

Electron Beam and Gamma Radiation Dose Dependency on the Degradation Products of Polytetrafluoroethylene (PTFE)

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Abstract

Gamma radiation induced degradation phenomena of polytetrafluoroethylene (PTFE) in the form of granules has been studied. The Electron Spin Resonance (ESR) technique was used to study radical generation and behavior under different conditions. The ESR spectra of PTFE irradiated at low or very high doses of irradiation were doublets, characteristic of peroxy radicals. Similarly spectra at different temperatures are also doublets. In order to study nature of free radicals, the *g*-values are calculated under different conditions and reason for variation in *g*-values is discussed. The PTFE irradiated with electron beam (ebeam) to 10 Mrad radiation dose contain some latent hyperfine structure in their ESR spectra. The hyperfine pattern was due to radicals $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F}_2\sim$ and/ or $\sim\text{CF}_2\text{-CF}_2\sim\text{OO}$ as revealed by computer simulations. The Fourier Transform Infrared (FTIR) spectra has shown the formation of functional groups such as $-\text{C}=\text{O}$, $-\text{COOH}$ and $-\text{COF}$ groups with the increase of radiation dose. The decrease in $-\text{C-F}$ stretching band intensity and the increase in $-\text{CF}_2$ group intensity is observed.

Keywords: Electron spin resonance (ESR), polytetrafluoroethylene (PTFE), gamma radiation, electron beam, degradation studies

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INTRODUCTION

The chemical structure of PTFE is type of polyethylene, where in the hydrogen atoms are replaced with Fluorine. The Fluorine atoms are larger than the hydrogen atoms and surround the carbon chain like a protective mantle. Therefore chemical resistance of the polymer is retained even at elevated temperatures. Though high crystallinity and low intermolecular forces render PTFE resistant to all solvents, PTFE is not a radiation resistant thermoplastic material. Tabata et al. [1] have reported two different ESR spectra for crosslinked and non-crosslinked PTFE sheets. The spectrum for non-crosslinked PTFE is attributed to $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F-CF}_2\sim$ (mid chain radicals) and the other one is determined by methylene type $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F}_2\sim$ radicals trapped in

the crystalline region of PTFE. The dynamic visco-elastic properties for crosslinked and original PTFE were studied by Oshima et al. [2]. These authors have proposed three types of motion in PTFE. Oliver et al. [3] have studied the molecular motion of PTFE using peroxy probes. Based on the variation in intensity of the 980 cm^{-1} FTIR absorption band, these authors have found 80% crystallinity in irradiated PTFE. Shimada et al. [4] have treated the chain end peroxy radicals of polyethylene on the surface of PTFE and studied the molecular motion. FTIR spectra of electron irradiated PTFE have been reported in detail by Fischer et al. [5]. Degradation of PTFE by electron beams leads to the formation of acid fluoride (COF) groups. The COF groups hydrolyze to carboxylic acid

groups (COOH) in the presence of atmospheric humidity. Nakayama et al. [6] have observed that, the effect of surface modification on Polytetrafluoroethylene (PTFE) by Ion irradiation. The results showed that low ion energy was produced polar groups components and high ion energy prevented the surface roughness. Zhongfeng et al. [7] have studied the tribological properties of PTFE sheet irradiated by electron beam. The results show that as the dose rate increases, the heat of crystallization and melting point of the cross-linked sheet of PTFE was decreased. After irradiated by Electron beam, PTFE cross-linked sheet became transparent and the friction coefficient increased with the absorbed dose. Sergei et al. [8] have observed that, the effect of irradiation on PTFE above the melting point. The result shows that there is a decreasing in porosity of irradiated PTFE above the melting point, due to the viscous flow produced by surface tension. Allayarov et al. [9] have observed that, thermal stability and phase behavior of PTFE by protons. The thermal stability of proton bombardment of PTFE was decreased. Taeger et al. [10] have reported that, the chemical compatibilization between irradiated PTFE and (PEEK) poly(ether ether ketone). Allayarov et al. [11] have studied the isotropic and anisotropic behavior of PTFE by gamma and laser Irradiated. The results showed that the powder and sheet of PTFE has amorphous nature and the melting point and glass transition temperatures of PTFE decreased as the radiation dose increased. Zhongfeng Tang et al. [12] have reported that, the crystal size and melting temperature of cross-linked polytetrafluoroethylene (XPTFE) was decreased with the increased radiation dose. Ye J and Pei Y [13] have studied that, the degradation effect of PTFE by electron beam irradiation at different doses and atmosphere conditions. They were observed the degradation effect was more on PTFE under electron beam irradiation. There was better degradation effect on PTFE sample at low dose rate compared to high dose rate under same atmosphere conditions. Electron beam irradiation on PTFE above melting point,

