

## Study of Doped Blend Film and Its Application as a Sensor Material

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### ABSTRACT

In this paper the results of thermally stimulated discharge current (TSDC) of thermoelectret of cinnamic acid doped polyvinyl chloride (PVC) polymethyl methacrylate (PMMA) blend film are presented. TSDC has been carried out in a temperature range 303K to 383K and at five different polarising fields. Results are discussed on the basis of space charge polarisation. these are further extended to the probable used as a sensor material.

**Keywords :** Polyvinylchloride (PVC), Polymethyl-methacrylate (PMMA), TSDC, Thermoelectret.

### I. INTRODUCTION

TSDC of polymers has become a widely used experimental technique for the investigation of various material parameters such as charge storage properties, determination of mean depth of the internal charge, activation energies of traps and trap structure of the material. Suzuki et al [1] studied characterisation of polymers with a considerable accuracy by using TSDC technique. Kellar et al [2] studied polyblends of PS and PMMA employing TSDC. The polyblends of PMMA and polyvinyl pyrrolidone (PVP) have been studied by Khare et al [3] Negau and Negau [4] obtained new results in thermally stimulated discharge current (TSDC) peak above room temperature. Sangawar [5] measured D.C. electrical conductivity of doped electrets of PS and PMMA. Belsare et al [6] studied iodine doped polyblends of PS and PMMA using TSDC. Burghate et al [7] measured thermally stimulated discharge

current (TSDC) and dielectric constant of semiconducting glasses.

The availability of new measuring techniques such as thermally stimulated discharge current [8] has freshened interest in achieving better understanding and application of the phenomena. The basic principle of this technique is to study the charge decay by heating the electret at a constant rate, and then to attempt to make use of these results in the design of a thermal sensor.

### II. METHODS AND MATERIAL

#### 2.1 Preparation of sample:

The polyvinyl chloride (PVC) of standard grade product supplied by Polychem Industries Mumbai and polymethylmethacrylate (PMMA) supplied by Dental Product of India Ltd., Mumbai were used for the study. The two polymers PVC (1.5g) and PMMA

(0.5g) were taken in the ratio of 3:1 by weight. The 1.5g of PVC in 20ml of tetrahydrofuran (THF) and 0.5g of PMMA in 10ml tetrahydrofuran were dissolved separately. After allowing them to dissolve completely the two solutions were mixed together. To prepare the cinnamic acid doped blend films, the cinnamic acid was taken in the percentage

weights Viz. 0.2%, 0.4%, 0.6%, 0.8% and 1.0%. Each was dissolved in 5ml of THF to produce cinnamic acid solution. The solution was later mixed with uniform solution of PVC and PMMA. The total volume of solvent was kept constant 35ml. The solution was heated at constant temperature 333K for the two hours to allow polymers to dissolve completely to yield clear solution. A glass plate (15cm x 15cm) thoroughly cleaned with hot water and then with acetone was used as a substrate.

To achieve perfect leveling and uniformity in the thickness of the film, the films were prepared on a thoroughly cleaned optically plane glass plate kept floating in a pool of mercury. The whole assembly was placed in dust free chamber maintained at constant temperature (313K). In this way the film were prepared by isothermal evaporation technique [9,10]. The film was subjected to 12hrs heating at constant temperature 323K and another 12hrs at room temperature to remove the traces of solvent. Finally, the film was removed from the glass plate. It was cut into small pieces of suitable size, which were washed with ethyl alcohol to remove the surface impurities.

## 2.2 Thickness measurement:

For greater accuracy and resolution, a compound microscope in conjunction with an occlusometer, which gives least count 13 $\mu$ m and 3.3 $\mu$ m at the magnification of 1:10 and 1:100 respectively was used. A small section of the sample was taken and mounted vertically to get a clear sectional view of the thickness. The films used for the present study are thickness about 80 $\mu$ m.

## 2.3 Electrode coating:

The electrode coating on the film of measured thickness was done by using the quick drying silver paste [11] supplied by Eltecks Corporation, Bangalore. A mask of circular aperture of 2.5cm diameter was used while coating to ensure uniformity in size of coated silver electrode.

## 2.4 Electret preparation:

The sample was mounted into the sample holder. The polarising field  $E_p$  was maintained for 30min between two faces at room temperature (303K). The electric field across the sample was removed and the sample was short circuited for 15min to remove the stray charges by wrapping the sample in an aluminum foil. The electrets were prepared at different polarising fields i.e.  $E_p = 37.5\text{ kV/m}$ ,  $75\text{ kV/m}$ ,  $112.5\text{ kV/m}$ ,  $150\text{ kV/m}$ ,  $187.5\text{ kV/m}$  respectively.

