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Study of PET corona electrets at atmospheric and lower pressures

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In the present paper poly (ethylene terephthalate) (PET) electrets, 40 µm thick, produced in a corona discharge, were studied. The electrets were charged to different initial surface potentials and were placed into a vacuum chamber under various pressures down to 0.1 mbar for 30 minutes. The surface potential was measured after charging the samples and after removal from the vacuum chamber. The thermo-stimulated discharge currents were recorded for electrets stored at atmospheric and low pressures. The time dependence of surface potential was also studied. The electrets, which have been placed preliminary under low pressure, showed higher time stability at room conditions.

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1. Introduction

PET is one of the polymers, which have been widely used for preparing electrets, because of its important commercial significance and its structure [1-7]. PET can be amorphous or semi-crystalline. Semi-crystalline PET samples can be obtained upon heating the amorphous films at elevated temperatures and subsequently quenching to room temperature or by biaxially stretching.

Producing of PET electrets is usually performed by a thermal method [4-6] or by charging in a corona discharge [3, 6, 7]. The formation of thermo-electrets is carried out by heating polymers to a given temperature, applying a high voltage and cooling to a room temperature. The corona discharge can be performed at room temperature or elevated temperatures and also in various gas media.

Mostly thermo-stimulated discharge current techniques [8] were used for studying the dielectric properties of PET electrets, due to the relative simplicity of the experimental setup and the high resolution of the method [3-6].

The influence of many factors on the electret behaviour has been studied. However, only a few publications were devoted to the influence of low pressure on charge decay of PET electrets [1-2]. The dependence of the charge decay on the pressure reduction has been studied and the charge decay was assumed to be a result of a sparking breakdown in air [2] or a desorption of ions from electret surface [1]. Furthermore, the thermo-stimulated currents in vacuum were recorded for two-sided metalized electrets [5, 9].

The aim of the present paper was to study the low pressure effect on the behaviour of corona charged PET electrets by thermally stimulated discharge currents and surface potential measurements.

2. Experiment

Nonmetalized samples of 40 µm, 36% crystalline poly (ethylene terephthalate) (PET) film were studied. The PET film was initially cleaned in alcohol in an ultrasonic bath for 4 minutes, rinsed in distilled water, and dried under room conditions. 30 mm diameter samples were cut from the clean film. All the samples were put onto the same diameter metal pads.

The charging of the samples in a corona discharge was carried out by means of a conventional corona triode consisting of a corona electrode (needle), a grounded plate electrode and a grid placed between them. The distance between the corona electrode and the grid was 10 mm and the distance grid to grounded plate electrode was 3 mm. The samples together with their metal pads were placed on the grounded plate electrode and were charged for 1 minute under room conditions. The voltage of the corona electrode was U_c=-5 kV. The electret surface potential was limited by the grid potential. Charging of electrets was performed under room conditions.

After charging the electret surface potential V_0 was measured by the method of the vibrating electrode with compensation, by which the estimated error was better than 5%.

Three groups of tests were performed. In the first group of tests after charging the electrets were placed into a vacuum chamber, consisting of isolated bases and a jar bell. Electrets were kept under a determined low pressure for 30 minutes. After that the electrets were removed from the vacuum chamber, their surface potential V was measured again and the normalized surface potential V/V_0 was calculated.

In the second group of tests the dependences of the electret surface potential on the time of storage under room conditions were studied for a month for two sets of samples. For the first set the electrets were stored under room conditions after producing and for the second one they were preliminary placed under a pressure of 0.1 mbar for 30 minutes.

In the third group of tests the thermally stimulated currents (TSC) spectra were recorded from a room temperature up to 160 ^{o}C for the two sets of samples. The short circuit TSC were measured with a voltmeter VA-j-52 (RFT) in a programme controlled oven. The heating rate was 1.95 K/min.

3. Results

For the first group of tests the dependences of the surface potential on the electret storage pressure for five sets of PET samples, charged in a negative corona were investigated. The samples were charged to the initial surface potentials limited by the grid potential values: -350 V, -500 V, -650 V, -800 V and -950 V respectively. Each sample set was studied within the pressure range from 1000 mbar to 0.1 mbar.

The dependences of normalized surface potential V/V_0 on normalized pressure p/p_0 for PET electrets charged in a negative corona are presented in Fig. 1. V_0 denotes the initial value of surface potential measured just after charging the electrets, and p_0 is the atmospheric pressure. Each point in the figure is a mean value from six samples. The error bars mark the confidence intervals for the mean values with 90% confidence level.

