

Measurements of interfacial charges in layered PTFE using step electroacoustic technique

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Abstract

The step-electroacoustic (SEA) technique was applied to measure interfacial charges in layered PTFE. Acoustic signals were measured from both sides of the sample and time-domain analysis was applied to eliminate the effect of reflections of acoustic waves at inner interfaces. The calibrated curves of space charge, electric field and potential in three layered specimen have been shown. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Space charge; Electroacoustic technique; SEA; PTFE; Multilayer structures

1. Introduction

The pulsed electroacoustic (PEA) method, also called the electrically stimulated acoustic wave (ESAW) technique, is a non-destructive acoustic method developed for the measurement of space charge distribution in solid dielectrics. The technique was originated by Takada, Maeno and co-workers [1,2] and was analysed by Bernstein [3]. Much progress has been made during the last ten years in the PEA or ESAW method [4]. The area of applications is reviewed in [5].

In order to obtain the measurements, a step voltage instead of a voltage pulse was applied to the sample in this work. In this case, the short-circuit current in the thin transducer is proportional to the local space charge density. The step version of the electroacoustic (EA) technique is defined here as the step electroacoustic (SEA) method.

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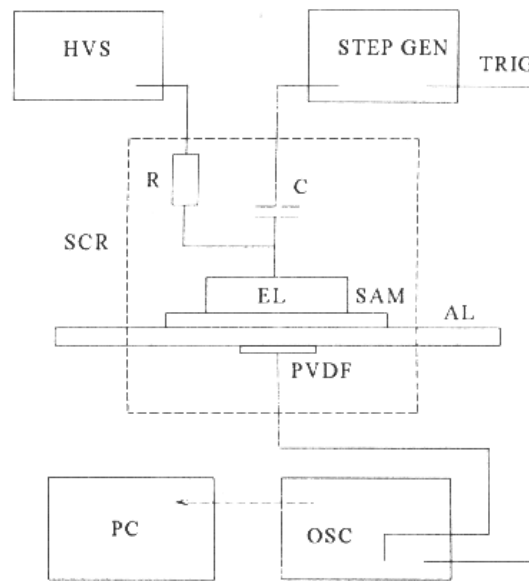


Fig. 1. The SEA measurement system.

2. Step electroacoustic technique

2.1. Experiment

The schematic view of the measurement system is shown in Fig. 1. The pressure wave generated by the step voltage (STEP GEN) applied to the sample (SAM) propagates through delay line (AL) towards the $9\ \mu\text{m}$ thick PVDF foil detector. Transducer and sample are acoustically matched on one side. The electret sample could be additionally polarised by dc polarising voltage u_0 within the range -4 to $+4\ \text{kV}$ from the high voltage source (HVS). The step voltage $u_s = \pm 3\ \text{kV}$ (with a risetime of $2\ \text{ns}$) was applied to the sample from a step generator (STEP GEN). High voltage coupling capacitor C separates dc and step sources. Electrical signals generated in a PVDF transducer, delayed in time ($3.1\ \mu\text{s}$), were measured using a digital storage oscilloscope (DSO), HP 54820A ($500\ \text{MHz}$, $2\ \text{GaSa/s}$). The time domain procedure was applied to analyse the signals using a personal computer (PC).

2.2. Measurements of space charge distribution in a three-layer structure

An electret sample (SAM) was composed of three PTFE foils with thicknesses $d_1 = 50\ \mu\text{m}$, $d_2 = 25\ \mu\text{m}$, and $d_3 = 50\ \mu\text{m}$ (Fig. 2). The outer sample surfaces were covered with Al by evaporation in vacuum. Interfaces between dielectrics were corona-charged with surface charge densities $+3\ \text{mC/m}^2$. The signal u_i measured at oscilloscope (input resistance $R = 50\ \Omega$) resulting from step voltage u_s applied to the sample is shown in Fig. 3, run 1. Within the transit time $t_p = t_6 - t_1 = 110\ \text{ns}$, the 6 peaks indicated as q_{si} can be observed. The 1st and 6th peaks are related to the

