

Formation of Blisters in Kapton Polymer by the Effect of 1.25 MeV Gamma Irradiation

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ABSTRACT: The surface morphology, chemical, optical, and structural response of 1.25 MeV gamma rays irradiation at various doses ranging from 16 to 300 kGy on Kapton polymer samples were studied by using scanning electron microscope (SEM), Fourier Transform Infrared (FTIR), ultraviolet/visible absorption (UV/VIS) and X-ray diffraction (XRD). The morphology study shows the blisters formation on the Kapton polymer surface due to 1.25 MeV gamma rays irradiation at ambient temperature. This observation provides a basis for the quantitative evaluation of FTIR results obtained for thermally stable polymer on the chemical bond deterioration with increasing gamma irradiation. The blistering mechanism is correlated with the internal gases (CO, H₂) released due to gamma radiation induced damages. The recorded UV-VIS spec-

trum shows a maximum absorption around the wavelength 540 nm. However, the nature of the spectra does not change due to gamma irradiation but a shift in absorption edge towards the higher wavelength side has been observed with increasing dose. The optical data shows an increase in the calculated band gap at the highest dose. The diffraction pattern of virgin sample shows that polymer is semicrystalline, but due to irradiation, a decrease in the peak intensity and FWHM and an increase in the crystallite size at the highest dose level of 300 kGy have been observed. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 2928–2937, 2011

Key words: gamma irradiation; blister; Kapton; SEM; FTIR; UV-VIS; XRD

INTRODUCTION

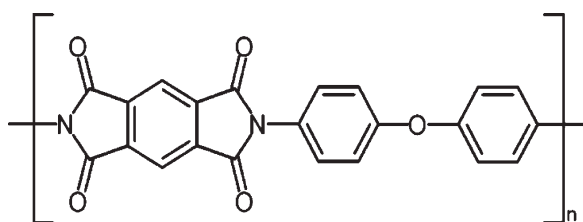
The development of polymers containing aromatic rings linked by various functional groups in the main chain (aromatic polymer) was extensively made during the 1970s to assist the production and the versatility of these polymers as they are used in the field of nuclear energy. Kapton is one of the heat resistive and technologically important polymer, which has the attracted attention of scientists and material engineers since last few decades. It has wide applications in different technologies.^{1,2} Kapton is the most radiation resistant polymer, it is quite suitable polymer specially for radiation application point of view. Satellites in space, face severe thermal vacuum radiation threats particularly when they are in space for longer time. This degrades many properties of their fabricated materials, especially those of outer most surface that face the maximum exposure. This may even cause the failure of the satellite in part or full. However, the flux and the type of

space radiations depend on the altitude of the satellite.³

It is noteworthy that now a days, the materials used in reactors is expected to get radiation dose beyond their tolerance limit in the life span of nuclear reactors.⁴ Polymeric materials are also being used in many ways in the containment building of the nuclear reactor, and in the worst case (e.g., liquid coolant failure accident etc.) such materials can get a radiation dose as high as 5×10^8 Rads.^{2,5} It is desired that the properties of these materials should not be changed even after such a high dose of irradiation. Hence, the qualitative as well as quantitative studies of the radiation damage of these so-called "radiation resistant" polymers like Kapton etc., at such high dose level, is gaining more and more importance. Blistering on the surface of metals and alloys due to ion implantation is a well-known phenomena.^{6,7} Besides its academic interest, it has great importance in nuclear and space technologies due to the fact that materials used are constantly exposed to radiation environment, which predominantly consists of various types of radiations. The well-established theory for blistering in metals may exactly fit for dielectrics, insulators and polymers due to the fact that they are electrical and thermal insulators. This gives rise to a complex situation as compared

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to the metals. One of the major differences in the effect of ion bombardment in metals and insulators is the quenching of the deposited charge. In case of metals, the deposited charge gets quenched immediately together with available free electrons, whereas in insulators it does not occur as such. The energy deposited in polymers due to ion bombardment leads to chemical changes and breakage of chemical bonds of polymer thereby releasing gases, which are independent of the nature of ionizing radiation. The electrostatic charge deposited during ion bombardment may play an additional important role in polymers.⁶ Although a lot of work has been done to investigate the effect of ion irradiation on polymeric materials but the dependence of effect parameters related to ionizing radiation has not been completely understood so far. Therefore, in this article, we report the results of surface morphology, chemical, optical, and structural changes produced by 1.25 MeV gamma ray induced modifications in Kapton polymer. The molecular structure of Kapton polymer is shown below.



MATERIALS AND METHODS

Materials

The Kapton polymer sheets of thickness of 250 μm were obtained from M/S Good Fellow, Cambridge, UK and were used without any further treatment.

Gamma ray irradiation

The polymer samples of size (1 \times 2) cm^2 were cut from the commercially available sheet. One sample was kept as virgin and the other five samples were subjected to irradiation of different doses. The samples were irradiated using 1.25 MeV gamma radiation source of Co^{60} in the radiation chamber (which is in the form of cylindrical chamber of 14-cm length and 10-cm diameter) with dose rate 4 kGy/h and source strength of 2 K Curie. The samples were irradiated in the dose range of 16–300 kGy at UGC-DAE Consortium for Scientific Research, Kolkata Center, Kolkata. The irradiated samples were characterized by using five different analytical techniques.

