

Space Charge and Electrical Conduction in Maleic Anhydride-grafted Polyethylene

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ABSTRACT

The space charge behavior such as charge distribution and electrical conduction of maleic anhydride grafted LDPE (LDPE-g-MAH) was investigated. Heterocharge was found in LDPE and it decreased with increasing MAH graft ratio in LDPE-g-MAH. Conduction currents also decreased with increasing MAH graft ratio. The relationship between the space charge behavior and the electrical conduction characteristics in LDPE-g-MAH is discussed.

1. INTRODUCTION

POLYMERS are widely used as insulating material because of their good electrical properties. Despite the well known advantages of polymer insulated HV transmission power cables in comparison with oil-filled and paper insulated cables, the use of polymer as an insulation for HV transmission power cables involves many problems, one of which is space charge accumulation in the insulation. Space charge in insulating material is known to affect electrical conduction and breakdown phenomena [1,2]. The accumulated space charge can greatly distort the original electric field distribution and sometimes even can cause breakdown of the insulation. It is, therefore, very important to understand its behavior to develop better insulating polymers and insulation design for HV transmission power cables with high reliability.

However, only a few quantitative studies have been made on space charge in dielectrics because of the difficulty in the direct measurement of space charge. Recently, two nondestructive direct techniques have been developed to measure space charge and electric field distribution in thick solid insulating material [3-7]. Many

results based on direct measurements include the effect of oxidation on space charge formation in oxidized polyethylene [8], the influence of crosslinking agent residues on space charge distribution in XLPE [9], and the effect of space charge on the dielectric breakdown and electrical conduction [10-12]. Most of these works are related to the understanding of nature or influence of space charge on solid dielectrics. Quite a few results are reported regarding the influence of chemical modification of polymers on space charge formation. It has been shown that the charge accumulation in polyethylene (PE) is reduced by the introduction of polar groups such as carbonyls, into the PE [13].

In this work, the chemical modification of LDPE by maleic anhydride grafting was performed and the influence of chemical modification on space charge formation and electrical conduction was investigated. An attempt also was made to explain the relationship between the space charge behavior and the conduction current characteristics in maleic anhydride grafted LDPE (LDPE-g-MAH).

2. EXPERIMENTAL

2.1. MATERIALS AND GRAFTING REACTION

The LDPE used in this study is the LDPE without additives supplied by Han Yang Chemical Corp. This LDPE has a density of 0.920 g/cm³, a melt index of 2.0 g/10 min, weight average molecular weight of ~ 144000 g/mol, and the molecular weight distribution (MWD) was $M_w/M_n \sim 8$. Maleic anhydride (Aldrich Chemical Co.) was purified by recrystallization from chloroform. The initiator, dicumyl peroxide (DCP, Aldrich Chemical Co.) was used as received. The grafting reaction was carried out in a twin screw extruder (Gottfert Corp.) at a constant rotating speed of 15 rpm and the barrel ($L/D = 25$) temperature profile of 150 to 220°C. The reaction mixtures were premixed in a Henschel mixer before they were fed into the extruder. The MAH graft ratio was adjusted not to exceed 0.50%wt to prevent the gelation by crosslinking during the grafting reaction.

2.2. ANALYSIS

In order to measure the content of MAH grafted on LDPE, the samples obtained after grafting reaction were predried to remove the unreacted MAH monomers in a vacuum oven at 60°C for 5 h. The dried samples were dissolved in xylene at a concentration of 2%wt. This solution was precipitated in acetone, filtered and dried again in a vacuum oven at 60°C for 24 h. The percentage of MAH grafted on LDPE was determined by titration [14] and elemental analysis. The contents of microgel which could be caused by crosslinking during the grafting reaction were measured by xylene extraction, but no gels were found.

The FTIR spectra of LDPE-g-MAH samples were collected using a Bomem MB-100 spectrometer at a resolution of 4 cm⁻¹. The melt indices (MI) of LDPE-g-MAH samples were measured at 190°C using a Toyoseiki extrusion plastometer with a load of 2160 g (ASTM D 1288; condition E). The melting temperatures and crystallinities of the samples were determined by using Du Pont DSC and 9900 thermal analyzer. Tensile properties were measured by Instron 4505 tensile tester.

