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# Space charge dynamic in polyethylene: from DC to AC stress

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**Abstract.** Time-resolved space charge profiles obtained in low density polyethylene under sinusoidal or square AC stress for frequencies going from DC to 10 Hz, temperature from 25 to 50°C and fields of 30 or 40 kV/mm are presented. It is shown that most of the features observed under DC stress (generation of carriers of the two polarities with non-symmetrical amount and velocity) are still active under AC stress. As a matter of fact, it is shown that a negative space charge builds up at 50°C within the whole insulation bulk even with relatively high stress frequency and for short stressing time (2 h). These results clearly show that space charge has to be considered under AC condition.

## 1. Introduction

Space charge in insulating polymers has been investigated thoroughly within the last three decades because of its influence on the internal field distribution and its relationship with electrical ageing mechanisms [1]. Most of these studies have been undertaken under DC for both the relatively easy implementation of the measurement, whatever the principle used, along with relatively efficient charge build-up in the bulk of the insulation, and hence ready demonstration of space charge effects.

Among lessons learnt from these measurements are the facts that charges often are generated by injection from the electrodes, that positive and negative carriers can coexist in the insulation bulk, and that the extent of the space charge regions depends on the stressing time. From these features, it can be anticipated that under AC stress for example, charges injected from the electrodes in one half-cycle may be extracted and/or recombined during the following half cycle without significant accumulation in the polymer leading to small net charge density, with localisation essentially close to the electrodes. Though the electric field distortion induced by space charge is probably mild under AC stress, energetic processes triggering material degradation [2] remain harmful. This can happen in situations where charges of opposite polarities recombine, or when “hot carriers” are scattered by atoms or molecules of the solid, leading to the generation of electronically excited states that are chemically reactive. These situations are encountered under DC stress when injected charges of opposite signs interact in the bulk due to charge transport, but AC is an ideal situation where injection/extraction at the electrode provides the conditions for carrier recombination in a thin layer at the contact.

The lack of data on space charge under AC or impulse stress is for a large part due to the difficulty for implementing such measurements. Of course, we are considering herein methods providing charge profiles under applied stress, meaning information as a function of the phase of the AC stress. Most of the set-ups developed for that purpose are based on the pulsed electro-acoustic technique (PEA) as it combines the possibility of high acquisition rate for charge profiles and measurement under applied high voltage. The principle of the PEA technique consists, in brief, in detecting acoustic waves generated by internal charges under the Coulombic influence of a pulsed electric field. The internal charge density is then deduced by signal processing and mathematic treatment [3]. Voltage pulses of relatively low amplitude are applied in order for it to act as probe without modifying the charge state during the measurement time. This is the reason why the acoustic response is averaged applying several voltage pulses [4]. The time for a charge profile acquisition, and hence the frequency of profile acquisition, depend directly on the frequency of the pulse generator (generally in the kHz range). When the frequency of the AC stress is relatively high, the applied voltage can not be necessarily considered as constant during the averaging time, so several periods of the stress are necessary to extract space charge profiles. So, technical difficulties are linked to the frequency of pulse generation, to the synchronization of pulses with the AC stress and to data transfer rate and storage.

In the PEA set-ups developed for the purpose of measurement under AC stress described in the literature [5-8], a digital oscilloscope is generally chosen for data storage, as is usually done for DC stress. However, limitations readily occur with such systems due to the low data transfer rate of the General Purpose Interface Bus (GPIB). This has led to the choice of alternative storage units like a 16 Mb memory data acquisition (DAQ) card [8] or box-car system that allows real time data acquisition and transfer at a high rate [5]. The common feature of all these systems is the triggering of pulse generation at specific positions (called point on wave) of the AC stress cycle. Generally, a limited number of these positions are used (to our knowledge, up to 16 so far) and bursts of pulses are

generated at the measurement point. The measurement time depends on the resolution expected on the phase of the AC stress and on the number of records (i.e. the response to a single voltage pulse) to average.