Surface morphology

The surface morphology of the polymer samples was characterized by scanning electron microscope

(SEM) (from JEOL, Model No. 3300) operating at 30 kV accelerating voltage. Surface of each sample was coated with a thin layer of gold (3.5 nm) by using the vacuum evaporation technique to minimize sample charging effects due to the electron beam.

Spectroscopic measurements

Chemical modifications were evaluated using Manufacturer Perkin-Elmer Corp., Norwalk, Connecticut, Model 1720, Fourier transform infrared spectrophotometer with 0.5 wavenumber resolution in the range (4300–500 cm^{-1}). The optical changes were analyzed by UV-VIS spectroscopy (UV 1601 PC) in the wavelength range 200–900 nm to observe the energy gap (E_g) variation with increasing dose.

Structural study

Structural aspects of Kapton were analyzed using X-ray diffraction technique. Powder XRD measurements were made on PW-1830 machine by using monochromatic CuK_α (8.04 keV and $\lambda = 0.154 \text{ nm}$) radiation in the range of Bragg angles 2θ ($5^\circ \leq 2\theta \leq 90^\circ$) at the scanning rate of $5^\circ/\text{min}$.

RESULTS AND DISCUSSION

To keep the treatment simple and unambiguous, the results of the present study have been discussed separately.

Surface morphology

Radiation induced blistering

Blistering is defined as the plastic deformation of the irradiated surface layer under cumulative influence of a large (critical) number of gas atoms that evolves near the surface region. The blisters formation involves the following three important stages: (i) bubble nucleation, (ii) bubble growth, and (iii) plastic yielding. Experimental and theoretical endeavors to understand these stages and to evolve the systematics of this phenomenon are set about since its discovery,⁷ however, very little has been achieved so far. Transmission electron microscopy of the bombarded specimens show bubbles of size ranging from the resolution limit of the microscope (10 \AA) to a few thousand angstrom.^{8–10} It is observed that the nucleation density of bubbles saturates after a certain radiation dose, and prolonged irradiation results only in their growth.⁹

As the radiation dose increases, the growing bubbles at a certain stage of their growth suddenly coalesce to form a disc shaped cavity parallel to the surface. When the gas pressure inside the cavity

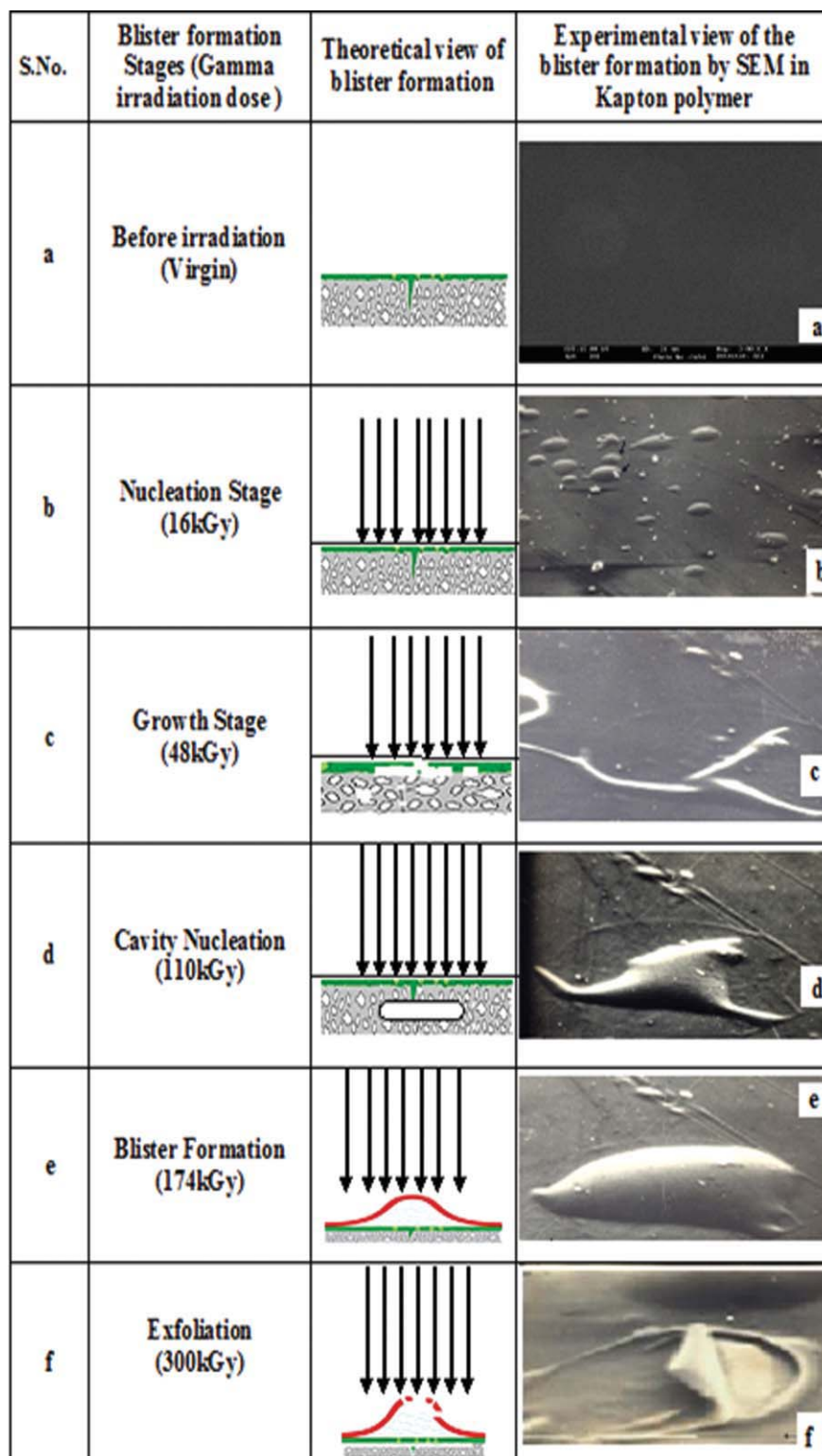


Figure 1 Schematic representation of different stages of blisters formation. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

