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Patterning piezoelectric thin film PVDF–TrFE based pressure sensor for catheter application

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ABSTRACT

We report a thin PVDF–TrFE (polyvinyledenedifluoride–trifluoroethylene) copolymer film pressure sensor, fabricated using standard lithography process for cost-effective batch process, film uniformity, and high resolution of polymer patterning. PVDF–TrFE copolymer, a semi-crystalline material, was spin-coated into thin films (1 μm thick) to tap the near β-phase formation. Pressure measurements demonstrated that the thin film (1 μm) show better performance compared to thick film (6 μm) with no electrical poling or mechanical stretching. Thin film devices showed higher β phase formation from Raman spectroscopy measurements, which translate into high piezoelectricity for sensing. The sensors can operate over a broad pressure range of 0–300 mmHg, with fast recovery time of 0.17 s, suitable for real time flow measurements in catheter applications.

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1. Introduction

Balloon occlusion of a major blood vessel is commonly used for both diagnostic and therapeutic purposes. Every year, clinicians in the U.S. alone place more than 1.5 million pulmonary artery catheters for the purpose of hemodynamic monitoring [1]. Balloon occlusion of the aorta has emerged as a tool for controlling life-threatening hemorrhage from the pelvis and lower extremities [2]. Currently, the pressure of the inflated balloon is left to the surgeon's experience or costly contrast based imaging techniques and is not dynamically controlled. Over inflation of the balloon results in a rare but lethal rupture of the occluded artery [3].

Patients with massive bleeding display rapidly changing hemodynamics and blood pressure. Hence the proper inflation pressure for successful occlusion changes with the patient's physiology. A highly compact pressure sensor would allow development of an interventional catheter that includes multiple points of pressure measurement, including inflation pressure as well as upstream and downstream blood pressure.

Over past decades, many types of microfabricated pressure sensors have been developed, and they can be categorized into three major groups, depending on their operating principles: capacitive, piezoresistive, and piezoelectric sensors [4–6]. Capacitive sensors are most commonly adopted in the clinical setting due to relative simplicity and less expensive fabrication, their high scalability for miniaturization, high sensitivity, and low power consumption, but are mostly applied to low pressures and require complex readout circuitry. Piezoresistive sensors, successfully landed into industry, offer great mechanical stability but they have some drawbacks, for instance, a significant power requirement, large temperature-dependence offset, nonlinearity, long-term instability in dynamic field conditions and impart undesirable stiffness to catheters. Piezoelectric sensors are commonly employed for the measurement of highly dynamic pressures.

The need for a pressure sensor on a catheter is not new and dates back to the 1970s [5,7]. So far, silicon based piezoresistive and capacitive pressure sensors have been employed for this application but with limited success due to above-mentioned reasons. Piezoelectric thin film transducer shows great promise as the implantable and biocompatible pressure sensors, implemented on a catheter for minimally invasive surgery.

In this paper, we design, microfabricate and characterize piezoelectric PVDF–TrFE (polyvinylidene fluoride–trifluoroethylene) based pressure sensor which can be later integrated with catheter for intravascular measurements. Recently, PVDF has gained increasing attention for the development of ultra-compact pressure sensors [8,9]. However, these sensors comprise of thick PVDF

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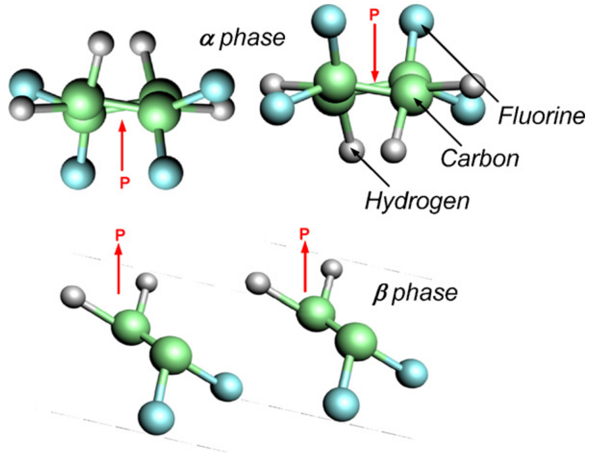

Fig. 1. The unit cell of bulk PVDF in α (top) and β (bottom) phases, combining fluorine (blue sphere), carbon (green sphere), and hydrogen (gray sphere). The α phase is antipolar, since the dipoles in the alternate chains cancel, while in the β phase they are parallel to each other. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

films with thickness of 10 μm and above. The present study is an extension of our previous work [10] on pressure sensor fabricated from 1 μm thin PVDF–TrFE films.

2. Sensor materials: thin film PVDF

Piezoelectric PVDF, a semicrystalline polymer, may exhibit any of its four phases, α, β, γ, and δ. Amongst these phases, β phase is important since it has largest effective dipole moment [11]. The key to achieving high piezoelectricity of the PVDF polymer is forming β-crystalline phase, which significantly improves sensor and actuator performance.