2.3. MEASUREMENTS OF CHARGE DISTRIBUTIONS AND ELECTRICAL CONDUCTION

Samples having 1 mm thickness for measurements of charge distributions were prepared by compression molding at 120°C for 10 min using a PET backing film. This condition remained unchanged for all the samples in order to avoid the effects of sample preparation conditions on the formation of space charge in LDPE [15]. Semiconductive films were prepared by curing at 180°C for 20 min

and then degassed to remove the volatile residues from the curing reaction in vacuum at 70°C for 100 h. Degassed semiconductive films were then attached to the sample by an iron. The raw material for semiconductive electrodes is the commercial product used for strand shield of medium voltage power cables, which is a mixture of olefin polymer with polar functional group, conductive carbon black, processing aids, and antioxidants. Since it has been reported that the constituents of semiconductive electrodes may change the charge distributions in polyethylene [5], the same type of semiconductive electrodes were used throughout this study. This semiconductive electrode was used in order to simulate the structure of power cables in which the insulation layer is sandwiched between two semiconductive layers, *i.e.* strand shield and insulation shield.

Charge distributions were measured by a pulsed electroacoustic (PEA) method. When dc voltage is applied to the sample, charge is generated in the sample. When a HV pulse is applied to the anode, the charges in the sample generate acoustic pressure waves. The waves go through the sample and the cathode at the velocity of sound, and then reach the piezoelectric device where the acoustic pressure waves are transformed into the electric signal, which can represent the space charge distribution. The measurement principle of this technique has been described elsewhere [7,16]. Measurements of charge distributions were carried out as follows: First, the voltage was applied for 30 min across ~ 1 mm thick sample with semiconductive electrodes. After the voltage application for 30 min, the sample was short-circuited and then immediately the charge distribution was measured. After all these processes had been completed, the voltage was ramped up to the next test voltage. The final voltage was 40 kV, which corresponded to an electric field of ~ 40 kV/mm. In this study, the charge distribution during the voltage application was not measured. Therefore, all the charge distributions described in this paper represent the residual charge remaining in the sample after the discharge.

Electrical conduction characteristics were investigated using ~ 50 μm thick film samples prepared by compression molding at 120°C for 10 min using a PET backing film. The degassed 50 μm thick semiconductive films were also used as electrodes. The apparatus used to measure the conduction currents is composed of power supply (Keithly 247), electrometer (Keithly 617) and test cell. Tests were carried out at temperatures from 30 to 85°C and test voltages were adjusted to be in the range of up to 36 MV/m. The current at 40 min after the voltage application was taken as the equilibrium current [17].

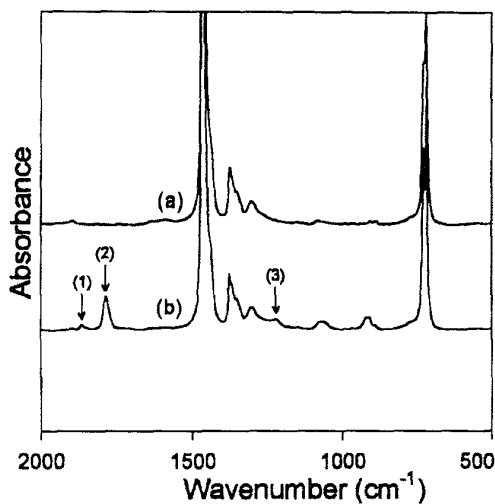


Figure 1.

FTIR spectra of (a) LDPE, (b) LDPE-g-MAH. (1) 1849 cm^{-1} , (2) 1794 cm^{-1} , (3) 1218 cm^{-1} .

3. RESULTS AND DISCUSSION

3.1. CHARACTERIZATION OF LDPE-g-MAH

LDPE-g-MAH has been characterized using a FTIR method. Figure 1 shows the FTIR spectra of LDPE and LDPE-g-MAH. It is found that there are three new peaks at 1849, 1794 and 1218 cm^{-1} in LDPE-g-MAH. The peaks at 1849 and 1794 cm^{-1} represent an anti-symmetric stretching and symmetric stretching modes of the C=O group. The peak at 1218 cm^{-1} corresponds to a stretching mode of the C-O-C group. The presence of these peaks indicates that the MAH has been grafted successfully onto polyethylene chains [18–20]. Their peak heights increased with the increase of MAH content in LDPE-g-MAH.