becomes large, the overlapping material layer is plastically deformed to produce dome shaped structure under the thrust of the gas pressure, known as exfoliation. Different stages of blisters formation are schematically shown in Figure 1. The optical micro-

graphs of virgin and irradiated Kapton samples (a) virgin, (b) 16 kGy, (c) 48 kGy, (d) 110 kGy, (e) 174 kGy, and (f) 300 kGy are shown in Figure 1. The Effects of 1.25 MeV gamma irradiation on them, clearly show sequential development of blisters

observed during the examination of the virgin and irradiated Kapton polymer samples under scanning electron microscope, an important observation made by us. The Figure 1(a) shows SEM micrograph of the virgin Kapton polymer sample. Smooth surface of pure Kapton with very small size of grains is observed throughout the scanned region. Figure 1(b) of SEM micrograph of gamma irradiated polymer sample at 16 kGy shows a number of small size blisters on the surface of Kapton polymer. Further investigations of 48 kGy and higher dose irradiated samples were also recorded using scanning electron microscope (SEM). These micrographs clearly show the surface roughness, due to gamma irradiation. Some of the interesting transitional sequences are illustrated in Figure 1(c–e). This is so called “Swan to Pig” transition taking place during the process of increasing irradiation dose. A swan like feature appears to be made of two different collapsed blisters interconnected by a thin channel shown in Figure 1(c), where the head and the body of the “Swan” are slightly swelled and separated, making the “Swan” rather fatter. It is evident that both the head and the body appear to be seeping inside these “swan” slowly and converting them into a blister in subsequent micrograph shown Figure 1(d). In the next micrograph of SEM shown in Figure 1(e), we observe that the head collapses into a swan which is totally separated from the body and swells further to look like a “Pig.” The boundary of the pig nearly touches the faint boundary of the original swan. From these micrographs, one can infer that the vertical movement of gaseous species or diffusion is a dominant factor as compared to the lateral movement diffusion. This may be due to the local structure of polymer beneath the skin and depend upon whether it is amorphous or crystalline zone and their respective contribution. Most of the gas transitions due to electron beam agitation during SEM take place in the transverse direction with respect to the plane of the sample i.e., the amount of volume swelled is not always constant. In the last micrograph of SEM shown in Figure 1(f), a typical exfoliation blister at 300 kGy gamma irradiation dose showing total exfoliation of the gas.

Fourier transform infrared spectroscopy

To investigate the reasons of blisters formation on Kapton surface, the FTIR studies were carried out for irradiated and virgin polymer samples in the wave number range 500–4000 cm^{-1} . The FTIR spectra of these samples taken in the transmission mode are shown in Figure 2. It is observed that there is no change in overall structure of the polymer, but only a minor change in the intensities have been observed, which implies that the interchain separa-

tion is not affected much by gamma irradiation. Most of the peak positions were found to be unchanged. Only the absorbance or transmittance value of the functional groups was found to change. It may be due to a decrease in the concentration of preexisting bonds or groups. The absorption bands at 3633 and 3485 cm^{-1} can be respectively, assigned to bonds N–H and O–H bond stretching vibration from absorbed water vapor alone rather than partially from alcohol functional groups in Kapton. The hydrogen bonded O–H stretching frequency of the alcohol functional group appears generally in the frequency range of 3700–3200 cm^{-1} . Infrared band at 3092 cm^{-1} in Kapton can be attributed to aromatic C–H stretching frequency and can be observed in the the frequency range of 3200–3000 cm^{-1} . The characteristic imides (NC=O) peak of polymer is observed at 2213 cm^{-1} , after irradiation the new peak is developed at 1601 cm^{-1} . This peak indicates the C=O stretching vibration. All of the absorption bands from 1600 to 725 cm^{-1} have been assigned to substituted phenyl. The absorption band at 3633, 3485, 3092, 2213, and 1601 cm^{-1} have been selected for this analysis, since they have a relatively large absorbance. It is found that the absorption band characteristics of all the above functional groups exist even after the irradiation upto the range (16–300 kGy). The minor change in the intensity of these functional groups is attributed to the emission of adsorbed gases just below the surface. It is inferred that the reduction in specific height is due to deterioration of these groups in the form of H_2 , CO, or CO_2 gases. The release of $\text{H}_2/\text{CO}/\text{CO}_2$ gas results in the blisters formation on the surface of thermally stable Kapton polymer. The minor changes in the peaks of the irradiated samples may be due to the breakage of one or two bonds in the ladder structure, but this will not change the overall structure of the polymer. From these observations, it may be concluded that the Kapton is highly resistant to radiation degradation.

