A New Route to Polymeric Transducers: 3D Printing of Polypropylene Ferroelectrets

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ABSTRACT

In the quest for new functional materials, ferroelectrets appeared in the late 1990s as a great surprise. Ever since, new preparation schemes and materials geometries have been proposed and demonstrated with several enhancements. Now, another promising approach for producing such materials is presented. Using 3D-printing technology and polypropylene (PP) filaments we were able to print, a two-layered film strucutre with regular cavities precisely distributed. Samples were prepared in a single printing process, i.e. without other printing steps, and their transducer behavior over time and at various temperatures and pressures were investigated in order to better understand the specific properties of the new structure and the applications-relevant effects of 3D printing and processing. Results revealed that the samples produced by this technique can exhibit a piezoelectricity similar to other PP ferroelectrets i.e. up to 200 pC/N, and that their temporal and thermo behavior are also in agreement with those better known samples. Finally the pressure analysis shown that these samples present a major sensibility around 20 kPa and 35 kPa, which is characteristic of the 3D samples.

Index Terms — 3D printing, polymer ferroelectrets, sensors and actuators, piezoelectrets, electret polymers, soft electro-active materials, functional materials, soft matter

1 INTRODUCTION

PIEZOELECTRICITY is a well-known phenomenon that has been observed on a variety of materials [1, 2] including crystals, ceramics, wood, bone and polymers. A symmetrybreaking polarization in the crystalline unit cell of a polar material or in the molecular chain of a natural or synthetic polymer can produce a direct as well as an inverse effect: Either an electrical response is observed when an external pressure is applied (direct effect), or a mechanical deformation is obtained as response to an electrical stimulus (inverse effect) [1]. The intrinsic piezoelectricity of several crystalline, ceramic and polymeric materials led to a range of applications in pressure sensors, acoustic transducers, hydrophone probes, ultrasonic microscopy, etc. [3]. As piezoelectricity is a tensor property that depends on the directions of electric polarization and mechanical stress or strain, the longitudinal effect in the thickness direction of a horizontal slab/film is maximized if both, molecular orientation (polar axis) and mechanical deformation are along the vertical *z*-axis [4]. In this case, the relevant tensor component is the piezoelectric d_{33} coefficient [1]. High d_{33} coefficients, on the order of hundreds of pC/N, have been observed only on a few materials, mainly piezo-ceramics [5]. Piezoelectric polymers, such as polyvinylidene fluoride (PVDF) and some of its copolymers, show lower piezoelectric d_{33} coefficients (typically around 25 pC/N), but also attractive properties such as low weight and high flexibility [4].

A second class of piezoelectric polymers emerged during the 1990s and is commercially known as electromechanical film (EMFi) [6]. The novel materials present a new paradigm for piezoelectricity and are now usually called *ferro- or piezoelectrets* [7-9]. Ferroelectrets have significantly enhanced our basic understanding of piezoelectricity, as they are either non-polar foamed polymers or polymeric structures with well-

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defined cavities, which received electrical poling. The standard example and still the "work horse" of ferroelectret research and transducer development are physically foamed polypropylene (PP) films whose piezoelectricity is caused by the macroscopic polarization resulting from space charges trapped on the inner surfaces of the foam cells [7-9]. The low density and the very low elastic modulus in the thickness direction of suitably prepared PP foams yield large anisotropic deformation under pressure resulting in piezoelectric d_{33} coefficients comparable to those of piezo-ceramics.

Commercially available PP-foam ferroelectret film, known as EMFi, which combines high piezoelectricity with softness, has taken a significant place in biomedical applications, such as measurements of cardiorespiratory signals [10]. However, the relatively low thermal stability of PP somewhat limits their practical application [11]. Furthermore, it has been shown that the sensitivity of the EMFi material varies across the film area, due to the random cellular structure of the material itself, as a consequence of the manufacturing process [12]. Consequently, the evolution of ferroelectrets went mainly in the direction of materials that are thermally more stable, such as fluoropolymers, and of template-based structures that are easier to control [13-17]. However, the use of the more difficult-toprocess fluoropolymers also led to new preparation routes for well-defined cavity structures [15-19]. The well-controlled cavity geometries proved to be much more uniform, which facilitates electric charging and allows tuning the frequency response of devices [19-21]. Thus, it also led to a better understanding of the charging process [20].

