Optimization of electret film forming method

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Abstract. The aim of this investigation was to develop a method of fabrication of electrets made from PTFE foil of a 0.5 mm thickness, from Ensinger Gmb H. Parameters allowing assessment of the electrets in terms of technical applications are: charge lifetime $\tau$ and equivalent voltage $U_z$. Measurements of electrets equivalent voltage $U_z$ has been carried out using compensation method. Assessment of lifetime required employing a method utilizing thermal stimulation of charge relaxation process. In the investigation, a continuous measurement of equivalent voltage in the condition of linear increase in temperature - $\text{TSU}_z$ was realized. It was stated that electret properties of a foil depend on the preparation conditions of the material and the forming temperature. The employed method of lifetime measurement enables verification of the applied procedure.

1. Introduction
For practical applications, electrets from polymeric materials are made by implantation of electric charge from an external source [1]. In the literature, there are described the methods for stability improvement of collected charge by chemical modification of polymer surface before the forming process or its treatment with a low-temperature plasma. Fabrication of electrets at elevated temperatures is also employed. A chemical modification of PTFE foil, 13$\mu$m, was reported in [2]. The modification was carried out by using tetraethoxysilane, tetrabutyl titanate and orthophosphoric acid. After modification, the foil was formed in corona discharge in an air triode system. Measurements of thermo-stimulated depolarization were performed at a linearly increasing temperature with a rate of 5°C/min. Chemical modification of the polymer led to an improvement of stability of the collected charge. An improvement of electret charge stability was also obtained by a treatment of the polymer surface with plasma of 20 W [3]. The plasma was developed in O$_2$/He, O$_2$ and H$_2$ atmosphere, at a supply voltage frequency of 13.56 MHz. The samples were formed in an air triode system. In [4], a method of electrets preparation from PTFE foil formed at temperatures of 30°C, 90°C and 150 °C, at a voltage of the corona electrode of 12 kV was presented. The tests of charge stability depending on temperature were performed after five-day storage of electrets in ambient environment. It was stated that the properties of electrets from PTFE foil depend on the forming temperature. In the paper, the effects of temperature and aging time of samples before the forming process and the temperature of charge implantation on the electrostatic properties of electrets have been presented.

2. The method of electrets forming
The investigations were carried out on samples made from PTFE foil of a 0.5 mm thickness, from Ensinger GmbH. The electrets were formed using corona discharge in an air triode system. A scheme of the set-up for forming the electrets is shown in Fig. 1. A sample with a one-side deposited electrode
was placed at a distance of 42 mm from a corona electrode with a radius of 50 μm. A grid was positioned at a distance of 10 mm over the sample surface.

Figure 1. A set-up for electrets forming.

Due to the need of deposition of an electrode on one of the PTFE surfaces, a chemical modification of this surface has been performed to improve its adhesive properties. The deposited electrodes were characterized by satisfactory adhesion to the substrate, both at the stage of forming at elevated temperature and during the thermo-stimulated tests. The electrode of the sample was grounded through a picoammeter as it has been shown in Fig. 1. The forming of electrets was carried out in air at room temperature (R.T.) T_0 and at temperatures of 100 and 150 °C, at a constant grid voltage U_s=1400 V and corona discharge current of 5 μA. Corona discharge voltage was set as +13kV. Before the forming process, the samples were aged for one or four hours. Temperature diagram of the sample before and during the forming process of the electrets at 100°C and 150°C is shown in Fig. 2. The sample was placed on the measuring setup where it was heated up to an assumed temperature for 30 min, and then annealed at this temperature for one or four hours (section t_1–t_2). After, the process of forming started, which was then continued until cooling of the sample. For all temperatures the same geometrical parameters of the measuring stand were maintained.

Figure 2. Diagram of temperature change before and during the forming process of electrets versus time.
3. Method of assessment of electret lifetime

Assessment of lifetime of charge \( \tau \) requires determining the dependence of electrets equivalent voltage at linearly increasing temperature [5]. An exemplary characteristics of thermo-stimulated equivalent voltage of electret with indicates characteristic parameters, necessary for lifetime assessment is shown in Fig. 3.

![Figure 3](image)

**Figure 3.** Characteristics of charge decay in PTFE electrets versus temperature.

The run of change in the charge (equivalent voltage) can be described by the Turnhout equation [6]:

\[
q_{SA}^{T(t)}(T) \approx q_{SO} \exp \left[ -\frac{kT^2}{bW\tau_0} \exp \left( -\frac{W}{kT} \right) \right]
\]  

where:
- \( q_{SA} \) – effective density of surface charge,
- \( q_{SO} \) – initial value of homocharge,
- \( b \) – rate of temperature change during investigation,
- \( W \) – activation energy,
- \( k \) – Boltzmann constant (1.38x10\(^{-23}\) J/K),
- \( \tau_0 \) – a constant,
- \( T \) – temperature.