UV–VIS spectral studies

The absorption of light energy by polymeric materials in UV and visible regions involves transition of electrons in σ , π , and n -orbitals from the ground state to the higher energy states. UV/VIS spectroscopy has become an important tool for investigating these electronic transitions. It is used to estimate the value of optical energy gap (E_g) in polymers. The results of absorption studies with UV/VIS spectrophotometer carried out on virgin as well as irradiated Kapton polymer samples are shown in Figures 3–7, respectively. The optical response of virgin and irradiated Kapton polymer samples at various doses

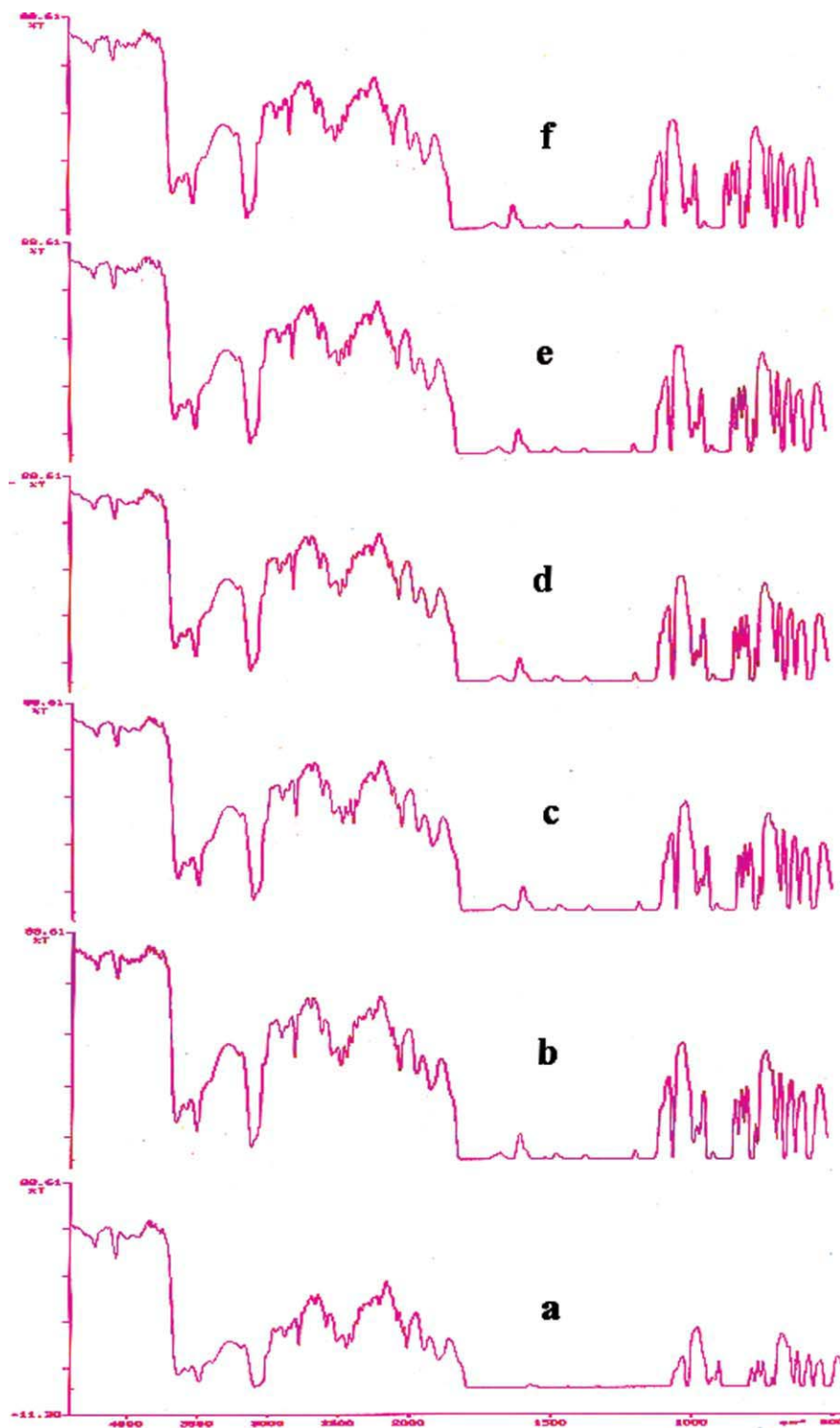


Figure 2 FTIR Spectra of virgin and gamma irradiated Kapton with various doses (a) virgin, (b) 16, (c) 48, (d) 110, (e) 174, and (f) 300 kGy. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

is shown in Figure 3. It is clear from the figure that initially the absorption edge is shifted towards the lower wavelength side with increasing the dose up to 48 kGy, then increases with dose towards the higher wavelength side. The absorption edges lies within the wavelength region 560–600 nm. This shift

in the absorption edge may be correlated with the formation of conjugated bonds showing the possibility of formation of carbon clusters. Further, this type of transition occurs in compounds containing non-bonding electrons and also in aromatic compounds due to bond cleavage and reconstruction.^{11–17}

