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X-ray diffraction (XRD), thermogravimetric analysis (TGA) and impedance spectroscopy studies of PM-355 as a function of proton fluence

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HIGHLIGHTS

- Effects of proton fluences on PM-355 polymer.
- Structural changes are induced after proton irradiation.
- Thermal stability increases with fluence.
- Nyquist plot showed decrease in electrical resistance with proton fluence.

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ABSTRACT

This study investigates the effect on structural, thermal and electrical properties of PM-355 as a function of different Proton fluence values having energy of 5 MeV. The reference and irradiated samples were studied using X-ray diffraction (XRD), thermogravimetric analysis (TGA) and impedance spectroscopy techniques. The XRD peak intensity in the irradiated sample, abruptly increased at the initial value of fluence (1×10^{13} ions/cm^2), and then decreases at higher proton fluences. This may indicates structural changes due to irradiation. The TGA analysis indicated an increase in the value of decomposition temperature as Proton irradiation fluence increases which may be attributed to an increase in thermal stability of the PM-355. Impedance spectroscopy revealed lower resistance value of the irradiated samples as compared to the reference sample. This could be thought of the formation of free radicals as a result of Proton irradiation.

1. Introduction

Solid State Nuclear Track Detectors (SSNTDs) are used in many ways such as energetic ions detectors and biological filters (Steckenreiter et al., 1997; Srivastava et al., 2002). Among all SSNTDs, CR-39, PM-355 and PM-500 have the same chemical composition: $C_{12}H_{18}O_7$ (polycarbonate of allyldiglycol). Their differences are mainly due to procedural dealings such as: plasticizing additives, curing cycles and quenching processes (Durrani and Bull, 1987) The PM-355 is mainly used for the detection of ions as heavy as Sulphur (S^{-1}) and as a result, it is mostly used in plasma experiments and recommended as a suitable systematic tool for the laser-plasma experiment (Szydlowski, 2003; Szydlowski et al., 2009). Heavy ion beam irradiation method is widely used to study polymeric SSNTDs to improve their physical and chemical properties for use in high technology applications (Radwan, 2009; Singh and Prasher, 2005). The comparison of the responses of different SSNTDs with hydrogen, helium ion and fast proton on track study have been also reported (Sadowski et al., 1994; Szydlowski et al., 1994) Irradiating polymers result into structural defects due to many effects such as chain scission, intermolecular cross-linking, bond breaking and the formation of free radicals. The structural defects lead to change in the optical, structural, thermal and electrical properties of the polymers (Durrani, 1982; Skladnik-Sadowska et al., 2002; Buford, 2005).

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Ion-induced structural and optical properties modifications for many other SSNTDs have been studied (Chakarvarti, 2009; Adhikari and Majumdar, 2004; Laranjeira et al., 2003; Kumar et al., 2012). In the present work, we report the structural as well as the electrical and thermal properties of PM-355 as a function of proton fluence. To the best of our knowledge this study is the first of its nature to investigate the thermal, electrical properties of PM-355 irradiated with different Proton fluences.

2. Experimental details

The PM-355 samples were irradiated at Western Michigan University 6 MV Van de Graaff accelerators USA. Samples made into circles of roughly 0.5 cm^2 were exposed to Proton fluence range of (1.0×10^{13} to1.0×10^{15} ions/$cm^{2)}$ having 5 MeV energy. The ion beam was passed through a gold foil to diverge the beam before passing through an 8 mm diameter circular collimator, which defines the area of the sample to be exposed to the ion beam. Prior to the beam irradiation, an image of the beam on the sample was obtained using photographic film. After exposure the samples were left in the scattering chamber at ~10^{-6} Torr for 24 h. The experimental results and data analysis were carried out at King Saud University, Saudi Arabia.