decreasing porosity and improved the mechanical strength of the sample. Shuling Liu [14] has studied the effect of irradiation on structural changes of PTFE in oxygen. They observed that the new groups of COF, COOH) and CF₃ were formed under heavy radiation dose exposure in oxygen. Xiaoyun Li, et al. [15] have studied the structural evolution of polytetrafluoroethylene (PTFE) and cross-linked PTFE irradiated by electron beam under stretching using synchrotron small- and wide-angle X-ray scattering SAXS/WAXS. The results showed that under higher radiation dose, the cross-linked density was higher, and the number of cross-links and network structure were formed. Zhaoxiang et al. [16] has reported the preparation, modification and application of PTFE membrane. Wyszowska et al. [17] has studied the effect of mechanical, chemical structure and optical properties of PTFE sheet by different gamma radiation doses. They observed that the properties of mechanical, density and crystallinity of the gamma irradiated PTFE was increased. Mohammadian-Kohol et al. [18] has observed gamma irradiation effect on the optical, structural and mechanical properties of PTFE sheet. The results showed that, the stiffness and the elastic behavior increased and plastic behavior and tear resistance of PTFE sheet was decreased as the absorbed dose rate increased.

In the present study, the PTFE in the form of granules is used. The PTFE is subjected to high energy radiation, such as electron beam (e-beam) and gamma rays from sterilization dose to high radiation doses. But the highly crystalline PTFE undergoes degradation with generation of free radicals, which is the cause of further deterioration in the PTFE properties. It is important to observe changes in PTFE, under the influence of high-energy radiation, particularly for medical applications because the formation of highly reactive acidic groups may cause harm to the end user. Hence these preliminary studies are under taken with a view to stabilize PTFE against high-energy radiation.

EXPERIMENTAL WORK

Materials and Irradiation

The PTFE in the form of granular powder obtained as a gift sample from Hindustan Fluoro Carbon Ltd. Hyderabad, INDIA, is of Hiflon 51A grade. The particle size of the sample was $\sim 400 \mu$. The granular powder samples are irradiated by gamma rays from Co^{60} source at the dose rate of $= 0.15 \text{ Mrad/hr}$ in air, at room temperature. A fixed amount ($\sim 50 \text{ mg}$) of PTFE is taken throughout for all EPR studies. The ESR spectra of the irradiated samples are recorded with a German made Bruker spectrometer operating at X-band frequencies and with 100 kHz modulation. The ESR spectra are recorded for all samples with the same receiver gain (5×10^4) and micro power (20 mW). The Diphenyl Picryl Hydrazyl (DPPH) is used as standard reference for recording ESR spectra of PTFE. The DPPH standard has a g -value of 2.00136 , which is used as reference g -value for calculating the g -values of the ESR spectra of PTFE. The FTIR spectra of unirradiated and irradiated PTFE have been recorded in KBr pellets on a NICOLET spectrometer and the DSC thermograms were recorded on Universal TA instrument.

Computer Simulations

ESR spectra recorded for irradiated PTFE under different conditions are analyzed by the method of computer simulations based on total curve fitting. Since the ESR spectrum is assumed to arise from the superposition of various component spectra, which arise due to the presence of various free radical species formed on irradiation of the polymer. By generating the component spectra assuming the ESR line to have either Lorentzian or Gaussian line shape equation, which contain magnetic parameters like relative intensity, line width, center of spectrum, hyperfine splitting constants and hyperfine lines arising due to adjacent magnetic nuclei. Various types of component spectra are generated and superposed in appropriate compositions to yield the experimental spectrum. Based on the values of n_i and m_i the chemical constitution of the free radical species

is identified. The method has been described previously in several papers [19–21].

RESULTS AND DISCUSSION

Electron Spin Resonance Studies

Effect of Radiation Dose

The ESR spectra of gamma irradiated PTFE have been recorded by irradiating the polymer at various doses. The spectra shown as curves 1 to 9 of Figure 1, refer to the irradiation doses of 1, 3, 4, 7, 10, 20, 30, 40 and 50 Mrad, respectively. The spectra observed for PTFE at different doses are almost doublets characteristic for peroxy radicals with some changes in the peak intensities. The spacing between two peaks is almost constant and it is around 6 G and the spread of the spectrum is around 70 G . The g -value of the two radicals corresponding to the peaks is around 2.02200 and 2.00976 respectively. All the ESR spectra are recorded under identical conditions. The area under the curve represents the total spins present in the polymer sample.