Various properties, such as melt index, degree of crystallinity, melting temperature, tensile strength and % elongation, of LDPE-g-MAH are summarized in Table 1. Melt index and % elongation decrease and tensile strength increases, whereas degree of crystallinity and melt temperature remain relatively unchanged when the MAH graft ratio increases to 0.5%wt. During the MAH grafting reaction using DCP as an initiator, the macroradicals can undergo chain linkage between the two polymer chains, resulting in formation of higher molecular weight chains [19]. This will contribute to the material properties of LDPE-g-MAH.

3.2. SPACE CHARGE DISTRIBUTIONS

Figure 2 shows the space charge distribution of LDPE and LDPE-g-MAH samples after the voltage of 40 kV is removed. Locations of the electrodes and the samples are indicated in the Figure. Two features are involved in

Table 1.
Material properties of LDPE and LDPE-g-MAH.

Sample	Graft ratio %wt	Melt index g/10 min	Cryst. %	Melt temp. °C	Tensile strength MPa	Elong. %
LDPE	—	2.0	38	111	13.5	635
LDPE-g-MAH	0.10	0.7	36	109	15.3	615
LDPE-g-MAH	0.21	0.6	37	109	16.0	615
LDPE-g-MAH	0.05	0.3	37	109	16.0	595
LDPE-g-MAH	0.17	0.3	36	109	17.5	575
LDPE-g-MAH	0.28	0.2	35	109	17.0	565
LDPE-g-MAH	0.50	0.2	36	108	18.0	565

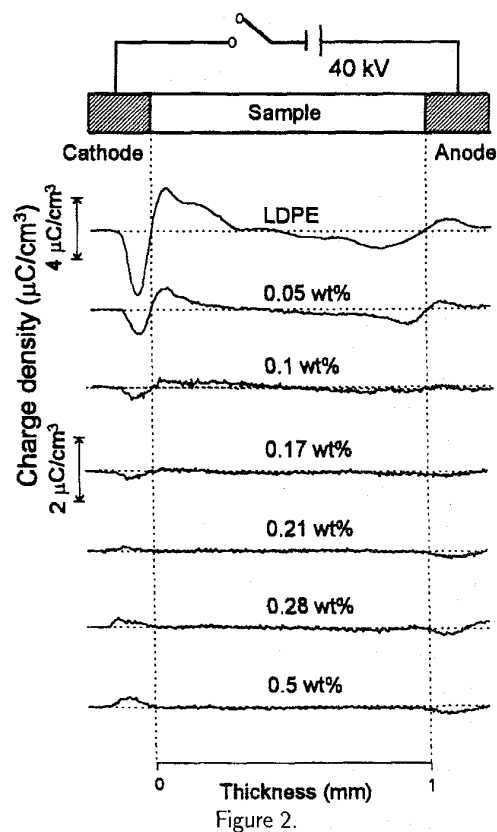


Figure 2.

Space charge distributions at 40 kV of LDPE and LDPE-g-MAH.

the space charge distributions. One is the peak induced at the electrodes. In LDPE, a clear negative charge peak at the cathode and a small positive charge peak at the anode are observed. These are the peaks for the charges induced at the electrodes, the polarities of which are opposite to those of bulk charges near the electrodes. The other is the attenuation of the signal from the anode. The peak intensities for the induced charge and the bulk charge at the anode side are small compared to those at the cathode side. This is because the signal is attenuated considerably while traveling through the bulk of the

sample to the piezoelectric detector. However, the signal from the cathode side is not attenuated significantly because the piezoelectric detector is adjacent to the cathode.

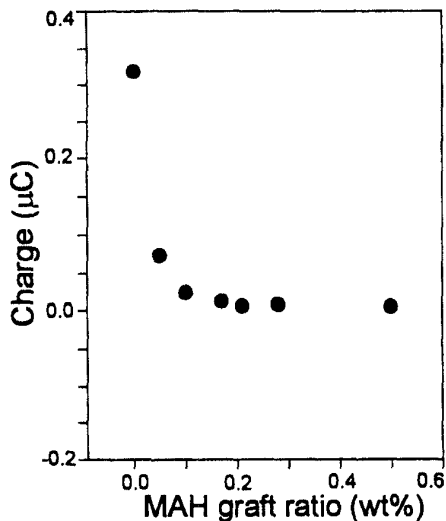


Figure 3.