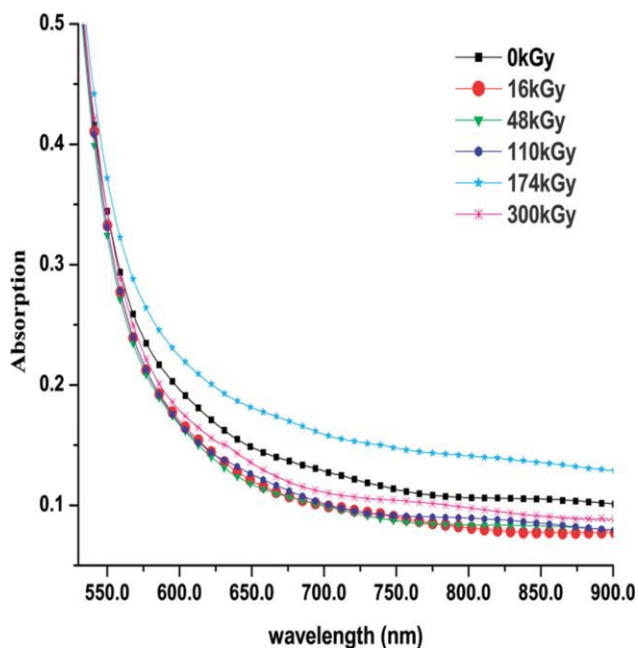


Figure 3 UV/VIS spectra of virgin and 1.25 MeV γ -irradiated Kapton polymers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Determination of band gap

The absorption edge of semi crystalline materials gives the measure of the band strength or band gap E_g , and the position of sharp absorption edge is

$$E_g = hc/\lambda_g \tag{1}$$

where h is the Planck's constant, c is the velocity of light, and λ is the wavelength. The present polymer obeys the rule of indirect transition as well as direct transition.¹⁸ The eq. (2) gives the relationship among

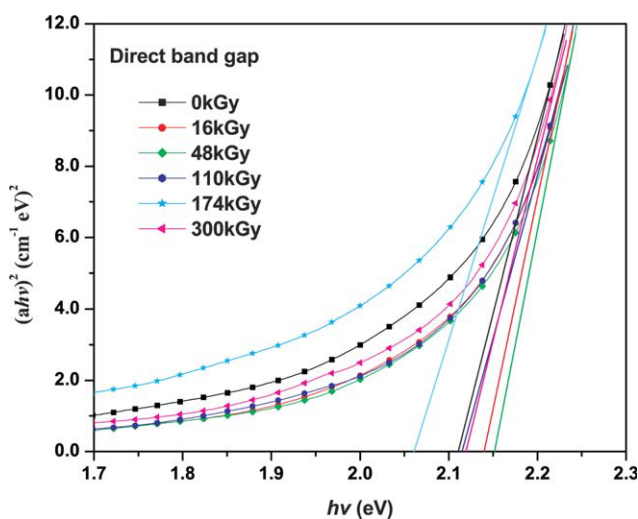


Figure 4 The dependence of $(\alpha hv)^2$ on photon energy ($h\nu$) for virgin and gamma irradiated Kapton polymers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

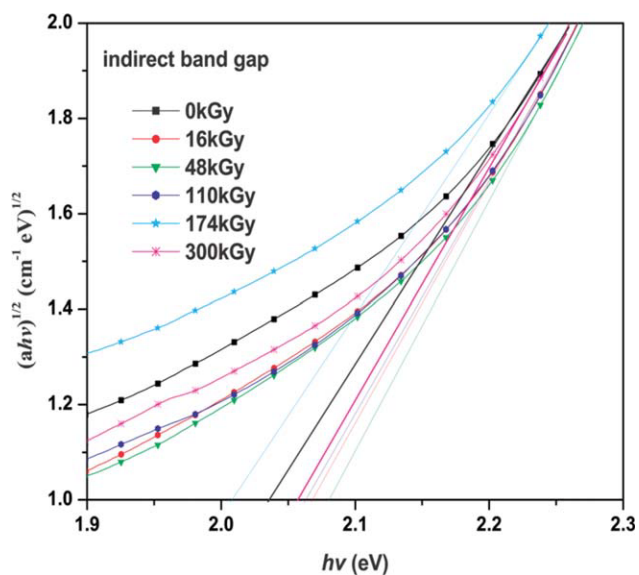


Figure 5 The dependence of $(\alpha hv)^2$ on photon energy ($h\nu$) for virgin and gamma irradiated Kapton polymers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

the optical band gap, the absorption coefficient and the incident photon energy as

$$\alpha(h\nu) = B(h\nu - E_g)^n / h\nu \tag{2}$$

where $h\nu$ is the energy of the incident photons, B is a constant, E_g is the value of the optical energy gap between the valance band and the conduction band, n is the power which characterizes the electronic transition, whether it is direct or indirect during the

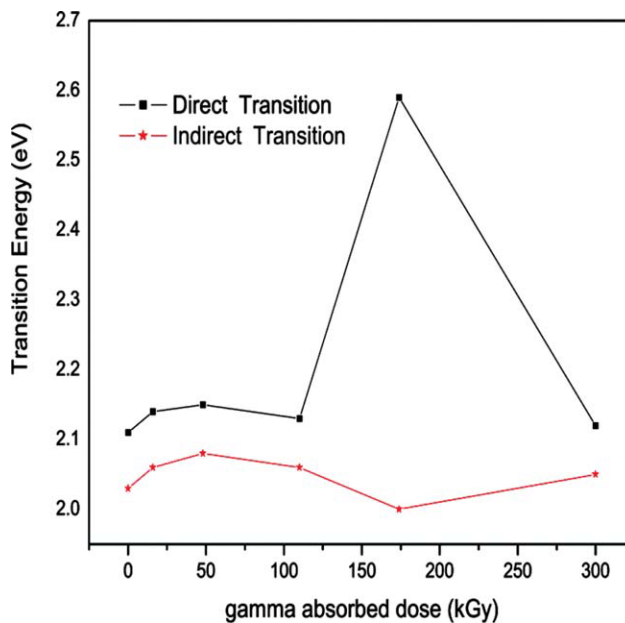


Figure 6 Plots for transition energy (eV) as a function of the gamma absorbed dose (kGy). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

