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C^{5+} (70 MeV) ion irradiation investigations on TSP current measurements in polyetheretherketone (PEEK)

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ABSTRACT

The thermally Stimulated Polarization Current (TSPC) is measured in pristine and irradiated samples of Polyetheretherketone (PEEK) at linear heating rate (2°C /min). The dielectric relaxation behavior of pristine and irradiated poly (ether ether ketone) (PEEK) are studied with the help of TSPC technique. The presence of ketone (>C=0) dipoles give rise to the TSPC maxima ~ 145 ^{o}C. The magnitude of TSPC peak (P_1) increases with the increase in fluence. The results obtained are compared with the results of Thermally Stimulated Depolarization Current (TSDC) technique and are in accordance with the TSDC analysis of irradiated samples of PEEK.

Keywords: *TSPC, TSDC, irradiation, poly (ether ether ketone) (PEEK), fluence.*

1. INTRODUCTION

The Thermally Stimulated Polarization Current (TSPC) is the name given to the current generated in construction of polarization i.e. Current generated when the dipolar structures orient in response to the application of the electric field [1]. The tool to investigate this current is called thermally stimulated polarization current (TSPC) technique. The TSPC is a complimentary technique of Thermally Stimulated Depolarization Current (TSDC) technique [2-6]. In TSPC, the spectra is affected by possible overlapping diffusion or injection phenomena, superposition of true conduction currents and higher noise level but it avoids overheating of the sample and eliminates the search for the optimum poling temperature as in case of TSDC [2-3]. In most of the materials, a spontaneous current (conduction current) is generated at high temperatures between the electrodes and the sample. However, the increase in current may also indicate the onset of a TSDC peak. Therefore, the important information about the temperature at which the conduction becomes significant is given by TSPC technique [3].

The present paper describes the dielectric relaxation behavior of pristine and irradiated poly (ether ether ketone) (PEEK) using the TSPC technique.

2. EXPERIMENTAL SECTION

The pristine and irradiated samples of PEEK of size 1x1 cm^2 were metalized on both sides (5 mm diameter) by silver paste and held in sample holder. The sample holder was suspended inside a properly earthed metallic chamber (Furnace). After reducing the transient current to almost negligible value at room temperature, the sample was heated at linear rate (2°C /min) with simultaneous application of constant dc electric field (EP) in series with a sensitive electrometer (Kiethley 6514) to measure the TSP currents. The slow heating rate is required to prevent temperature gradient within the sample, temperature difference between sample and surroundings as well as for obtaining a fine resolution in TSPC spectra [7].

3. RESULTS SECTION

3.1. Pristine Sample. The variation of the TSPC in pristine sample of PEEK with temperature at poling field E_P (200 kV/cm) is illustrated in Figure1. The current peak (P_1) is observed ~ 145 ^{o}C. A steady rise in current with temperature is observed beyond 160 ^{o}C. This result is in accordance with the non-isothermal charging mechanism of a polymer in which the poling current is assumed to have two components behaving differently as a function of temperature. The dipolar orientation as a transient process which results in the formation of a peak and the conduction current due to the motion of equilibrium charge carriers increasing continuously with temperature. In PEEK, the ketone (>C=0) dipoles linked with the main chain contribute to the polarization of the polymer. The presence of ketone (>C=0) dipoles give rise to the TSPC maxima ~ 145 ^{o}C which is also confirmed by TSDC spectra [8]. Therefore, the presence of P_1 peak is attributed to the dipolar nature of ketone (>C=0) group.

Vanderschueren and Gasiot [9] reported that the TSPC will be characterized by peaks roughly similar to those appearing in TSDC measurements but they will obviously appear in a reverse direction, at least at the beginning of the spectrum. Simmons and Taylor [10] and Devaux and Schott [11] proposed a trapping model according to which, the TSPC peaks without reversal and similar to TSDC peaks can also be observed, provided at least one of the electrodes is non-ohmic. The magnitude of TSPC peak is large as compared to the TSDC peak at same condition of poling field (E_P) which is attributed to superposition of true conduction

currents and higher noise level in TSPC spectra and buildup during the polarizing period of ionic space charge which results in reducing the forming field [9].

3.2. Irradiated Sample. The variation of the TSPC of C^{5+} swift heavy ion irradiated samples of PEEK with temperature at different fluences ($1x10^{10}$ ions/cm^2, $1x10^{11}$ ions/cm^2 , $1x10^{13}$ ions/cm^2) and poling field E_P (200 kV/cm) is illustrated in Figures 2- 4. In irradiated samples of PEEK, the magnitude of TSPC peak (P_1) increases as compared to pristine sample of PEEK. Also, the magnitude and broadening of the peak increase with the increase in fluence.

The increase in magnitude of TSPC peak (P_1) in irradiated samples of PEEK as compared to pristine samples of PEEK is attributed to two mechanisms: one is the increase in dipole concentration within the polymer and another is increase in either electronic or ionic mobile charge concentration. The ion irradiation leads to degradation of polymeric chains, chemical bond cleavage and creation of free radicals [12]. Subsequent chemical reactions after irradiation cause creation of excessive double bonds, increasing the dipolar nature and cross-linked structures [13-14]. The number of dipoles increases with the increase in fluence and hence the magnitude of TSPC peak (P_1) increases with the increase in fluence.

The broadening of peak is a direct function of dipole concentration and distribution in structural ordering parameters which determine the free-volume relaxations required by the main-chain rearrangements [2]. Irradiation of polymers produces excited states, ions and free radicals which result in rearrangements and formation of new bonds. The effects of these reactions are the formation of oxidized products, grafts, scission of main chain (degradation) or cross-linking. These effects are more pronounced with the increase in fluence. Therefore, the broadening of TSPC peak (P_1) increases with the increase in fluence. These results are in accordance with the TSDC analysis of irradiated PEEK samples [15].