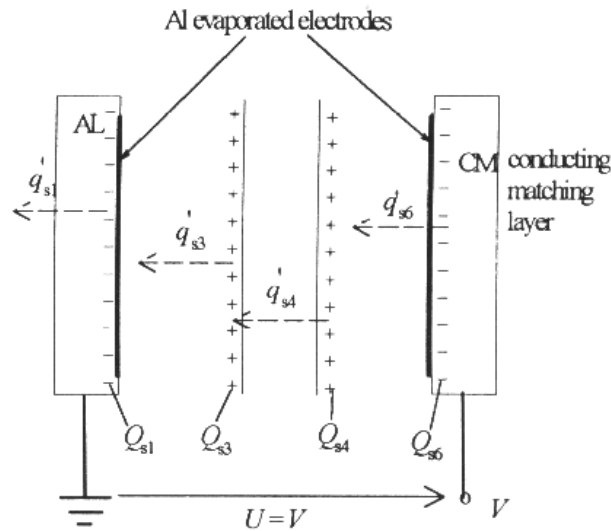


Fig. 2. Propagation of acoustic waves in a PTFE sample with the charges at interfaces.

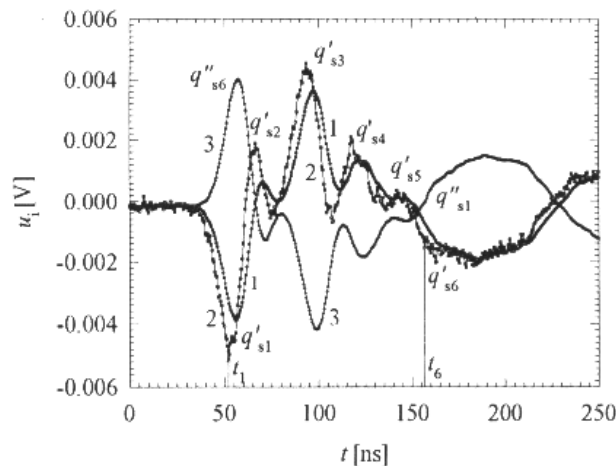


Fig. 3. The SEA responses. The time lines at t_1 and t_6 correspond to the position of electrodes: 1: $u_s = -3 \text{ kV}$, $u_0 = 0$; 2: $50i_{sc}$ (signal 1 after processing); 3: $u_s = 3 \text{ kV}$, $u_0 = 0$, sample converted.

electrode charges. The 3rd and 4th peaks are related to the charges at polymer/polymer interfaces. The origin of the small 2nd and 5th peaks is not clear at the moment. The space charge distribution in the sample is symmetric, so peak amplitudes were expected to be the same. The lower amplitude of the peak q'_{s4} in comparison to q'_{s3} and q'_{s6} to q'_{s1} is a result of acoustic wave damping and their reflections at interfaces.

In the next experiment the same sample was reversed in position, i.e., the sample electrode, which was at AL is now at CM (Fig. 4). Difference in the potential across the sample was not changed. After the sample was reverted, the amplitude of the 1st positive peak, originated from electrode 6 (q''_{s6} in Fig. 3, run 3), became the same as the

