Dielectric Relaxation and Schottky Conduction of IR Laser Irradiated Makrofol-DE Polycarbonate

Taha A. Hanafy

Physics Department, Faculty of Science, Fayoum University, Fayoum 63514, Egypt

ABSTRACT: The effect of IR laser pulses on the dielectric properties, X-ray diffraction, Fourier transform infrared spectroscopy (FTIR), and *I–V* characteristics of Makrofol-DE 1-1 CC polycarbonate has been investigated in the temperature and frequency ranges of 300–375K and 10 kHz–4 MHz, respectively. IR laser fluencies are set from 0.47 to 7.07 J/cm^2. Three relaxation processes namely, α, β, and γ have been obtained. The first process is due to the micro Brownian motion of the segmental parts of the main chain. The second process was assigned to the local motion of the dipoles within the crystalline phases of the sample. The third one was attributed to the crank shaft motion along the main chains of the Makrofol.

X-ray diffraction and FTIR spectroscopy have revealed that the irradiation of Makrofol at the fluence range 0.94–7.07 J/cm^2 causes crosslinking formation inside the sample. Reduction in crystallinity of Makrofol at higher IR laser doses is due to the crosslinking formation taking place within polymer matrix. At higher voltage, the conduction mechanism of Makrofol-DE was identified as Shottky type. The attained results suggested strongly the applicability of Makrofol-DE in IR laser pulses dosimetry. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 124: 1–8, 2012

Key words: dielectric properties; FTIR; X-ray; sensors

INTRODUCTION

Solid-state nuclear track detectors (SSNTDs) are being widely used in variety of application through the field of radiation dosimetry due to several advantages. Various techniques have been performed on radiation measurements in these SSNTDs and it has been proved that these detectors are good due their ability to record heavy ionized particles, neutrons, and laser.[1–3] Radiation detection depends basically on the manner in which the incident radiation interacts with the material content and the type of detector used. The probability of occurrence of any particular kind of interaction depends on the type and energy of the incident radiation as well as category of the absorber medium.

Laser energy is lost mainly by electron excitations. The absorbed photon energy is transferred directly into excitations of lattice vibrations. A hot spot is generated, which results in thermally activated fragmentation of polymer.[3,4] This phenomenon has aroused considerable interest. The most spectacular effects involve a change of phase in the absorbing material due to the scissions or crosslinking formation. Scissions of chemical bonds of polymer under the influence of heat are the result of overcoming bond dissociation energies. At the same time, it is also possible to observe the formation of chemical bonds between different molecules (intermolecular crosslinking) in the main chain. Moreover, radiation effects on dielectric properties are particular interest to science and technology and they have many applications in modern engineering.[5–8]

Dielectric relaxation spectroscopy (DRS) is a useful method to investigate structure–property relationships of polymers. This method is sensitive to molecular fluctuation of dipoles within the system. For polymers, these fluctuations are related to the molecular mobility of groups, segments, or wholly polymer chains as well as which are shown up as different relaxation processes. Structural transitions in polymers are generally accompanied by changes in relaxation properties. Also, the space charge limited current (SCLC) is a powerful tool for the study of conduction phenomenon in polymers. It has been successfully used in obtaining general information about the localized defect states in the forbidden gap. The charge into the insulator in response to applied electric field is trapped at these localized defects. The presence of these trapping sites in the forbidden gap strongly affects the current–voltage (*I–V*) characteristics of the material.

In the present work, dielectric properties, *I–V* characteristics, X-ray diffraction, and FTIR spectra of Makrofol-DE polycarbonate were performed, before

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Correspondence to: T. A. Hanafy (tahanafy2@yahoo.com).
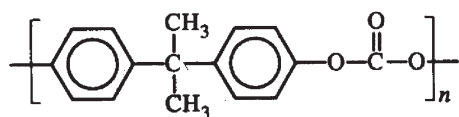
Scheme 1 The chemical structure of Makrofol polycarbonate.

and after irradiation with different fluencies of infrared laser pulses, to study laser–polymer interactions. Further, the study allowed us to investigate the possibility of using Makrofol-DE polycarbonate in constructing simple sensors for infrared laser irradiation.

EXPERIMENTAL

Makrofol-DE 1-1 CC is a bisphenol-A polycarbonate of chemical composition ($C_{16}H_{14}O_3$). It is manufactured by Farbenfabriken Bayer A. G., Leverkusen (Germany), with an average thickness of 300 μm, density of 1.23 g/cm^3, and the glass-rubber transition (T_g) of 423K. The chemical structure of BCC is shown in Scheme 1.

Makrofol polycarbonate samples were exposed to laser pulses for different exposure doses between 0.00 J/cm^2 and 7.07 J/cm^2, using an infrared pulsed laser of 5 W power (Model No. SSL3, USA). The unit is capable of producing 2000 pulse per second with pulse duration of 200 ns at 904 nm. The laser beam was in the form of a circle 1.8 cm in diameter, of Gaussian intensity profile across the circle. All irradiation processes are performed in atmospheric air.