## 2.5 Measurement of thermally stimulated discharge current (TSDC):

After electret formation the sample holder assembly was placed in high temperature furnace. The sample was short circuited through a sensitive picoammeter (Model DPA III scientific equipment, Roorkee, having accuracy + 1 pA) for the measurement of discharge current. Then the sample was heated at a uniform rate (1.25 $^{\circ}$ C/min) from 303K to 383K. The discharge current was measured for sample. The temperature was recorded by mercury thermometer having an accuracy + 1K.

## III. RESULTS AND DISCUSSION

TSDC thermograms of PVC-PMMA polyblend films doped with cinnamic acid are presented in the form of graphs figs (1 to 5) between discharge current versus

temperature at various poling fields and constant poling temperature of 333K.

The nature of the graphs is similar with following characteristics features.

1) In almost all cases, initially a large current is observed to decay at a faster rate over a certain range of temperature. This can be explained to be due to freeing of charge carriers in shallow traps near the surface region. Subsequently these must be moving towards the injecting electrodes resulting in the initial decay.

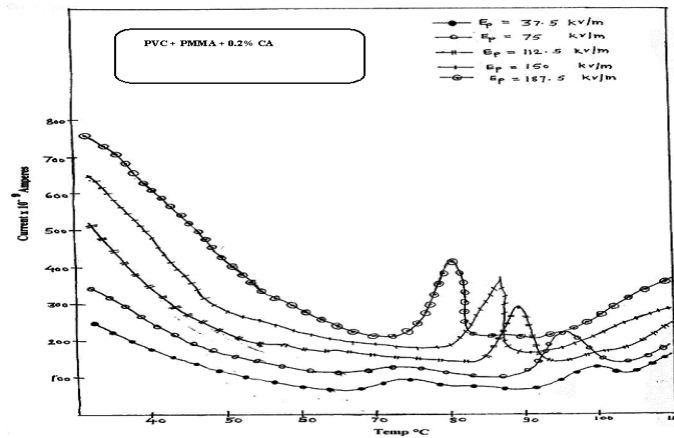


Figure 1: TSDC for sample 1 (current Vs temperature plot.)

2) In general, a single current peak is observed namely the current peak in the range of about 80°C to 95°C. The height of the peak increases with polarising field at each dopant concentration and also increases with concentration of dopant cinnamic acid.

The polarisation in polymer arises on account of orientation of dipole and trapping of charge carriers in different traps [8, 12, 13]. The

dielectric relaxation in PMMA is a high temperature – relaxation near its  $T_g$  between about 90°C to 98°C. That in PVC near its  $T_g = 87^\circ\text{C}$ . Hence in the polyblend the current peak should occur in this vicinity. Since the present study has been done at higher temperature the low temperature peak near

about 30°C, associated with disorientation of polar side groups has not been observed.

The addition of PVC in PMMA produces a heterogeneous structure and plasticisation effect, which is further assisted by dopant cinnamic acid. Plasticization causes loosening of structure and hence charge carriers injected in the bulk may pile up at the phases boundaries, providing greater mobility to the charge carriers resulting in increase of peak current.

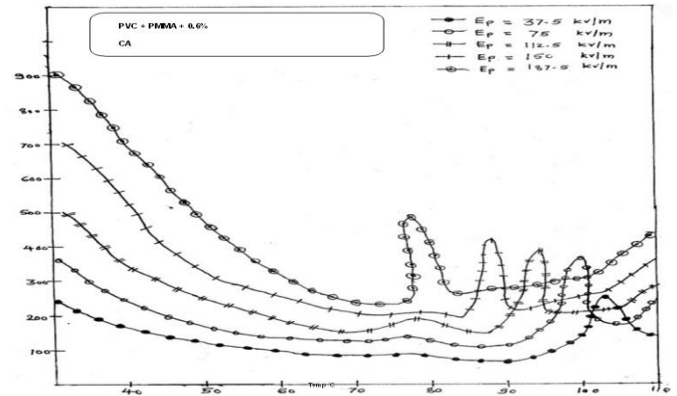


Figure 2 : TSDC for sample 2 (current Vs temperature plot.)

3) A linear relation between peak current and polarisation field is observed i.e. the magnitude of peak currents increases with the magnitude of the polarising field  $E_p$ . The higher value of  $E_p$  produces ionization of large number of impurities centres there by enhancing the current.