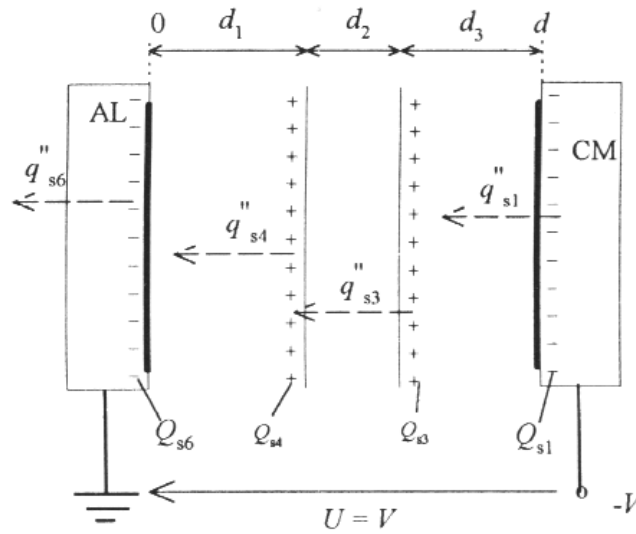


Fig. 4. Propagation of acoustic waves in inverted PTFE sample. The voltage difference between electrodes is the same as in Fig. 2.

amplitude of the 1st negative peak originated from electrode 1 (q'_{s1} in Fig. 3, run 1). The first negative peak originated from electrode q'_{s1} (run 1) is similar to the positive peak from electrode q''_{s6} (run 3, sample converted).

The measured signals u_i are related to short-circuited current i_{sc} (Fig. 3, run 2) of the PVDF transducer. The following relation was used to calculate:

$$Ri_{sc} = u_i + RC \, du_i/dt, \tag{1}$$

where $C = 0.1 \text{ nF}$ is the capacitance of the transducer. The short-circuited current of the thin detector is related to space charge distribution in sample $q_v(z)$ along the sample thickness z according to the relation,

$$i_{sc}(t) = Ku_s q_{v(z=vt)}, \tag{2}$$

where v is the sound velocity in a sample and K is a constant.

2.3. Compensating the effects of attenuation and reflections of acoustic waves

In order to decrease the effects of attenuation and reflections of acoustic waves on the measurement results the following procedure was applied. Signal 3 from Fig. 3 was converted in time according to the relation $u_{i\text{conv}}(t) = u_i(t_1 + t_6 - t)$. Then the positive peak q''_{s6} (run 3, Fig. 3) was located at t_6 , and the positive peak q''_{s1} at t_1 . The other peaks q''_{s2} – q''_{s5} of converted run 3 were located at the times t_2 – t_5 , where the peaks of run 1 appear. The difference between run 1 and converted run 3 of the curve converted in time (not shown) gives signal u_i , in Fig. 5. The processed signal $50i_{sc}$ after the application of Eq. (1) also shown in Fig. 5. The 6 distinct peaks can be seen from signals in Fig. 5.

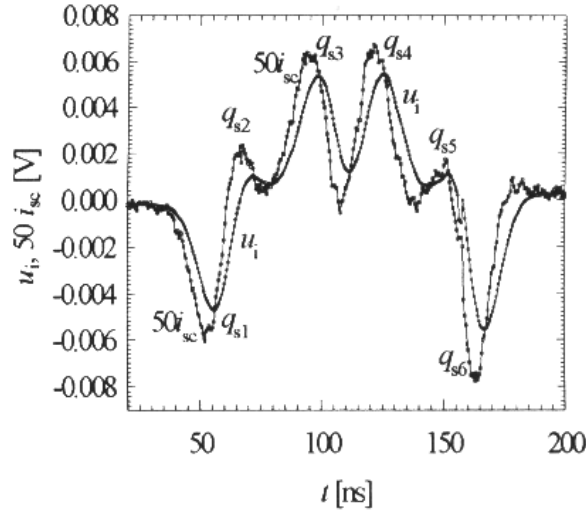


Fig. 5. u_i is the difference between signals measured from one side of the sample from the other side of the sample but after conversion of the second run. $50i_{sc}$ the processed curve related to the short-circuited current.

