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An Optical Technique Based Mark Tracking for Electrical Field Induced Mechanical Strain Measurement in Thin Polymer Films

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Abstract: An optical non contact measurement technique based on the tracking of successive positions of computerized markers has been developed which enables one to characterize the electric field induced strain response of the plane gold-metalized surfaces in thin organic insulating films. The present study was made on two microstructures of films: a virgin amorphous material and some semi-crystalline samples 70μm thick were obtained by annealing the amorphous one at annealing temperatures of 170°C at 5, 60 and 120 minutes using Differential Scanning Calorimetry (DSC). The test results demonstrate that the newly developed method is capable of detecting displacement of selected markers when the sample is subjected to the application of a dc high voltage. The field-induced mechanical strain measurements have been performed as a function of time and then analyzed with respect to the applied electric field. The observed strain levels varied from $\sim 10^{-3}$ to 10^{-2}. Moreover the influence of crystallinity on the electric field induced strain mechanical response is discussed.

Keywords: Mechanical strain, Dielectric properties, Thin polymeric films, Non destructive optical technique, Mark tracking.

1 Introduction

Due to many advantages of polymeric materials there is a constant effort to develop high performance based electro active polymer for electromechanical transducer applications, and to provide electrical insulation as a dielectric in manufacturing of capacitors. In these applications, the polymeric material behavior under a dc electric field is of prime concern in the material selection. Currently, it still remains a great challenge to reliably determine the strain induced by a dc electric field in thin polymer films without any constraint imposition.

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Poly (Ethylene 2, 6 Naphthalene dicarboxylate) (PEN) is a thermoplastic polyester which combines thermal stability, degradation resistance, low permeability, with excellent mechanical and dielectric properties [1-3]. It can be used as a base film support for very thin gauge electromagnetic tapes such as audio, video and computer tapes and also in capacitors. In this field, it should be mentioned that PEN has a dielectric permittivity smaller and dielectric loss factor lower than that of PET. During the last two decades, relatively few papers about PEN have been published, which deal mainly with semi-crystalline bi-axially stretched samples. The PEN chain structure is close to that of PET but PEN has naphthalene instead of a phenyl group in the repeating unit which leads to a stronger rigidity of the macromolecular chain. It is used recently in electrical engineering as an insulator, as in the case of energy transport cables, or a dielectric as in capacitor manufacturing. The characterization of field induced strain of this polymer film gained early interest in the literature [4, 5] in order to improve the comprehension of phenomena that might lead to the electric breakdown. Based on DSC measurement technique, a thermal cycle of crystallization was carried out. Different specimens were obtained in this way, starting with as-received amorphous polymers.

The recent development of the image processing system demonstrated the high sensitivity of the captured numerical image of gold-metalized flat surface of the tested sample. The image reveals small, contrasting spots. These spots are identified with computerized marking. The displacement of spots when the sample is subjected to electric field allows us to measure in real time the electric field induced strain in thin polymeric samples based on the computer tracking of successive positions of the four markers.

The results from computing software indicates that the present experimental set up can indeed be used reliably for improving this measurement method in order to characterize the electric field induced strain in PEN samples.

2 Experimental method

Experimental samples

PEN samples were obtained from DUPONT TEIJIN FILMS (Luxembourg) in the form of amorphous films with a thickness of 70μm. Studies were made on the virgin amorphous material and three semi-crystalline samples obtained by annealing the amorphous one at annealing temperatures of 170°C at 5, 60 and 120 minutes. To guarantee a better electrode/polymer contact, the test samples were metalized by gold coating using a S150B plasma sputter coater. Electrodes of 20 mm diameter and 30 nm thickness were thus obtained on both sample sides.

Thermal treatment

The thermal analysis of PEN films was performed using a TA Instruments device of type DSC 2010. The as-received amorphous thin films were first maintained at 170°C for 5, 60 and 120 minutes. Then, these samples were cooled down to 30°C with rate of -10°Cmin-1 and the DSC measurements have been carried out from 30 to 300°C with a heating rate of 10°C min-1. This technique allows us to determine the glass transition (Tg) and the melting point temperature (Tm) of the sample.

Experimental apparatus and procedure

The Fig. 1 shows the diagram of the experimental device developed to quantify the electric field induced strain measurement by using an optical technique based on the tracking of successive position displacement of four computerized markers by DEFTAC software [6], associated with an optical system formed by an optical microscope connected to a charge coupled device (CCD) camera (resolution 768 pixels by 576 pixels with 256 gray levels) provided by MATROX - METEOR II, image processing card permitted the observation of the sample surface in reflected light.

