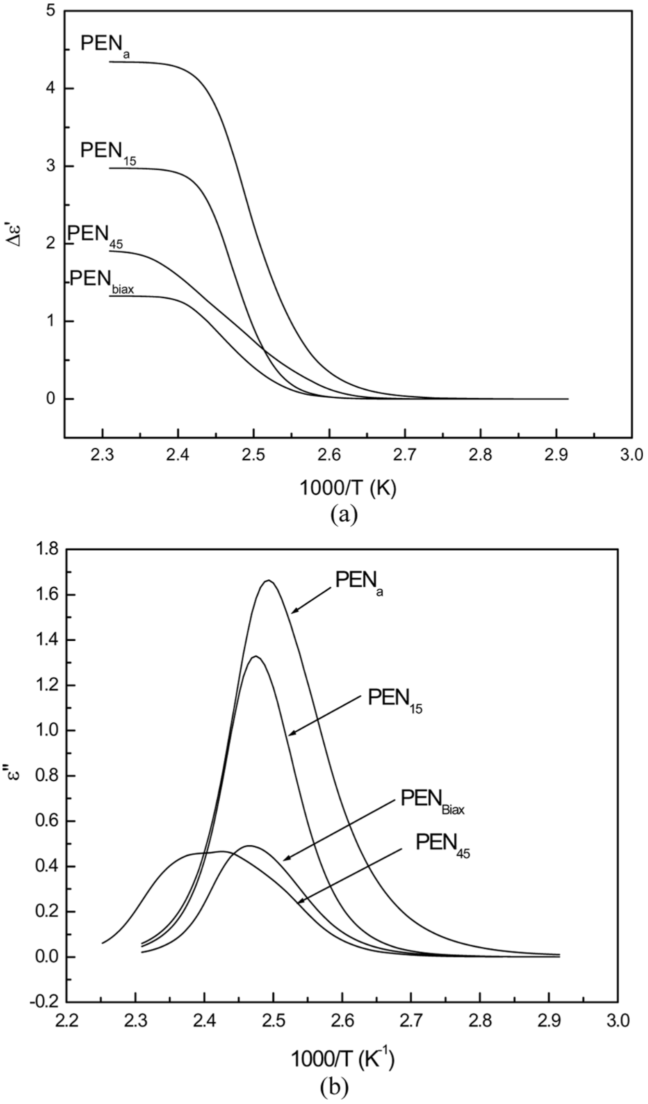


Figure 5. Evolution vs. temperature for each elementary peak of the real and imaginary parts of the dielectric permittivity in the PEN$_a$ sample: (a) evolution of $\Delta\varepsilon'$ and (b) evolution of ε''.

Figures 5(a) and (b) show, as an example, the evolution of $\Delta\varepsilon'$ and ε'' versus 1000/T for each elementary peak in the PEN_a sample. The superposition of the values of $\Delta\varepsilon'$ and ε'' for each elementary peak permits deducing the evolution of the dielectric parameters for the complex spectrum of TSC.



(a)

(b)

Figure 6. Evolution vs. temperature of the real and imaginary parts of the dielectric permittivity in the PEN with different crystallinities: (a) evolution of $\Delta\varepsilon'$ and (b) evolution of ε''.

Figure 6(a) displays the evolution of the real part $\Delta\varepsilon'$ of the dielectric permittivity of each sample. We observe that $\Delta\varepsilon'$ decreases when the crystallinity degree increases. It can be noted that the dielectric maximum losses are situated in the vicinity of the glass transition temperature of the material for each sample (Figure 6(b)). Also, the increase of the crystallinity rate of the material causes the decrease of the dielectric losses. In fact, when the crystallinity rate of the material increases, the molecular mobility decreases. This behavior can be explained, by the fact that the decrease of this latter induces a reduction of energy dissipation. Thus, it results in a diminution of the dielectric losses in the material. Similar results were obtained for PET by Bacharan.[31]

On the other hand, we notice that although the crystallinity rates of PEN_{45} and PEN_{biax} are brought closer, a difference in the shape of ε'' curves of the two samples can be observed. This difference could be due to the difference between the modes of crystallization. In fact, PEN_{45} was obtained by a heat treatment, which induces a spherulitic structure observed by Lee et al.,[32] who used polarized optical microscopy (POM). However, PEN_{biax} was obtained by a mechanical treatment (biaxial stretching).

Figure 7 displays the evolutions of the Cole and Cole diagrams for each studied sample. The obtained plane plots are not a semicircle but skewed. This distortion is due to the dispersion of the relaxation mode. The dotted curve represents the change that we observe in the case of a nondistributed process.

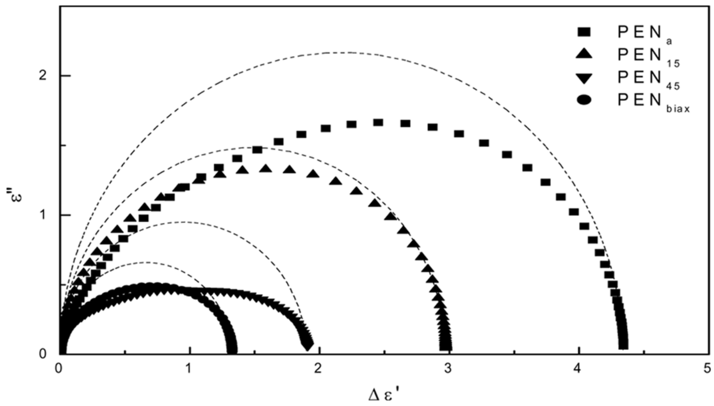


Figure 7. Comparison of the Cole-Cole plots for PEN with different crystallinities.

CONCLUSION

We have reported on the dielectric characterization of PEN by the use of the thermally stimulated currents technique (TSC).

According to the obtained results, it can be concluded that:

- Measurements of TSC spectra carried out with the amorphous and semicrystalline PEN revealed broad peaks around glass-rubber transition temperature of the materials. The maximum intensity of the obtained peaks represents a net dependence with regard to the crystallinity rate of the material and the crystallization mode.
- The complex spectra were resolved into elementary peaks, characterized by one relaxation time. A compensation phenomenon was observed at a temperature T_c, which depends on the crystallinity rate of the materials. This compensation phenomenon is attributed to the existence of an order within the material in its amorphous phases.
- The real and imaginary parts of the dielectric permittivity at low frequency ($f = 10^{-3}$ Hz) have been deduced. A net dependence of the dielectric losses with regard to the crystallinity rate is observed. A net decrease of the imaginary part (ε'') of the dielectric permittivity is observed when the crystallinity rate increases, because the decrease of the molecular mobility in the material induces a reduction of the dissipation of energy in the material.

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