The ESR spectra of irradiated PTFE are an asymmetric doublet and assumed to consist of two components namely symmetric and asymmetric components. They are distinguished by their characteristic line shape and exhibit g -anisotropy. Further the components are observed at high and low magnetic fields of ESR spectrum. In this context Matsunaga and Shinohara [22] have recorded ESR spectra of irradiated PTFE under different conditions. They have proposed the existence of two types of peroxy radicals. The radical with the g -value around 2.00976 is called the symmetric component (due to mid-chain peroxy radicals), while the other one is with high g -value of 2.02200 is called the asymmetric component (due to end chain peroxy radicals). As such the ESR spectra of PTFE, gamma irradiated in the dose range of $1\text{--}50 \text{ Mrad}$ have been analyzed. It is observed that, with the increase of radiation dose the formation of end chain peroxy radicals increases. Formation of peroxy radicals in irradiated PTFE can be explained as follows. The bond energy of the C-C bond is less than

that of C-F bond. As the C-F bond energy is larger, there is less possibility of cleavage of these bonds on irradiation. Hence, the formations of the end chain radicals are more probable. The intensity variation of the ESR doublet with the increase in gamma dose is shown as curve 1 (P₁) and curve 2 (P₂) of Figure 2.

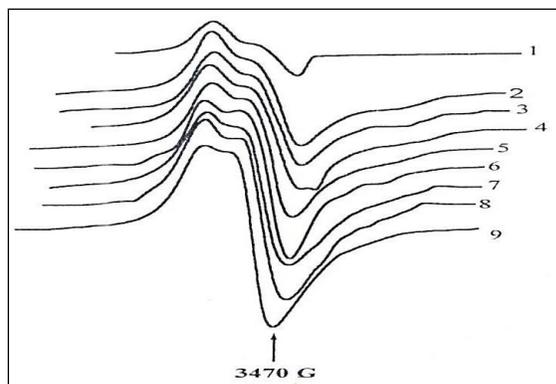


Fig. 1: The ESR Spectra of Gamma Irradiated PTFE at Various Doses 1, 3, 4, 7, 10, 20, 30, 40 and 50 Mrads.

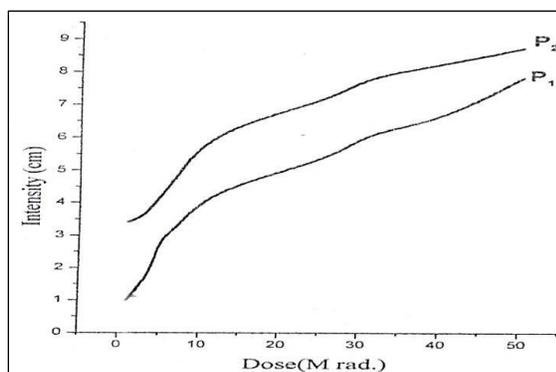


Fig. 2: Variation of ESR Intensity Against Radiation dose. P₁-Symmetric; P₂-Asymmetric Component of the ESR Spectrum.

The intensity variation of both the peaks (P₁ & P₂) has been observed. The peak P₁ corresponds to the symmetric component of the ESR spectrum and the peak P₂ correspond to the asymmetric component of the ESR spectrum. However, the increase in intensity of P₂ is much higher than that of P₁ with the radiation dose. Therefore, the increase in irradiation dose leads to the formation of a higher number of

asymmetric peroxy radicals. Therefore, as the radiation dose increases the formation of radicals of the type $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F-CF}_2\sim$ and $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F}_2$ takes place. But the formation of $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F}_2$ radicals is more probable and these radicals abstract atmospheric oxygen and converts to the corresponding peroxy radicals. Hence the P₂ peak intensity increases with the radiation dose (Table 1 and Figures 3–4).

Table 1: *g*-values at Different Radiation Dose.

Radiation Dose (Mrad)	$g_1 \times 10^{-7}$	$g_2 \times 10^{-7}$	$g_3 \times 10^{-7}$
1	2.00695	2.00386	2.00017
3	2.00695	2.00386	2.00017
4	2.00695	2.00386	2.00017
7	2.00695	2.00386	2.00017
10	2.00695	2.00386	2.00017
20	2.00695	2.00387	2.00017
30	2.00695	2.00387	2.00017
40	2.00695	2.00386	2.00017
50	2.00695	2.00387	2.00017

Effect of Temperature

The ESR spectra of irradiated PTFE are recorded in the temperature range of 77 K–470 K. The resolution of spectral lines depends on temperature of observation, i.e., spectra at low temperatures are poorly resolved; while high temperature spectra have good resolution. The spectra, basically has doublet structure, with some hyper fine structure. The computer simulations were employed to analyze the spectra, as discussed in the earlier section. The magnetic parameters employed to simulate the spectra are as given Table 2.

Table 2: Magnetic Parameters of Irradiated PTFE.