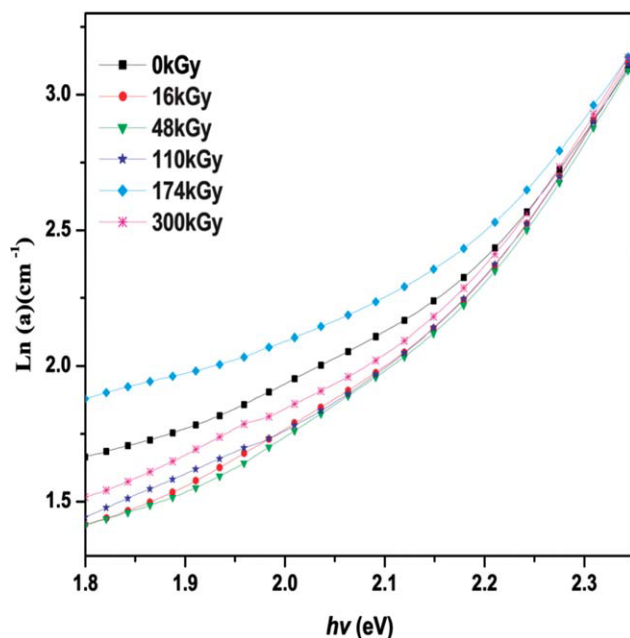


Figure 7 The dependence of natural logarithm of α on photon energy for virgin and gamma irradiated Kapton polymers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

absorption process in the k -space. They value of n as $1/2$, $3/2$, 2 , and 3 stands for direct allowed, direct forbidden, indirect allowed and indirect forbidden transitions, respectively.

The variation of $(\alpha h\nu)^{1/2}$ with photon energy ($h\nu$) for Kapton polymer is shown in Figure 4. The value of the band gap was determined by taking the intercept on the x -axis. It is clear that the value of optical indirect band gap increases from 16 to 48 kGy and decreases from 110 to 174 kGy and again increases at 300 kGy. The variation of energy gap with the irradiation dose is provided in Table I.

The variation of $(\alpha h\nu)^2$ with photon energy ($h\nu$) for Kapton polymer is shown in Figure 5. It is clear that like the value of optical indirect band gap the value of optical direct band gap also increases from 16 to 48 kGy, decreases at 110 kGy, again increases at 174 kGy and finally decreases at the highest dose.

In these studies, the results were obtained by plotting $(\alpha h\nu)^{1/2}$ and $(\alpha h\nu)^2$ as a function of the photon energy ($h\nu$), respectively. Taking into account the linear portion of the fundamental absorption edge of the UV/VIS (shown in Fig. 3) such plots have been presented in Figures 4 and 5, respectively. By extrapolating the straight parts of this relation to the $h\nu$ axis, indirect and direct band gaps have been determined for virgin and gamma irradiated Kapton polymer samples. The different transition energies are shown in Figure 6 and are also provided in Table I. This, in turn, clearly indicates the simultaneous existence of indirect and direct band gaps in the Kapton polymer samples with decreasing tendency at higher gamma ray dose. These results confirm that the irradiation produces faults in Kapton polymer structure (band rupture, free radical, etc.) which consequently increase the electronic disorder responsible for the creation of a permitted state in the forbidden (interdict) band or the deformation of valence band. Furthermore, the values of indirect band gap have been found to be lower than the corresponding values of direct band gap as given in Table I. The coexistence of indirect and direct band gaps in the Kapton polymer has not been reported so far, but such results have been observed in some other materials.^{19–21}

Urbach rule

The absorption coefficient near the band edge for noncrystalline materials shows an exponential dependence on the photon energy ($h\nu$) and is given by the expression known as Urbach formula²²

$$\alpha(\nu) = \alpha_0 \exp(h\nu/E_u) \quad (3)$$

The exponential absorption edge (known as Urbach's rule) observed in most materials is interpreted in terms of thermal fluctuations in the energy band gap. In eq. (3), α_0 is a constant, E_u is an energy which is interpreted as the width of the tail of localized state in the forbidden band gap, ν is the frequency of radiation, and h is Planck's constant. The

TABLE I
The Variation of the Band Gap Energy and Urbach's Energy in the Virgin and Gamma Irradiated Kapton, Along with the Number of Carbon Atoms (N) Per Conjugated Length

γ -radiation dose (kGy)	Direct band gap energy (eV)	Indirect band gap energy (eV)	Urbach's energy (eV)	Direct (N)	Indirect (N)
0	2.11	2.03	0.30	9~	9~
16	2.14	2.06	0.28	8~	9~
48	2.15	2.08	0.28	8~	9~
110	2.13	2.06	0.78	8~	9~
174	2.59	2.00	0.32	7~	9~
300	2.12	2.05	0.25	8~	9~

