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Variation of Lexan Polycarbonate Properties by Electron Beam

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ABSTRACT: The modifications in microstructural, optical, and photoluminescence properties of the Lexan polycarbonate (bisphenol-A-polycarbonate) films exposed to different electron doses have been studied using UV–vis spectroscopy, Fourier transform infrared spectroscopy (FTIR), positron annihilation lifetime spectroscopy, photoluminescence spectroscopy, and scanning electron microscopy (SEM). The obtained UV–vis spectroscopy results showed decrease in optical energy gap, optical activation energy, and increase in number of carbon atoms per cluster with increase in electron dose. The chemical changes in electron lifetime study with optical measurement is obtained, and electron irradiation-induced microstructural modifications within the polymer is understood. SEM result shows the degradation of Lexan polymer after electron irradiation. The mechanical properties and average molecular weight of Lexan decrease after irradiation, whereas average number of chain scissions per original polymer molecule increases. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

KEYWORDS: polycarbonate; electron beam irradiation; UV-vis spectroscopy; FTIR; SEM; crosslinking; chain scission

Received 25 May 2011; accepted 26 February 2012; published online **DOI: 10.1002/app.37589**

INTRODUCTION

The physical and chemical properties of polymers can be modified by electron beam irradiation. When electron beam is allowed to pass through a polymeric material, the drastic changes in properties such as optical, structural, free volume and thermal have been observed due to crosslinking, chain scission, chain aggregation, molecular emission, etc. The chain scission and crosslinking occurs simultaneously when polymer is irradiated by electron beam. The chain scission process decreases the molecular weight and increases solubility. The crosslinking process results in formation of chemical bonds between two adjacent polymer molecules which increases the molecular weight of polymer. Both processes can significantly alter the physical and chemical properties of polymers.^{1,2}

Lexan polycarbonate (Lexan) is a promising polymer having lot of desirable properties for different applications. The high transparency of Lexan in the visible spectral range makes it an excellent substitute of glass substrate in variety of applications such as aircraft canopy, solar cells, solid state nuclear track detector, etc. Polycarbonate detectors such as Lexan are generally used for recording fission fragments tracks. Nowadays, radiation processing of polymers has gained lots of interest due to its potential applications in various fields. A very few reports are available on the radiation processing of Lexan polycarbonate.

In amorphous polymers (like Lexan), characterization of free volume plays an important role. One way to get information about the free volume accurately is positron annihilation lifetime spectroscopy (PALS) as it allows the detection of free volume holes on an atomic scale.^{3,4} A brief description of the PALS techniques is given below: When an energetic positron from radioactive source enters a condensed medium-like polymer, it thermalizes by losing its energy in a very short time, then annihilates with an electron of the medium. Annihilation usually takes place from different positron states viz., free annihilation, or from a localized state (trapped state) or from a bound state called positronium (*p*-Ps, spin antiparallel), which annihilates with a life time of 0.125 ns and *ortho*-positronium (*o*-Ps, spin parallel), which annihilates with a lifetime of 140 ns in free space. In condensed matter, the *o*-Ps annihilates predominantly through a fast channel with an electron of the surrounding medium possessing an opposite spin; a process called pick-off annihilation and the *o*-Ps lifetime gets reduced to a few nanoseconds. Each of these annihilation processes has a characteristic lifetime. In polymers, the *o*-Ps lifetime is an important parameter since positronium is trapped and annihilated in free volume sites, and hence it is related to the mean size of free volume holes in the polymer matrix.⁵ In this study, electron beam-irradiated Lexan samples are characterized by UV–vis spectroscopy, Fourier transform infrared spectroscopy (FTIR), PALS, photoluminescence spectroscopy (PL), and scanning electron microscopy (SEM) techniques to understand the modifications brought out by irradiation in Lexan films.

EXPERIMENTAL DETAILS

Electron Beam Irradiation

Lexan films (Grade 125) manufactured by General Electric Company having thickness 200 μ m were irradiated at doses of 100 and 225 kGy sealed in polyethylene bags. The irradiation was carried out at Microtron Centre, Mangalore University, India, using 8-MeV electron beam having pulse current 20 mA; pulse repetition rate 50 Hz; pulse width 2.3 μ s at room temperature. The dose delivered to the sample was measured using current integrator calibrated against appropriate radiation dosimeters.