A very promising approach for preparing ferroelectrets with well-defined cavities was recently demonstrated with the successful 3D-printing of acrylonitrile-butadiene-styrene (ABS) polymer structures that are in principle suitable as ferroelectrets [22]. The procedure consisted of printing two separate thin films- one containing ridges ring-shapes on a flat surface and the other one being a single flat film. The ABS films, 3Dprinted from BS filaments, were joined together to form a 2layer sandwich with internal cavities. After electrical charging, the sandwich structure exhibits a piezoelectric behavior similar to that of typical ferroelectrets. However, the complicated multistep preparation procedure and the use of the ABS polymer with its low thermal charge stability have resulted in difficult-tocontrol structures and in electrically rather unstable ferroelectrets, respectively. The 3D-printing method for fabricating ferroelectrets was further improved using PP filaments in a single-step printing process [23]. In such method, the electric dipoles were formed by non-controlled gaps between two orthogonal printed layers.

In continuation of this seminal work, we introduce a new preparation route for 3D-printed ferroelectrets with PP and a single printing step. The main difference is that a third middle layer was added to enhance cavities control. Temporal-and thermal-stability studies of the piezoelectric behavior of the 3D-printed and electrically charged/poled PP ferroelectrets were carried out for comparison with well-known PP-foam ferroelectrets. In addition, the static-pressure dependence of the resulting piezoelectric d_{33} coefficients was investigated, for various sensor and actuator applications.

2 3D-PRINTED POLYPROPYLENE FERROELECTRETS

3D-printing first requires a virtual model of the object to be replicated or created – usually obtained by means of a suitable computer-aided design (CAD) software. Second, materials-specific 3D-printing parameters such as temperatures, speeds and filling patterns need to be implemented into the software that controls the printing process. For *electro-active* polymers, the process is not very different from conventional 3D-printing for passive use.

As a first step, a digitized model of a square film/slab with side lengths of 40 mm each and with a height of 300 μ m was designed with the software Autodesk Inventor. After defining the exact geometry of the desired structure, the Simplify 3D program was chosen for controlling the printing due to its ease-of-use parameters setup, which allows print layers with different geometrical parameters, leading to a vast range of possibilities in creating distinguished cavity patterns. As reported previously, it is particularly important to control the shape size and distribution of the cavities in ferroelectrets, as their electrical charging, resonance frequencies and resulting piezoelectricity depend on the cavity dimensions [21, 24].

The printing parameters were adjusted to produce two solid outer layers with 100 μ m thick, completely filled with material and an additional central layer, also 100 μ m thick, with a fill factor of 80%, printed in a triangular pattern as schematically shown in Figure 1. Figures 2a and 2b show micrographs of real 3D-printed PP structures with the triangular pattern. Figure 2c depicts a typical cross-section with two cavities.



(a) (b) (c) **Figure 1.** Schematic representation of the layer sequence for the 3D printing process: (a) bottom layer, (b) central layer with a fill factor of 80% (triangular pattern) and (c) top layer (partially cut away to expose layer b).



Figure 2. (a) Top view of the bottom and central layers, showing the triangular patterns of the central polypropylene layer. (b) SEM image (top view). (c) Cross section showing two of the 3D-printed cavities.