Temperature (K)	Relative intensity (G)	Line width a_i (G)	Centre of Spectrum X_i (G)	Hyperfine Splitting		n_i	m_i
				A_i	B_i		
77	14	16	3230	23	12	2	5
130	8.4	14	3230	23	12	2	5
150	7	12	3230	23	12	2	5
170	5.6	9	3230	23	12	2	5
220	4.9	7	3230	23	12	2	5
270	4.2	6	3230	23	12	2	5
300	3.5	5	3230	23	12	2	5
320	2.8	4	3230	23	12	2	5
370	2.1	3	3230	23	12	2	5
420	1.4	2	3230	23	12	2	5
470	0.7	1	3230	23	12	2	5

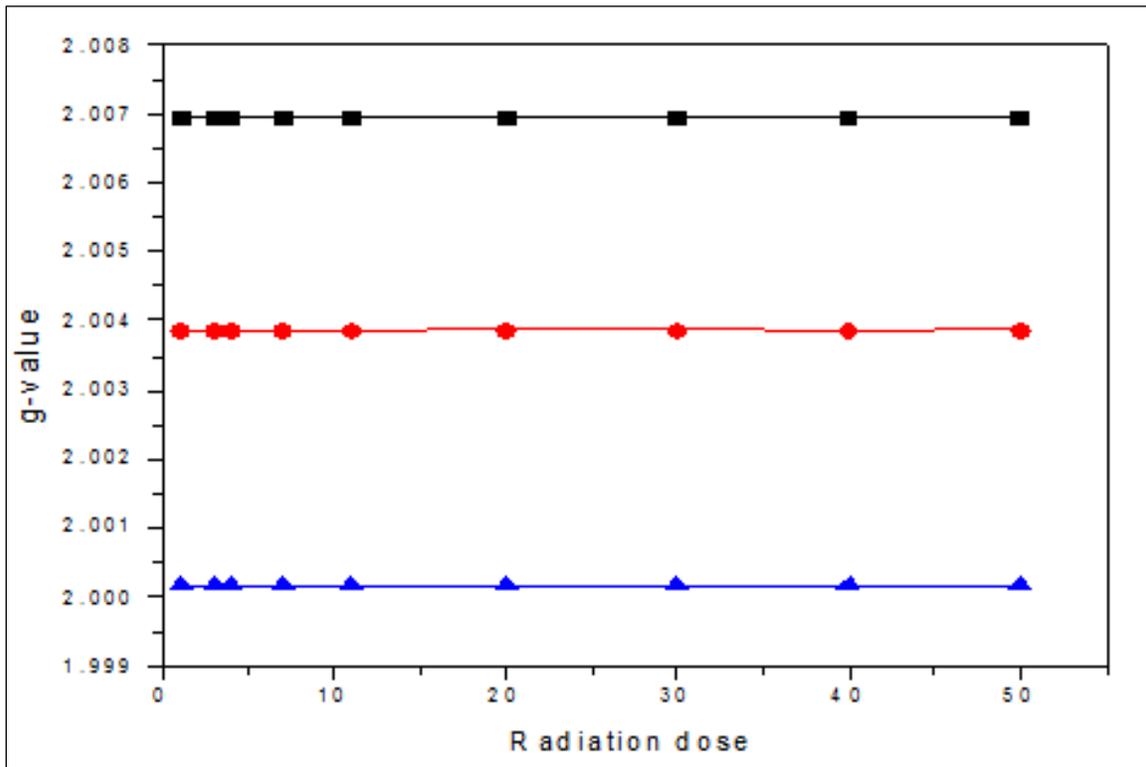


Fig. 3: Variation of g-value with Radiation Dose.

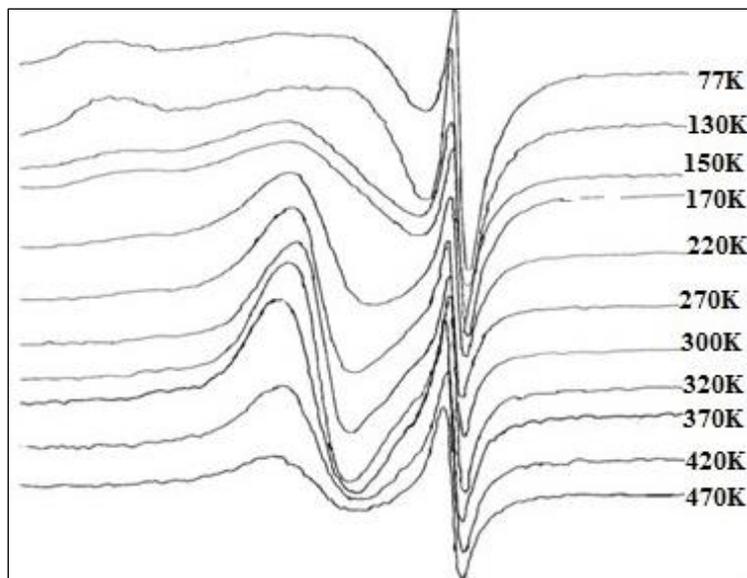


Fig. 4: The ESR Spectra of Gamma Irradiated PTFE at Various Temperatures.