Polymer Characterization

UV-vis absorption spectra were recorded in the wavelength range from 250 to 450 nm keeping air as a reference, using Shimadzu spectrophotometer (model UV-1800 PC). FTIR spectra were recorded in the wave number range 400-4000 cm⁻¹ having a resolution of 4 cm⁻¹, using NICOLET spectrophotometer (model 5700). The barrier properties of polymeric materials is strongly dependent on their molecular architecture and presence of free-volume or microvoids. If the free-volume cell/microvoid size in the polymer is higher, the barrier efficiency of the membrane against different molecules will be inferior. The free-volume cell/microvoid size in the polymers was measured using PALS using a conventional fast-fast coincidence system with a time resolution of 220 psec. A 17 Ci ²²Na source, which was deposited on 0.127-mm-thick Kapton foil and sandwiched between two discs of the sample ($\sim 1 \text{ mm thick}$) under study, was placed between two BaF2 scintillation detectors. Each spectrum contained more than 10⁶ counts that were accumulated in \sim 1.5–2 h. The measured positron lifetime spectra were analyzed using the computer program PATFIT88.6 Photoluminescence spectra of pristine and electron-irradiated Lexan films were recorded in the wavelength range 400-2000 nm using Jobin Vyon Horiba Spex Fluoromax-3 fluorometer at excitation wavelength of 534 nm. Surface morphology of pristine and electron-irradiated Lexan samples was studied by means of SEM (model JEOL JSM 6390LV). The intrinsic viscosity ($[\eta]$) of pristine and electron-irradiated samples were measured using ARG2 Rheometer, TA instruments at 25°C. The average molecular weight (M_{ν}) was calculated from the intrinsic viscosity using the following equation:⁷

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$$[\eta] = 1.23 \times 10^{-4} \, M_V^{0.83} \tag{1}$$

The tensile strength and percentage of elongation of Lexan samples were measured using LLOYD Instruments (model LRX plus) at a speed of 50 mm min⁻¹.

RESULTS AND DISCUSSION

The chemical structure of Lexan and the possible chemical reaction after electron irradiation is as shown in Figure 1. This can be explained in the following way: an early investigation by Zimmermann et al.⁸ reports that the C-O bonds adjacent to the carbonyl group in the Lexan polycarbonate polymer chain are the vulnerable bonds, lacking the resonance stabilization of the phenyl group. The most important volatile product during Lexan polycarbonate degradation is CO₂, and hence carbonyl linkage is the most reactive group in the polymer.⁹ On irradiation, the chain scission must have occurred mainly at the C-O bonds adjacent to the carbonyl group. Thus, on irradiation, the carbonate group absorbs energy selectively, and as such Lexan polycarbonate undergoes chain scission with the formation of free radicals such as phenoxy-type and phenyl radicals initially [as shown in Figure 1(a)] and thereafter crosslinking of the free radicals may occur. A study on viscosity of UV-irradiated polycarbonate¹⁰ is supportive evidence to the earlier description. An experimental study of photoirradiation of polymers¹¹ reveals that an increase in viscosity is due to crosslinking. Hama and shinohara¹² reported that photolysis of C-O bonds in polycarbonate yields phenyl- and phenoxy-type radicals which are thought to be associated with conjugated double bonds and formed by crosslinking of the phenyl radicals. It is also known that most of the aryl esters and carbonates on photolysis undergo photofries rearrangement,¹³ the product formed are due to the rearrangement of oxygen atoms. If Lexan polycarbonate undergoes such a rearrangement on irradiation it results in the formation of photostabilizers such as phenyl salicylate and 2,2'-dihydroxybenzenephenone [as shown in Figure 1(b)].

UV-Vis Spectroscopy

UV-vis absorption spectra of 8-MeV electron-irradiated Lexan along with pristine are as shown in Figure 2. Polymeric materials absorb light energy in the ultraviolet and visible region and promote the electron in σ -, π -, and *n*-orbital from the ground state to higher energy states which are described by molecular orbital.¹⁴ The following types of electronic transitions are involved in the ultraviolet and visible regions, $\sigma \to \sigma^*$, $n \to \sigma^*$, $n \to \pi^*$, and $\pi \to \pi^*$. The optical transitions that result from the presence of impurities have energies in the visible part of spectrum; consequently the defects are referred to as color centers.¹⁵ The pristine Lexan has an absorption edge at 285 nm which is assigned to the $n \to \pi^*$ transition. On increasing the electron dose, the absorption edge shifts toward longer wavelength region. This red shift in the absorption edge can be explained as follows. The electron irradiation of Lexan induces bond breaking and dehydrogenation of the polymer chains (i.e., evolution of hydrogen atoms as hydrogen molecules). This in turn produces conjugated double bonds or chromospheres groups. The conjugated double bonds increases with increase in electron dose, resulting in the red shift of the absorption edge.¹⁵



Figure 1. Schematic representation of chemical reaction induced in Lexan after electron irradiation.

