Volume 4, Issue3 (2016) 538-541 ISSN 2347 - 3258
International Journal of Advance Research and Innovation

Analysis of Specific Charge Storage Properties in Polymer Films with Halogen Treatment

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Article Info
Article history:
Received 02 April 2016
Received in revised form
20 May 2016
Accepted 28 May 2016
Available online 15 October 2016

Keywords
Crystalline structure**;** Polypropylene film; thermally stimulated; thermal current

Abstract
The electrical conduction in thick Polypropylene film with different crystalline has been studied different temperatures & at different potential gradients. In different cases the charge storage properties of a super charged BOPP film & un-oriented PP films have been studied.
Now a special study on TOPP is taken. An initial study has been made of radiation induced oxidation of Polypropylene (PP) in both its additive-free form and one containing discrete amounts of stabilizer. Samples of PP were irradiated in a $^{60}Co_{\gamma}$ source to various doses .The field dependence of current shows that the conduction mechanism in this polymer is ion hopping. In these films the thermally stimulated charge decay and open and short circuit thermal current were measured.

1. Introduction

Electrical conduction in polymeric materials is governed by generation and transport of charge carrier. Since carrier transport is influenced by the polymeric structure, electrical conduction in various polymers has been studied in relation to their structures by a number of authors.
Polymer electrets with good charge storage stability are important device applications charge trapping plays a key role in charge storage processes in polymers. The charge trapping can occur at the crystalline amorphous boundaries at chain folding, in cavities and at impurities. Bopp polymer films have better physical properties in the two directions of the film as compared to un-oriented and uniaxial oriented film. It is because of the distribution of chain axis orientation in the machine, transverse & some angled direction.
The oriented stretching of crystalline polymers is a widely used method for the preparation of super strong and high module polymer foils, fibres and other materials with useful properties. Because of that many investigations are directed to the study of the structural transformations of crystalline polymer systems at a uniaxial stretching. The oriented polypropylene films are extensively used as dielectrics in capacitor designs due to their good electrical &mechanical properties. HV capacitors are almost importance in high power traction motors, such as in electrical locomotives. During the last decades considerable progress has been made in capacitor technology impregnates avoiding air enclosures in the capacitor rolls were optimized. The effect of radiation on electrical conduction and mechanical properties of BOPP has been reported.
The storage of charge has been studied in many polymers because of its applications in devices such as microphones, gas filters, under water transducers, radiation dosimeters and piezoelectric & pyro electric detector. A good attention has been gives to the charge storage properties of BOPP. In this paper, studies of charge rigidity quantity properties in most commonly used BOPP &TOPP films are shown special techniques TSCD &TSC were used to obtain useful information about the mechanism of charge rigidity, stability & distribution in BOPP & in some other oriented polymer films, X-ray and differential scanning calorimetric scans of BOPP were analysed for structural information.

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2. Experimental

A sample film of nearly 55μm thickness was obtained by National Physical Laboratory New Delhi. These films were extruded and oriented in machine direction (elongation 250%),in transverse direction (elongation 150%) & at angle 45˚ (125%). The thickness of the unoriented polypropylene film was 55μm. A square sample of side 3 cm were cut from a roll of the film. It was chemically cleaned ultrasonically with aceton and dried in super atmosphere. The sample film were heated in an oven at various temps for about 2-2.5 hours & then cooled rapidly at room temperature. Multi Aluminium electrodes of thickness 30nm & area 3.14 cm^2 were vacuum deposited on surface (back electrode) of the sample.
These samples were negatively corona charged through nano matalized surface for 150 sec at slightly more than room temperature and at elevated temperature (30 to 85˚C) the aluminium side grounded. The corona voltage was maintained at -13 KV and the surface potential was controlled with the help of a grid kept at a negative potential and held ~2.5mm over the surface of the sample for post irradiation sample were irradiated in vacuum at the temperature of liquid nitrogen and then exposed to the room air after samples had received on appropriate dose.
For the open TSC measurement the upper non conducting electrode was at a distance 2.5 mm from the sample short circuit TSC measurement with both the electric shorted. TSC (open and short circuit) and TSCD measurement were carried out in the temperature range 32˚C to 189˚C at a constant heating role 0.055˚C/sec, using a specially designed appropriate digital temperature controller. A monroeisoprobe electrostatic voltmeter model 173-1 with

probe was used to measure the surface potential. The TSC & TSCD were recorded on a strip chart recorder.