Computer Simulation

The hyperfine pattern observed in the ESR spectrum of PTFE irradiated at 10 Mrad is shown in Figure 5. In order to analyze the

complex hyperfine pattern, computer simulations are employed. Curve 1 of Figure 5 is the component triplet spectrum, which is simulated with values of $n_i = 2$, $m_i = 5$ and $A_i = 23$

G, $B_i = 12$ G. The radicals contributing to the formation of the triplet component in an ESR spectrum of irradiated PTFE may be of the type $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F}_2$. The curve 2 is doublet which is attributed to $\sim\text{CF}_2\text{-CF}_2\text{-O}\dot{\text{O}}$ and $\sim\text{CF}_2\text{-CF}(\text{O}\dot{\text{O}})\text{-CF}_2\sim$ i.e. both end chains and mid chain peroxy radicals. The curve 3 is component singlet spectrum.

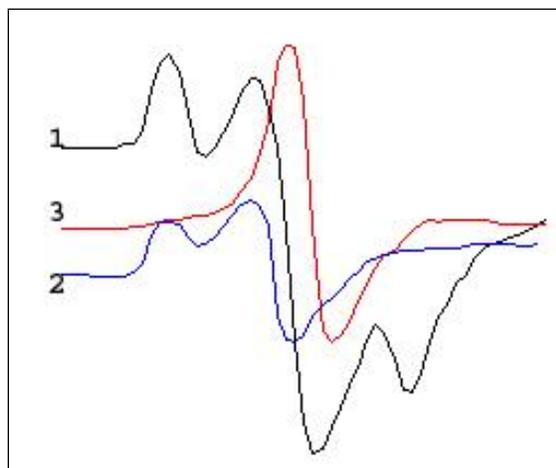


Fig. 5: The ESR Spectra of PTFE Irradiated at 10 Mrad.

The ESR spectrum of PTFE above room temperature have a doublet characteristic of peroxy radicals, but the peak corresponding to symmetric mid chain peroxy radicals have very less intensity and the asymmetric end chain peroxy radicals have more intensity. The peroxy doublet of irradiated PTFE consists of symmetric ($\sim\text{CF}_2\text{-CF}(\text{O}\dot{\text{O}})\text{-CF}_2\sim$) and asymmetric ($\sim\text{CF}_2\text{-CF}_2\text{-O}\dot{\text{O}}$) components with different g-values. Under such conditions, contribution of each component has been evaluated the formation of $\sim\text{CF}_2\text{-CF}_2\text{-O}\dot{\text{O}}$ radicals is more pronounced at high temperatures.

The ESR spectra at different temperatures are also simulated with almost same values of magnetic parameters. Peroxy doublet and component singlet are almost present at all temperatures, but concentration of macroradicals is different. The Component spectrum due to macroradicals at different temperatures is shown in Figure 6.

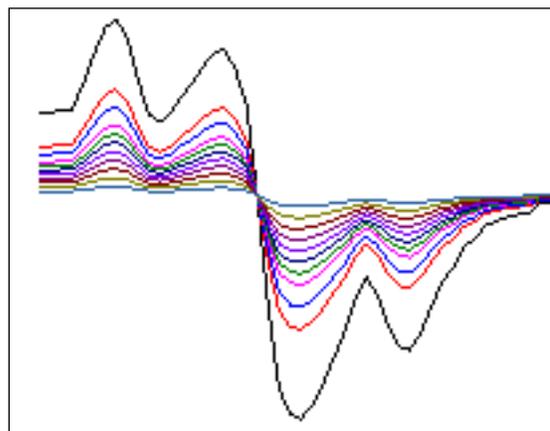


Fig. 6: The Simulated ESR Spectra of Irradiated PTFE at Different Temperatures.

Evaluation of g-value

The ESR spectra of irradiated PTFE possess characteristic line shape corresponding to the peroxy radicals. To study the variation of g-anisotropy with temperature, g-values are calculated at different temperatures and listed in Table 3. A graph is drawn to show the variation of g-value against temperature and the same is shown in Figure 7.

Bloch Analysis

To evaluate activation energy associated with free radical decay, Bloch analysis is applied [23, 24]. For this purpose line widths at different temperatures are calculated to find the value of τ as listed in the Table 4. A plot of \log of inverse of (τ) against inverse of temperature is drawn as shown in Figure 8. The curve is straight line and the value of slope gives activation energy. For Gamma irradiated PTFE, the average value of the slope is around 389 K J/mole; while low temperature slope is 358 kJ/mole and high temperature slope is 421 kJ/mole. Corresponding graphs are as shown inset of Figure 8. The results suggest that inter molecular interactions might have occurred due to crosslinking, preventing the recombination of free radicals. The curve is a straight line. From the slope of curve, the activation energy associated with free radical decay is evaluated. For irradiated PTFE the average value of E is around 389 K J/ mole.

Table 3: Variation of g-values with Temperature.