Fig. 1 shows atomic structure of α-phase PVDF and β-phase PVDF respectively. The crystal structure for β-phase PVDF show how the dipoles are aligned parallel to each other to create surface charge separation under stress or compression. Due to the random orientation of the dipoles in the α-phase, the dipoles cancel out each other resulting in no net surface charges [4]. Untreated PVDF itself cannot have β phase without delicate mechanical stretching or electrical poling processes. Additionally, due to its incompatibility with the standard lithography process, many alternative fabrication methods such as screen printing and shadow mask process [11] have been developed.

Our approach offers a practical option to overcome these limitations, and offers cost-effective batch process with high film uniformity and high resolution of polymer patterning towards enhanced sensor performance using standard lithography fabrication. Further, we hypothesized that, without any mechanical stretching or poling, a thinner film should exhibit higher piezoelectricity compared to a thicker film. The output voltage V produced by a piezoelectric sensor is given by [2]:

$$V = F \cdot g_{3n} \cdot d/A$$

where, F, g_{3n} are the applied force, appropriate voltage coefficient; d and A are the thickness and area of the film, respectively. Hence, a thicker film should have higher output voltage if the film exhibits good β-phase structure. But a thinner film may have higher crystallinity and hence higher voltage coefficient.

To verify the above, we fabricated two different thicknesses of PVDF films and characterized using Raman spectroscopy and FTIR (Fourier Transform Infra Red) spectroscopy. Samples were spin coated on silicon wafer and the absorbance due to silicon was subtracted from a background spectrum. Raman spectroscopy was done using Renishaw InVia Raman spectroscope using green laser (514.4 nm) with exposure of 30 s.

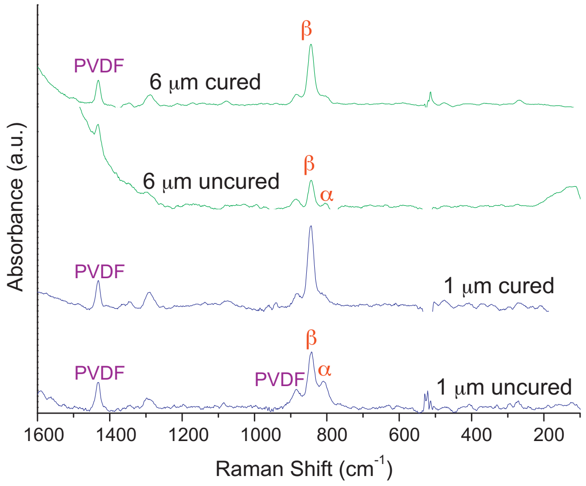

Fig. 2. Raman spectroscopy measurements for the various PVDF–TrFE films used in the present study, showing enhancement of beta phase on curing of films.

Fig. 2 showing the Raman spectroscopy peaks for the various PVDF film thicknesses are highly distinct. We notice a clear sharpening in the β-phase crystal phase (840 cm^{-1}) on curing the 1 μm and 6 μm thick PVDF–TrFE films. Further, we notice that the α-phase crystals (800 cm^{-1}) in the PVDF–TrFE films decreases or get eliminated as the film is heated and recystallized. This phenomenon has also been reported elsewhere [12]. The intensity of the peak from PVDF absorbance (1430 cm^{-1}) remains unaltered, however. Raman spectrum shows mainly the presence of the β-phase crystal domains throughout the films. Less noticeable, however, is the presence of the α-phase crystal structures also. Fourier Transform Infra-Red (FTIR) absorption spectrum for the various PVDF films fabricated was also done in the present study (data not shown). The plots showed similar nature as that from Raman spectroscopy, confirming the presence of beta phase formation in the membrane.

3. Device fabrication

Two different designs of electrodes on PVDF were fabricated: (1) parallel type: electrodes on the same side, where the electrode can be further patterned into dual and quadruple design; and (2) sandwich type: PVDF thin film sandwiched between the electrodes. The PVDF–TrFE lithographic based fabrication process for sandwich structure is illustrated in Fig. 3. Starting with a silicon wafer, photoresist was patterned to define the bottom Al electrode (2000 Å thick) using the lift-off process. PVDF–TrFE was spin coated on the Al-patterned wafer. Then another patterned photoresist was used as a Reactive Ion Etch (RIE) mask to pattern the PVDF–TrFE thin film. The dry etch was performed under 100 sccm oxygen gas environment with 200 W RF power and 5 mT pressure. The PVDF–TrFE thin film etch rate was 150 nm/min. The photoresist mask was etched simultaneously with an etch rate of 100 nm/min and dry etch was continued until the photoresist mask was etched fully. Top Cu electrode (2000 Å thick) was patterned by wet etch and the photoresist mask was removed by dry etch. Using this recipe, we fabricated single, dual, triple and quadruple membrane geometries in the following bottom electrode sizes: 1 × 1 and 1.5 × 1.5 cm^2. Further, parallel electrode devices were also patterned using just the top mask to pattern the electrodes on one side of the membrane.

Fig. 4 shows film thickness for PVDF–TrFE films that can be prepared versus spin speed. Three different solutions, having