To avoid the experimental difficulties of these methods, we have presented recently [9] a new method based on a standard PEA test cell and a new signal processing method that exploits the properties of the Hilbert transform for phase recognition purpose. Here, the position of the voltage pulses along the phase of the AC stress, and therefore of the corresponding acoustic signal records, are estimated *a posteriori*. Only minor modifications of the PEA test benches used for DC stress measurements are necessary for adaptation to AC stress measurements, as triggering of pulse generation at specific positions of the AC stress is no longer necessary. A much higher resolution along the phase of the AC stress is achieved.

The present contribution discusses results obtained on low density polyethylene, a material which is well documented regarding space charge build-up under DC stress. The behaviour of space charge is presented, going from DC, to low frequency (1mHz) AC stress and up to 10Hz AC stress, for temperatures in the range 20 to 50°C. It is shown that the same processes, namely injection of carriers at the both electrodes and a lower mobility for negative than for positive carriers seem to hold irrespective of the temperature (though processes are accelerated) and frequency. We first recall the principle of the measurements under AC stress by the new method.

## 2. Materials and PEA set-up

### 2.1. Material

Samples considered herein are low density polyethylene -LDPE- films containing an antioxidant (Santnox®). Film samples in the form of 70 mm diameter disks and 190 µm thickness were obtained by press-molding LDPE pellets for 10 min at 140°C. The cooling down was achieved at an average rate of 4°C/min.

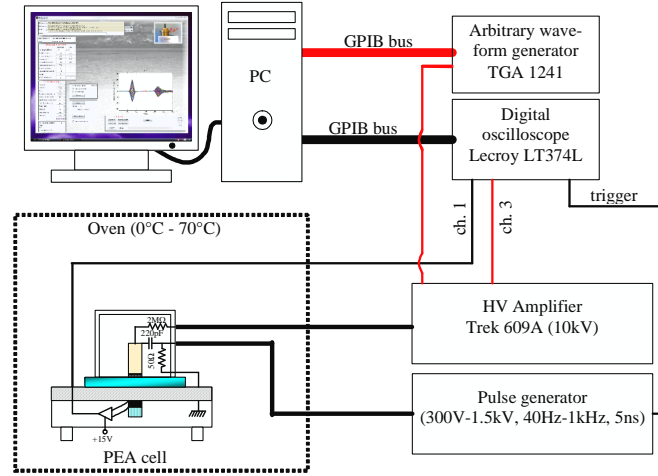
Measurements have been carried out without prior metallization of the films, so that the electrodes were those of the PEA set-up: a semi-conducting –SC- material (carbon black-doped polymer) as upper electrode and aluminium as ground electrode. One fresh sample has been used for each measurement. In all the space charge density patterns presented therein, the SC electrode is at the position  $x=190\ \mu\text{m}$  and the Al electrode is at  $x=0$ .

### 2.2. PEA set-up

Figure 1 shows the set-up used for PEA measurements under AC stress. It is based on a PEA cell provided by TechImp®, Italy. The AC stress is provided by a HV amplifier fed by an arbitrary waveform generator. Care has been taken about the effects of the coupling circuitry between the upper electrode and the HV source. Considering the output impedance of the amplifier, the values of the coupling components of the PEA cell and the sample capacitance of 10 pF, we have estimated as 8.7° and 1% respectively the phase shift and the voltage attenuation between the output of the amplifier and the voltage effectively applied to the sample for a frequency of 50 Hz. These remain acceptable values provided the phase shift is taken into account.

Measurements have been carried out both under DC and AC stress. For DC stress or low frequency AC stress (<50 mHz), the variation of the applied voltage is small within the measurement time (defined as the time required for averaging a minimum of 50 records). Then, the oscilloscope proceeds to averaging of records as is usually done under DC stress.

The measurement time is just the period of the AC stress, ranging from 20 to 1000 s for frequencies in the range 50 mHz to 1 mHz. In order to limit the amount of data to be stored, the pulse frequency is reduced when going to low frequency for the HV stress. The applied voltage is measured using the third channel of the oscilloscope.