To study the effect of electron beam on optical energy gap (E_g) of Lexan which is an important tool for the optical properties investigation, the absorption coefficient (α) was calculated from equation given as: ^{15,16}

$$I(t) = I_0 e^{-\alpha t} \tag{2}$$

where I_0 and I(t) are the intensities of the incident and transmitted light, respectively, and *t* is the thickness of film. The absorption coefficient (α) is related to the photon energy (*hv*) by the relation:¹⁷

$$\alpha h v = A (h v - E_g)^r \tag{3}$$

where *A* is a constant, E_g is the optical energy gap, and *r* is an index that can take the values 1/2, 3/2, 2, or 3 depending on the type of transition responsible for the absorption. The allowed direct transition is simply the electron transform

vertically from the top of the valance bond to the bottom of conduction band without change in momentum; whereas the nonvertical transitions are normally forbidden.¹⁵ The simplest way to deduce the type of transition is to check the value of *r* which fits hv to αhv with a straight line relationship.¹⁰ In our study, $r = \frac{1}{2}$ fits well which means an allowed direct transition. Figure 3 shows the correlation of α^2 with photon energy (hv) for both pristine and electron-irradiated Lexan samples. Extrapolating the linear part of these curves to the *hv*-axis yields the corresponding E_q value. For pristine Lexan, the E_g was found to be 4.36 eV, and it decreases with increase in electron dose; finally it reached 4.19 eV for 225 kGy. The regression coefficient has been found to be 0.9791 for pristine Lexan and 0.9821 for 225-kGy irradiated Lexan for the fitted lines. The decrease in E_g value may be due to the production of free electrons, radicals and ions, which may decrease the resistivity (increase in conductivity) of the polymer by increasing the number of charge carriers.



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Figure 2. UV-vis spectra of Lexan irradiated with different doses of 8-MeV electron beam.

For further investigation, one can determine the optical activation energy using Urbach rule:^{17,18}

$$\alpha = B \exp(h\nu/E_a) \tag{4}$$

where *B* is a constant and E_a is optical activation energy. Figure 4 shows the dependence of Ln (α) on the photon energy (*hv*) for pristine and electron-irradiated Lexan samples. From the slope of the obtained straight lines, one can calculate the E_a value. The values of E_a for all the samples are listed in Table I. It was found that the values of E_a decreases with increase in electron dose. This decrease in E_a value is attributed to the increase in the defects. This will be evident from positron lifetime results presented in the next paragraphs. These defects lead to the formation of lower energy states resulting in the increase in number of charge carriers in the conduction band.⁹ The decrease in both E_g and E_a values can be attributed to the formation of carbon clusters near the polymer surface^{18,19} possibly due to energy transferred from the electron beam.



Figure 3. The relation between α^2 and the photon energy for Lexan irradiated by 8 MeV electron with different doses.



Figure 4. Variation of Ln (α) with the photon energy for Lexan irradiated by 8 MeV electron with different doses.

According to Robertson relation,¹³ the number of carbon hexagon in a cluster is:

$$E_g = 2\beta/(N)^{1/2} \tag{5}$$

where 2β is the band structure energy of a pair of adjacent π sites. Fink et al.¹⁸ have pointed out that the Robertson relation underestimates the cluster size in irradiated polymers, and hence the cluster structure was assumed to be like a buckminsterfullerene (a C₆₀ ring instead of C₆) and relation becomes:

$$E_a = 34.3/(N)^{1/2} \tag{6}$$

The number of carbon atoms (N) per cluster can be calculated using above relation, and they are listed in Table I. From Table I, it can be seen that the number of the carbon atoms per cluster increases with increase in electron dose. This is attributed to the slowdown of energetic ions in polymeric matrix by the interaction with the electronic system of target atoms, which produce atomic electrons by excitation or ionization leading to cleavage of bonds and hence release of hydrogen. As a result, with increase in electron dose, the surface layer may be transformed in to a hydrogenated amorphous carbon.^{21,22}