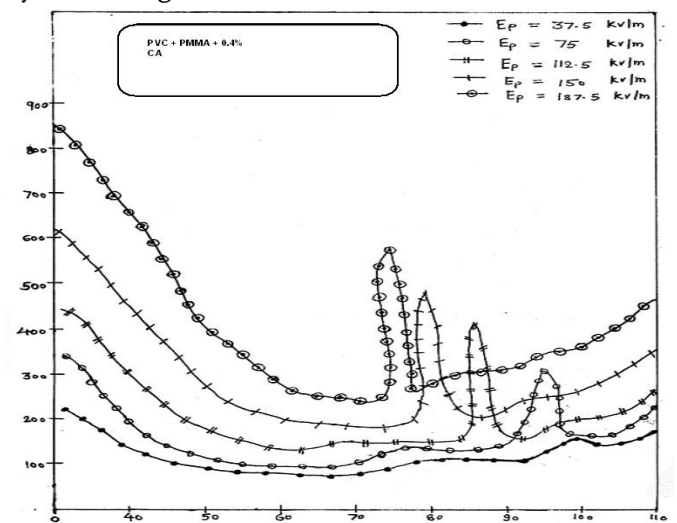


Figure 3 : TSDC for sample 3 (current Vs temperature plot.)

4) Further as the dopant concentration increases, the magnitude of peak currents also increases. This is evident because dopant introduces impurity centre, ionization of which helps to make available large number of carriers to enhance current.

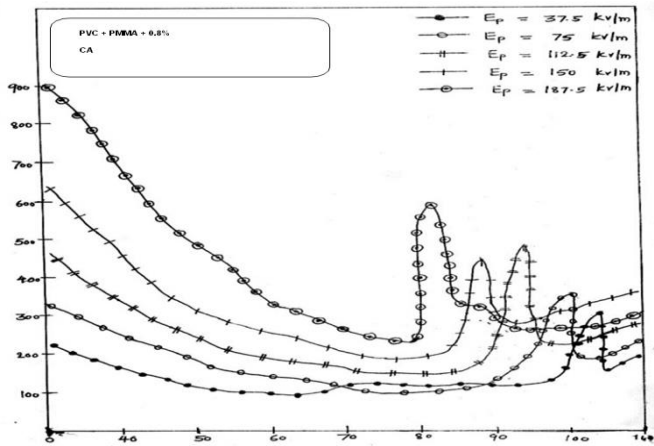


Figure 4 : TSDC for sample 4 (current Vs temperature plot.)

The TSDC of the polyblend thermoelectret, all show (Figs. 1 to 5) a current peak in the range of temperatures 80 to 95°C, in all samples. The sample with 1% cinnamic acid doping (Fig. 5) shows maximum peak current. Thus, the current response to temperature, of this electret, in this temperature range will be extremely helpful in the design of a thermoelectret as a temperature sensor. This, all the while is more important, because most of the sensors available commercially operate

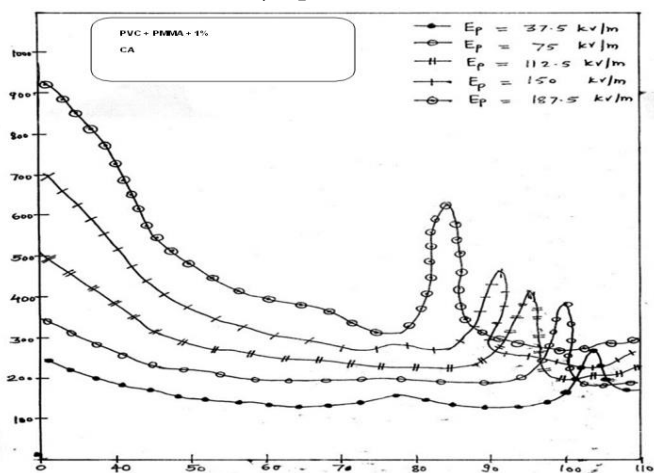


Figure 5 : TSDC for sample 5 (current Vs temperature plot.)

[14] at elevated temperature upto 300°C. Some aspects like electrical conduction in undoped films have already been reported [15]. In addition to this the present TSDC study is being presented here. Mishra et al [14] have reported semiconducting polymeric thin film sensor for detection of toxic gases the selection of the material in proper proportion and optimization in the design and fabrication are very likely to yield a thermal sensor. This can further be extended to the detection of toxic acids and also to control environmental pollution.

#### IV. CONCLUSION

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