**Figure 1.** Experimental arrangement used for space charge measurements under AC stress.

For higher frequency, acquisition over several periods of the AC stress is necessary. The oscilloscope, which is set into the sequential trigger mode, acquires the signal in the form of sequences, each of them containing 4000 segments. A segment represents the response to a single pulse, i.e. a record. Two input channels of the oscilloscope are used: one for the acoustic signal and one for measuring the applied voltage. Once a pair of sequences has been acquired, they are transferred into memory blocks of the oscilloscope. Three pairs of sequences can be stored representing 12000 records. Afterwards, the data are transferred to the PC and processed. In order to identify the phase of the AC stress for each record, we use a property of the Hilbert transform, which is to enable to transform a real data sequence  $v(t)$  in an analytic signal  $s(t)$  that contains the information on the original phase signal [10].

$$s(t) = v(t) + j \hat{v}(t) = A(t)e^{j\phi(t)} \quad (1)$$

where  $\hat{v}(t)$  corresponds to the Hilbert transform of  $v(t)$ , which is the applied voltage in our case:

$$\hat{v}(t) = \frac{1}{\pi} \int_{-\infty}^{+\infty} \frac{v(\tau)}{t - \tau} d\tau \quad (2)$$

Then we obtain the phase corresponding to a given voltage value from:

$$\phi(t) = \arctan \frac{\hat{v}(t)}{v(t)} \quad (3)$$

In this way, voltage values recorded at unknown times during several periods of the signal can be sorted out: each data point value is identified by a unique value of the instantaneous phase, and hence the position in a period at which a given record has been acquired can be unambiguously identified considering  $\phi(t)$ . Then, acoustic signals comprised between equally spaced intervals of the phase of the AC stress are averaged. Finally, space charge profiles are processed according to the standard equation relating the acoustic response and the space charge profile [3].

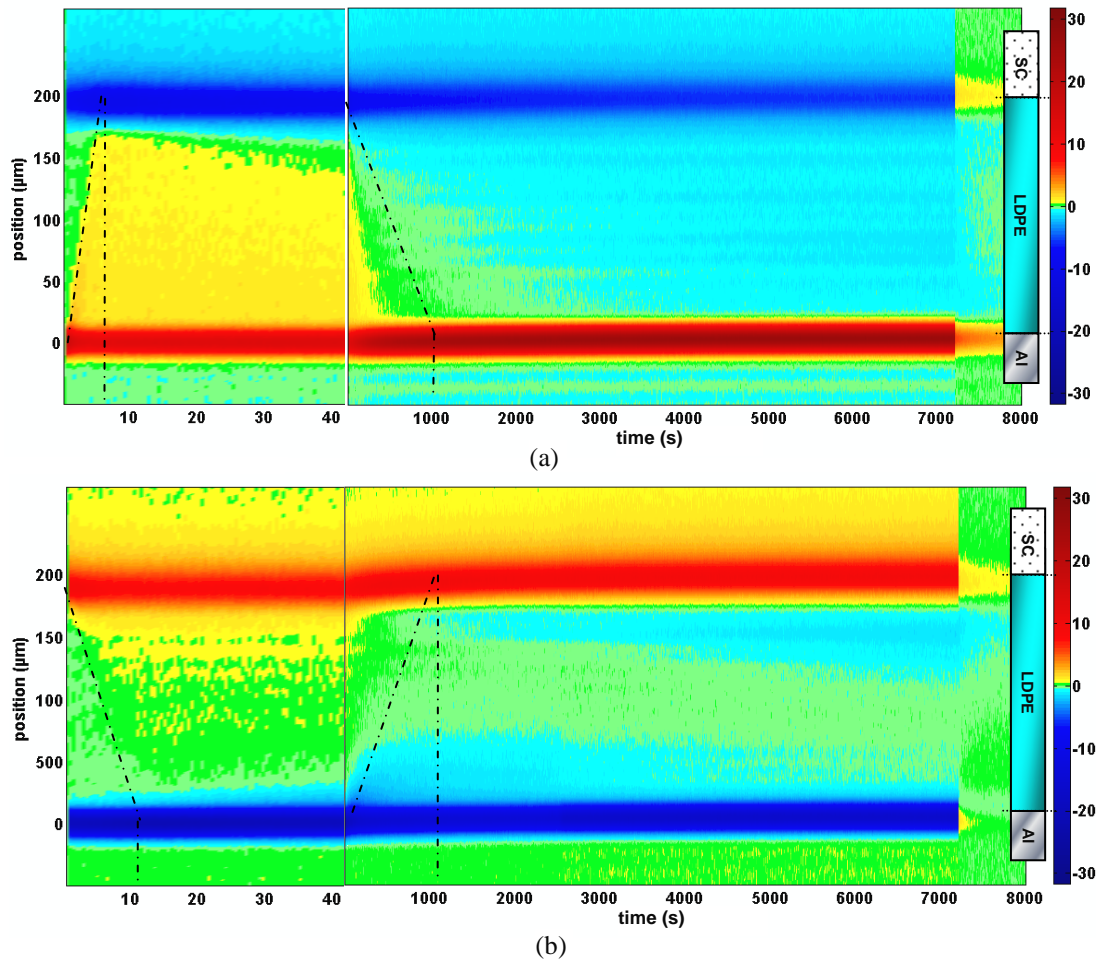
More detail on the hardware and limitations of the method are given in [9]. Results presented herein were obtained with a resolution of 200 space charge profiles per period of the AC stress. Each profile is issued from the averaging of about 60 acoustic signal records. The acquisition time of 12000 acoustic records, including transfer time to memory blocks of the oscilloscope, is roughly 20 s using a 1kHz pulse generator. This corresponds to a number of periods of the AC stress ranging from 200 periods at 10Hz AC stress to 2 periods at 100mHz for obtaining one phase-resolved space charge map as shown in the following.