Temperature (K)	$g_1 \times 10^{-7}$	$g_2 \times 10^{-7}$	$g_3 \times 10^{-7}$
77	2.0448	2.0402	2.0268
130	2.0448	2.0402	2.0268
150	2.0448	2.0402	2.0268
170	2.0448	2.0402	2.0268
220	2.0448	2.0402	2.0268
270	2.0448	2.0402	2.0268
300	2.0448	2.0402	2.0268
320	2.0448	2.0402	2.0268
370	2.0448	2.0402	2.0268
420	2.0448	2.0402	2.0268
470	2.0448	2.0402	2.0268

Table 4: Parameters used in Bloch Analysis.

Temperature (K)	$1/T \times 10^{-3}$	θ	$\tau \times 10^{-3}$	$1/\tau$	$\log(1/\tau)$
77	12.98	33	-	-	-
130	7.6	30	2.842	351.75	2.546
150	6.6	28	2.694	371.07	2.569
170	5.88	26.5	2.597	385.04	2.585
220	4.54	25.3	2.528	395.53	2.597
270	3.70	24.32	2.453	407.60	2.61
290	3.3	23.48	2.412	414.49	2.617
320	3.12	22.75	2.372	421.42	2.624
370	2.70	22.1	2.349	425.66	2.629
420	2.38	21.52	2.319	431.19	2.634
470	2.12	21	2.286	437.35	2.64

Fourier Transform Infrared (FTIR) Studies

The FTIR spectra of pure and irradiated PTFE have been recorded to ascertain the chemical changes brought about by the radiation and the same are shown in Figure 9. The spectra of irradiated PTFE possesses absorption bands at 2911-2891 2816-2811, 2360-2336, 1278-1147 cm^{-1} and at 3550, 1721-1675 cm^{-1} . The new bands at 3550 cm^{-1} and 1721 cm^{-1} are attributed to formation of -OH and -C=O groups on irradiation. A variation of the intensity with the radiation does have also been observed for the 2811, 1721-1675, 1275-1147 cm^{-1} band. These studies indicate that the intensity at 2811 cm^{-1} i.e. the C-F stretching intensity is decreased due to abstraction/dissociation of fluorine from polymer molecule. On the other hand an increase in the absorption band at 1885 cm^{-1} indicates an increase in the concentration of -COF groups with radiation does, due to abstraction of fluorine atoms from the crystalline phase of PTFE. The abstraction of atmospheric hydrogen by peroxy radicals results in the formation of -COOH groups in the irradiated PTFE.

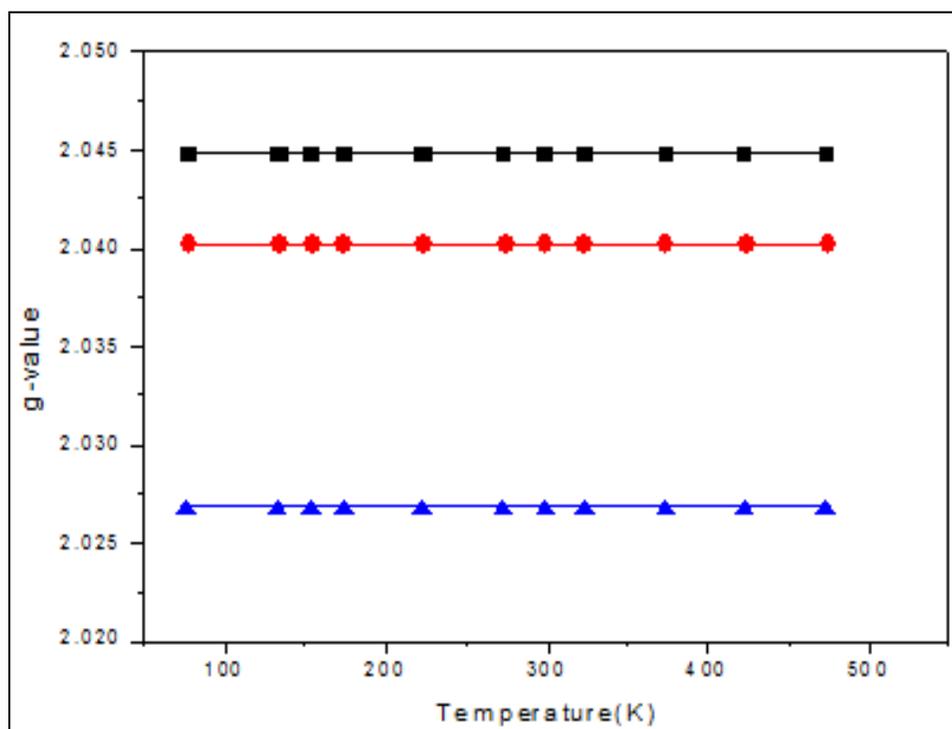


Fig. 7: Variation of g-values Against Temperature.