2.4. Correlation between measured signals to interfacial charges

The validation of the above procedure can be based on a simple model, in which reflections at interfaces play the major role in attenuation of acoustic waves. The attenuation propagation coefficient for PTFE equals 184 m^{-1} at 2 MHz [6]. It is dependent on frequency, but there is no reliable data for higher frequency. Using the above value, the attenuation on the sample thickness can be estimated at about 2%. Therefore, it has no practical meaning. Sample interfaces are not flat but rough. Therefore, the acoustic waves crossing the interfaces due to the reflections and scattering are weaker. The above effects can be evaluated from the relations between measured signals q_{si} and interfacial charges Q_{si} :

$$q'_{s1} = k_q T_{AP} Q_{s1}, \quad (3a)$$

$$q'_{s3} = k_q (1 + \Gamma_{AP}) Q_{s3} / 2 = k_q T_{AP} Q_{s3}, \quad (3b)$$

$$q'_{s4} = k_q T_{AP} T_{PP}^2 Q_{s4}, \quad (3c)$$

$$q'_{s6} = 2k_q T_{AP} T_{PP}^2 T_{PM} Q_{s6}, \quad (3d)$$

where $T_{AP} = Z_{AL} / (Z_{AL} + Z_P)$ is the transmission coefficient at AL/polymer interface, $\Gamma_{AP} = (Z_{AL} - Z_P) / (Z_{AL} + Z_P)$ is the reflection coefficient, $T_{PM} = Z_P / (Z_P + Z_M)$ is the transmission coefficient at polymer/matching electrode, T_{PP} is the quotient of the transmitted to incident wave at polymer/polymer interface and k_q is a constant. Similar equations can be written for the situation in Fig. 4, as an example for signal q''_{s1} shows:

$$q''_{s1} = -2k_q T_{AP} T_{PP}^2 T_{PM} Q_{s1}. \quad (4a)$$

Differences q_{si} between signals q'_{si} and q''_{si} for separate peaks equal:

$$q_{s1} = k_q T_{AP}(1 + 2T_{PP}^2 T_{PM})Q_1, \quad (5a)$$

$$q_{s3} = k_q T_{AP}(1 + T_{PP}^2)Q_3, \quad (5b)$$

$$q_{s4} = k_q T_{AP}(1 + T_{PP}^2)Q_4, \quad (5c)$$

$$q_{s6} = k_q T_{AP}(1 + 2T_{PP}^2 T_{PM})Q_6. \quad (5d)$$

It is necessary that $1 + 2T_{PP}^2 T_{PM} = 1 + T_{PP}^2$ for the signals to have the same weight. It is fulfilled if $Z_P = Z_M$, i.e., the acoustic impedance of polymeric sample equals the acoustic impedance of conducting matching layer CM. This condition was fulfilled in the experiment and $T_{PP}^2 \approx 0.65$. Therefore, the measured peaks are related to the interfacial charges in a sample.

3. Results and discussion

The processed curves (as shown in Fig. 5) were integrated doubly and then they were calibrated. Distribution of potential versus sample thickness is shown in Fig. 6. Next the electric field calibrated profiles were obtained by differentiation of potential profiles (Fig. 7). The space charge density profiles were obtained from the field profiles (Fig. 8). The position was estimated from acoustic transit time through the sample thickness. The interfacial surface charges in the sample obtained by integration of separate peaks are shown in Fig. 9.

Acoustic description of the propagation of pressure waves considers the incident waves only in this work. The oblique waves can also affect the amplitude of the signals. The wave that comes from the charge Q_{s3} and propagates to the right may be reflected at surface 4 and can give a contribution to a signal at time t_5 , that

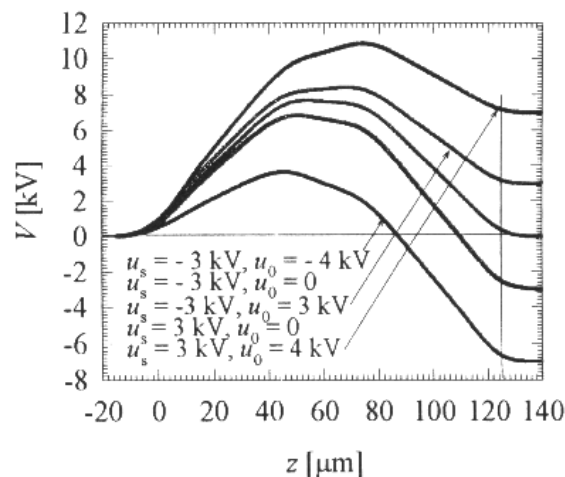


Fig. 6. Potential profiles along the sample width.