The X-ray scans were obtained with a diffractometer, using Ni filter Cuk$_\alpha$ radiation of wavelength 0.155nm. Differential Scanning Calorimetry (DSC) measurements were made at 0.155˚C/sec using a thermal analyser. The weight of the sample was 13 mg.

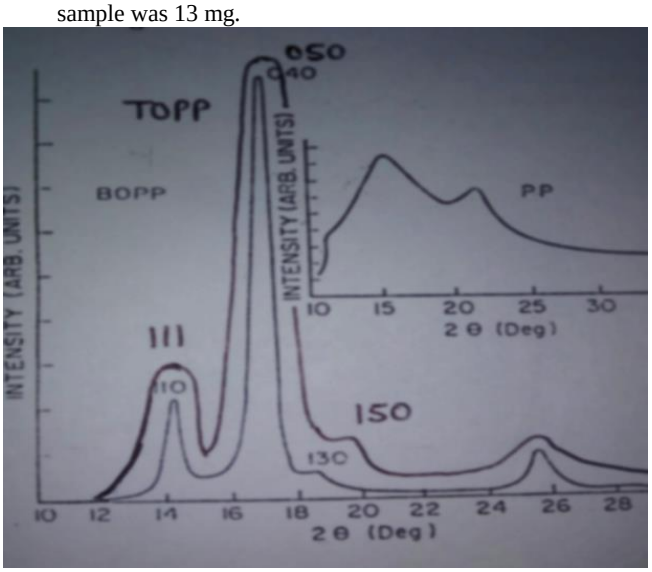

Fig.1:- X-ray Scan for BOPP & TOPP

3. Result & Discussion

Figure 1 insect shows the X ray scans for BOPP &TOPP& PP films. X-rays scans of BOPP indicate monoclinic α from crystallites with same peaks absent. The absence of some of peaks could be due to orientation of the crystalline phase. For unoriented PP (figure 1 in set) the peaks are broader which indicates a higher amorphous content than BOPP. Broadening of the peaks could arise from the defects between crystallites and/or imperfections in ordered phase. The inset of figure 1 shows a typical diffraction pattern of PP with a sematic structure. The crystallinity was calculated using Hinrichsen's method where the true diffraction intensity is taken to be a above curve drawn along the above of peaks to the total area crystallinity values of 73% of 69% and 37% where obtained TOPP, BOPP & PP respectively. The crytallite size was calculated using scherrer's relation $t=0.9\lambda/\beta Cos\theta hkl$

Where t is the crystallite thickness that is perpendicular to the hklplane,λ the X-ray wavelegth, β the peak width in radians and θ_{hkl} the Brgg's angle. The average cystallite size for TOPP & BOPP corresponding to 050 & 050 planes was 17nm.

Fig. 2 shows a differential scanning calorimetric curve for BOPP & TOPP. It has a sharp endothermic peak at 182˚C and a small peak at 165˚C. The sharp peak can be attributed to melting of α crystallites of BOPP & TOPP and the small peak to the oriented amorphous region as suggested by Samuels [1-2]

