

X-RAY DIFFRACTION ANALYSIS OF SOLID STATE POLYMERIC RADIATION DETECTORS TREATED WITH LOW- AND HIGH-LET ION BEAMS

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Introduction

The solid state thin polymeric films find applications in many diverse fields including their extensive use as polymeric radiation track detectors. The ion beam induced nano-scale damage in these materials has been successfully employed to improve the response of radiation detectors and to explore new dimensions in nano-technology [1]. Since the polymers primarily contain the particles of nano-aggregates which are typically nano-scale irregular crystals, crystallites or amorphous particles, the high-energy ion beam treatment of these materials enhances their applications by selectively modifying their physico-chemical properties. The swift ions interact predominantly through inelastic scattering while traversing any polymeric medium and produce excited/ionized atoms. This results into the modification of physical and chemical response of the polymers via chain-scission and cross-linking [1-4].

In the present investigation, three important polymeric detectors, viz. polyethylene terephthalate (PET), polypropylene (PP) and polytetrafluoro ethylene (PTFE) are irradiated to both low-LET (Linear Energy Transfer) electrons and high-LET proton beams. The X-ray diffraction (XRD) analysis of these irradiated samples shows significant modification in their crystalline nature, shift of peak position, peak width and intensity as compared to the unirradiated (pristine) ones. The average crystallite sizes are calculated by applying the Scherrer formula:

$$\tau = K\lambda/\beta \cos \theta \quad \dots(1)$$

where, τ : crystallite size (Å),

K : constant or shape factor which varies from 0.89-1.39, with an average = 1 for polymers,

λ : wavelength of the x-ray beam = 1.54 Å for Cu-K α ,

β : FWHM (radian),

θ : peak position, obtained from X-ray diffractograms.

Using the defined values for K and λ , the equation reduces to:

$$\tau = 1.54/\beta \cos \theta \quad \dots(2)$$

Experimental

Different stacks of PET, PP and PTFE were irradiated with 2 MeV electron (low-LET) and 62 MeV proton (high-LET) beams in the Ion Beam Laboratory of HMI, Berlin. The beams were directed perpendicularly on the surface of polymer stacks. XRD patterns were recorded using the Cu-K α ($\lambda=1.54$ Å) radiation with 8.04 keV energy from the Rigaku $\theta - 2\theta$ X-ray spectrometer. A rotating anode source and a Sodium

Iodide scintillation detector have been used. The value of 2θ ranged from 3° to 90°.

Results and discussion

Polyethylene terephthalate (PET)

The XRD-diffractogram for the pristine and irradiated PET are shown in Fig.1 and the parameters assigned to the peaks are tabulated in Table 1. The Fig.1 shows a sharp peak at $2\theta = 26.26^\circ$ denoting the crystalline nature of the polymer. The peak is shifted to $2\theta = 26.38^\circ$ and $2\theta = 25.92^\circ$ for the electron and proton irradiated samples respectively. For low-LET (electron) irradiation, the shift in peak position, decrease in intensity and reduction in average crystallite size (%) indicate a destruction of the crystal structure, which are attributed to essentially the chain scission after irradiation. The average crystallite size of the electron irradiated sample is reduced by about 22%, whereas a slight reduction (~0.1%) is observed for the proton irradiated sample. However, for high-LET (proton) irradiation, three new peaks (A, B, D) emerged at $2\theta = 9.46^\circ$, 13.18° and 28.63° after proton irradiation, which might be due to the newly formed fine polymer crystallites in the amorphous zone of PET. Fig.2 represents the perfect lattice of the pristine PET and the disturbed lattice of PET due to proton irradiation. Since the diffraction pattern depends upon the internal structure of the polymer, the observed changes in the X-ray diffraction spectra represent the growth in order of the original lamella (crystalline) structure of PET, thus, leading to a considerable increase in its crystallinity after proton irradiation.

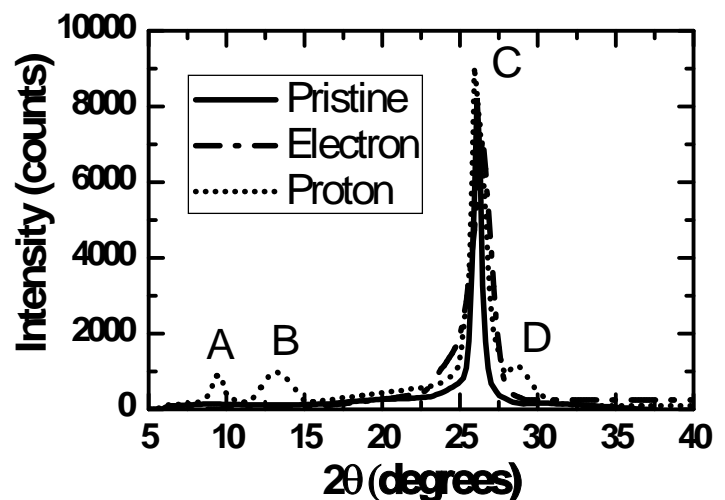


Fig.1. XRD spectra of pristine, electron- and proton-irradiated PET.