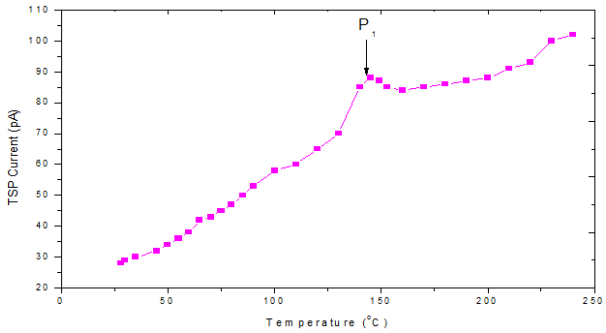

Figure 1. Variation of the TSPC of Pristine PEEK with temperature at poling field E_P = 200 kV/cm.

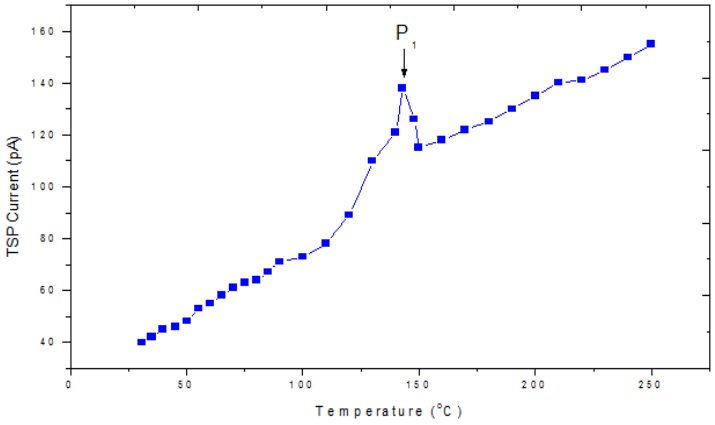

Figure 2. Variation of the TSP current of C^{5+} ion irradiated sample of PEEK with temperature at fluence $1x10^{10}$ ions/cm^2 and poling field (E_P) = 200 kV/cm.

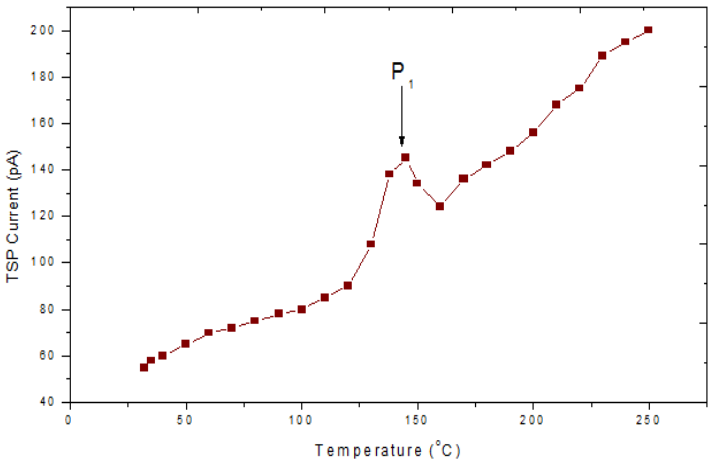

Figure 3. Variation of TSP current of C^{5+} ion irradiated sample of PEEK with temperature at fluence $1x10^{11}$ ions/cm^2 and poling field (E_P) = 200 kV/cm.

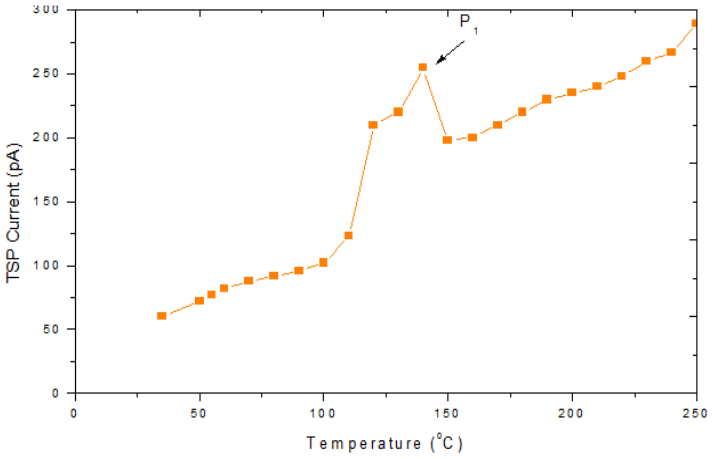

Figure 4. Variation of the TSP current of C^{5+} ion irradiated sample of PEEK with temperature at fluence $1x10^{13}$ ions/cm^2 and poling field (E_P) = 200 kV/cm.

4. CONCLUSIONS

The TSPC is a complimentary technique of TSDC technique. The comparison of TSPC and TSDC spectra confirms the nature of the peak. The presence of ketone (>C=0) dipoles in PEEK give rise to TSP current maxima ~ 145 ^oC which is in accordance with the TSDC analysis of PEEK. The TSPC peak is obtained without reversal and is similar to TSDC peak but with enhanced magnitude. The number of dipoles increases with the increase in fluence and hence the magnitude of TSPC peak (P_1) increases with the increase in fluence. The broadening of TSPC peak (P_1) increases with the increase in fluence. These results are in accordance with the TSDC analysis of irradiated samples of PEEK.

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