Charge near the cathode as a function of MAH graft ratio in LDPE-g-MAH.

In the bulk of LDPE, positive charge near the cathode and negative charge near the anode are observed, which indicates the formation of heterocharge. Heterocharge in LDPE may be due to the polarization and/or migration of the impurities and the ions. This heterocharge decreases as the MAH graft ratio in LDPE-g-MAH increases, as shown in Figure 2. At high MAH graft ratio, the positive induced charge peak is found to develop at the cathode. It implies that there are negative charges near the cathode. These positive charge peak intensities increase as the MAH content increases, which means that homocharge increases as the MAH content increases. The MAH content dependence of charge near the cathode can be seen more clearly in Figure 3. This Figure shows a decrease of heterocharge with the increase in the graft ratio. Above a certain value, $\sim 0.20\%$ wt in this particular case, the residual charge becomes negligible. These results may be clear indications that the MAH reduces the heterocharge in LDPE.

3.3. ELECTRICAL CONDUCTION OF LDPE AND LDPE-g-MAH

Equilibrium current density J vs. electric field E characteristics are shown in Figure 4 for the LDPE and in Figure 5 for 0.28%wt MAH. Higher current densities are observed at higher temperatures and electric fields. In particular, the current density and the slope in $\log J$ vs. $\log E$ decrease as the MAH content increases, as shown

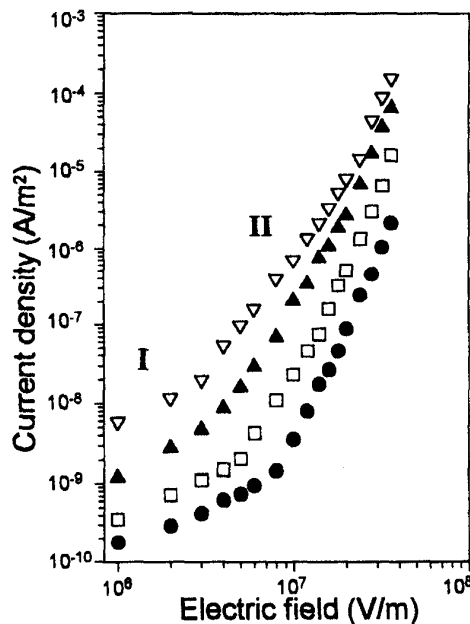


Figure 4.

J vs. E curves of LDPE at various temperatures. 30°C (●), 50°C (□), 70°C (▲), 85°C (△).

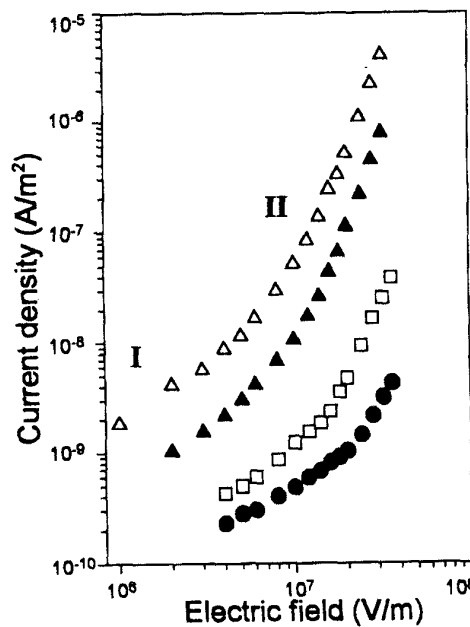


Figure 5.

J vs. E curves of LDPE-g-MAH (0.28%wt MAH) at various temperatures. 30°C (●), 50°C (□), 70°C (▲), 85°C (△).

in Figure 6. It was also found that the equilibrium current density is proportional to the n -th power of electric field.

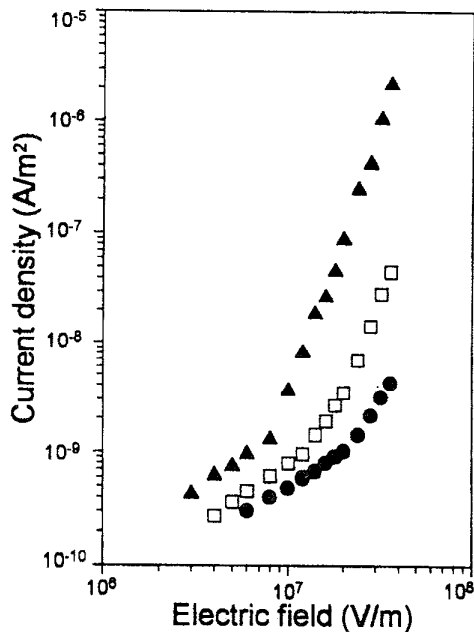


Figure 6.
 J vs. E curves of LDPE and LDPE-g-MAH at 30°C. LDPE (\blacktriangle), 0.05%wt MAH (\square), 0.28%wt MAH (\bullet).