Fourier Transform Infrared Spectroscopy

As the electron irradiation of polymeric system leads to change in bond length, bonded groups or even orientation groups or

 Table I. Optical Parameters for Lexan, Irradiated by 8 MeV Electron at Different Doses

Dose (kGy)	Optical energy gap (E _g in eV)	Optical activation energy (E _a in eV)	Number of carbon atoms (N)
0	4.36	1.11	62
100	4.28	0.94	64
225	4.19	0.86	67



Figure 5. FTIR spectra of pristine and irradiated Lexan films.

molecules cause some changes in molecular orientations. The nature of chemical modification caused by electron irradiation on Lexan can be studied using FTIR measurements. Figure 5 shows the FTIR spectra of pristine and electron irradiated Lexan films. From Figure 5, it is observed that for pristine Lexan, the bands for methyl (-CH₃) group appears at (A) 2976 cm⁻¹ (v_{as}) , (B) 2876 cm⁻¹ (v_s) , (C) 1469 cm⁻¹ (δ_{as}) , and (D) 1366 cm^{-1} (δ_s). The (E) carbonyl (-C=O) stretching band absorbs at 1760 cm^{-1.23} Apart from these, on irradiation new band appears at (F) 1599 cm⁻¹, (G) 1496 cm⁻¹, (H) 1012 cm⁻¹, (I) 942 cm⁻¹, and (J) 824 cm⁻¹ corresponding to C=C. Apart from this, the intensity of the peak band corresponding to -C=O stretch at 1760 cm⁻¹ decreases slightly on irradiation. This may be due to chain scission at the carbonate site with the probable elimination of carbon dioxide or carbon monoxide. On increasing the electron dose, the rate of chain scission increases, as a result, the formation of carbon double bonds also increases. These small carbon enriched units may agglomerate into carbonaceous clusters due to their possible hydrophobic interaction and packing of similar segments at appropriate portion of polymer chain where the degradation occurs. From the overall discussion of FTIR, it can be inferred that the chemical changes arises due to the rearrangements of ions to form a stable product after chain cleavage, which is the dominant process at lower dose, and crosslinking is the dominant process at higher dose.

Positron Annihilation Lifetime Spectroscopy

The measured positron annihilation lifetime spectra for pristine and electron-irradiated Lexan films were analyzed in terms of three lifetime component τ_1 , τ_2 , τ_3 , and corresponding intensities I_1 , I_2 , and I_3 . Here, τ_1 is the shortest lifetime component with intensity I_1 belongs to the self annihilation of *para*-positronium (*p*-Ps) atoms and free annihilation of positrons in the bulk samples. τ_2 is the intermediate lifetime component with intensity I_2 , and is due to the positrons trapped in the defects present in the crystalline regions or trapped in the crystalline amorphous interface regions. τ_3 is the longest lived component with intensity I_3 and is attributed to the *o*-Ps atoms in the free volume sites of amorphous regions of polymer via pick-off annihilation.

The *o*-Ps lifetime (τ_3) can be used to obtain the radius *R* of the free volume hole using the semiempirical relation²⁴ given by:

$$(\tau_3)^{-1} = 2[1 - (R/R_0) + 1/2\pi \text{Sin} (2\pi R/R_0)]$$
(7)

where $R_0 = R + \Delta R$ and ΔR is the fitting parameter and a value $\Delta R = 0.166$ nm was determined by fitting eq. (6) with experimental τ_3 values to data from molecular materials like zeolites with known hole sizes. Here, *o*-Ps lifetime in the electron layer of thickness ΔR is the spin-averaged Ps lifetime of 0.5 ns. The free volume size (V_f) can be calculated as:

$$V_f = (4/3)\pi R^3$$
.