Makrofol polycarbonate samples were cut into square pieces and coated with silver paste to achieve ohmic contacts. The dielectric constant (ε') measurements were carried out with a Hioki (Ueda, Nagano, Japan) model 3532 High Tester LCR, the accuracy of which for measuring the capacitance was of the order of 0.0001 pF. The DC electrical current was measured using a Keithly 487 picoammeter. The temperature of the sample was measured by a k-type thermocouple connected to a Degi-Sense Digital Thermometer (Edison, NJ) with accuracy ±1K.

X-ray diffraction measurements were carried out using D8 Advance diffractometer manufactured by Bruker AXS, Germany, equipped with a primary Johansson-type monochromator which offer a pure CuK$_{\alpha1}$ radiation. The diffraction pattern is recorded in the 2θ range from 4° to 50° with scanning speed of 2° per min using pure CuK$_{\alpha1}$ (wavelength 1.5405 Å) in a Bragg–Brentano geometry (θ/2θ).

FTIR spectra of nonirradiated and the irradiated Makrofol samples were recorded using model Shimadzu 8201 PC. All the measurements were done in the range of 4000–400 cm^{-1}. The spectra were obtained for the absorbance of the polymer as a function of wavenumber with resolution 1 cm^{-1} and accuracy better than ±4 cm^{-1}.

RESULTS AND DISCUSSION

Dielectric properties

The temperature dependence of ε' and the dielectric loss factor ε''; for nonirradiated Makrofol-DE polycarbonate in the frequency range of 10 kHz to 4 MHz, is shown in Figure 1(a,b). The figure shows that ε' increases smoothly with temperature. This can be due to the increase of the mobility of the dipoles of Makrofol sample. Consequently, the dipoles have sufficient energy to orient themselves easily in the direction of applied electric field. Moreover, the dependence of ε' on temperature confirms the orientational distribution of polymer chains in the crystalline and the amorphous phases inside the polymer.[9] The behavior of ε'' exhibits a broad peak at 323K as in Figure 1(b). This peak becomes narrow and moves towards lower temperature with increasing frequency. This behavior can be assigned to sub-glass relaxation process or β-relaxation. This process can be attributed to the local motion of the dipole within the crystalline phases of the sample.

The peak height shift of β-relaxation to lower temperature more likely lies in the fact that the length of the order phases reaction is reduced. This agrees with some suggestions in the literature that β-peak
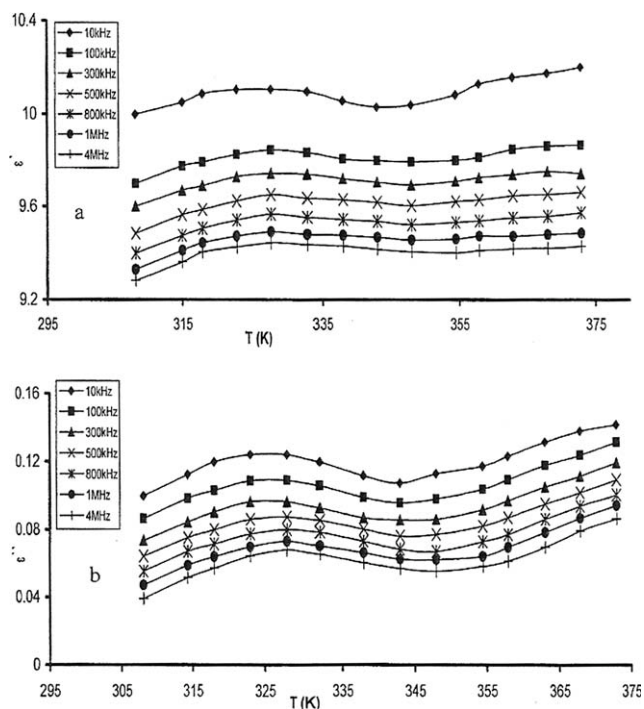

Figure 1 Temperature dependence of: (a) dielectric constant ε', and (b) dielectric loss factor ε''; at fixed frequencies for nonirradiated Makrofol-DE.
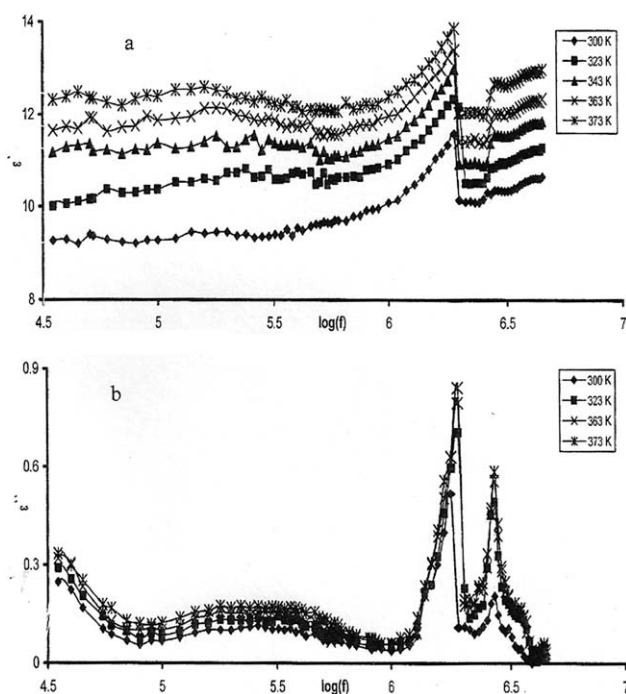
Figure 2 Frequency dependence of: (a) dielectric constant ε', and (b) dielectric loss factor ε''; at various temperatures for nonirradiated Makrofol-DE.