Figure 3 shows the TSCD of TOPP (Curve a), BOPP (Curve b) and PP (Curve c) films negatively charged to an initial surface potential of 390 V at room temperature TSCD half value temperature ($T_{0.5}$) of 116˚C of TOPP, 120˚ for BOPP & 98˚C PP was obtained. The higher crystallinity (69.5%) of TOPP& (67.6%) of BOPP films. An increase in $T_{0.5}$ with crytallinity has been observed in polyethylene & polypropylene & laminated paper by other authors [1]. However it is not certain at present to what extent the difference in crystalline structure between TOPP, BOPP and PP contributes to the observed increase in the specific charge stability of TOPP& BOPP. Since $T_{0.5}$ is measured to the charge storage stability in polymers, it follows that TOPP shows better charge strong hence BOPP & BOPP shows better charge storage then PP. Hence TOPP was chosen for further studies. For TOPP the dependence of TSCD on initial surface potential was also investigated the $T_{0.5}$ was found to be low at high surface potential. At 1200 V, $T_{0.5}$ was 89˚C which is 30˚C lower than 380V (curve a fig.3) showing the decrease in charge storage stability with surface potential.

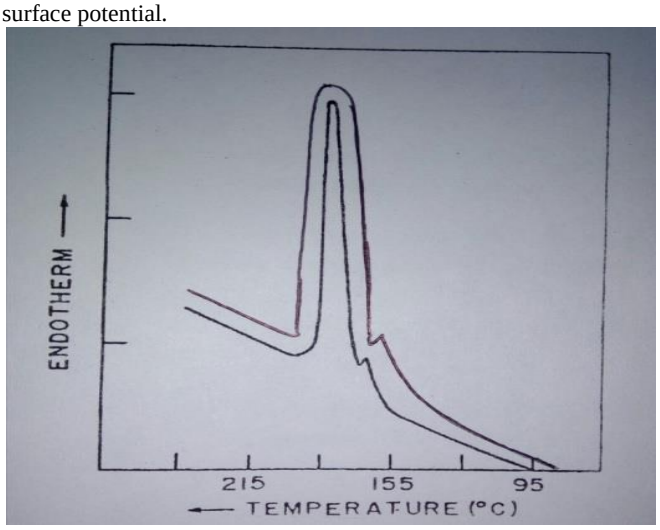

Fig.2-Differential Scanning Calorimeter for BOPP &TOPP

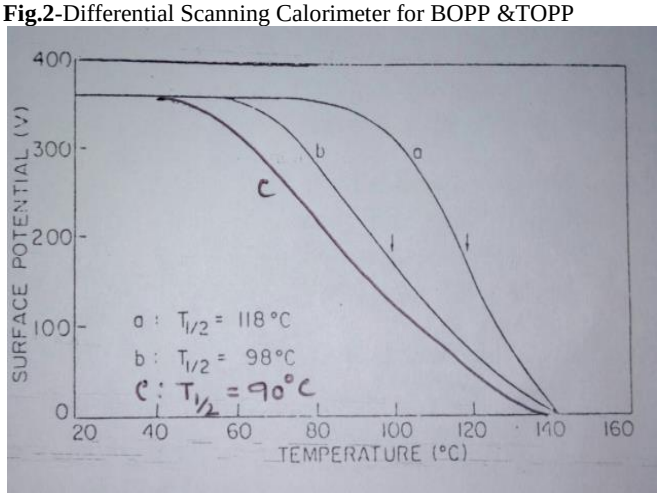

Fig 3:-TSDP of TOPP, BOPP& PP. Samples negatively corona charged at room temperature.

Figure 4 shows typical short circuit TSC for TOPP& BOPP film negatively charged to initial surface potential of 370V (curve a) at 500 V (curve b) & 850V (curve c). Curve a of fig. 4 gave two different peaks at 73˚C & 144˚C. The TSC observed at 850V (curve b) showed for peaks at 41˚C, 73˚C, 144˚C & 180˚C. The current reversal will depend on the charge penetration and spatial distribution of charge within the distribution there lies a zero field plane. Charges trapped above the zero field plane will come out towards the top

electrode whereas the charges below this plane will move towards thSe lower electrode. The TSC is the algebraic sum of two opposite current components and current reversal can occur. The reversed peak position shifts towards lower temperature (144˚C at 500V and 180˚Cat 850 V) with increased field. This peak can be attributed to the space charge in deep traps. On the other hand, the lower temperature peaks may be due to charges trapping at shallow traps.