3. Results and discussion

To study the effects of proton irradiation on PM-355, X-ray diffraction measurements were carried out on the samples with CuK_{α} radiation $_{(1.54\ Å)}$ in the range of Bragg's angle 2θ ($15 < \theta < 60°$). Kumar et al. (2012) have clearly reported in their published work that the pristine sample of PM-355 is partly Crystalline polymer which has dominant amorphous phase, Alsalhi et al. (2017) have very recently stated that the reference sample of the film in question (PM-355) is partially crystalline although it has a single abroad peak. It is believed that the reference is partially crystalline (low crystallinity.) with a dominant amorphous phase. The X-ray diffraction patterns of the reference and the irradiated PM-355 polymers are shown in Fig. 1. Interestingly after first irradiation (1×10^{13} ions/cm^2) the intensity of the peak of the reference abruptly increases which demonstrates the increase in the percentage crysallinity of the PM 355. It is also observed that the peak position shifted slightly to the larger angle up to 5.0×10^{13} ions/cm^2 and then shifted to the lower angle at higher proton fluences (1×10^{14} to 1×10^{15} ions/cm^2). The observed changes in X-ray diffraction pattern after irradiation are attributed to the disordering of the original structure of PM-355 (Tayel et al., 2015).

To study the effects of proton irradiation on the thermal stability of PM-355 polymer, TGA were performed in the temperature range from room temperature to 600 °C, at a heating rate of 5 °C min^{-1}. It has been observed that the response of the PM-355 detector decomposes into two main breakdown stages, Fig. 2.

The figure shows that onset temperature of decomposition T_o, increases with increasing irradiation fluence, indicating higher thermal stability of the PM-355 after proton irradiation which may be due to cross-linking process. The values of onset temperature of decomposition T_o, were calculated using the TGA thermograms and are given in Fig. 3 as a function of the proton fluence with the first point being that of the reference sample.

Impedance spectroscopy has been a useful technique for measuring electrical properties and indicating operational mechanisms of polymeric materials. Fig. 4 shows the plot of two measured complex impedance values, Z_{real} and Z_{imag} (Nyquist plot). It is observed that the measured resistance values at each fluence level form a single semicircle and are fluence dependent. The values shown in the figure shows that the PM-355 reference sample has the highest resistance which decreases as the fluence is increased. This may be associated with the formation of free radicals by the proton irradiation.

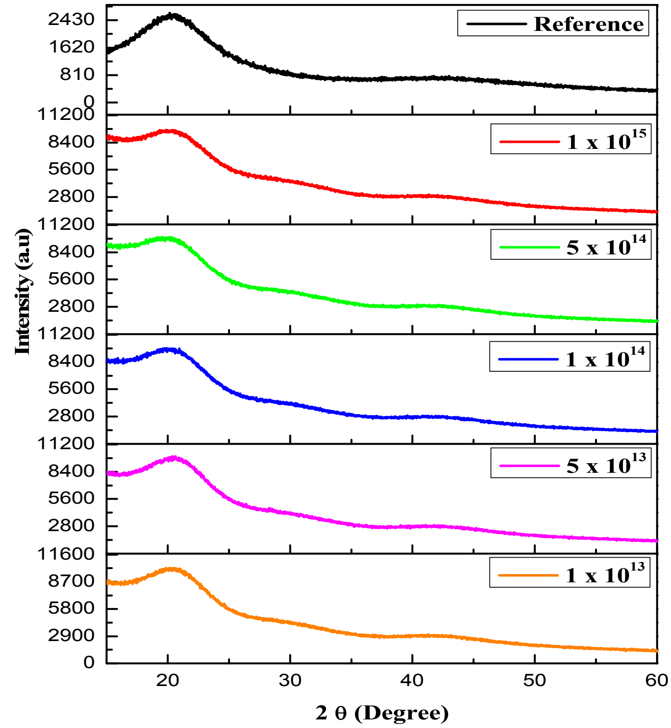

Fig. 1. XRD spectra of reference and proton irradiated PM-355 SSNTDs.