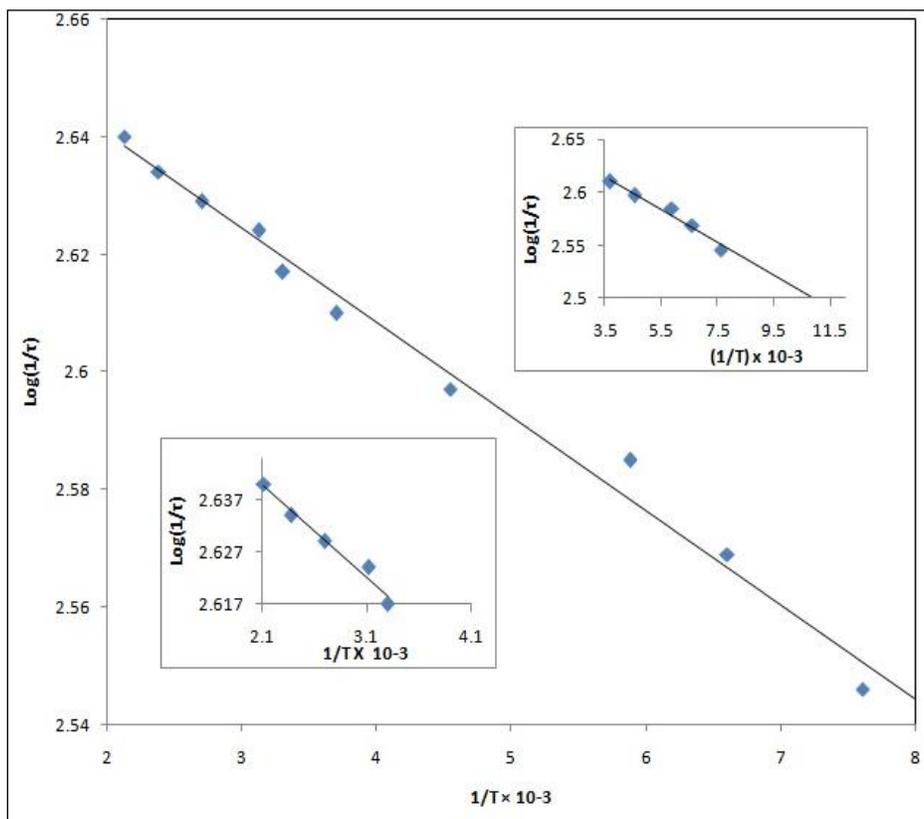


Fig. 8: Variation of $(1/T)$ against $\log(1/\tau)$.

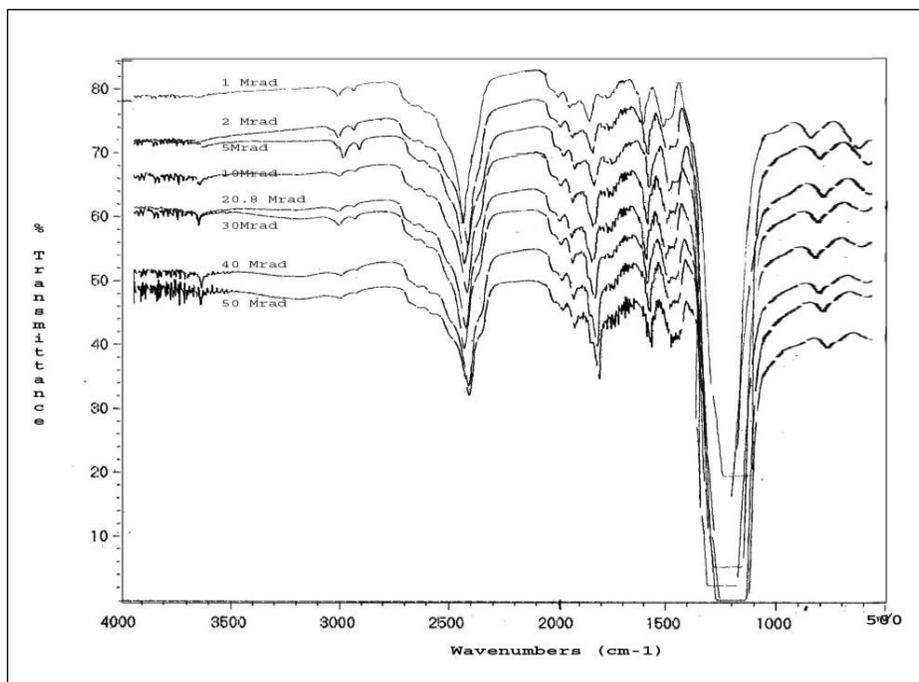


Fig. 9: FTIR Spectra of Unirradiated and Irradiated PTFE.