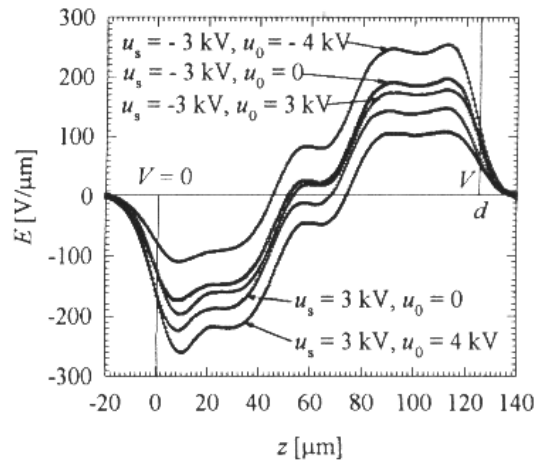


Fig. 7. The electric field profiles.

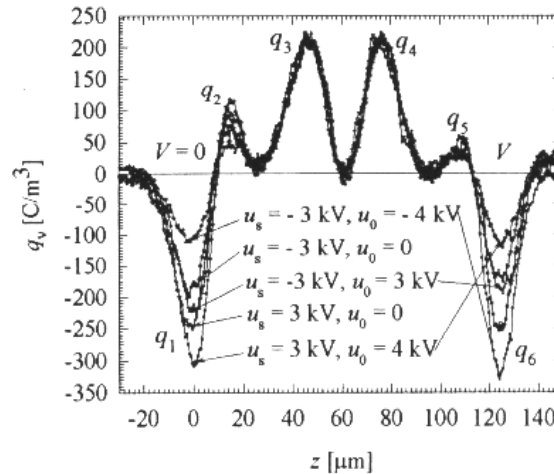


Fig. 8. Space charge density profiles.

corresponds to the peak q'_{s5} . Therefore, the peaks q_{s2} and q_{s5} may originate in part from the oblique waves. But the peaks q'_{s2} and q''_{s5} cannot be an effect of oblique waves but they can come from heterocharges that accumulate near the electrodes. The above assumption is also reflected in Fig. 9. The sum of electrode charge Q_{s1} and heterocharge Q_{s2} at electrode/polymer interface equates the charge $-Q_{s3}$ at $u_s + u_0 = 0$.

4. Conclusions

The two-side SEA technique compensates the effects of acoustic wave reflections at inner interfaces in layered specimens, which increasingly affects the reliability of measurements compared to one-side SEA method. The measuring procedure allowed

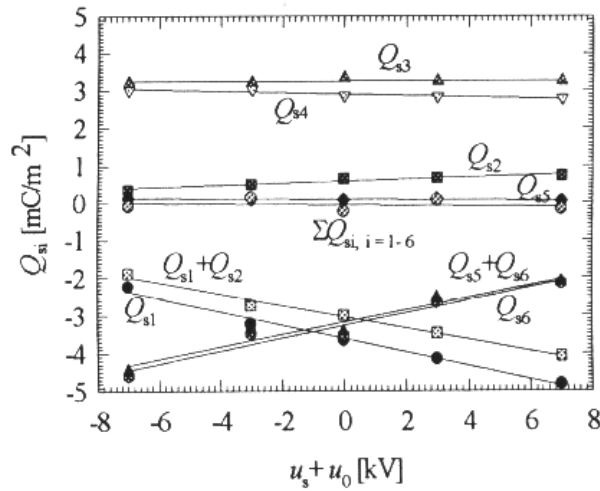


Fig. 9. Surface charge densities at interfaces versus the sum of step and dc voltages.

to obtain the reasonable profiles of space charge, electric field and potential distributions in three layered PTFE specimens with charges at interfaces.

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