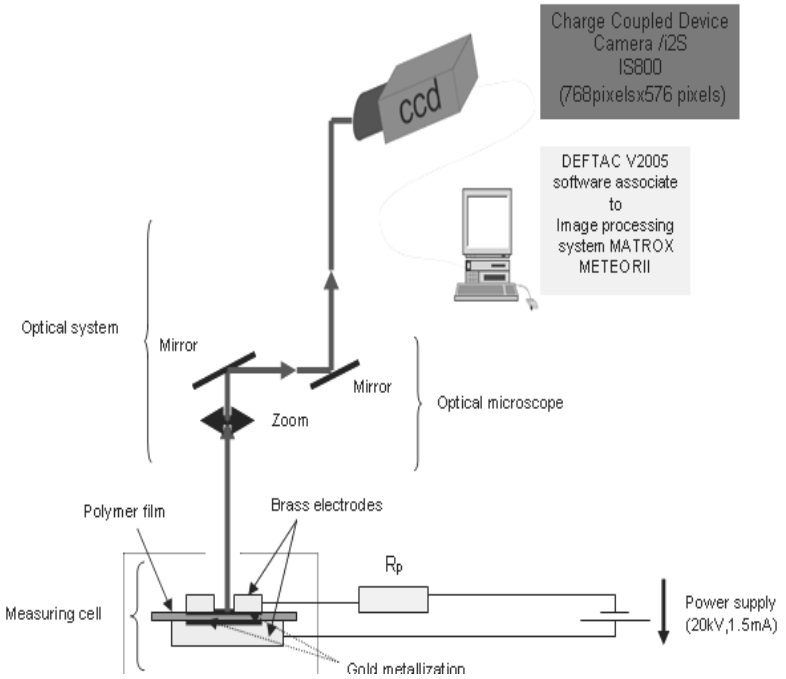

Fig. 1 – *The schematic diagram of the experimental device.*

The prepared sample was placed between two external brass electrodes. The upper one, the negative, was constructed as a hollow cylinder of 12 mm diameter. This permitted the use of a flexible light source to illuminate the upper face of the sample. The lower electrode, the positive, was connected to a high dc voltage source (HCN 35-20000; 20kV and 1.5mA limited current) with controllable output. The samples were placed between electrodes in the measuring cell; the electrodes were short-circuited a few hours before the testing in order to eliminate the initial charges existing on the sample faces before applying the electric field [7].

In order to minimize the influence of the environmental conditions, measurements were carried out at the room temperature at atmospheric pressure, and for relatively short durations. The field-induced mechanical strain measurements were performed as a function of time and then analyzed with respect to the applied electric field.

Mechanical strain measurement

Spots observed on gold metalized sample surface are selected and marked by using the computing software (Fig. 2).

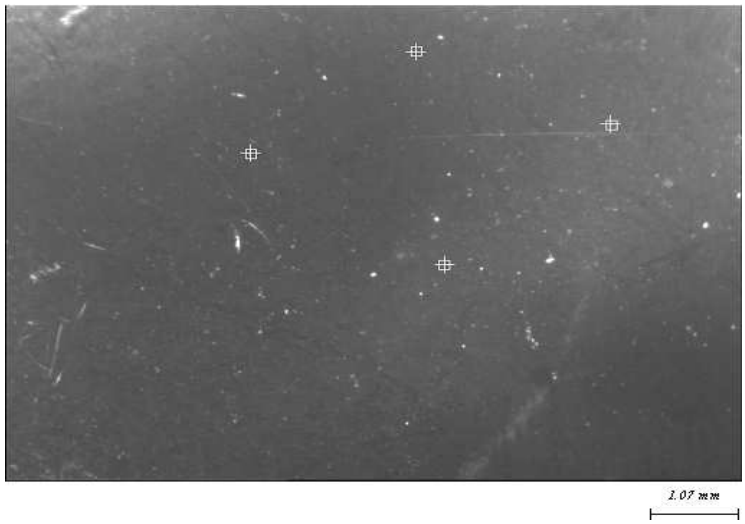

Fig. 2 – *Disposition of four markers on gold–metalized amorphous* PEN *sample.*

The principle of mark tracking is to calculate the geometric centre of the spot from a rectangular zone (the research zone). The experimental technique of strain measurement consists of four markers, forming a cross, positioned on the sides of a parallelogram using the Lagrangean formalism [8] for the deformation of a parallelogram during the application of electrical field. Five induced strain components are measured. These components are ε_x, ε_y, and ε_{xy}, which are

associated with the directions parallel to the x, y, and xy -shearing axes, respectively and are related to image plane of CDD camera [9]. The ε_1 and ε_2 components are associated with the principal a direction at which γxy is null. We have assumed the homogeneity of the deformation on the measurement base, and as a result, the reported values are averages [10].