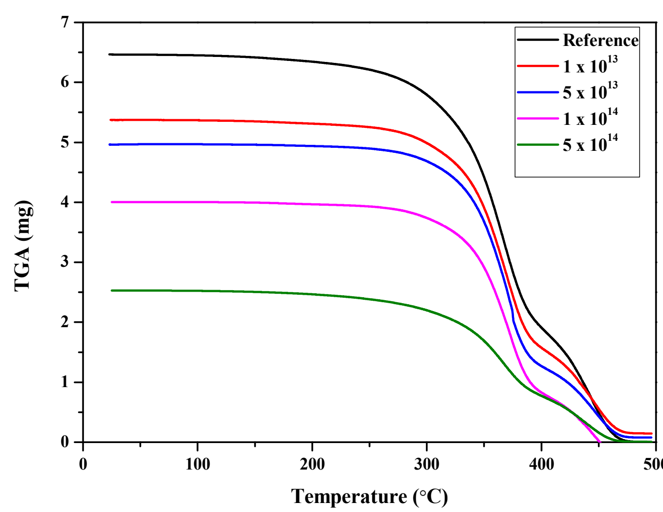

Fig. 2. Thermogravimetric analysis of PM-355 samples as a function of proton irradiation fluence.

4. Conclusions

The structural changes seen in the XRD analysis of the PM-355 Polymer as result of Proton fluences may be attributed due to the chain scission and intermolecular cross-linking. The increase in the

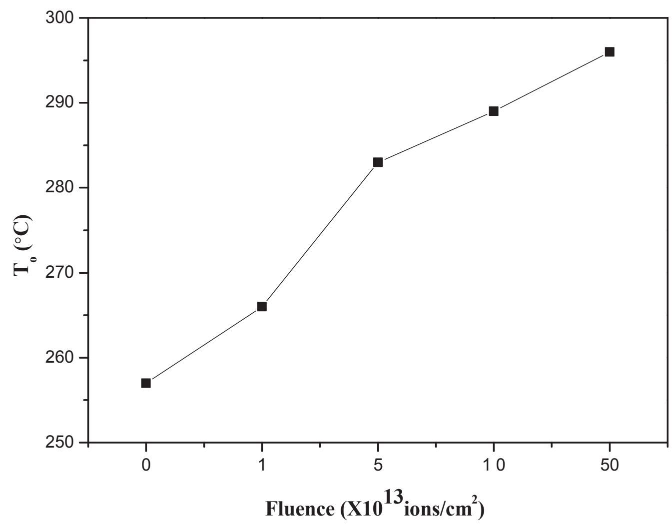

Fig. 3. Plot of decomposition temperature as a function of Proton fluences on the the of PM-355 samples (1st point is the reference sample).

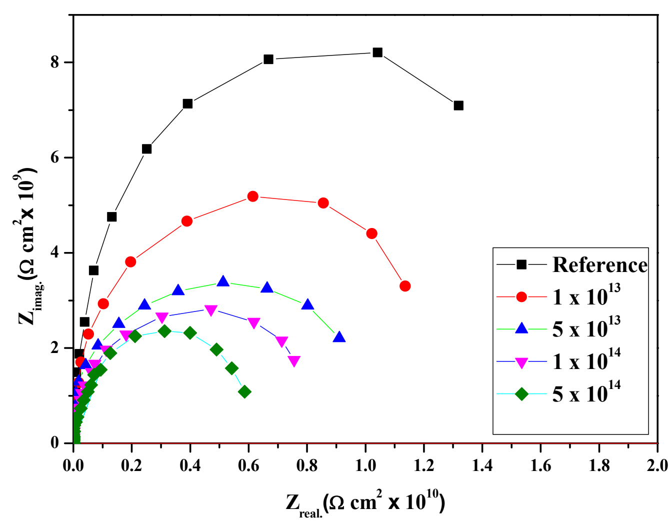

Fig. 4. Nyquist plot for the reference and proton irradiated PM-355 samples.

value of decomposition temperature as obtained by the TGA analysis indicates higher thermal stability of the PM-355. Impedance spectroscopy revealed lower resistance value of the irradiated samples as compared to the reference sample. This could be thought of the formation of free radicals as a result of Proton irradiation. The reported results may contribute to heavy ions dosimetry.

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