shifts to lower temperature as the length of the segment chain in motion decreases.[10] In addition, if the loss peak is relatively broader below glass transition, T_g, it shows β-relaxation. Also, β-relaxation process is associated with the local motion determined by rotational or vibrational fluctuations of side groups. So, the polarization of the two CH_3 groups plays an important role for this process. In addition, assignment of the molecular motions associated with mid-temperature range relaxation is attributed to the β-relaxation mode.

The frequency dependence of ε' and ε'' for nonirradiated Makrofol-DE at fixed temperatures of 300, 323, 363, and 373K is shown in Figure 2(a,b). It is clear that the values of ε' increase slowly with increasing temperature. This behavior can be discussed as follows: at low temperature the thermal energy absorbed by the dipole is small so a small number of dipoles can rotate with small angle. When the temperature is increased the dipoles have sufficient energy to orient themselves easily in the direction of the electric field. On the other hand, it can observed from Figure 2(b) that ε'' of Makrofol exhibits three relaxation processes namely, α, β, and γ-relaxation process at 316 kHz, 1.9 MHz, and 2.7 MHz, respectively. The α-relaxation process can be attributed to the micro-Brownian motion[7] of the segmental parts in the polymer chains. At the same time the carbonyl (C=O) groups that are directly

attached to the main chain of Makrofol cannot move independently. So, the polarization of C=O groups play an important role for this relaxation process. γ-relaxation process, 2.7 MHz, may be assigned to the crankshaft motion along the main chain of the backbone structure of the sample.[11]

The temperature dependence, at 10 kHz, and frequency dependence, at 300K, of ε' for Makrofol-DE before and after irradiation with 0.47, 0.94, 1.42, 1.89, 2.83, 4.24, 5.66, and 7.07 J/cm^2 of IR laser fluence are displaced in Figure 3(a). It is obvious that the values of ε' are increased with laser fluence up to 0.94 J/cm^2 and then decreased with any further increasing of laser fluence up to 7.07 J/cm^2. The effect of radiations on polycarbonates is primarily chain scission. However, at increased doses, active sites or branching point created by scission may lead to intermolecular crosslinking.[12] It was found that, both scission and crosslinking coexists and either one may predominant depending not only on the chemical structure of the polymer but also on the conditions of irradiation.[13]

Polymers absorb laser energy in a destructive manner, which is bond cleavage, either by hydrogen abstractions and its elimination from a chain or by the cleavage of the chain itself. Each of these events typically results in the formation of free radicals. These radical species may then further react with advention polymer or with oxygen. The formation of radicals and their oxidation reaction may continue with time, resulting in fracture and discoloration
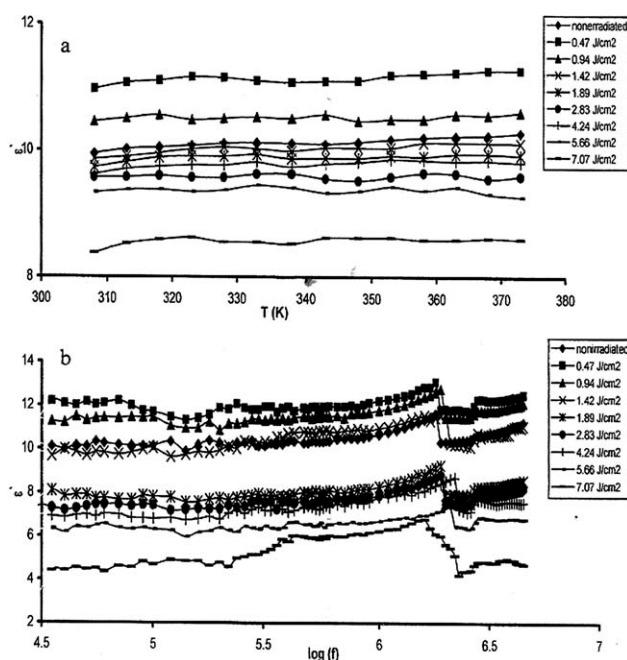