3 Results and Discussions

DSC measurements have been carried out from 30 to 300°C, in order to characterize the glass transition, the melting point and the crystallization degree of the material. The results obtained with as-received material as well as with the amorphous one. The glass transition, clearly observable in the scan corresponding to the amorphous sample, lies at approximately Tg 123°C. This amorphous sample crystallizes between 160 and 220°C, preceding the fusion of the material at 268°C. The scan corresponding peak, which indicates a high degree of crystallinity in the material. The main endothermic peak, located at 268°C, corresponds to the fusion of the crystalline phase and is preceded by a small endothermic peak probably associated with a solid state transition in the material (Fig. 3). According to the extrapolated heat of fusion for a pure PEN crystal (103.3J.g-1) and the total heat flow involved in the endothermic peaks, a crystallinity degree of about 4.17% is estimated for the as-received material, **Table 1**.

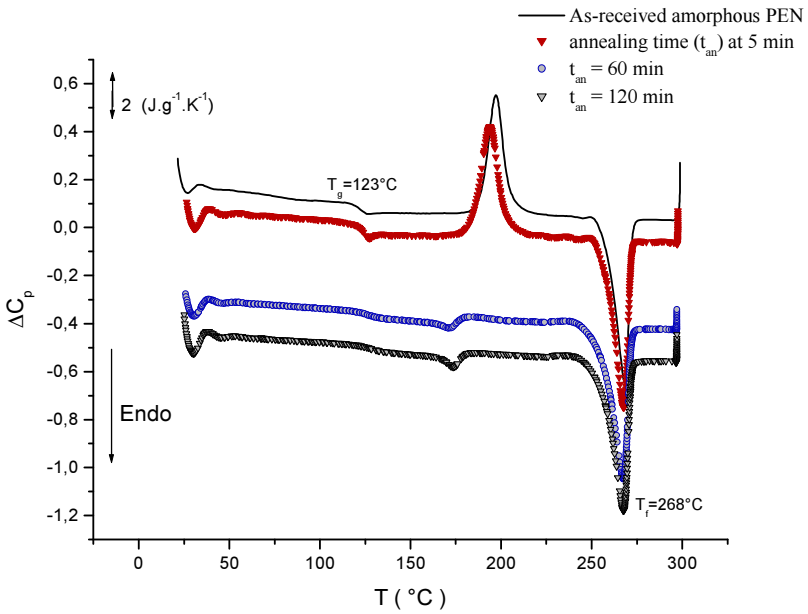

Fig. 3 – DSC *thermograms of* PEN *samples annealed at* 170°C.

The accuracy of the strain measurement is a function of the marker position and the distance between the spots. The measurements of the noise level were carried out for an as-received PEN sample 70 μm thick on a mechanical deformation without an electric field. The measurements of the noise level were carried out for an as-received PEN sample on a mechanical deformation without an electric field. Figs. 4a and b show the measured components of the noise levels corresponding to the distant markers (average distance 2.475mm between 2.40 and 2.55mm; Fig. 2).

Table 1

Annealed as-received amorphous PEN *samples.*

t_a (min)	T_m (°C)	ΔH_c (J/g)	ΔH_m (J/g)	χ %
As-received	268.20	-33.02	37.31	4.17
5	267.68	-32.72	39.71	6.76
60	267.67	+1.64	43.15	43.31
120	267.89	+3.05	42.80	44.34

These figures show that in the case the noise levels were very small and corresponded to the predicted error (2.10-4). For This result we could neglect the effect of the weight of the higher electrode. Moreover the measured mechanical strain deformations in this study corresponded only to the strain induced by the electric field application.

To quantify and analyze the deformation level resulting from applied high dc voltage, PEN films were subjected for periods of 200 seconds at gradually increasing 0.5 kV constant step of high applied voltage. The deformation of the film was recorded with constant CDD camera using a sampling rate (one image per two and half seconds). An attempt was made to compare the level and the evolution of field–induced mechanical deformation in the as-received amorphous and partially crystalized PEN samples.