The results for PTFE and PP, as given in Table 1, will be discussed in the paper along with their

corresponding diffractograms. The details of the polymers used for this study and the XRD technique will also be presented in the paper.

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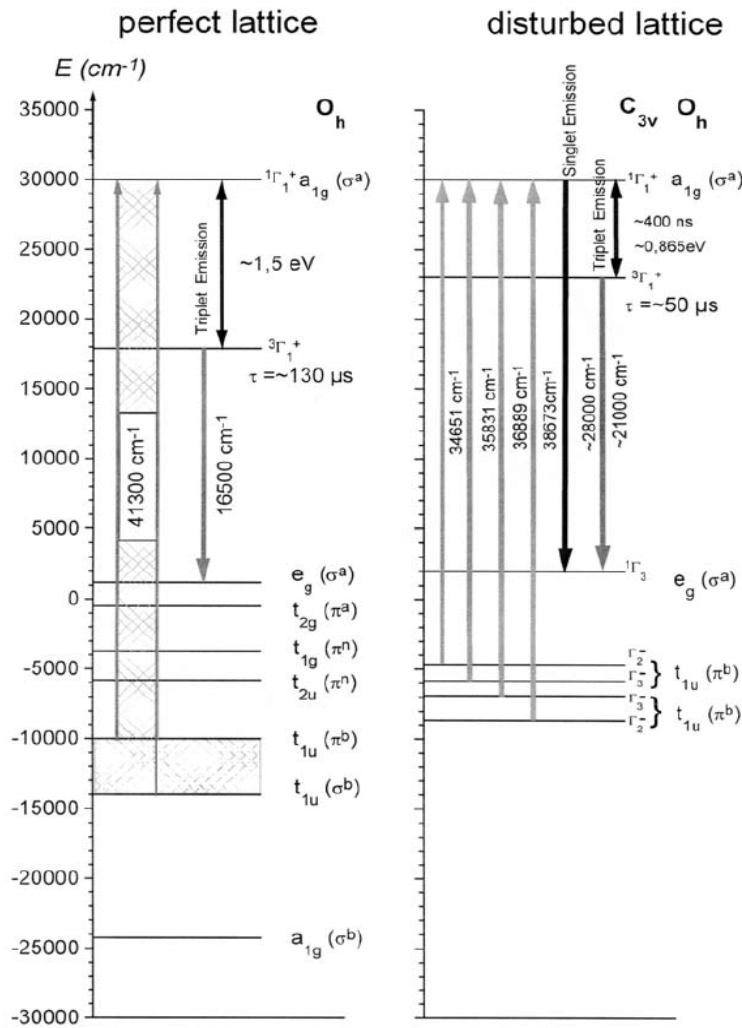


Fig.2. XRD diagram showing the perfect lattice of pristine PET and modified lattice by proton irradiation.

Conclusion

For electron irradiation: about 22% and 8% reduction in is noticed in average crystallite size of PET and PTFE respectively, but 28% increase in PP. For proton irradiation: 0.1% reduction in PET, but 29% and 49% increase in average crystallite size are observed for PTFE and PP respectively.

References

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Table 1. Position 2θ ($^\circ$), FWHM ($^\circ$) and crystallite size τ (\AA) of pristine, electron irradiated (e^-) and proton irradiated (H^+) PET, PTFE and PP samples.

Polymers	2θ ($^\circ$)	FWHM ($^\circ$)	τ (\AA)
PET			
Pristine	26.26	0.7	129.4
e^-	26.38	0.9	100.6
H^+	9.46	0.13	681.1
	13.18	3.4	26.1
	25.92	0.7	129.3
	28.63	1.2	75.9
PTFE			
Pristine	21.79	2.6	34.6
	36.00	0.4	231.9
	42.00	2.9	32.6
e^-	18.10	2.8	31.9
	37.00	0.3	310.1
	43.00	0.3	316.1
	75.00	0.5	222.4
H^+	17.90	2.0	44.7
	36.99	0.3	310.1
	42.00	0.3	325.9
	72.57	0.3	364.9
	75.54	0.3	372.1
PP			
Pristine	14.20	0.9	98.8
	18.00	0.9	99.3
	18.60	1.3	68.8
	25.60	0.5	181.0
	28.70	0.9	101.2
e^-	9.50	0.2	442.7
	14.00	0.8	111.1
	16.90	0.7	127.4
	18.60	0.7	127.7
	25.60	0.5	181.0
	28.70	0.7	130.1
H^+	9.50	0.2	442.7
	14.00	0.7	127.0
	16.90	0.6	148.7
	18.50	1.3	68.8
	23.60	2.4	37.6
	25.60	1.1	82.3
	28.60	0.3	303.5
	31.70	4.4	20.8
	35.70	0.2	463.5