### 3. Experimental results and discussion

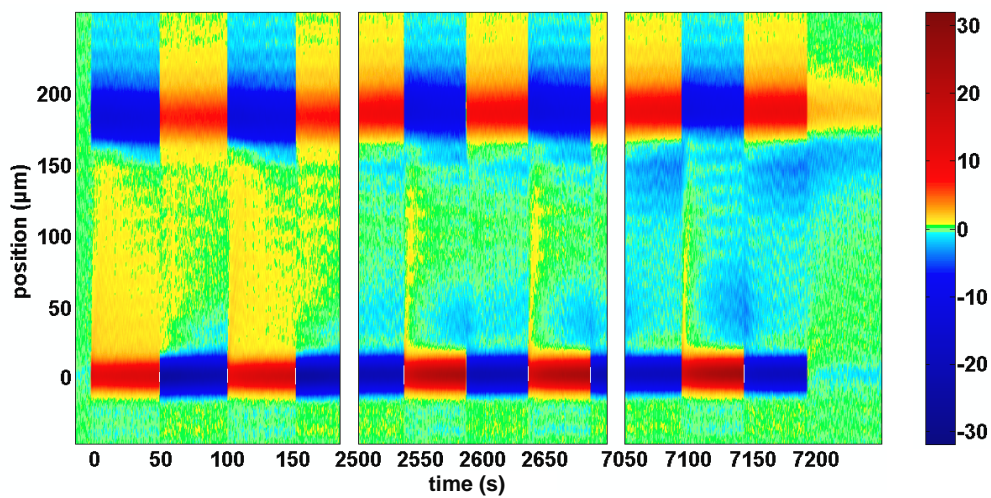
#### 3.1. DC stress and low frequency square stress