The slopes in $\log J$ vs. $\log E$ curves are summarized in Table 2 for the LDPE and modified polyethylenes. The slope at low electric fields is ~ 1.0 for both LDPE and modified polyethylenes. At high electric fields, the slopes change to higher values, typically 3.3 to 4.5 for the LDPE and 1.9 to 3.3 for modified polyethylenes. The slope for LDPE decreases as the temperature increases, as reported by others [21–24]. As shown in Table 2, these slopes decrease with increasing MAH content. Typical values are 2.7 to 3.3 for 0.28%wt LDPE-g-MAH and 1.9 to 2.3 for the 0.50%wt LDPE-g-MAH. This n -th power relationship between current density and electric field may be an indication of the space charge limited conduction (SCLC) model, as reported by others [24, 25].

Table 2.

Slopes in a $\log J$ vs. $\log E$ plot for LDPE and LDPE-g-MAH.

Temp. °C	LDPE		LDPE-g-MAH			
	R.I	R.II	0.28%wt		0.5%wt	
			R.I	R.II	R.I	R.II
30	1.0	4.5	0.9	2.7	0.9	1.9
50	1.1	3.8	1.1	3.4	1.0	2.4
60	–	–	–	–	1.2	2.3
70	1.1	3.6	1.2	3.3	1.1	2.3
85	1.1	3.3	1.1	3.3	1.4	2.3

Complementary evidence for the SCLC mechanism were also found from the time and thickness dependence of

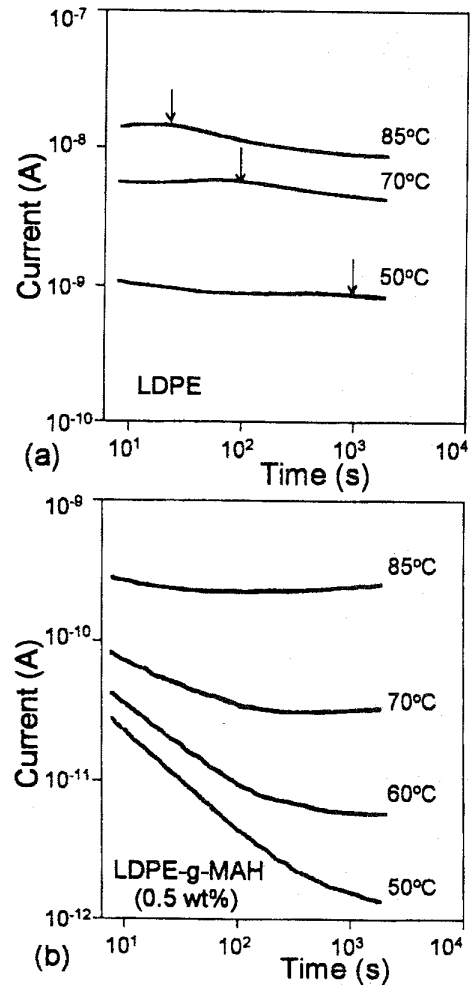
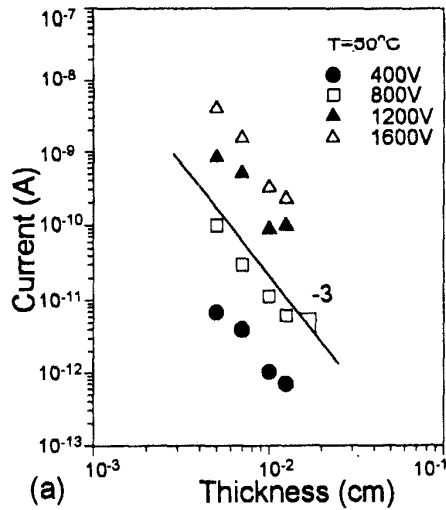


Figure 7.