The evolution of the equivalent induced strain by the electric field shows three zones. Indeed, initially there is a region of very low deformation until an electrical field threshold is attained after which an increasing induced strain level is observed. Finally, there is a diminution of the strain that could be produced by a local densification of the material. This behavior suggests the presence of a thermally dominated mechanism attributed to the propagation of local break-downs caused by large local fields in micro-voids. It is rather a mechanism of electrical aging having a mechanical origin. In order to explore the influence of crystalinity on the equivalent induced strain in treated films, the results given in Fig. 5 reveal higher deformation levels in case of amorphous samples. Furthermore; for annealing time 60 and 120 minutes, the crystalinity degree is

constant respectively 43.31% and 44.34%. But the local strain observed for $t_a = 120$ min is lower compared to $t_a = 60$ min, at the high electric field range. This can be explained by a more ordered microstructure that has higher mechanical strength. This morphology increases the stiffness of the polymer, hence its mechanical properties.

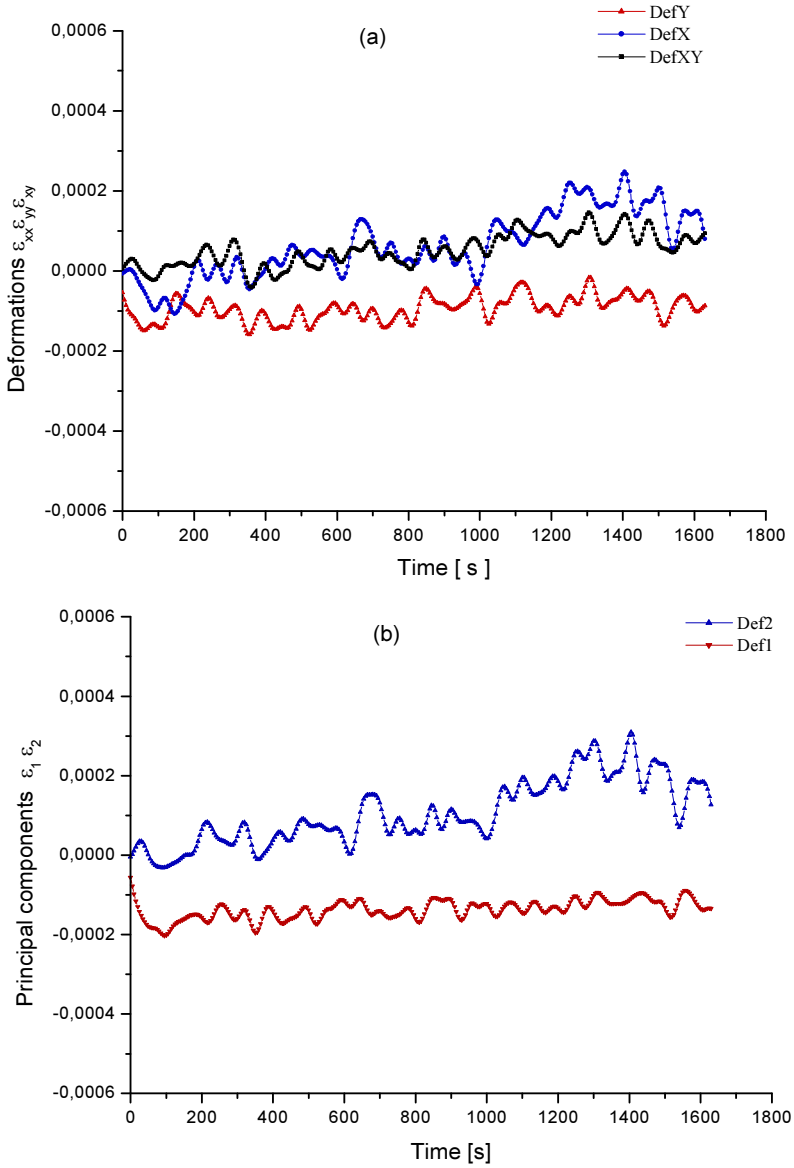

Fig. 4 – *Strain components of the noise levels* (electric field = 0).