Figure 3 (a) Temperature dependence of dielectric constant ε' at 10 kHz, and (b) frequency dependence of dielectric constant ε' at 300K for Makrofol-DE before and after irradiation with different IR laser fluencies.
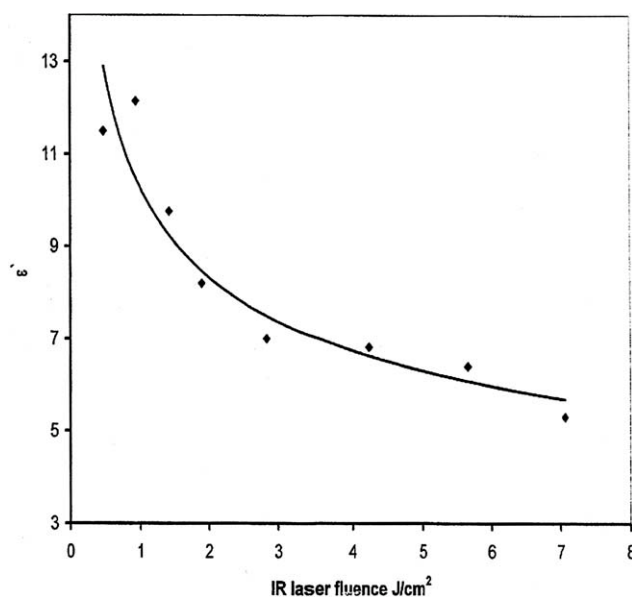
Figure 4 Variation of the dielectric constant ε' of Makrofol-DE, at 35 kHz and 300K, with different IR laser fluencies.

due to crosslinking formation. This reason may be responsible for the decrease of ε' of the investigated sample.

Figure 4 shows the variation of ε' of Makrofol-DE sample against the IR laser fluence, at 300K and 35 kHz. It is clear that the values of ε' of Makrofol-DE give us a good proportionality with IR laser fluence range of 0.47–7.07 J/cm^2. Thus, one can suggest that the dielectric constant ε' of Makrofol-DE can be used as a new technique to estimate IR laser pulses fluence.

X-ray diffraction

Most polymers are not entirely crystalline. The chains or parts of chains that are not part of the crystals obviously have no order to the arrangement of their chains. So, a crystalline polymer really has two components, the crystalline and amorphous phases. The existence of amorphous phase leads to the appearance of characteristic amorphous halos in X-ray diffraction pattern. Moreover, it was found that the crosslinking and scission formation inside the polymer are accompanied by the existence of amorphous phase and crystalline phase, respectively.[15] To study the effect of IR laser fluence on the degree of ordering of Makrofol-DE, X-ray diffraction measurements were carried out. Figure 5 illustrates
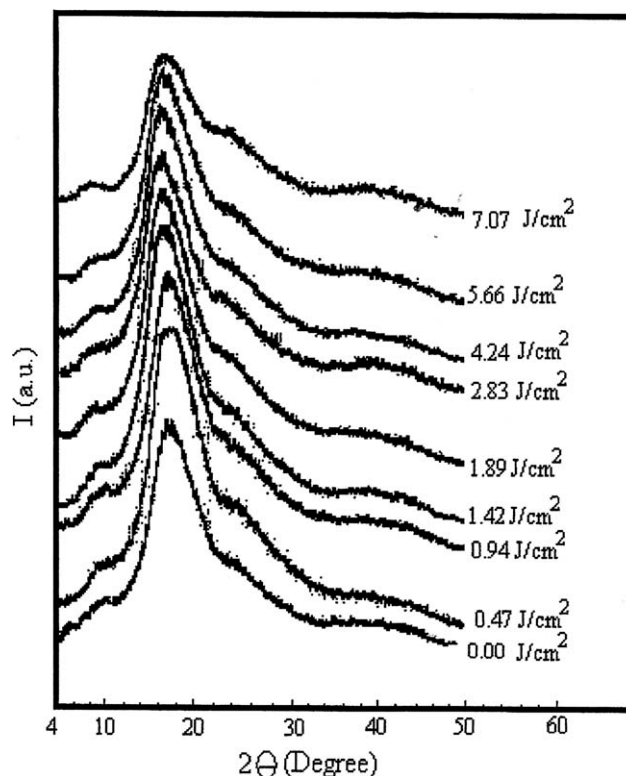
usually concentrated at polymer surface.[8] This leads to enhance of the dielectric constant ε' of the investigated sample. So, one can suggest that, Makrofol sample which irradiated with 0.47 and 0.94 J/cm^2 of IR laser fluence undergoes the main chain scission.

In other words, the direct effect of the laser is to excite the carbonyl groups, which exist within the backbone structure of the polymeric material, to form other free radicals. Then, the α-relaxation of the sample may originate from the polarization of C=O groups and the normal segmental motion of the fragments for the main chain. Also, phenyl groups align themselves in such a way and participate in cooperative motion of the main chain. Moreover, the polarization of the two methyl groups, which attached directly to the main chain, plays an important role in the β-relaxation process of the Makrofol sample.