For 3D printing, PP filaments from 3DX filaments (Brazil) with a diameter of 1.75 mm (3DX filaments) were fed into a 3D printer (model GTMax 3D Core A1 with a printing tip of 0.4 mm). It is important to note that the samples were printed in a one-step process at a room temperature of approximately 28 °C over a stick surface. After printing, Al electrodes were attached to the sample to provide electrical contacts for charging and for measuring piezoelectricity. Electrical charging/poling was performed in direct contact by applying a DC voltage of 3.0 kV for 10 s at room temperate. Application of an electric field of this magnitude results in dielectric barrier discharges (DBDs) inside the 100 μ m high cavities, leading to a quasipermanent polarization consisting of charge layers with opposite polarity, as reported in the literature [2, 13, 14].

Self-adhesive aluminum (Al) films used as electrodes on the outer layers was previously tested to ensure that the observed piezoelectricity originates from the electric charges trapped in the cavities of the PP structure and not from possible trapping in the adhesive tape itself. It is important to mention that, although the 3D samples were designed with an overall thickness of 300 μ m, they turned out to have a total thickness of approximately 150 μ m, indicating that shrinking during cooling needs to be taken into account.

3 EXPERIMENTAL DETAILS

Electromechanical materials in general and piezoelectrics in particular can either exhibit an electrical response due to a mechanical stimulation (direct effect) or generate a mechanical deformation due to an electrical excitation (inverse effect). The direct and the inverse effects are usually characterized quantitatively with a piezoelectric coefficient, which in case of highly anisotropic ferroelectret films is the thickness or d_{33} component of the relevant piezoelectric tensor. In order to determine the d_{33} coefficient of the 3D-printed samples, the experimental setup shown in Figure 3 was designed and built. The setup consists of an actuating device and a measuring system. The actuator itself is essentially an electromagnetic coil stimulated by and an alternating electrical signal, which pulls the neodymium magnets (and thus reducing the pressure on the



Figure 3. Schematic representation of the setup implemented for measuring piezoelectric d_{33} coefficients. A designed electric coil push and pull a set of magnets that produces a mechanical pressure over the sample. The electrical signal, which is produced as a response is recorded with an electrometer.

sample) and pushes them back (compressing the sample). The electrical response produced by the oscillating force is measured by an electrometer (Keithley model 617) coupled to a digital oscilloscope (Agilent model DSO-X-3024A).

The magnets were placed over the top aluminum electrode that was in directly contact onto the 3D-printed samples. In total, the weight of the aluminum electrode and the magnets was 23.3 g. The coil with an inner diameter of 20 mm, containing 2,000 turns of standard galvanized wire AWG 26, was mounted in an external holder, which height could be adjusted. To drive the coil, a function generator (Tektronix model CFG253) producing an electrical signal modulated as a rectangular signal with a frequency of 1 Hz and peak voltages of up to 13.2 V, pushed and pulled the magnets with an resulting force ΔF of 400 mN directly applied at the sample. The total weight of the (non-actuated or actuated) magnets, sample and bottom electrode was measured before and after actuation by use of a scale underneath the bottom electrode, so that the change of the force ΔF acting on the sample could be determined with a precision of \pm 0.01 p (*i.e.* approximately \pm 0.1 mN). The electrometer in its charge amplifier mode was used to integrate the resulting electric current from the sample. With the charge variation $\Delta Q (Q_1 - Q_2)$ produced during quasi-static stimulation and the force stimulation ΔF (measured weight or force), the quasi-static piezoelectric coefficient d_{33} was calculated dividing $\Delta O by \Delta F$, assuming that the electrode area (Se) and the surface area (St) are the same.

Figure 4, shows measurements over time of a polyvinylidene-fluoride (PVDF) sample used for calibrating the system and indicates how ΔQ was obtained. Q_1 is defined as the charge accumulating during the time period of force application, whereas Q_2 is the charge accumulation upon removal of the quasi-static force. It is important to note that both charge values Q_1 and Q_2 are obtained from stable regions of the charge curve. ΔF was calculated by multiplying the measured weight change ΔG with the gravitational acceleration $g = 9.8 \text{ m/s}^2$. The d_{33} coefficient was calculated as a mean value of the measurements made during ten quasi-static loading/unloading cycles.