Figure 2 shows typical space charge results obtained during 2 h under 30 kV/mm DC stress at 40°C considering positive and negative polarity for the upper electrode. In figure 2(a), in the first instants of voltage application, a front of positive charges is injected from the lower aluminium electrode and drifts up to the upper SC electrode within about 5 s (see dotted lines construction). In the course of time, positive charges are progressively replaced by negative carriers which ultimately occupy the all insulation bulk, a behaviour typical of LDPE [11]. The apparent transit time for negative carriers is much longer than for positive ones, being of the order of 1000 s. Very similar processes were observed when changing the polarity of the applied voltage (cf. figure 2(b)). The transit times were of the same order as those given above. The main difference was in the amount of injected charges which was lower for positive carriers. The difference in the nature of the electrodes is likely to explain such results, aluminium being more efficient for injecting positive carriers than negative one, and conversely for the SC [12]. At the end of the experiment, the bulk-averaged space charge density was close to -1 C/m<sup>3</sup> in both cases. The stress frequency has been progressively increased. Let us consider first the behaviour under 10 mHz square voltage, see figure 3.

During the first half-period of stressing under 10 mHz square voltage (t=0-50s), processes at play appear similar to those described in figure 2. Positive charges are massively injected at the lower electrode and already negative carriers accumulation due to injection from the upper electrode can be distinguished at the end of the half-period. After the first polarity reversal (t=50-100 s), a front of positive charges injected from the upper electrode can be distinguished though the density is less than for the first half-period. In the meantime, negative charge injection occurs at the lower electrode. So, basically the processes are similar to those observed under continuous DC stress with one or other polarity. During the second period of the square stress, the pattern is about the same as for the first period.



**Figure 2.** 3D representation of space charge profiles obtained under  $-30$  kV/mm (a) and  $+30$  kV/mm (b) DC stress applied for 2 hours at  $40^{\circ}\text{C}$  in LDPE. SC electrode to the top. Color scales for the charge density (in  $\text{C}/\text{m}^3$ ) given to the right. The plot to the left enlarges the response in the first instants of voltage application. The voltage has been set to zero after 7200 s.



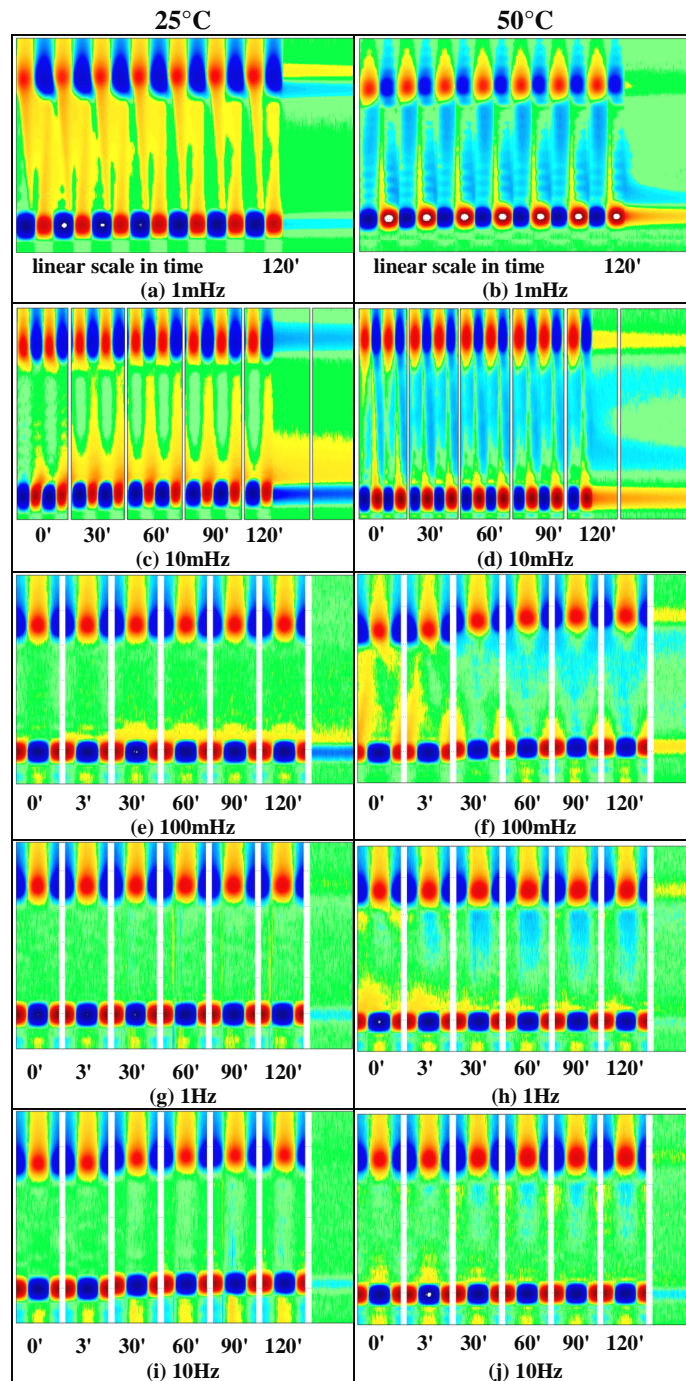
**Figure 3.** Space charge profiles obtained in LDPE at  $40^{\circ}\text{C}$  under square voltage of  $\pm 30$  kV<sub>max</sub>/mm at a frequency of 10 mHz. The patterns represent profiles obtained just after voltage application, after 45 min of stressing and after 2h of stressing. After 7200 s, the voltage has been set to 0.