However, the decrease of ε' for Makrofol sample that irradiates with any further IR laser fluence up to 7.07J/cm^2 could be related to the crosslinking formation inside the sample. Such crosslinking is achieved by recombination between any two radicals, such as methyl, carbonyl (C=O), and phenyl groups, or by coupling of two neighboring chains through the elimination of hydrogen atoms from C—H bond. Crosslinking formation leads to diminishing of the number of the carbonyl groups and to decrease the free volume inside the sample.[7,14] So, both phenyl and CH$_3$ groups has little chance to rotate or to align themselves with the direction of the applied electric field. Then, the polarization of C=O, phenyl, and methyl groups may be decreased

Figure 5 X-ray diffraction patterns of the Makrofol-DE before and after irradiation with different IR laser fluencies.
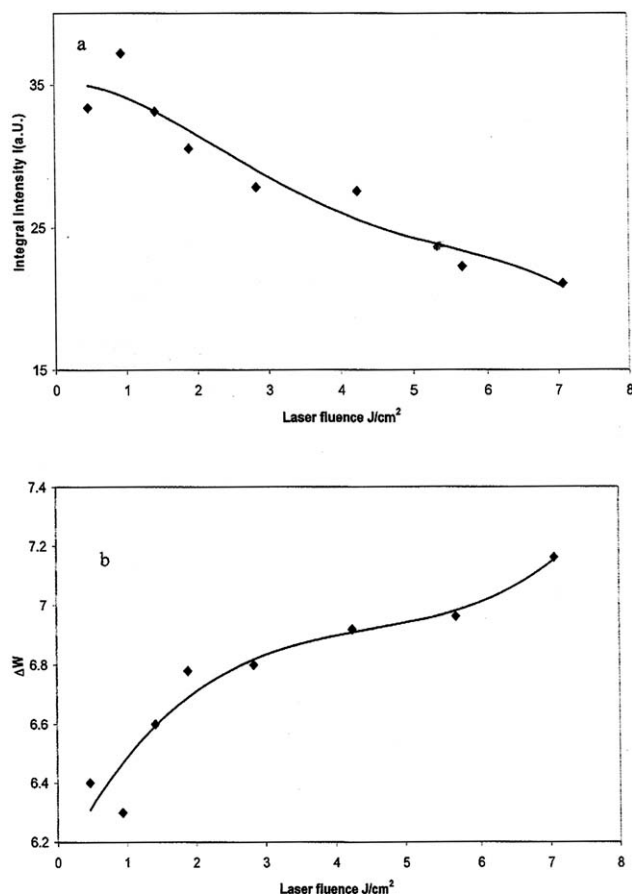
Figure 6 Variation of (a) the integral intensity I and (b) the full Width at half maximum intensity ΔW of the main diffraction peak of Makrofol-DE with different IR laser fluencies.

where ΔW is the peak's width at the half of maximum intensity and λ is the wavelength of the X-rays. Figure 6(b) shows the variation of ΔW with the laser fluence. The results indicate that ΔW shows an opposite trend to that of the integral intensity, where it decreases until a minimum value for the irradiated sample with 0.94 J/cm^2 of IR laser beam is reached, after that it increases with the increase of the laser fluence up to 7.07 J/cm^2. The increase in integral intensity indicates an increase in the crystallinity of the polymer samples which can be attributed to degradation induced by laser irradiation. This scission can reduce the number of entanglements per molecule. Chain scission can also act to relieve intermolecular stress in the amorphous regions and increase the chain mobility. The increase in mobility permits some molecules to re-crystallize because crystalline state is thermodynamically stable state.[17]

On the other hand, the decrease in the integral intensity at the fluence range 0.94–7.07 J/cm^2 denotes a decrease in the amount of crystalline phase in the samples and the crystalline structure (lamella) has been destroyed. This could be attributed to the crosslinking, which changes the previously regularly arranged portions into nonarranged ones by forming new bonds between the neighboring chains. This means that crosslinking is the predominant effect in this fluence range.
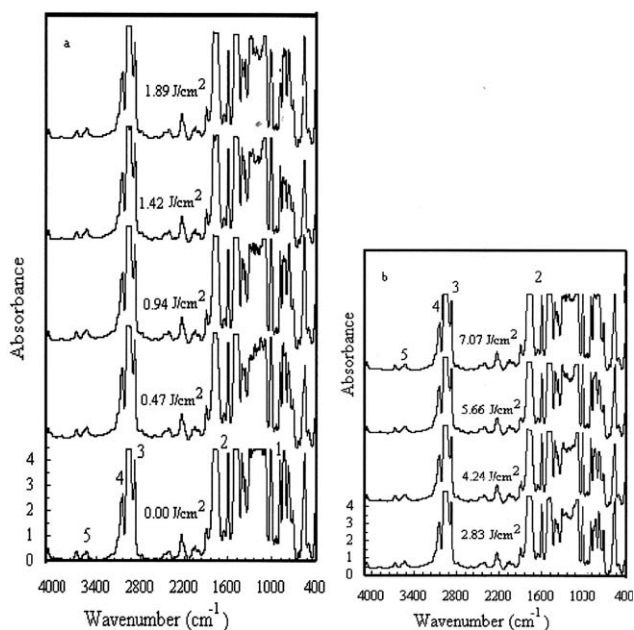
the X-ray diffraction of Makrofol sample before and after irradiation with IR laser fluence from 0.47 to 7.07 J/cm^2. From the figure it is clear that the X-ray diffraction pattern of the investigated samples are characterized by halos extending in the 2θ range 10°–34°. The profile of the halos shows that the Makrofol is a partly crystalline polymer with a dominant amorphous phase. The area under these halos is proportional to the integral scattering intensity of the X-rays.