The results represented in Fig. 1 show that for each set of samples there is a narrow region of pressures, in which the surface potential sharply decays. The potential sharply decay region is shifted to the lower pressures when the initial surface potential value decreases. It can be also seen that under pressure less than 1 mbar the surface potential stays constant excepting the case of -350 V initial surface potential.

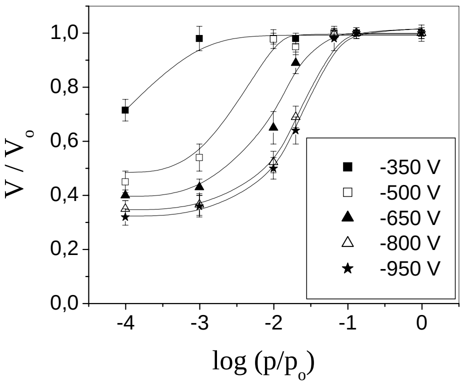

Fig. 1. Dependence of the surface potential on pressure for PET films negatively charged to different initial surface potential V_0 *.*

For the second group of tests the dependences of normalized surface potential on the time of storage under room conditions are represented in Fig. 2. The error bars mark the confidence intervals for the mean values with 90% confidence level.

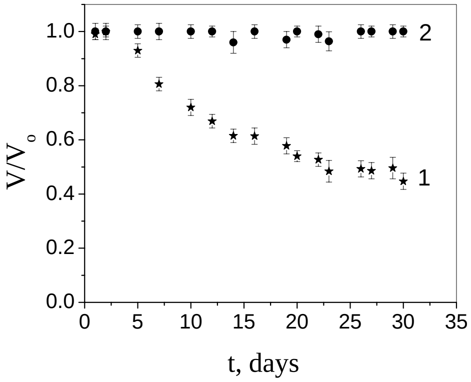

Fig. 2. Time dependence of surface potential for negatively charged PET films.

Curve 1 corresponds to the electrets which were stored under room conditions after producing. Curve 2 corresponds to the samples which were preliminary placed for 30 minutes under a low pressure of 0.1 mbar. The surface potential measured just after removal of the electrets from the vacuum chamber is assumed to be the initial value. It can be seen that the electrets, which preliminary have been placed under low pressure, are more stable.

For the third group of tests the dependences of the short-circuit thermally stimulated currents for PET samples charged in a negative corona are depicted in Fig. 3. Curve 1 shows the results for samples, the current of which is recorded just after charging them. Curve 2 presents the results for samples preliminary placed under low pressure of 0.1 mbar for 30 minutes. TSC was registered after removing the samples from the vacuum chamber.

As it can be seen from Fig. 3 both curves have two peaks. The first peak is in the negative range at temperature about 80 ^{o}C and for the samples preliminary stored under 0.1 mbar pressure is narrower and shifted to a higher temperature. The second peak is observed in the positive range at a temperature around 120 ^{o}C and practically coincides for the two curves.

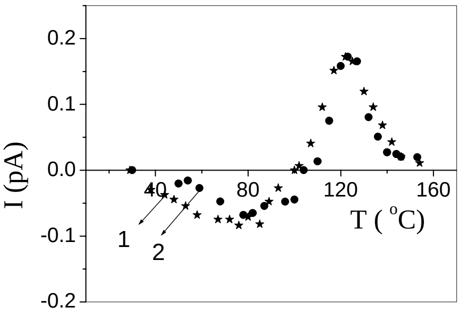

Fig. 3. Thermally stimulated current spectra for negatively charged PET films.

4. Discussion

The results displayed in Fig. 1 show that the sharp surface potential decay region depends on both initial surface potential and pressure under which the electrets were kept. It was shown [10] that for PP and PTFE electrets only one generalized curve could be drawn to represent the dependence of the normalized surface potential on the ratio p/V_0. Such generalized curve for the results displayed in Fig.1 is plotted in Fig. 4 with the coefficient of determination $R^2 = 0.92$ (the values at grid potential -350V are not included in the Fig. 4). In Fig. 4 the dependence of the normalized surface potential V/V_0 on the normalized ratio $(p/V_0)^* = (p/V_0)/(p_0/V_0^*)$, where p_0 is atmospheric pressure and $V_0^* = 1000\,\mathrm{V}$, is represented. The error bars mark the confidence intervals for the mean values with 90% confidence level.