After 45 min of stressing under 10 mHz square voltage, negative charges tend to be dominant and have penetrated in the bulk of the insulation. Positive charges appear only at polarity reversal and are detected for a short time only (to be more precise, as only the net charge is measured, the positive charge density exceeds the negative ones only short after polarity reversal). After 2 h of stressing, the dominance of negative carriers is further reinforced. They are detected at roughly the same position as after 45 min of stressing under 10 mHz square voltage, but the density increases significantly. Compared to the density after 2 h of permanent DC stressing, the charge density is less. What is also remarkable is the fact that the negative charge density is at maximum at about 50  $\mu\text{m}$  from the positive electrode (cf. profiles obtained in volt-off, 3rd pattern) in every half period.

### 3.2. Sinusoidal stress

The above results have shown that, depending on the time interval considered, space charge profiles can be dominated either by positive or negative carriers. As will be shown below, the net charge density being measured is continuously decreasing when increasing the frequency. Therefore, the applied field has been increased slightly (40 kV/mm) for the purpose of investigating the SC behavior as a function of frequency under sinusoidal stress. Figure 4 shows a set of SC profiles obtained under sinusoidal stress for frequencies ranging from 1 mHz to 10 Hz and for temperatures of 25 and 50°C. In all the cases the stress was applied for 2 h. For measurements carried out at 1 mHz, space charge profiles have been acquired continuously. For measurements at 10 mHz, the profiles were obtained within two periods of the AC stress, in steps of 30 min. The two periods are represented in each pattern. For 100 mHz and on, several periods of the AC stress have been used to produce one pattern. The Hilbert transform has been used to sort out acoustic signals and to build patterns representing one period of the AC stress. In this case, the first profile in the pattern corresponds to the time when the upper electrode is at  $-V_{\text{max}}$ . Some profiles obtained after voltage removal are also represented in all the cases.

Charge patterns obtained at 1 mHz are appealing, switching from positive charge- to negative charge-dominated profiles depending on the temperature. For  $T=25^\circ\text{C}$ , positive charges are injected alternatively from the upper and from the lower electrode. Even though the sinusoidal stress does not permit a fine estimation of the transit time, the latter can be roughly estimated as about 500 s from the velocity of the charge front close to AC voltage peak. As the stressing time increases, the positive charge density tends to be less. Also, negative charges are detected adjacent to the upper electrode during depolarization. Therefore, it can be inferred that the decrease in the density of positive charge is due to the injection of negative carriers and their slow propagation in the bulk.



**Figure 4.** Space charge patterns obtained in LDPE at 25°C (left) and 50°C (right) under sinusoidal AC stress of  $40 \text{ kV}_{\text{max}}/\text{mm}$  crest field. The color scale for charge density is the same as that of figures 2 and 3. The stressing time is 2 h in all instances. Some patterns are shown in volt-off at the end of the stressing period in each case.