The integral intensity I(a.u) of the halos as a function of the laser fluence is shown in Figure 6(a). It is noticed that the integral intensity of the irradiated samples increases up to a maximum value for the irradiated sample with 0.94 J/cm^2 of IR laser and then decreases with the increase of the laser fluence up to 7.07 J/cm^2. Since the halo's width at the half of maximal intensity ΔW is inversely proportional to the crystallite size, approximate indicative values of ΔW were calculated using the Scherrer equation[16]:

$$L = (0.89\lambda)/(\Delta W \cos\theta) \qquad (1)$$

Figure 7 FTIR spectra of Makrofol-DE before and after irradiation with different IR laser fluencies with the characteristic bands are: 889 cm^{-1} (1), 1778 cm^{-1} (2), 2877 cm^{-1} (3), 3047 cm^{-1} (4), and 3533 cm^{-1} (5).
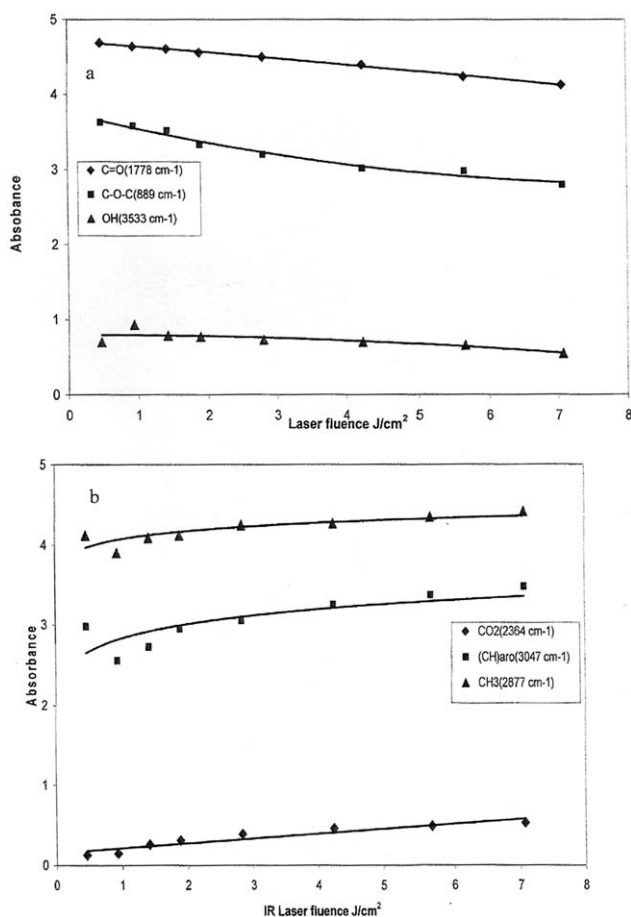
Figure 8 (a,b) Variation of the absorbance measured at characteristic band of Makrofol-DE with different IR laser fluencies.

FTIR spectral analysis

FTIR spectra of the Makrofol-DE sample before and after irradiation with different IR laser fluencies as mentioned before are shown in Figure 7(a,b). It is observed that the half band width of the infrared absorption was slightly affected by the laser dose. Makrofol is an aromatic polycarbonate and its functional groups include methyl, phenyl ring, carbonyl, ether, and hydroxyl. The absorbance of different bands coming from the function group exhibits the same trend with the laser fluence. So, each characteristic groups of polycarbonate is represented by only one wave number.

Figure 8(a,b) shows the variation of the absorbance with the laser fluence. It is clear that the absorbance at 1778 cm^{-1} and 889 cm^{-1} decreases with the increase of the laser fluence up to 7.07 J/cm^2; this indicates that the breaking of the C=O and C—O—C bonds is achieved. The absorbance at the wavenumber 2877 cm^{-1} and 3047 cm^{-1} shows a decrease in the intensity of C—H bond until a minimum value for the irradiated sample with 0.94 J/cm^2. The inten-

sity of CH_3 bond is proportional to the laser fluence within the range of 1.42–7.07 J/cm^2.

The intensity of the peak corresponding to OH group, at 3533 cm^{-1}, exhibits an opposite trend to that of C—H bond. These results indicate that scission takes place at the carbonate site with probable elimination of carbon dioxide/carbon monoxide and formation of hydroxyl groups. The increase of hydroxyl groups in the fluence range of 0.47–0.94 J/cm^2 means an increase in the end groups of macromolecules. This indicates that the degradation process prevails at these fluence ranges. At the fluence range of 0.94–7.07 J/cm^2, an opposite trend has been observed due to the formation of bonds through crosslinking mechanism.