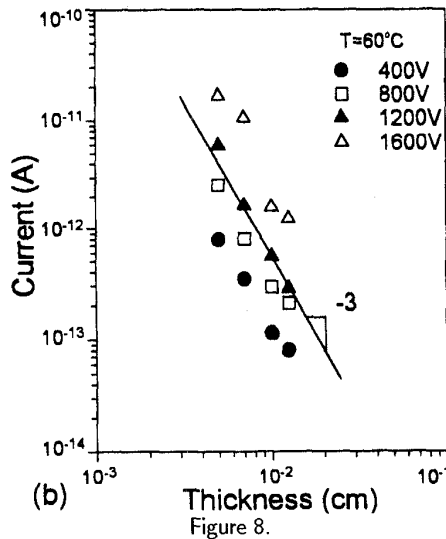
Time dependence of charging currents at 1200 V. (a) LDPE, (b) LDPE-g-MAH (0.50%wt MAH).

charging currents. Typical time dependence of charging currents is shown in Figure 7, where (a) is for the LDPE and (b) is for the 0.50%wt LDPE-g-MAH. The current maxima in $\log i$ vs. $\log t$ curves are observed in the LDPE. The current peaks shift to a shorter time when the applied fields or the temperature increase and they show similarities to the transient SCLC peaks proposed by Many and Rakavy [26]. Charge mobilities were calculated from the peak time (arrows in Figure 7(a)) using the equation of transient SCLC model with $4.2 \times 10^{-15} \text{ m}^2/\text{Vs}$ at 50°C, $2.3 \times 10^{-14} \text{ m}^2/\text{Vs}$ at 70°C and $7.2 \times 10^{-14} \text{ m}^2/\text{Vs}$ at 85°C, and at 1200 V (24 MV/m). Figure 7(b) shows no peaks in 0.50%wt LDPE-g-MAH. The reason for this may be that the existence of traps probably obscures the peak. Figure 8(a) and (b) show the thickness dependence of conduction current for the LDPE and 0.50%wt LDPE-g-MAH. Although an extensive range of

sample thicknesses was not covered due to the instrumental limitation, the current density is proportional to the -3 power of sample thickness, which could be an indication of the SCLC model.



(a) Thickness (cm)



(b) Thickness (cm)

Figure 8. Thickness dependence of conduction currents. (a) LDPE, (b) LDPE-g-MAH (0.50%wt MAH)

In case of SCLC with a single set of traps, the effective charge mobility can be calculated from the Child law [27] by using $\epsilon_r = 2.25$ for all samples. Calculated effective charge mobilities at 30 and 50°C were plotted as a function of MAH content in Figure 9. For both temperatures, the charge mobility decreases as the MAH content increases. Typical values at 50°C and 1200 V (24 MV/m) are $5.3 \times 10^{-15} \text{ m}^2/\text{Vs}$ for LDPE and $1.2 \times 10^{-17} \text{ m}^2/\text{Vs}$ for 0.50%wt LDPE-g-MAH, indicating that the MAH molecules reduce the charge mobility. The value for LDPE is

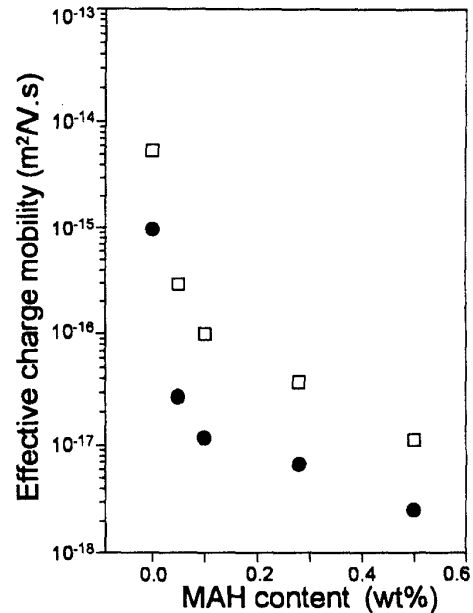


Figure 9. MAH content dependence of effective charge mobility at 1200 V. 30°C (●), 50°C (□).

comparable to the value calculated from the peak time shown in Figure 7(a) and the value reported by others [21, 28, 29].