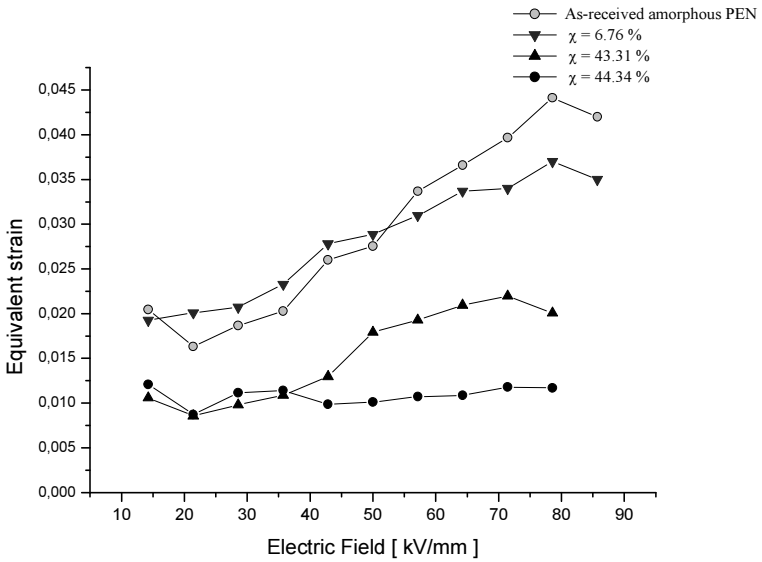

Fig. 5 – *Influence of crystalinity percentage on the equivalent induced strain.*

4 Conclusion

In this experimental study, the assessment of the field-induced mechanical strain in PEN thin films with different degrees of crystalinity was investigated by using an original optical technique. The experimental results showed that samples with small crystallinity percentage were more vulnerable to the equivalent field-induced strain compared to semi-crystalline samples. This technique enables us to predict practical application behavior of solid insulating materials used under operating conditions involving electrical stress. The analysis of the strain characteristics indicates that this novel measurement technique can indeed be used reliably for giving a clear concept of breakdown mechanisms and aging phenomena in a polymeric material.

5 References

[1] S. Buchner, D. Wiswe, H. G. Zachmann: Kinetics of Crystallization and Melting Behavior of Poly (Ethylene-2, 6-Naphthalene Dicarboxylate), Polymer, Vol. 30, pp. 480-488, 1989.

[2] J. C. Canadas , J. A. Diego , M. Mudarra, J. Bellana, R. Diaz-Calleja , J. A. Sanchis, C. Jaimes: Relaxation Study of Poly (Ethylene-2, 6-Naphthalene Dicarboxylate) by TDSC, DEA and DMA, Polymer, Vol. 40, pp. 1181-1190, 1999.

[3] J.J. Martinez-Vega, N. Zouzou, L. Boudou, J. Guastavino: Molecular Mobility in the Amorphous and Partially Crystalline PEN after Isothermal Crystallization, IEEE Transactions on Dielectrics and Electrical Insulation, Vol. 8, pp. 776-784, 2001.

[4] P.R. Mamy, J. Martinez-Vega, J.C. Dupre, N. Bretagne: Quantification of Mechanical Deformations Induced by an Electric in a Semi-Crystalline Organic Insulator, Journal of Applied Polymer Science, Vol. 93, pp. 2321-2321, 2004.

[5] Chavez-Lara, J.F.; Martinez-Vega, J.J; Zouzou, N., Guastavino: Influence of Morphology on Electric Conductance in Poly (Ethylene-2, 6-Naphthalene Dicarboxylate), Journal, Macromol Symp, 2004, 212, pp. 485-490, 2004.

[6] J.C. Dupré, V. Valle, F. Brémand: Mode d'emploi du logiciel DEFTAC V2005, Laboratoire de Mécanique des Solides, Université de Poitiers, 2005.

[7] B. Zegnini , L. Boudou, J.J Martinez-Vega: Quantification of Mechanical Deformation Induced by DC Electric Field in Poly (Ethylene-2, 6-Naphthalene Dicarboxylate) using Optical Technique, Macro 2006 - 41st International Symposium on Macromolecules Proceedings, Rio De Janeiro, Brazil, July 16-21, 2006.

[8] N. Bretagne, J.C Dupré: Mesures de Champs de Déformations par suivi des Marqueurs Pour l'étude des Couplages Thermomécaniques, XVéme Congrès Français de Mécanique, Nancy, 3-7 Septembre 2001.

[9] P.R. Mamy: Etude de la Réponse Mécanique des Isolants Organique Solides au Cours de l'Application d'un Champ Electrique: Cas du PET Semi Cristallin, PhD Thesis, Paul Sabatier Université, France, 2004.

[10] B. Zegnini , L. Boudou, J.J Martinez-Vega: An experimental Study of the Mechanical Strains Induced by a DC Electric Field in Amorphous and Partially Crystallized PEN films, ICSD2007-IEEE International Conference on Solid Dielectrics, Winchester, Hampshire, United Kingdoom, July 8th-13th, 2007, ISBN: 1-4244-0751-6, pp. 246-249.