I–V characteristics

Figure 9 shows I–V characteristics of Makrofol-DE sample before and after irradiation with different IR laser fluencies at constant temperature of 300K. The data reveal that Makrofol sample which irradiated with 0.47 and 0.94 J/cm^2 exhibits maximum current due to the degradation and the increase of the crystal ratio inside the sample. However, the current of Makrofol sample is decreased with the increase of the laser fluence up to 7.07 J/cm^2. This can be attributed to the crosslinking formation and the decreased crystal ratio inside backbone structure of Makrofol-DE. Also, I–V characteristics have two regions. One at lower voltage 10–55 V with slope approximately 1 (i.e., $I \propto V$) and the conduction is ohmic due to the
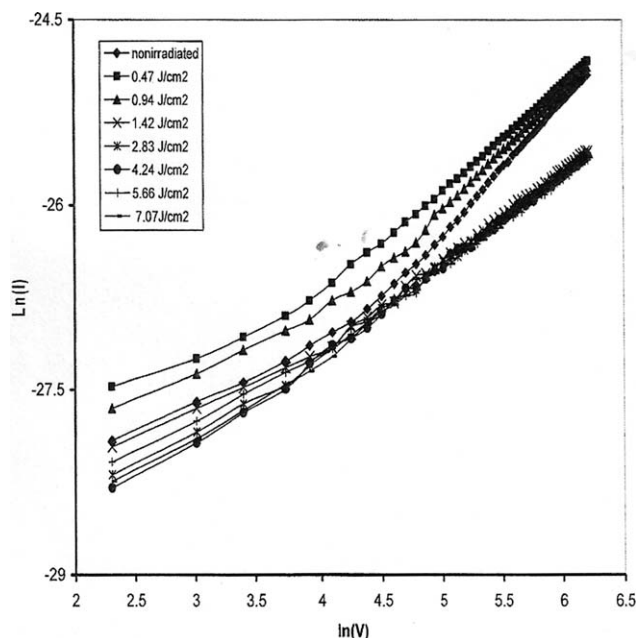

Figure 9 I–V characteristics of Makrofol-DE at 300K before and after irradiation with different IR laser fluencies.
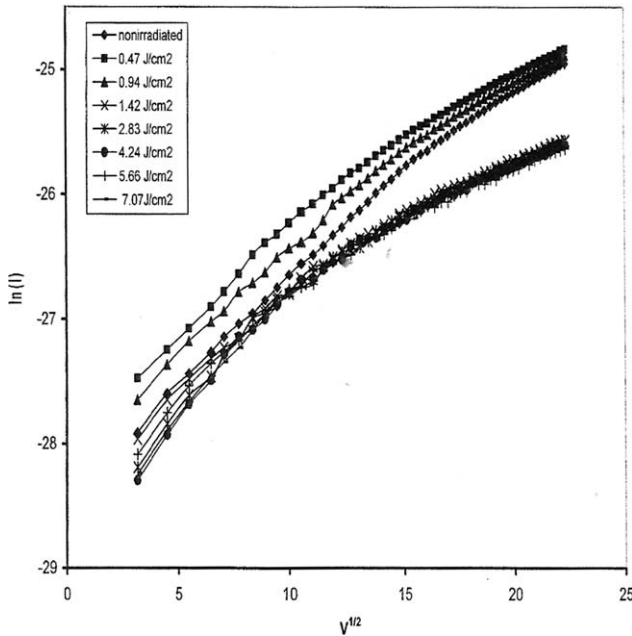
Figure 10 The relation between ln(I) versus $V^{1/2}$ for Makrofol-DE before and after irradiation with different IR laser fluencies.

intrinsic conductivity of the material. This indicates that the thermal activated carriers controlled the current of the sample.[18] At higher voltage range, approximately 150–500 V, a square power law (i.e., $I \propto V^2$) was obtained. The voltage dependence of the current in second region leads to the conclusion that the conduction mechanism of Makrofol-DE is due to SCLC dominated by a discrete trapping level. In addition, the conduction mechanism may be either Shottky or Poole-Frenkel type.[19]

For Schottky effect, the current density can be estimated according to the thermoionic emission function:

$$J = J_s \exp\left(\frac{\beta_s V^{1/2}}{kTd^{1/2}}\right) \qquad (2)$$

TABLE I
The Values of β in (eV V$^{-1/2}$ m$^{1/2}$) for Makrofol Sample Before and After Irradiated at Different IR Laser Fluencies

Laser fluence (J/cm^2)	β (eV V$^{-1/2}$ m$^{1/2}$) × 10^{-5}
0.00	3.56
0.47	3.08
0.94	3.58
1.42	2.20
1.89	2.20
2.83	2.20
4.24	2.20
5.66	2.20
7.07	2.20

where k is the Boltzman's constant, T is the absolute temperature, V is the applied electric voltage, d is the sample thickness, and J_s is the saturation current density which can be represented by:

$$J_s = AT^2 \exp\left(\frac{\Phi_s}{kT}\right) \qquad (3)$$

where A is the Richardson's constant, and Φ_s is the Schottky barrier height.

