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Direct piezoelectric responses of soft composite fiber mats

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Recently soft fiber mats electrospun from solutions of Barium Titanate (BT) ferroelectric ceramics particles and polymeric acid (PLA) were found to have large (d_{33} \sim 1 \text{nm/V}) converse piezoelectric signals offering a myriad of applications ranging from active implants to smart textiles. Here, we report direct piezoelectric measurements (electric signals due to mechanical stress) of the BT/PLA composite fiber mats at several BT concentrations. A homemade testing apparatus provided AC stresses in the 50 Hz-1.5 kHz-frequency range. The piezoelectric constant d_{33} \sim 0.5 \text{nC/N} and the compression modulus Y \sim 10^{4}-10^{5} \text{Pa} found are in agreement with the prior converse piezoelectric and compressibility measurements. Importantly, the direct piezoelectric signal is large enough to power a small LCD by simple finger tapping of a 0.15 mm thick 2-cm\(^{2}\) area mat. We propose using these mats in active Braille cells and in liquid crystal writing tablets. © 2013 AIP Publishing LLC [http://dx.doi.org/10.1063/1.4802593]
28 μm thick PVDF film sensor obtained from Images SI Inc. (Staten Island, NY), were placed on top of each other, and were sandwiched between the plate of the heater and the flat foot of the piston so that the fiber mat was slightly (<1%) compressed to ensure full contact between the speaker and the sample during the measurements.

The frequency dependence of the piezoelectric current $\dot{I}$ of the PVDF film with known $\dot{d}_{33} = 33pC/N$ and $I_s$ of our samples with the unknown $d_{33}$, were measured as a function of frequency of the applied stress. Because the forces acting both on the sample and the piezoelectric standard are the same, the calculated $d_{33}$ is independent of the size ratio of the sample and the PVDF sensor, and

$$d_{33} = \dot{d}_{33} \cdot I_s/\dot{I}. \quad (1)$$

The frequency dependences of the charge constants of 0.15 mm thick films at several BT concentrations are shown in Figure 3.

One can see that $d_{33} < 10pC/N$ of the pure PLA mat is practically negligible compared to the charge constants of the fiber mats containing BT particles. The measured $d_{33}$ appears to be roughly proportional to the BT concentration, although they have slightly different frequency dependences. It is important to note that the measured values for the 42 wt. % composite are in good agreement with the values obtained by Morvan et al.\textsuperscript{16} from the converse piezoelectric measurements.

In Figure 4 we plot the strain dependence of the measured short circuit currents at a constant frequency (1 kHz).

![FIG. 1. SEM (a) and OM (b) images of a 15 wt. % BT in PLA fiber mat electrospun across 25kV potential difference between the nozzle and the collecting ITO coated glass plate placed 10cm apart from each other. The scale bars correspond to 5 μm and 50 μm in (a) and (b), respectively.](image)

![FIG. 2. Schematic of the apparatus used for applying an alternating stress to a fiber mat and measure the signal from accelerometer and samples.](image)

![FIG. 3. The frequency dependence of the piezoelectric charge constant $d_{33}$ for 0.15 mm thick PLA fiber mats containing 0, 15, and 42 wt. % of BaTiO$_3$ particles.](image)

The strain was determined by the voltage signal $V_a$ of the accelerometer fixed to the sample. Knowing the sensitivity $b = 6.08 \times 10^{-4} V/mm$ of the accelerometer, the displacement $\Delta s$ of the top substrate could be obtained as $\Delta s = V_a / [b \cdot \omega^2]$, where $\omega$ is the angular frequency of the vibration. We see that even as small as 0.04% strain of 0.15 mm thick and 2 cm$^2$ area fiber mat with 42 wt. % BT concentration is able to generate 2 nA of electric current. Similar current levels were obtained recently on a M13 phage-based piezoelectric generator, but with 6% of strain.\textsuperscript{22}

Calculating the induced polarization from the short circuit current $I_s$, measured in the samples of area $A$ as $P = I_s/(\omega \cdot A)$, and the stress $T$ from $\Delta s$ as $T = Y \cdot (\Delta s/A)$, where $Y$ is the compression modulus of the sample with thickness $l$, $d_{33}$ can be related to $Y$ as $d_{33} = \frac{I_s}{V_a} = \frac{b \cdot l \cdot \omega}{V_a \cdot Y}$. With these the compression modulus can be calculated from the measured $I_s$, $V_a$, and $d_{33}$ values, as

$$Y = \frac{I_s}{V_a} \frac{b \cdot l \cdot \omega}{d_{33} \cdot A}. \quad (2)$$

Combining Eqs. (1) and (2), we see that $Y$ can be obtained also from $\dot{I}$ of the PVDF sensor and its known charge constant $\dot{d}_{33}$, as
Using this equation, the compression moduli $Y$ of the mats were obtained from the measured frequency dependence of $\omega$, and plotted in Figure 5.

It can be seen that the compression moduli are on the order of $10^{-100}$ kPa, which are much smaller than the compression modulus of the PVDF standard of $\approx 3$ GPa, therefore the displacement $\Delta s$ deduced from the accelerometer signal corresponds to the thickness variation of the fiber mats. The observed compression moduli are largest for the pure PLA (300–500 kPa), and smallest (15–30 kPa) for the mat containing 42 wt. % BT particles. This probably is related to the increased width of the fiber when the BT particles inserted, which results in a decrease of the number of nodes of the fiber mesh. We note that the Young modulus of the 42 wt. % BT mat obtained from these direct piezoelectric measurements are in good agreement with $16$ kPa reported based on DC compression measurements (measuring variation of the film thickness as a function of weights added on the top of the mat). The significance of these compression modulus values is that they are on the order of the Young moduli of biological tissues, such as muscle, brain, etc. This mechanical impedance matching thus maximizes the energy conversion between the fiber mat and the soft load, and make them ideal in applications, such as in active Braille, noise insulators, energy harvesters and in liquid crystal writing tablets.

In order to illustrate this latter possibility, we made a simple prototype where a single pixel LCD panel was connected to the electrodes of a 0.15 mm thick and 2-cm$^2$ area fiber mat sandwiched between two ITO glasses. When a finger taps the mat, the LCD pixel switches to a black (homeotropic) state (Figure 6). This shows that the generated voltage on the liquid crystal pixel is over 3 V. From this prototype, it was estimated that a 1 mm-thick fiber mat would provide enough voltage to switch a cholesteric liquid crystal writing tablet (Boogieboard$^\text{TM}$) commercialized by Kent Displays, Inc, thus making it completely power-free.

To summarize, we have measured the effective piezoelectric charge constant of electrospun fiber mats consisting of BaTiO$_3$ nanoparticles. We found good agreement with the recent converse piezoelectric measurements and demonstrated that thin (0.15 mm) mats can switch a liquid crystal (LC) display by a finger tap, making them promising for powering some liquid crystal displays and other applications.

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