At 50°C and 1 mHz, positive carriers can be distinguished only at the beginning of the second half of each period when the lower electrode is at positive polarity and are apparently injected from the lower electrode. Except for that instant, negative charges are dominant and are alternatively injected at the higher or lower electrode. In this case, there is no evident change in the density of carriers with the

repetition of the stress (in the limit of 8 periods considered here), like if a kind of steady situation regarding the accumulation of charges was reached.

When the frequency is increased, the amount of positive charges detected at 25°C clearly decreases, and the more injecting character of the lower electrode observed for measurements under DC stress at 40°C is confirmed. At 10 mHz, positive charges have not enough time to cross the insulation within a half period (50 s) due to their slow transit time. They seem not to be extracted at the lower electrode, and therefore tend to accumulate in front of this electrode. When the frequency is further increased (100 mHz), positive charges are detected only adjacent to the lower electrode. They are no longer detected at higher frequency.

Considering frequency effects on data at 50°C, one observes that negative charge occupy less and less the volume of the insulation when going from 10 mHz to 10 Hz. Consequently the formation of positive space charge regions can be detected. Positive charges cross the insulation in the half period of the 100 mHz frequency. As the frequency increases, their apparent penetration depth is less and the positive charge appears confined close to the lower electrode. As observed previously for the square voltage at 40°C, the negative charge density tends to grow as a function of the stressing time.

### 3.3. Discussion

Despite the fact that measurements have been carried in different conditions regarding stress frequency, waveform and measurement temperature, a common frame for the interpretation of space charge formation from DC to AC stress can be drawn. First, the ensemble of the behaviour appears interpretable considering only positive and negative carriers injected from the electrodes. When ionic species are present, the charge distribution under DC stress reveals the formation of symmetrical space charge domains accumulating nearby the electrodes but with opposite polarities (i.e. heterocharges accumulation). In this case, the accumulation time is associated with the migration time of the ionic species in the bulk [13]. The fact that the space charge phenomenon in LDPE is dominated by carrier injection has been reported in several papers [14-16]. The different behaviour in terms of injected positive and negative charges is due to the difference in the nature of the electrode, aluminium vs. SC, as already pointed out in paragraph 3.2.

With the repetition of AC cycles applied to the material, more and more charges accumulate in the bulk. This means that at least within the moderate stress durations considered here (2 h), the rate of charge annihilation in the bulk (by extraction at the electrodes or by recombination with carriers of opposite polarity) is smaller than the rate of charge injection at the electrodes. The amount of charge stored appears decreasing with an increase of the stress frequency and the charge stays closer to the interfaces. This last feature is expected if the transit time is longer than the half period of the HV stress. However, one would expect a growing intensity of the charge density adjacent to the electrode. This is actually not the case. This complex behaviour can be linked in part to the bipolar nature of transport and to the differences in the generation and transport features for positive and negative carriers.

The lower electrode (made of Al) appears more efficiently injecting for positive charges than the SC electrode. Consequently, the transit features of positive carriers can be more easily characterized when this electrode is at positive polarity. Very clearly, positive carriers move much faster than negative ones, and so it is whatever the temperature. From the data available, we have roughly estimated the

transit times for electrons and holes for a r.m.s. field of roughly 30 kV/mm. The data are reported in table 1. Apparent mobilities that can be deduced are of the order of  $2.5 \cdot 10^{-13}$  and  $1.2 \cdot 10^{-11} \text{ m}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$  for electrons and holes, respectively, at 50°C. They are lowered by a factor 400 and  $10^4$ , respectively, at 25°C. Activation energies were not estimated as the variation of mobility with temperature did not follow an Arrhenius law.

**Table 1.** Estimation of the charge transit time as a function of temperature for positive and negative carriers (field  $\approx 30$  kV/mm, distance = 190  $\mu\text{m}$ ).