The measuring device was calibrated with a commercial PVDF film (25 μ m thick) from Arkema-Pieozotech S.A.,



Figure 4. Charge variation ΔQ on the PVDF-sample electrodes from quasistatic mechanical actuation through electro-magnetic excitation.

France, which has a nominal piezoelectric coefficient d_{33} of 20 pC/N according to the manufacturer and which is provided with chromium-gold (Cr-Au) electrodes on both sides. Ten measurements were performed with this sample to determine its piezoelectric coefficient d_{33} . For each measurement, the sample was removed from and re-inserted into the measuring system at random positions. The procedure resulted in a value of (21 ± 2.8) pC/N.

4 RESULTS AND DISCUSSION

The quasi-permanent electro-mechanical properties of ferroelectrets rely on the electret material's ability to retain electric charges for long periods of time even at high temperatures. Therefore, researchers in this field search for polymers that are electrically more stable and can trap more charges of both polarities in order to enhance piezoelectricity. PP films are the most widely used material for the production of ferroelectrets and their piezoelectric properties are well known. However, the use of PP filaments for the 3D printing of ferroelectrets is a new strategy.

Therefore, an obvious concern about the novel 3D-printed ferroelectrets is their long-term stability. Thus, measurements of the piezoelectric coefficient d_{33} were performed for several days at room temperature and under atmospheric pressure. Figure 5 shows these results, which indicate a considerable piezoelectric coefficient d_{33} of almost 200 pC/N, but also show a significant decay of the 3Dprinted PP ferroelectrets over the first 20 days after charging with an average loss of 40% of the initial piezoelectricity. After 3 weeks, all samples seem to have reached a stable d_{33} value (129.52 pC/N) of approximately 60%. An exponential fitting given by $d_{33} = 129.52 + 116.15e^{(-0.311t)}$, where t is the time variable and -0.311 is the d_{33} constant decay. These results are in good agreement with those reported in [25], which decays of around 36% were noticed on 70 µm thick cellular-foam PP ferroelectret films over similar time periods.



Figure 5. Decay of piezoelectricity in 3D-printed PP ferroelectrets at room temperature (R.T.) during three months after charging. About 40% of the initial piezoelectric d_{33} coefficient is lost during the first three weeks.

Another essential aspect of all electrets is thermal stability, specially with ferroelectrets made from PP since it has been reported that they begin to lose charges (and piezoelectricity) at temperatures above 50 °C [25]. In order to check the relevant behavior on 3D-printed ferroelectrets, the short-term thermal stability was determined at stepwise increasing temperatures. Initially, the d_{33} coefficient was measured at room temperature (R.T., 25 °C). Then, the samples were kept in an oven at 30, 40, 60, 80 and 100 °C for one hour each. After annealing, the samples were cooled down to room temperature, and d_{33} was determined again. Cooling the samples before measuring d_{33} is important because the temperature also changes Young's modulus, leading to an increase in piezoelectricity [25]. The results from the stepwise thermal analysis are presented in Figure 6, and it can be clearly seen that the piezoelectricity remained stable up to temperatures of at least 60 °C. It can also be seen in Figure 6 that the piezoelectric d_{33} coefficient decays by an average of about 17% after samples had been annealed at 80 °C for one hour. Above 80 °C, the decay becomes even steeper so that only a little bit less than 40% of the initial piezoelectricity is still observed after one hour at 100 °C. The thermal behavior shown in Figure 6 is again close to what was observed on foamed PP ferroelectrets [25].

However, in the earlier measurement of cellular-foam PP ferroelectrets, the piezoelectric coefficients were measured at the respective elevated temperature, and the observed increase of d_{33} is due to the smaller Young's modulus at higher temperatures. The difference demonstrates the importance of taking the total electromechanical behavior of ferroelectrets into account, as their charge stability usually decreases with temperature, while their elastic modulus slightly increases.