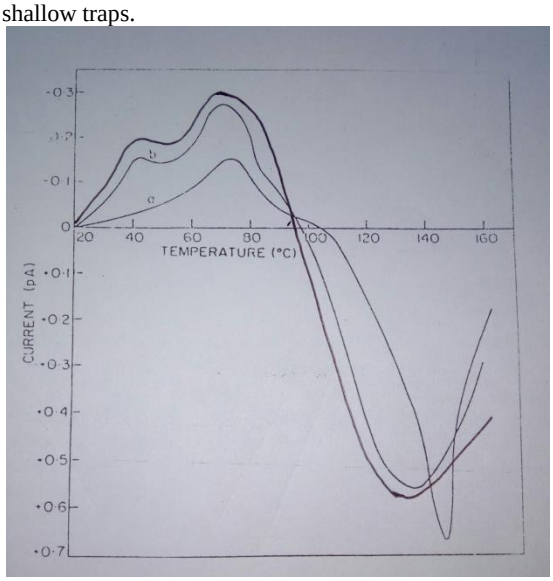

Fig.4-Three Typical Short Circuit TSCCurves Charged to initial surface potentials of -300V(curve a),-800V(curve b)&1200 (curve c)

For TSC measurement bias an electrode metal is used because of its small thermal effect on the sample films during the vacuum deposition. A sample film with circular bi electrode which was clamped between two Teflon rings, was corona charged surface potential of 220-234V at room temperature for 3 min. open circuit. TSCS of these charged films were recorded from room temperature to 170˚C at a heating rate of 3.1-3.3˚C/min.

Impurities, such an antioxidants, residual catalysts, residual and process residues have been shown to present in commercial polypropylene film [ ] such types of impurities can lie for the amorphous phase and/or at interface of crystalline and amorphous regions and can act as carrier traps. It has been shown that impurities (or additives) act as carrier taps in polyethylene (12, 13). The impurities (or additives) pay an important role in improving conducting polymers. Halogen (I_2, Br_2 &F_2) in TOPP, BOPP & PP assists electrons & holes to jump from a chain
to a neighboring. One which results in an increase in electrons & hole mobility.

PP antioxidant doped & BOPP. Impurity effects seems complicated for instance, one of the antistatic agents which are the source of ions has been pointed out to from electric localized level in TOPP & BOPP and to enhance electron injection via localized levels from cathode. Some impurities affects both ionic & electronic conduction.

The enhancement of conducting & TSC have been reported antistatic agent doped

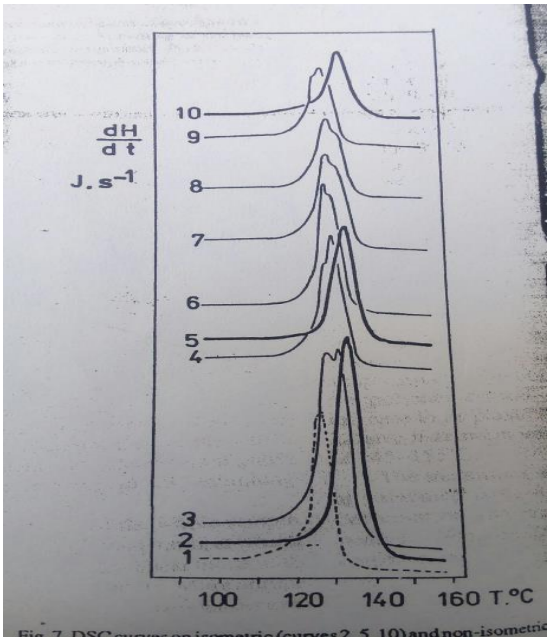

Fig 4A:-DSC curves on isometric (2, 5, 10) & non isomeric (curves 3, 4, 6-9)