For Poole-Frenkel effect, the current density can be estimated according to the lowering of coulombic potential barrier when intersects with the electric field:

$$J = J_o \exp\left(\frac{\beta_{PF} V^{1/2}}{kTd^{1/2}}\right) \qquad (4)$$

where J_o is the low field current density, β_S and β_{PF} are the Schottky and Poole-Frenkel field-lowering coefficients, respectively. Moreover, the coefficient β is an essential factor that determines the magnitude of coulombic field. The β_S and β_{PF} values are theoretically calculated by[20]:

$$2\beta_s = \beta_{PF} = \left(\frac{e^3}{\pi\varepsilon\varepsilon_o}\right)^{1/2} \qquad (5)$$

where e is the electronic charge ε and ε_o are the electric permittivity of the sample and the free space, respectively.

By taking the high frequency dielectric constant as 10.3, the calculated values of β_S and β_{PF} are

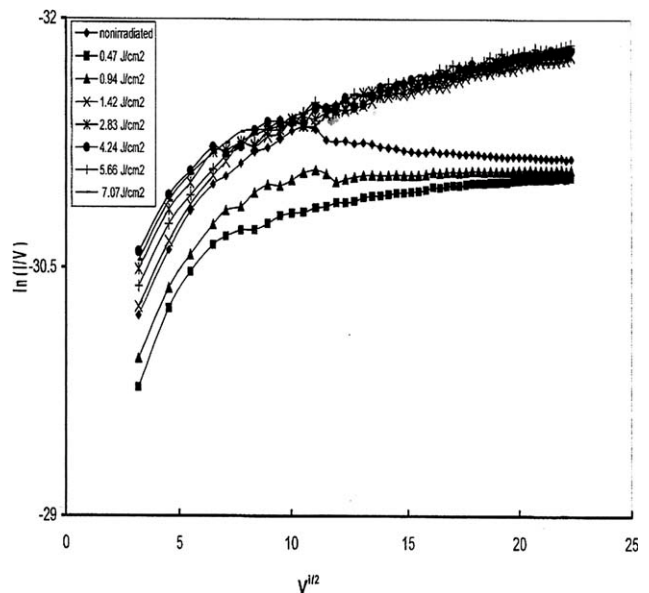**Figure 11** The relation between ln(I/V) versus $V^{1/2}$ for Makrofol-DE before and after irradiation with different IR laser fluencies.

2.4 $\times$ 10^{-5} and 4.8 $\times$ 10^{-5}eV V$^{-1/2}$ m$^{1/2}$, respectively. The experimental values of β are calculated from Figure 10 and listed in Table I. It is noticed that the experimental values of β are closer to the calculated $β_S$ value. So, one can suggest that the dominant conduction mechanism for all Makrofol samples is Shottky type. From another point of view, the interpretation of the conduction mechanism from the experimental data may be done by using the measured slope of ln(I) versus $V^{1/2}$, Figure 10, and ln(I/V) versus $V^{1/2}$, Figure 11, plots. If the obtained curve of Figure 10 is linear, the conduction mechanism is considered to be Schottky (electrode limited) process. Also, if measured values of Figure 11 is linear, the conduction mechanism is consider to be Poole-Frenkel (bulk limited) process. From Figures (10 and 11), it clear that, the curves in Figure 10 are more linear than present in Figure 11. This is sustaining the suggestion that the conduction mechanism for all Makrofol-DE samples is of Shottky type.

The obtained results of ε′, Figure 4, X-ray diffraction, Figure 6, and FTIR spectroscopy, Figure 8, showed a good proportionality through the investigated range, 0.47–7.07 J/cm^2, of IR laser pulses fluence. As mentioned earlier, the aim of the present work is to investigate the possibility of using Makrofol DE polycarbonate in constructing simple sensors as a new technique for IR laser dosimeter. In this technique, one can estimate the IR laser pulses fluencies for the range from 0.47 up to 7.07J/cm^2. The outcome results of this work give a good signal for the application of IR laser pulses.

CONCLUSIONS

The effect of IR laser pulses on the dielectric properties, X-ray diffraction, FTIR spectroscopy, and I–V characteristics of Makrofol-DE was interpreted. The frequency dependence of ε″ allow us to detect three relaxation transitions, α, β, and γ. Makrofol-DE sample that irradiates with 0.47 and 0.94 J/cm^2 of IR laser fluencies undergoes chains scission which causes the increase of ε′ and the increase of

crystalinity of the sample. However, the higher fluencies of IR laser up to 7.07 J/cm^2 causes the crosslinking formation within the Makrofol sample. The crosslinking was assumed to be responsible for the reduction of ε′ and the reduction of crystalinity of the polymer. The conduction mechanism for the investigated samples was assumed to be Shottky type. The behavior of ε′, X-ray diffraction, and FTIR spectroscopy of Makrofol-DE 1-1 CC has good proportionality with IR laser pulses fluence. This could be employed as a new dosimeter technique.

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