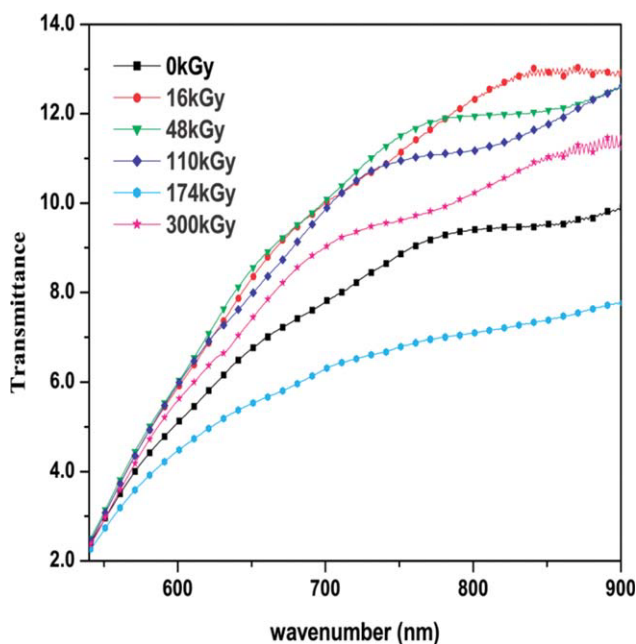


Figure 8 Gamma radiation dose dependence of optical transmittance spectra of virgin and Irradiated Kapton polymer. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

origin of E_u is supposed to be due to thermal vibrations in the lattice.²³ The logarithm of the absorption coefficient $\alpha(\nu)$ was plotted as a function of the photon energy ($h\nu$) for irradiated Kapton polymer samples with different doses of gamma rays as shown in Figure 7. The value of the Urbach energy E_u in each case was calculated by taking the reciprocal of the slope of the linear portion in the lower photon energy region of these curves and is listed in Table I. The decreases in Urbach's energy in the case of Kapton polymer may be due to a decrease in the crystalline nature of the polymer at highest dose.

The number of carbon atoms per conjugation length N (i.e., the number of carbon hexagonal atoms in a cluster) can be calculated by Robertson relation.²⁴ Given by

$$N = 2\beta\pi/E_g \quad (4)$$

Here 2β gives the band structure energy of a pair of adjacent π sites. The value of β is taken to be -2.9 eV as it is associated with $\pi \rightarrow \pi^*$ optical transitions in $-\text{C}=\text{C}-$ structure. A shift in the absorption edge can be attributed to an increase of the conjugation length. The number of carbon atoms per conjugation length is found to increase up to 9 at the highest dose i.e., 300 kGy. The increase of unsaturated conjugation length in polymer samples results in their discoloration i.e., the Kapton polymer samples turn light yellow from transparent with increasing gamma radiation dose. Gamma radiation can induce the for-

mation of unsaturated bonds in polymers and contribute to coloration, since as a rule, the conjugated double bonds are responsible for the color of most organic compounds.^{12,21} The optical transmission spectra of both virgin and irradiated polymer samples show some variation in transmittance with increasing dose. They also have very low UV cut off range 480–490 nm. In terms of percentage of transmission, it is