Also assuming that the relative intensity I_3 reflects the number of free volume holes in the polymer material, the relative free volume fraction F_{ν} can be calculated as $F_{\nu} = CI_3 V_f$ where C is a constant whose value is taken as 0.0018 Å⁻³ and V_f is the average free volume size. Figure 6 shows the variation of o-Ps lifetime and free volume size V_f against electron dose. From this figure, it can be seen that both o-Ps lifetime τ_3 and free volume size V_f decreases up to 100 kGy and then increase slightly for 225 kGy. This o-Ps lifetime variation is sensitive to local molecular environment and is directly correlated to the size of free volume region in which it is localized. This decrease in τ_3 may also be due to sudden rupture of chemical bond in Lexan and formation of conjugated bond. It can be seen from the UV-vis studies that the carbon clusters increases with increase in electron dose. Therefore, on irradiation, τ_3 decreases and hence free volume size shrinks. Lee et al.²⁵ showed that when PMMA is irradiated by high LET produce high concentration of free radicals over many neighboring molecular chains, facilitating track overlap, and enhancing crosslinking over scission, whereas low LET affects only a simple molecular chain, leading to chain scission. It can be seen in FTIR results, the irradiation of Lexan results in the formation of conjugated bonds and free radicals. After 100 kGy, it seems that the chain-scission process starts dominating over the crosslinking process and thereby increasing free volume size.



Figure 6. Correlation of *o*-Ps lifetime and free volume size V_f with electron doses.





Figure 7. Correlation of *o*-Ps Intensity and relative free volume fraction F_{ν} with electron doses.

Figure 7 shows the variation of o-Ps intensity (I_3) and relative free volume fraction (F_{ν}) with electron dose. In polymers, the o-Ps intensity is proportional to the relative concentration of free volume sites and is influenced by temperature, doping, chemical nature, electric field, and electron irradiation. It is observed from Figure 7 that both o-Ps intensity and F_{ν} increases



Figure 8. Variation of (a) I_1 as a function of electron irradiated dose in Lexan and (b) I₂ as a function of electron-irradiated dose in Lexan.

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27.5-(a)

27.0-

26.5-

26.0

25.5

Figure 9. Correlation of (a) o-Ps intensity plotted against optical energy gap (E_q) of irradiated samples and (b) free volume size plotted against optical energy gap (E_g) of irradiated samples.

for an electron beam of dose 100 kGy, which suggests the redistribution of the molecules. This would effectively reduce the free volume size by splitting the larger size free volume into



Figure 10. Photoluminescence (emission) spectra for pristine and 8-MeV electron-irradiated Lexan.



Figure 11. SEM images for (a) pristine and 8-MeV electron-irradiated Lexan at doses (b) 100 and (c) 225 kGy.

smaller ones and increases the number of such holes as the crosslinking hinders the molecular chain mobility. Therefore, a decrease in τ_3 and V_f is observed.

From Figure 8(a), it can be seen that the intensity I_1 decreases from 46.37% for pristine sample to 38.75% for 100 kGy dose and then increases to 43.37% for 225 kGy dose. Figure 8(b) shows that the intensity I_2 increase from 29.51% for pristine sample to 33.96% for 100 kGy dose and then decrease to 23.68% for 225 kGy dose. The lifetime component τ_2 decreases



Figure 12. Stress-strain curves for Lexan samples as a function of the electron dose.

from 0.48 ns for pristine sample to 0.43 ns for 100 kGy and then increases to 0.44 ns for 225 kGy dose. These variations of positron parameters indicate the changes in the microstructure of polymer on electron irradiation.

To correlate the positron data with the optical data, free volume size and *o*-Ps intensity are plotted against optical energy gap (E_g) of irradiated samples, and are as shown in Figure 9(a,b). It is found from Figure 9(a), initially I_3 increases up to certain value and then decreases with increase in E_g . It is observed from Figure 9(b) that initially V_f decreases by a small value and then increases suddenly with increase in E_g . This variation in I_3 with E_g is due to the random arrangement of the bonds in polymer matrix reduces the mobility of excess electrons to such an extent that they are within the range of the coulombic attraction of a positron, which facilitates the positron–electron recombination and Ps formation.