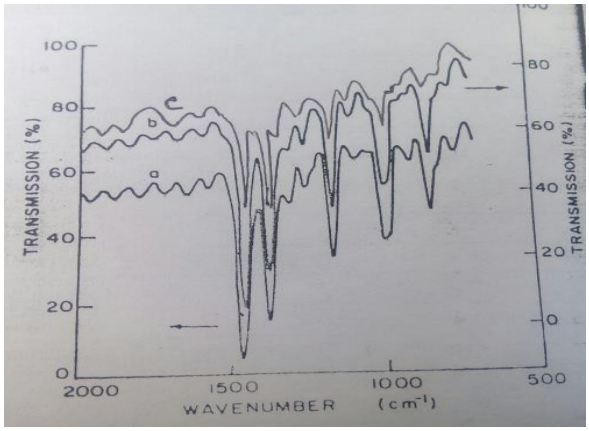

Fig4B:-infrared of virgin (spectra a), hexane treated (spectra b) BOPP &TOPP

The peak at 72 ˚C may be attributed to impurity traps. It is known the triaxially oriented films & Biaxially oriented films contain that tie molecules in amorphous region which are mobile above 37.9˚C . The DSC measurements (Fig.2) reveal. The presence of oriented amorphous region, so it is reasonable to assume the presences that tie molecules in TOPP & BOPP. The peak at 40˚C may arisefrom traps at that tie molecules in amorphous region.

Figure 5 shows typical short of circuit TSC for TOPP & BOPP film negatively charged at 450 V and at three elevated temperatures 40˚, 60˚ & 70 ˚C (curve a, b, c) The height of lower temperature peak decreased with increase in the poling temperature. This may be due to charge in spatial distribution of charges in the sample caused by less trpping at high temperature. The reversed peak height increases with poling temperature existence of space charge and detrapping of space charges gives size to these peaks.

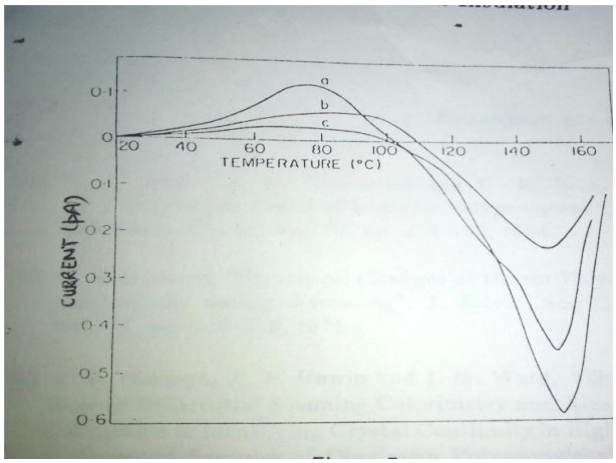

Fig 5:-Typical short circuit TSC curves for BOPP carona charged to initial surface potential of -400V and at three elevated temperature of 40,60& 70˚C (curve a,b,c)

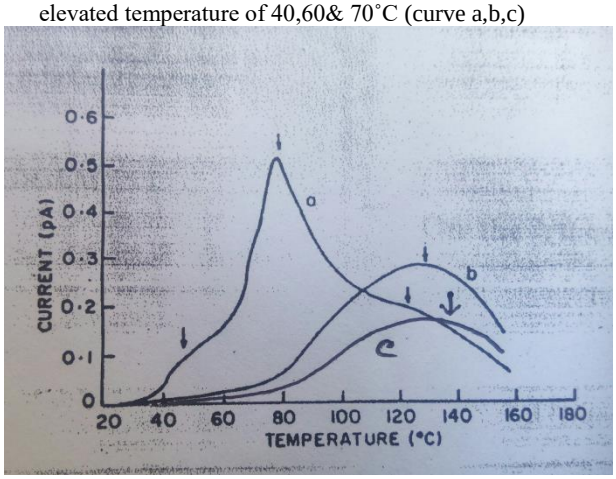

Fig 6:-Open –circuit TSC of BOPP carona charged to initial surface potentials of -400,-800,-1000 &-1200v (curve a, b, c &d respectively) at room temperature.