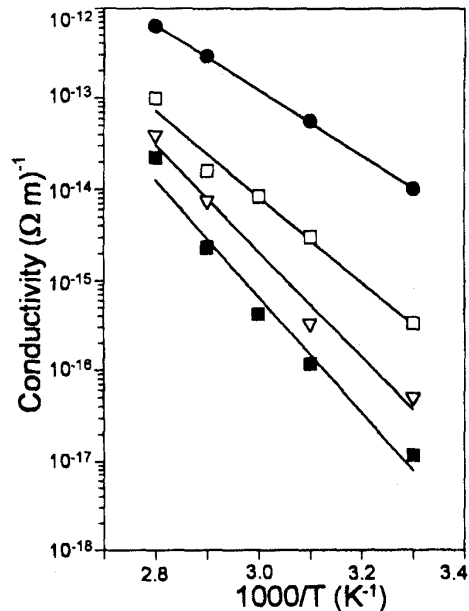


Figure 10.

Temperature dependence of conductivity at 1200 V. LDPE (●), 0.05%wt MAH (□), 0.28%wt MAH (▽), 0.50%wt MAH (■).

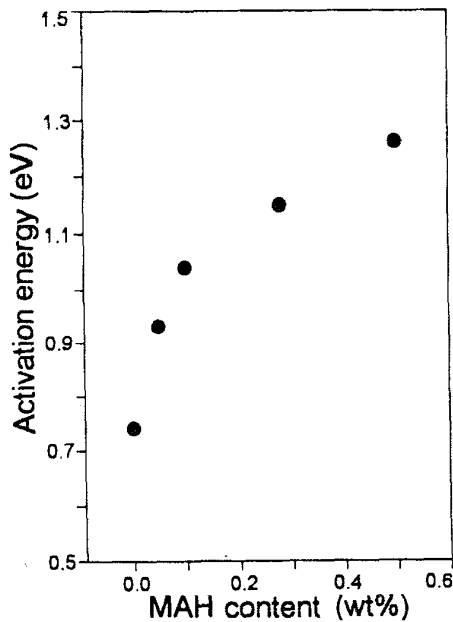


Figure 11.

MAH content dependence of activation energy at 1200 V.

Figure 10 shows the temperature dependence of conductivity and Figure 11 shows the activation energy calculated at 1200 V (24 MV/m). The conductivity decreases, while the activation energy increases, when the MAH content increases as shown in Figure 10 and 11.

All these results suggest that the migration of charge (electrons) is restricted by the MAH molecules acting as trapping sites, the extent of which becomes more considerable as the MAH content increases.

3.4. CORRELATION OF CONDUCTION CURRENT WITH SPACE CHARGE DISTRIBUTION

It has been found throughout this study that the MAH grafted on LDPE reduces the heterocharge, conduction currents and effective charge mobility. It can be assumed that both heterocharge and homocharge are developed at the same time when the voltage is applied. The measured space charge distributions are determined by the process which is dominant at the particular state of measurement. The fact that both current density and effective charge mobility decrease with the MAH grafting suggests the negative charges (injected electrons) are trapped and become immobile due to the MAH. This is quite possible because the MAH, a strong electron acceptor, has carbonyl groups and a ring structure acting as trapping sites. The net result is the decrease of heterocharge at higher MAH content (Figures 2 and 3).

It is of interest to note that conduction currents decrease as the amount of heterocharge decreases. Two

explanations may be possible. One may be that the migration of mobile charge to form heterocharge contributes to the conduction currents. In this case, the more heterocharge, the higher the conduction current. The other may be that the heterocharge accumulated at the electrode/sample interface increases the local field near the electrode, and thus the charge injection is enhanced. As more charge is injected, the local field enhancement decreases because the heterocharge becomes smaller. Thus it may be expected that the field enhancement at the interfacial region becomes low and the charge injection becomes less, with the net result being a decrease of conduction currents. A similar observation has been reported by Kikuta *et al.* [12].

4. CONCLUSIONS

THE study on the space charge and conduction behavior in LDPE-g-MAH reveals the following conclusions.

The heterocharge in LDPE decreases with increasing MAH content grafted on LDPE. The residual charge becomes negligible at MAH content > 0.2%wt.

The conduction current and effective charge mobility of LDPE-g-MAH decrease with increasing the MAH content.

All these features were attributed to the enhanced homocharge trapping at MAH in LDPE-g-MAH

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