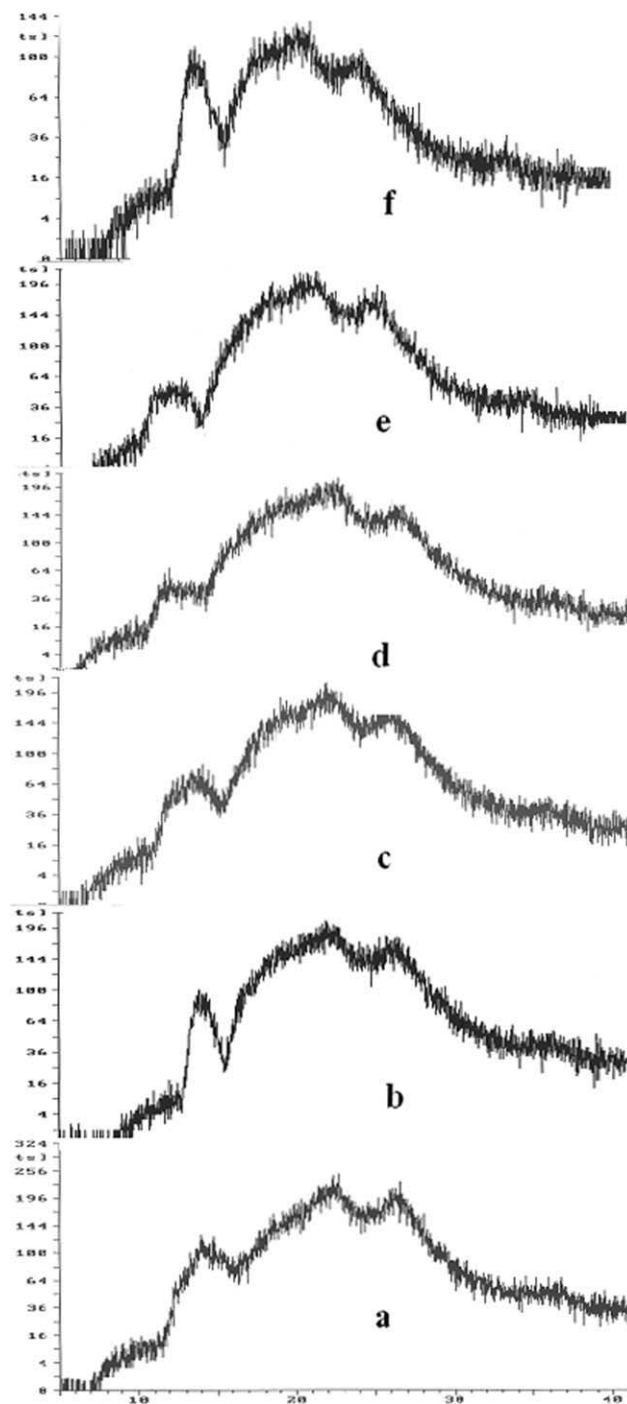


Figure 9 X-ray diffraction patterns of Kapton (a) virgin and irradiated (b) at 48 kGy, (c) 110 kGy, (d) 142 kGy, (e) 174 kGy, and (f) 300 kGy Kapton polymer.

TABLE II
X-ray Diffraction Spectra of 1.25 MeV Gamma Irradiated Kapton Polymer at Different Doses

S. No	Angle of peak (2θ)	Dose (kGy)	FWHM (β)	Crystallite size (L)	Peak intensity	Strain $\epsilon = (\beta \cos \theta)/4$	Dislocation $\delta = (15 \epsilon/aD)$
1	14.02	0	0.960	083.49	50	0.2328	0.52
2	14.52	16	0.800	100.14	52	0.1936	0.33
3	13.55	48	0.800	099.10	34	0.1944	0.35
4	15.10	110	0.960	083.49	28	0.2317	0.51
5	13.32	174	0.480	174.68	20	0.1165	0.12
6	13.75	300	0.400	200.10	32	0.0971	0.08

observed that the optical transmission for both virgin and irradiated polymer samples initially increases in visible as well as in infrared regions. The increase in optical transparency indicates the decrease in band gap. Further, the optical transmission is found to decrease with increasing dose, it indicates the increase of band gap as shown in Figure 8.

Structural study

The XRD spectra of both virgin and irradiated Kapton polymer samples were recorded in the angular range $0 < 2\theta < 40$ at different doses of radiation (16, 48, 110, 174, and 300 kGy). They are shown in Figure 9(a–f). Figure 9(a) show the spectrum of virgin polymer sample. This is semicrystalline in nature, it has a crystalline peak at $2\theta = 14^\circ$ and an amorphous humps at $2\theta = 22^\circ$ and 26° . This semicrystalline nature of polymers arises due to the systematic alignment of polymer chain folding.²⁴

Figure 9(b) shows the XRD pattern of gamma irradiated Kapton polymer sample at 16 kGy, the peak intensity is found to increase. Figure 9(c,d) show that peak intensity decreases in the irradiated samples at 48 and 110 kGy. Again, the peak intensity increases at 174 kGy and also at the highest dose (300 kGy) as shown in Figure 9(e,f), respectively. The full width at half maximum (FWHM) is generally associated with the crystallite size which can be obtained from Scherrer's formula

$$L = K\lambda/\beta \cos \theta \quad (5)$$

where $K = 1$, $\lambda = 1.54 \text{ \AA}$, and $\beta = \text{Fwhm}$ in radian.

The strain (ϵ) value can be evaluated using the relation

$$\epsilon = (\beta \cos \theta)/4 \quad (6)$$

The dislocation density (δ) may be calculated by using the formula:

$$\delta = 15 \epsilon/(a \times D) \quad (7)$$

Various observed and calculated parameters, such as the peak intensity, strain, dislocation, FWHM, and

crystallite size with irradiation dose are provided in Table II. The results indicate the occurrence of recrystallization in Kapton polymer samples due to increase of gamma irradiation dose.

CONCLUSION

Modifications in Kapton Polymer samples under 1.25 MeV gamma radiation source of Co^{60} have been studied by means of SEM, FTIR, UV-VIS, and XRD. The following conclusions have been drawn:

- The blisters formation on the surface of the Kapton is ascribed as a result of rupture of chemical bonds and the formation of low molecule gases. These gases are accumulated inside the polymer at a depth where maximum radiation damage takes place. When the pressure of accumulated gas crosses the mechanical strength of Kapton polymer, it deforms and results in blisters formation. The emission of low molecular gases (i.e., CO and H₂) is also confirmed with the FTIR analysis of irradiated Kapton samples.
- The average of peak intensity is found to increase with increasing dose. It shows that crystalline nature of the Kapton polymer increases. The crystallite size is also found to increase with increasing dose.
- The peak intensity, strain (ϵ) and dislocation (δ) are found to show recovery characteristics with increasing dose.
- The optical band gap (E_g), and Urbach energy (E_u) were determined from the optical absorption spectra. It is concluded that the values of indirect band gap are lower than the corresponding values of direct band gap in both virgin and gamma irradiated Kapton Polymer samples. Also the values of band gap (E_g) changes with the increase of gamma irradiation dose, due to the photo-degradation of Kapton polymer and the formation of defects and clusters in the material, while the cluster size (number of carbon atoms N per conjugated length) increases with the increase of absorbed dose.

In all the four experimental studies such as SEM, FTIR UV-VIS, and XRD, the results show recovery behavior with radiation dose. This may be due to scissioning and crosslinking of the polymer chains which can be correlated with the recovery of original characteristic with increasing dose.

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