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Effects of temperature on electromechanical properties and ultrasonic performance of piezoelectric cellular PP films

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ABSTRACT

Ferroelectrets are a recent addition to the family of piezoelectric polymers. Ferroelectrets are space-charge electrets with a heterogeneous and usually cellular foam structure. They are flexible, available in large areas and show strong piezoelectric response. Due to their low acoustic impedance they have strong application potential in air-borne ultrasonic transducers. Here, the influence of temperature on the electromechanical and ultrasonic properties of polypropylene (PP) ferroelectrets was addressed. PP ferroelectrets were subjected to repeated temperature variations. The characterization was performed by means of dielectric resonance spectroscopy (DRS) and Laser-Doppler Vibrometry (LDV). Profound variations in the piezoelectric properties during thermal cycling were observed and correlated to the changes in the elastic properties. Exposure to elevated temperatures resulted in a reduction in the piezoelectric response and an increase in the elastic stiffness constant which also shifted the thickness-extension resonance peaks towards higher values. Large differences in piezoelectric properties were discovered between the first and the subsequent cycles. To decrease the influence of temperature cycling on the piezoelectric properties of PP ferroelectrets, annealing was used to advantage. Annealing treatments with different durations and different annealing temperatures were investigated. A particular procedure yielding foam stiffening and a sufficiently high piezoelectric activity was determined and was employed for further investigations. The piezoelectric properties of the annealed ferroelectrets varied much less when compared to non-annealed ferroelectrets. Characterization of thermally cycled annealed and non-annealed films revealed relatively homogeneous phase and amplitude distributions of the surface-vibration below the resonance, which resembles a piston-like response. Piston-like response was not significantly deteriorated by thermal cycling below the resonance frequency. This was corroborated for all samples by measuring the acoustic directivity of them in the farfield, at frequencies below the resonance. At the resonance, much higher distortions of the response were observed as revealed from the phase images.

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1. Introduction

Piezoelectric materials are well known and widespread in sensors, piezoelectric motors, ultrasonic transducers and a plethora of other applications. The electromechanical properties of piezoelectric materials are strongly affected by temperature. Piezoelectric ceramics are one of the most widespread piezoelectric materials which show thermal stability over broad temperature range [1,2]. Piezoelectric polymers [3] are inferior to piezoelectric ceramics in terms of thermal stability. However, piezoelectric polymers offer distinctive advantages such as flexibility, resistance towards mechanical stress or processing from the liquid state.

A very recent addition to the family of the piezoelectric polymers are ferroelectrets, which are typically space-charge electrets with a heterogeneous, usually cellular structure [4,5]. Showing strong piezoelectric response and being affordable at low cost, large areas and showing typical advantages of polymers (lightweight and flexible), they have attracted considerable scientific attention. Thermal stability of ferroelectrets is mainly governed by the electret properties of the matrix material. Ferroelectrets have been produced from various polymers, such as thermoplastic cycloolefin compounds (COC) [6,7], poly(ethylene terephthalate) [8], polyethylene-naphthalate [9], fluoroethylenepropylene [10] or polycarbonate [11,12]. However, charged cellular polypropylene (PP) electrets have so far received the biggest part of scientific

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attention and were commercialized. In addition, cellular PP ferroelectrets are particularly well suited as airborne ultrasound transducers due to their strong piezoelectric response, very low acoustic impedance and resonance frequencies falling into the lower ultrasound region [13–17].

Polymer sensors are subjected to wide range temperature fluctuations in real-life conditions. Polymers soften at elevated temperatures [18]. Consequently, electromechanical [19–21] and ultrasonic properties [22–24] of piezoelectric polymers are strongly influenced by temperature. The influence of temperature on electromechanical properties of cellular PP has been reported to some extent [19–21,25–30]. However, studies on the electromechanical and ultrasonic properties of PP ferroelectrets after exposure to repeated temperature changes are still lacking.

PP ferroelectrets were subjected to repeated temperature variations and the piezoelectric properties were obtained by means of the dielectric resonance spectroscopy (DRS). Profound variations in the piezoelectric properties during thermal cycling were observed and correlated to the changes in the elastic properties. Large differences in piezoelectric properties were discovered between the first and the subsequent cycles. To decrease the influence of temperature cycling on the piezoelectric properties of PP ferroelectrets annealing was used to advantage. Annealing treatments with different durations and different annealing temperatures were investigated. A particular annealing procedure was employed for further investigations. Annealed piezoelectric films were subjected to thermal cycling and characterized at room temperature before and after each thermal cycle. Results were compared to the non-annealed, thermally cycled transducer films. The piezoelectric properties of the annealed ferroelectrets varied much less when compared to non-annealed ferroelectrets. Laser Doppler Vibrometer (LDV) characterization revealed a rather uniform phase and amplitude distribution of the surface vibration points for all the piezoelectric films at frequencies below the resonance. Piston-like response was not significantly deteriorated by thermal cycling below the resonance frequency. This was corroborated for all samples by measuring the acoustic directivity of them in the far field, at frequencies below the resonance. At the resonance, significant increase in the surface vibration velocity was observed. Simultaneously, much higher distortions of the response were observed as revealed from the phase images. The effect of the temperature cycling on the homogeneity of the resonance distribution was clearly acknowledged.