T (°C)	25	40	50
holes	$\approx 500$ s	$\approx 10$ s	$\approx 0.5$ s
electrons	$\approx 10000$ s	$\approx 800$ s	$\approx 25$ s

Our results highlight several points raised in the literature on the matter of space charge under AC stress. The oldest one concerns the accumulation of space charge in the bulk of the material under AC stress. It is generally considered that most of the carriers injected at electrodes during a half cycle are ejected during the next half cycle, so the net balance of charge on a cycle is zero [17]. It is shown in this work that there is an unbalance between charge injection and extraction leading to a slow accumulation of charge with stressing time, all the more the temperature is high, and this behaviour is evidenced in the range of the industrial frequency. The maximum stressing time in our experiments is 2 h and bulk space charge accumulation is clearly shown at a frequency of 10 Hz and a temperature of 50°C. Wang *et al.* [18] reported a upper limit frequency of 20mHz for space charge accumulation in polyethylene. The present results are in some respect in accordance with this statement considering measurements at 25°C. However, increasing temperature undoubtedly breaks the rule with negative charge accumulation clearly observable at 10Hz and 50°C. Measurements up to 50Hz are achievable with the actual PEA hardware with careful prevention of surface and corona discharges in the environment of samples [6, 9]. As measurements can be achieved safely up to 6kV voltage only at 50Hz [9], they were not attempted in the present set. However, we can reasonably anticipate that long enough stressing time at the industrial frequency would also lead to charge accumulation. The residual charge has indeed been measured after several (14) days of application of 50Hz AC stress to samples of polyethylene at temperatures in the range 45-90°C using the thermal step method [19]. It was found that the space charge, with a density in the range of  $1 \text{ C} \cdot \text{m}^{-3}$ , was maximum for a temperature of 60°C, and enough to induce a residual field  $>10$  kV/mm. Our own results are consistent with those and give a real-time picture of the accumulation process.

Another feature that is revealed by our experiments is the bi-polar nature of space charge formation under AC. Due to the fact that the PEA technique measures only the net space charge density (like all the other methods for probing space charge distribution), the actual density of carriers is much higher than what is measured because of the superposition of the distribution of positive and negative carriers. This fact gives a direct interpretation to the origin of electroluminescence phenomena under AC stress. It has been proposed that they are due to alternative injection of carriers of opposite polarities at the electrodes that recombine the trapped charge injected during the previous half cycle, leading to excited states with radiative decay [20]. The experiments are in agreement with such explanation. At low frequency, when the carrier transit time is smaller than the half period of the AC

cycle, conditions for recombination phenomena are similar to the DC case, i.e. front of carriers of opposite polarities interact in the bulk. By increasing the frequency, the carrier transit time becomes larger than half the period and the recombination process is taking place near the electrodes. In the range of industrial frequencies, recombination domains are of small extension in the vicinity of the electrodes and are ultimately confined to the contact region. This scheme is in agreement with the frequency dependence of electroluminescence in LDPE that is constant per cycle in the frequency range between 1 and 100 Hz, at room temperature and 40 kV/mm [21]. In these conditions, charge transfer is confined to the interfacial region.

#### 4. Conclusion

Time-resolved space charge measurements were carried out on low density polyethylene with a sinusoidal AC stress for frequencies ranging from DC to 10 Hz. The same processes, namely injection of carriers at the both electrodes and a lower mobility for negative carriers seem to hold irrespective of the temperature (though processes are accelerated) and frequency. For the same stressing duration (i.e. 2 hours) the quantity of net stored charge decreases when the frequency is increased. Though in most of the cases a steady situation is not reached regarding space charge patterns, after stressing for 2 h negative charges are effectively the dominant carriers or reasonably tend to become so, depending on experimental conditions. Although space charge effect under AC stressing is generally considered as unimportant, we have shown that charge accumulates with time and its dynamic has to be considered as it provides the conditions for carrier recombination. With current data at moderate frequency, matter is already provided for a better understanding and modelling of charge transport and storage under alternating stress.

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