Characteristics \( q_{SA}^{T(t)}(T) \) (Fig. 3) shows an inflection point at temperature \( T_n \). By determining tangent to the curve at the inflection point it is possible to establish the activation energy from the following equation [6]:

\[
W = -\frac{kT_n^2}{q_{SA}} \frac{dq_{S}^{T(t)}(T)}{dT} \bigg|_{T=T_n}
\]  

where:
- \( k \) – Boltzmann constant (1.38x10\(^{-23}\) J/K),
- \( T_n \) – temperature at inflection point,
- \( U_{in}(T_n) \) – voltage at inflection point,
- \( \tan \alpha \) - tangent to the curve at inflection point.
For the data obtained from the characteristics shown in Fig. 3 (value of voltage at inflection point \( U_n = 707 \text{ V} \), temperature at inflection point \( T_n = 570 \text{ K} \)), tangent to the curve at the inflection point and activation energy have been determined as \( \tan \alpha = -33.33 \text{ V/K} \) and \( W = 1.35 \text{ eV} \), respectively. It has been stated that the lifetime of a charge implanted to dielectric can be assessed based on equation (3), assuming that \( T_e \) is a temperature at which the equivalent voltage \( U_z \) takes the value of \( U_{z\text{max}}/e \), \( U_{z\text{max}} \) is the maximum value of equivalent voltage and \( e \) is a base of natural logarithm [6]:

\[
\tau(T) \approx \frac{k T_e^2}{b W} \exp \left( \frac{W}{k} \left( \frac{1}{T} - \frac{1}{T_e} \right) \right)
\]

where:
- \( T_e \) – temperature, at which \( U_z \) takes the value of \( U_{z\text{max}}/e \),
- \( T \) – temperature,
- \( b \) – rate of temperature change during investigation, \( b = 0.033 \text{ K/s} \),
- \( W \) – activation energy,
- \( k \) – Boltzmann constant

The lifetime of the electret charge, based on the data shown in Fig. 3 was evaluated as \( 10^6 \) years.

4. Results of measurements
The characteristics of equivalent voltage decay of the charge of electrets made from PTFE foil of 0.5 mm thickness, annealed for four hours at a given temperature and formed either at room \( T_0 \) or annealing temperature (Table 1) are shown in Fig. 4. The activation energy and lifetimes \( \tau \) have been collected in Table 1.

![Figure 4. Equivalent voltage versus temperature for different temperatures of electrets forming.](image)

The obtained characteristics provide information about stability of the charge collected in the electret material while the analysis of the decay rate allows us to assess the charge lifetime in the electrets [7].
Table 1. Activation energy and lifetime of electrets

| No. | Annealing temperature $T_w$ [°C] | Annealing time [min] | Forming temperature $T_f$ [°C] | $T_n$ [K] | $\eta \alpha$ [V/K] | $W$ [eV] | $T_e$ [K] | $\tau$ (days) |
|-----|---------------------------------|----------------------|---------------------------------|----------|-----------------|--------|---------|--------------|
| 1.  | -                               | -                    | $T_o$                            | 543      | 8               | 0.35   | 563     | 14.11        |
| 2.  | 100                             | 240                  | $T_o$                            | 563      | 22.2            | 1.34   | 568     | $2.77 \times 10^8$ |
| 3.  | 100                             | 60                   | 100                              | 533      | 10              | 0.34   | 556     | 13.05        |
| 4.  | 100                             | 240                  | 100                              | 583      | 30              | 1.46   | 584     | $3.97 \times 10^9$ |
| 5.  | 150                             | 60                   | 150                              | 566      | 10              | 0.38   | 566     | 25.37        |
| 6.  | 150                             | 240                  | 150                              | 587      | 40              | 1.47   | 571     | $3.59 \times 10^9$ |

On the basis of the results collected in Table 1, we can draw the following conclusions:

- Four-hour annealing of the samples at elevated temperature preceding the forming process led to obtaining electrets with a long lifetime (items 2, 4, and 6 in Table 1),
- The value of annealing temperature in the four-hour annealing almost has not changed the resulting lifetime,
- One-hour annealing, independently of the value of temperature, has not extended the lifetime. The lifetime of these electrets is comparable with the lifetime of electrets formed at room temperature, without preliminary annealing.

5. Conclusion

In the paper, results of investigation of selected electrostatic properties of electrets made from PTFE foil of 0.5 mm thickness have been presented. The effects of temperature and annealing time of the samples before the forming process as well as temperature at which charge implantation took place, on the lifetime of the electrets have been analyzed. It has been stated that:

- The lifetime of electrets made from PTFE foil of 0.5 mm thickness depend on the time of samples annealing before their forming,
- The lifetime of the electrets practically does not depend on the temperature of long-term annealing.

6. References

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