Photoluminescence Spectroscopy

The formation of defects and chromophores and the presence of impurities, additives, and unsaturation can be studied using photoluminescence.²⁶ The photoluminescence spectra for pristine and electron-irradiated Lexan samples is as shown in Figure 10. When excited around 534 nm, the emission spectra show a strong peak at 632 nm, i.e., 1.96 eV and a weak peak at 680 nm, i.e., 1.82 eV. If defect concentration is more, the luminescence intensity also will be high. In general, the lattice parameter can be correlated with the concentration of defects in the polymer. Movement of the atoms or ions from the lattice sites results in the creation of defects. On irradiation, a small

Table II. Average Molecular Weight (M_V) , the Average Number of Chain Scission per Original Polymer Molecule $(M_{V0}/M_V - 1)$, Tensile Strength, and Percentage of Elongation in Electron-Irradiated Lexan films

Dose (kGy)	M _V	$M_{ m VO}/M_{ m V}-1$	Tensile strength (MPa)	Percentage of elongation
0	17,000	0	58.67	8.54
100	11,200	0.52	56.15	6.19
225	6155	1.76	50.46	5.25

increase in intensity of both peaks shows that the changes in lattice parameters changes slightly.

Scanning Electron Microscopy

The electron-irradiated polymers surface show a visible color change from transparent to yellow and the intensity of the color increases with increase in electron dose. These changes in the color of the electron bombarded samples may be due to formation of carbon clusters resulted from the energy transferred from the incident electrons to the polymer.¹¹ Figure 11 shows the SEM images of the pristine and electron irradiated Lexan samples at different doses. It is observed from Figure 11(b) that the surface of Lexan irradiated with 100 kGy shows microscopic blisters of different sizes. The formation of blisters is possibly due to the chain scission on the surface of Lexan polymer. The chain scission is responsible for the evolution of gases from the surface of polymer which leads to significant surface roughening. Some of the scissioned polymer chains may further crosslink. This may change the free volume on the surface due to which the surface appears imperfect after electron irradiation. On further increase in electron dose, almost all the network structure was found to be broken as shown in Figure 11(c) and its surface roughness increases.²⁷

Mechanical Properties

The tensile stress–strain curve of Lexan samples for different electron dose is as shown in Figure 12. The obtained values of tensile strength and percentage of elongation are listed in Table II. From Table II, it is observed that the tensile strength and percentage of elongation decreases with increase in electron dose indicating that the electron irradiation of Lexan changes its mechanical properties from a ductile material to brittle one. This evolution of the mechanical properties of Lexan with the radiation dose is in agreement with some of the results reported in literature.²⁸ The decrease of tensile strength and percentage of elongation of the samples can be linked to the degradation of the polymer material by the electron irradiation.

Molecular Weight Variation

The estimated values of average molecular weight (M_V) of pristine and irradiated samples using eq. (1) are listed in Table II. The average number of chain scissions per original polymer molecule can be estimated using the equation given by:

$$\frac{M_{V0}}{M_V} - 1 \tag{8}$$

The values estimated are given in Table II. It is found from Table II that the average molecular weight decreases after irradiation. This decrease in average molecular weight is ascribed to chain scission. This is supported by increase in average number of chain scission per original polymer molecule as can be seen in Table II. This change in molecular weight of Lexan after irradiation supports the changes in mechanical properties.

CONCLUSIONS

The following conclusion may be drawn from the studies on electron beam irradiation of Lexan polymers. The E_{g} and E_{a} values were found to decrease with increase in electron dose due to the formation of carbon clusters near the polymer surface due to the electron beam bombardment. The FTIR spectroscopy shows the bond-breaking processes and the formation of conjugated double bonds after irradiation which supports UV-vis spectroscopy studies. The changes in τ_3 , V_6 and I_3 as seen from PALS results reveal that chain scission process dominates over the crosslinking process in Lexan after 100-kGy electron beam irradiation. The correlation of PALS with optical measurement gives the information about how the carbonaceous clusters and vacancy defects are distributed in Lexan films after irradiation. PL result reveals that defect sites increases with increase in electron dose. SEM result shows the blister formation in polymer sample after electron irradiation. The tensile strength, percentage of elongation, and average molecular weight of Lexan decreases after electron irradiation, whereas average number of chain scissions per original polymer molecule increases. This study suggests the possibility of applying these types of films as a chemical sensor in the detection of ionic species and also in the field of space.

ACKNOWLEDGMENTS

The authors acknowledge the financial assistance by the Board of Research in Nuclear Science, Department of Atomic Energy, Government of India. The authors are also thankful to University Science Instrumentation Centre, Karnatak University, for the help during characterization of samples. The authors thank research scholars and staff of the Microtron Centre for their help and support during the course of this work.

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