2. Experimental

Samples were prepared from commercial cellular polypropylene PP films (type VHD40, Treofan, Germany) with an initial thickness of 40 mm and initial density of 600–650 kg/m^3. Samples were expanded in order to optimize the electromechanical properties [31]. An expansion pressure of 50 bar and a time under pressure of 30 min were used for all expansion procedures. After this, samples were kept in an oven (model T6060, Heraeus, Germany) at 60 °C for 30 s. Expanded cellular films were metalized on both sides with round aluminum electrodes of 100 nm thickness and 16 mm diameter. Electrical charging of the samples was performed in a direct contact (−7 kV) by means of a high voltage generator (model PNC30000, Heinzinger, Germany).

Electromechanical properties were obtained from the measurement of piezoelectric resonances by means of dielectric resonance spectroscopy (DRS). The dielectric spectra were recorded on freely vibrating samples by means of a precision impedance analyzer (model Alpha, Novocontrol, Germany) over the frequency range of 0.01–1.5 MHz and at different temperatures by means of a temperature control system (model Quatro, Novocontrol, Germany). From the frequency-dependent real part of the capacitance, the thickness-extension resonance frequency $f_{\mathrm{r,TE}}$, the elastic stiffness constant c_{33} and the piezoelectric d_{33} coefficient were obtained via a least squares fit [32].

Thermal treatment of piezoelectric films was performed in the range from −60 to 100 °C. Thermal cycling was performed in the temperature range from −60 to 80 to −60 °C. Temperature was changed in 10 K steps with a 0.5 K/min gradient. During the annealing, samples were placed in a vacuum oven (model T6060, Heraeus, Germany) preheated to the defined temperature.

The velocity profile of the transducer prototypes was measured using a laser Doppler vibrometer – LDV (OFV5000 –OFV 505, Polytec Inc., Germany) while excitation was carried out using a chirp signal with a frequency content from 20 to 800 kHz [17]. The samples were glued on a flat solid aluminum substrate (used as lower electrode) by means of a Z conductive plastic tape (50 μm thick). The inverse d_{33} coefficient which was obtained from LDV measurements is calculated as:

$$d_{33} = \frac{t(f)}{V_{ac}(f)}$$

where, t is the vibration amplitud at a given frequency f of operation. V_{ac} is the corresponding alternating voltage applied to the sample in frequency domain. The amplitude of the vibration is obtained by dividing the velocity spectrum from LDV measurement by the angular frequency:

$$t(f) = \frac{v(f)}{2\pi f}$$

$v(f)$ is the frequency domain version of the obtained time response of the sample to the chirp excitation signal.

Acoustic directivity of the samples was measured using a calibrated microphone (1/8 in, 4DP, GRAS, Denmark), which has a flat frequency response between 10 Hz and 140 kHz, located on principal axis at 30 cm from the transducer. A rotation unit allowed us to sample the beam at angular steps of 1°. At frequencies higher than 140 kHz the microphone sensitivity decreases. In spite of this, it is still feasible to use the microphone to obtain the normalized directivity of the samples at those frequencies. A low pulse repetition frequency was employed to avoid the creation of a standing field. The emitting sample and the microphone were located sufficiently high from the floor level to avoid echoes bouncing from the floor to the microphone.

Measured far field radiating pattern of the samples was compared to that of an ideal circular baffled piston, which can be calculated according to:

$$D\left(\theta\right) = \frac{2J_1\left(ka\sin\theta\right)}{ka\sin\theta},$$

where k, a and θ are, respectively, the wavenumber, the radius of the piston and the angle at which the directivity pattern is calculated. J_1 is the Bessel's function of first kind and order 1.

3. Results and discussion

The influence of temperature on the response of the piezoelectric cellular PP film in the temperature range from −60 to 100 °C is shown in Fig. 1. Upon temperature increase from −60 °C up to approximately room temperature, a slow but steady increase of piezoelectric activity was measured. The increase in the piezoelectric response was due to the softening of the material, which will be discussed later. A much stronger increase in the piezoelectric response was obtained in the temperature range from 20 to 70 °C. The peak of piezoelectric response was reached at about 70 °C. At even higher temperatures, a steep decline in piezoelectric activity was observed. A similar trend was reported for commercially

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