Figure 6. Remaining piezoelectricity of 3D-printed PP ferroelectret samples after annealing at stepwise increasing temperatures for one hour each.

In view of the fact that a major area of ferroelectret applications is in acoustic-pressure sensors, the influence of different external pressures on the electrical response of 3Dprinted PP samples was investigated by means of a pressure varying device based on the pneumatic loading method (PLM). The measuring system is employed to apply a direct pneumatic pressure to the samples electrodes, which causes a deformation of the ferroelectret cavities that leads to a change of electric-field distribution from the internally trapped electric charges and thus, to an electrical signal at the electrodes. In the present measuring system, the sample is slightly stretched over an electrically grounded contact ring with an open diameter of 1.5 mm, in order to leave one side of the sample exposed to the pneumatic pressure from pressurized air that can be released into the sample chamber via a control valve. Another pneumatic piston ensures continuous electrical contact with the top electrode as well as sinusoidal excitation of the piezoelectric response from the sample, and a pressure gauge (SMC model ISE30) is used to adjust the pressure amplitude applied to the piston to a value of 20 kPa. The response of the ferroelectret sample is recorded via a Keithley electrometer model 6517B in charge mode. The pressure in the PLM setup was varied in equal steps from 5 kPa to 35 kPa. The results are presented in Figure 7, indicating that the samples are sensitive to pressure variations in the range between 20 kPa and 35 kPa. The reduction on the piezoelectric effect is mainly caused by the constant compression of the cavities, which become stiffer with increasing static pressure.



Figure 7. Influence of increasing pressure on the piezoelectric coefficient d_{33} of 3D-printed polypropylene samples.

5 CONCLUSIONS

In this paper 3D-printing was successfully demonstrated as a suitable technology for preparing soft functional materials such as ferroelectrets. In order to achieve reasonably stable ferroelectret samples for eventual sensor and actuator applications, the standard ferroelectret polymer, polypropylene (PP) was employed as printing material. Advantages of this approach are not only an almost unlimited range of possible geometrical structures, but also the possibility to test various filling patterns with a spectrum of resulting properties. It was shown that PP ferroelectrets can be produced, using standard 3D-printing parameters without the necessity of modeling complex structures. The electro-mechanical behavior and the stability of the novel 3D-printed ferroelectrets are not much different from those of the foam- or template-based structures described in the literature. The 3D-printed samples exhibit similar piezoelectric d_{33} coefficients of around 200 pC/N and show a decay of 40% of their initial piezoelectricity after a period of 20 days at room temperature and a decay of 17% after being exposed to a temperature of 80 °C for one hour. From the dependence of the piezoelectricity on static pressures between 5 kPa to 35 kPa, it can be stated that the 3D-printed PP ferroelectrets are sensitive to pressure variations. The results indicate that 3D-printed ferroelectrets are suitable for sensor applications with similar limitations as those found on other PP ferroelectrets. Nevertheless, a reliable 3D-printed functional material can be achieved in a single printing process on the basis of a very simple structural design.

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Werner Wirges was born on 23 January 1962 in Bonn (Germany). He was trained as a plumber before studying Physical Engineering at the Aachen University of Applied Sciences. He graduated as Dipl.-Ing. in April 1988. Between 1988 and 1992 he worked at the Heinrich Hertz Institute für Nachrichtentechnik, where he was in charge of the production and coating of viscoelastic control layers. From 1992 to 1997 he worked on the technology of thin film polymer systems for non-linear optical applications. This work was focused on the production and poling of polymer multi-

layers. At the same time he started his work on plasma deposition of new fluoropolymers for optical communication. Until April 2000, he was employed in two industrial projects on passive optical components (thermooptical switches and polymeric waveguide filter elements). His work is being continued in Potsdam since May 2000 in the group of Prof. R. Gerhard, where he focuses on production and characterization of non-polar fluoropolymers.



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