Figure 6 shows the open circuit TSC of TOPP & BOPP films negatively corona charged to initial surface potential of 450, 850, 1100 & 1250 V (curve a to d respectively). These curves are derivatives of their corresponding TSCD. So that their peak positions correspond to TSCD in flection point. Thus their peak temperature are close to $T_{0.5}$. The open circuit TSC spectra exhibit four peaks at temperature around 47˚C 85˚C & 122˚C & 180˚C.The shallow peak near 47˚C & the large peak at 85˚C in all possibility are the same peak were due to a dipolar mechanism, then a reversal in the open circuit TSC would be observe . It shows the absence of hetrocharge in the sample. From the potential heating method the activation energies for 47˚ C & 85˚C peaks were found to be 0.62 & 1.31eV respectively.

Figure 7 indicates the higher temperature peak is attributed to a space change mechanism. The peak at 85˚C gives more with initial surface voltage them this space charge (122˚ C).The ratio of these peak currents Ip (85˚C) / Ip (122˚C) increases with initial surface potential. The TSCD measurement of high initial surface potential gave a low value of $T_{0.5}$ therefore showing the low charge storage stability for higher surface potential and hence for a good charge storage stability, the ratio of peak currents should be small.

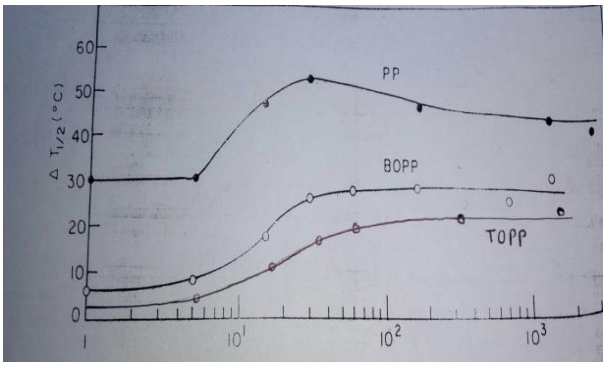

Fig7:-Increase in half value temperature vs hexane treatment time initial surface potential=400V

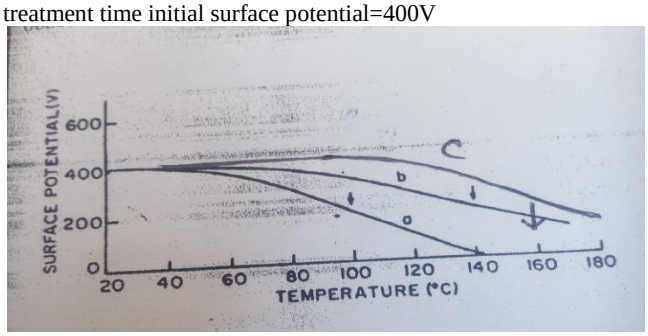

Fig8:-comparison of TSCDs of virgin (curve a) and hexane treated PP (curve b) arrows indicate $T_{1/2}$

4. Conclusions

I. It is seen that the aggregate structure parameters of TOPP, BOPP,& PP film such as crystallinity, orientation & morphology etc. determined with infrared spectroscopy agree with these obtained by the X-ray diffraction method.

II. The key point to improve breakdown & tensile strength & stability of these film is to increase crystallinity, orientation & molecular weight of these films.

III. A TOPP film is better than BOPP which is better than PP film.

IV. TOPP film has better charge storage properties than BOPP & unoriented PP film.

V. The charge storage is predominately due to impurity trapping & space charge mechanism.

VI. The polar functional groups induced in TOPP & BOPP & PP by irradiation have been shown to affect the dielectric response of the material loss.

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