From Fig.4 the midpoint of the range within which sharp surface potential decay occurs could be calculated and the following value is obtained: 0.019 mbar/V. It should be noted that in ref.[10] the values 0.024 mbar/V and 0.022 mbar/V for PP and PTFE electrets respectively are reported. Therefore, the values for PET, PP and PTFE are of the same order and differ for different sample materials.

Fig. 1 and Fig. 4 show that at a pressure less than 1 mbar the surface potential stays practically constant. That is why atmospheric pressure and 0.1 mbar pressure were chosen for the next experiments.

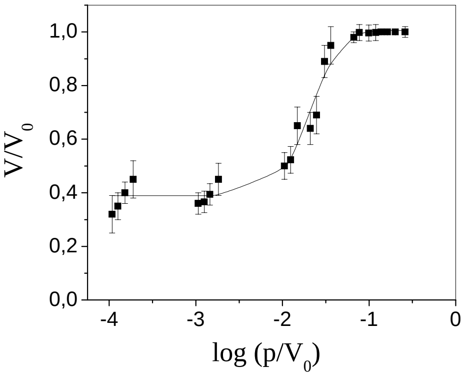

Fig. 4. Generalized curve for negatively charged PET films drawn for the data of Fig. 1.

As it could be seen from Fig. 2 the surface potential for the electrets preliminary kept at 0.1 mbar holds the same value for a month under room conditions. At the same time the surface potential decreases with time for samples which were not preliminary kept at low pressure. According to [7] phenolic – OH, carbonyl and carboxyl acid (COOH) have been identified as the functional groups incorporated onto the surface and low-molecular-weight oxidized materials have been also observed in the form of a globular morphology on the surface of the PET film electrets. Hence, charge traps of different depths were available on the electret surface. When the electrets were kept under low pressure liberation of charges from shallow traps occurred and no charge detrapping was observed during subsequent keeping under room conditions. Therefore, placing the electrets initially under low pressure for one month can be used as a method for stabilizing the electret charges.

The two thermo-stimulated current spectra, presented in Fig. 1 show two well distinguished peaks – a negative low-temperature broad peak in the temperature range from 30 ^{o}C to about 100 ^{o}C and a positive higher-temperature peak in the temperature range from 100 ^{o}C to 140 ^{o}C. The low-temperature broad peak is believed to be due to a charge release from traps and charge dissipation as temperature increases. One can expect that the thermo-stimulated current peak for electrets which have been preliminary stored at pressure of 0.1 mbar has to shift to higher temperatures.

The mean activation energies for the low-temperature peaks were calculated by the initial rise method. Their values were: 0.46 eV for the electrets preliminary kept at 0.1 mbar pressure for 30 minutes and 0.31 eV for the electrets, thermo-stimulated current of which was recorded just after charging. The calculated activation energies showed that when electrets were preliminary kept at 0.1 mbar low pressure charge detrapping from shallow traps occurred and that was why the mean activation energy increased.

With regard to the second higher temperature peaks at about 120 ^{o}C their magnitudes and positions were the same for the two curves and were not depend on pressure. It was most likely to be due to cold crystallization but this assumption should be verified.

5. Conclusions

In the present paper nonmetalized samples of 40 µm, 36% crystalline poly (ethylene terephthalate) (PET) films were studied at atmospheric and lower pressures. Three groups of tests were performed: the behaviour of electrets under reduced pressures; the time dependences under room conditions for electrets preliminary kept at 0.1 mbar pressure and electrets which did not placed under low pressure, and TSC spectra within the temperature range from room temperature to 160 ^{o}C.

The results obtained for PET electrets confirm the supposition advanced for PP and PTFE that the surface potential decay depends on the ratio of pressure to initial surface potential.

When the electrets were kept under low pressure liberation of charges from shallow traps occurred and no charge detrapping was observed during subsequent keeping under room conditions. The TSC spectra showed two well distinguished peaks. The low temperature peak was believed to be due to a charge released from the traps. The calculation of activation energies by the initial rise method confirmed the assumption that under low pressure charge detrapping from shallow traps occurred and the mean activation energy was increased. Therefore, preliminary placing the electrets under pressure less than 1 mbar can be used as a method for stabilizing the electret charge.

Acknowledgements

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