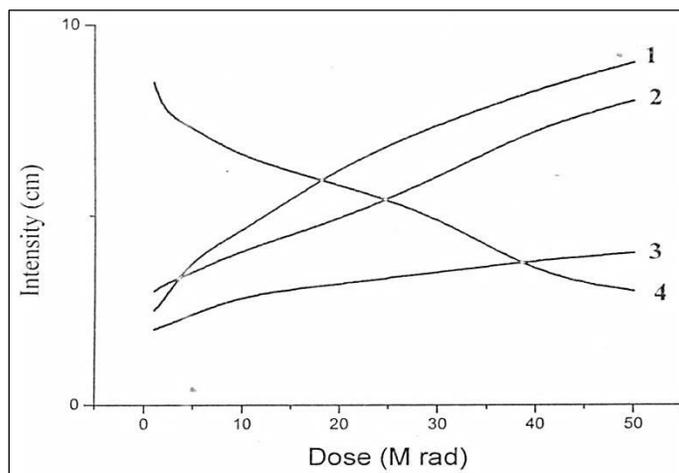


Fig. 10: Intensity Variation of FTIR Absorption Bands of Curve 1:1278 cm^{-1} , Curve 2:1721 cm^{-1} , Curve 3: 1885 cm^{-1} and Curve 4: 2811 cm^{-1} .

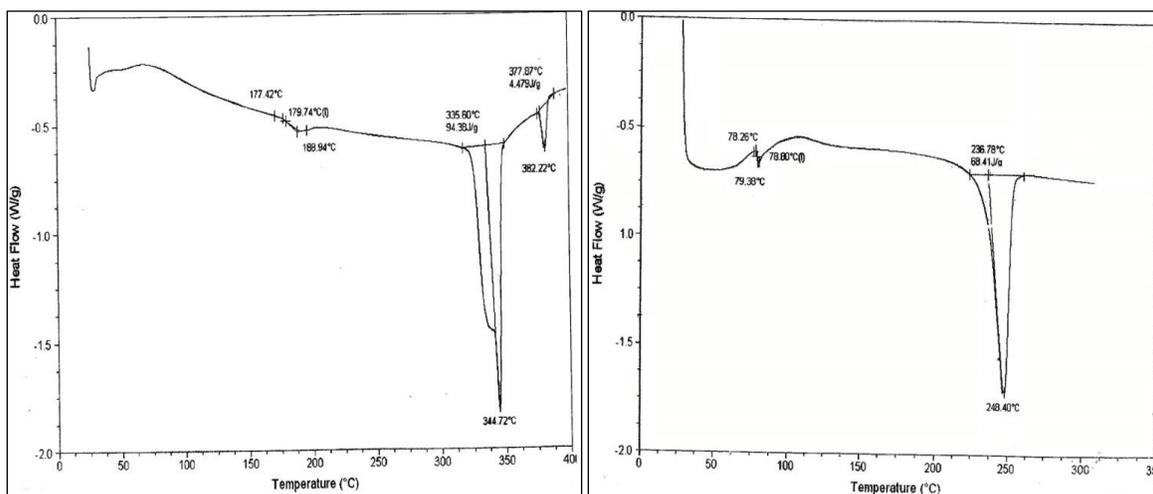


Fig. 11: DSC Thermograms of Unirradiated and Irradiated PTFE.

The FTIR spectra of unirradiated and irradiated PTFE have been recorded at various doses. The spectrum of irradiated PTFE has shown absorption bands at 2911-2891, 2816-2811, 2360-2336, 1885-1846, 1721-1675, 1555-1503, 1459-1416 and 1278-1147 cm^{-1} . Among these bands the intensity variation of the 2811 cm^{-1} absorption band decreases with the increase in radiation dose and the intensity of the 1885, 1721-1675 cm^{-1} and 1278-1147 cm^{-1} absorption bands has increased with the increase of radiation dose as shown in Figure 10. Curves 1, 2, 3 and 4 in Figure 10, represent the variation of FTIR intensity at the 1278,1721,1885 and 2811 cm^{-1} bands respectively.

Differential Scanning Calorimetry (DSC) Studies

Thermal properties of unirradiated and irradiated PTFE are investigated by recording the DSC thermograms and the same is shown as Figure 11. The Unirradiated PTFE has shown a first order transition or base line shift around 80°C and second order transition corresponding to melting transition around 250°C. On irradiation the width and position of melting peak decreased indicating the effect of irradiation.

CONCLUSIONS

The increase in end chain peroxy radicals with the radiation dose is observed for the first time

by the ESR method. The formation of $\cdot\text{CF}_2\text{-}\dot{\text{C}}\text{F-CF}_2\cdot$ radicals is small due to the high reactivity of fluorine atoms abstracts oxygen diffused in crystalline part and hence the component of a spectrum corresponding to $\sim\text{CF}_2\text{-}\dot{\text{C}}\text{F-CF}_2\cdot$ radicals is not observed with computer simulations. The end chain radical formation is increased as the formation of mid chain radicals leads to crosslinking and some of the mid chain peroxy radicals might be converted to various stable functional groups. As for electron beam irradiated PTFE, the formation of bands corresponding to -OH stretching of -COOH groups appear in gamma irradiated PTFE. The increase in radiation dose causes an increase in the formation of acidic -COF groups in PTFE.

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