

Material Properties

Preparation and characterization of porous poly(vinylidene fluoride-trifluoroethylene) copolymer membranes via electrospinning and further hot pressing

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ABSTRACT

Ferroelectric polymers, including polyvinylidene fluoride homopolymer and poly(vinylidene fluoride-trifluoroethylene) [P(VDF-TrFE)] copolymer, have been investigated extensively due to their potential application in sensors, actuators, transducers and energy harvesting. Recently, porous ferroelectric polymers have stimulated much interest because such materials have advantages of light weight, low acoustic and mechanical impedance, and high voltage sensitivity. In this work, P(VDF-TrFE) porous membranes were prepared for the first time by electrospinning and subsequent hot pressing. The morphology of the resultant electrospun membranes (nanofibers with or without beads) can be varied depending on the concentration of polymer solution used during the electrospinning process. Differential scanning calorimetry, wide-angle X-ray diffraction and Fourier-transform infrared confirmed the automatic formation of β crystallites in the P(VDF-TrFE) electrospun porous membrane. Based on piezoelectric strain coefficient measurement, dielectric constant measurement and dynamic mechanical analysis, it was found that the hot pressed P(VDF-TrFE) electrospun membrane with beads not only had a relatively high d_{33} value compared with those of other electrospun P(VDF-TrFE) membranes, but also the advantage of low dielectric constant and high flexibility.

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1. Introduction

Ferroelectric polymers, such as polyvinylidene fluoride (PVDF) homopolymer and poly(vinylidene fluoride-trifluoroethylene) copolymer [P(VDF-TrFE)], have attracted much attention because of their unique piezoelectricity. The piezoelectricity resulting from the strong molecular dipoles within the polymer chains can transform mechanical energy into electrical energy, which makes ferroelectric polymer a potential material for sensors, transducers, actuators and electrical energy harvesting from environmental vibration such as human motion [1–3]. Generally speaking, piezoelectric materials can be divided into two main groups:

ceramics and polymers. Although piezoelectric ceramics have been used successfully in many applications, they have some obvious drawbacks: limited deformation, brittleness, high mass density and toxicity when containing Pb. These limitations can be overcome using piezoelectric polymers, which are lightweight, spinnable, soft and flexible.

PVDF has five crystallite phases originating from three different chain conformations, including *TGTC'* for α and δ phases, *TTTGTTC'* for γ and ϵ phases, and *TTTT* for β phase [4–6]. Although α is the most common and stable phase, only the β -phase (all-trans) has very strong piezo activity. Therefore, many efforts have been carried out to improve the β -phase content in PVDF using various techniques, including the addition of clay as filler, mechanical stretching and electrospinning [7–10]. Electrospinning is a simple but effective technology for fabricating polymeric nanofibers by

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electrostatically driving a jet of polymer solution out of a nozzle onto a metallic collector. This technique can be used, not only to fabricate the porous and flexible membrane, but also to effectively induce β phase crystallites in PVDF directly from solution in one step.

The induction of porosity in the ferroelectric polymers may meet the demand of some special application. For example, Manuspiya et al. added bubbles in PVDF film to reduce its acoustic and mechanical impedance, which is important for piezoelectric hydrophone devices [11]. Moreover, some researchers attempted to incorporate piezoelectric material into clothing as the power source for wearable electronic devices [1,2]. In this case, a porous piezoelectric polymeric membrane should be a better choice owing to its good permeability of air and water vapour when compared to solid piezoelectric film.

P(VDF-TrFE) is one of the most important PVDF copolymers as it can form β -phase crystallites automatically at room temperature. To achieve low dielectric constant, low elastic stiffness, low density and resultant high voltage sensitivity, P(VDF-TrFE) copolymer nanotubes have been developed in the past [12–14]. However, little work has been carried out so far to fabricate electrospun P(VDF-TrFE) copolymer nanofibers and membranes, which are inherently flexible, lightweight and permeable. In this study, the fabrication process and properties of electrospun P(VDF-TrFE) copolymers nanofibres and membranes were investigated for the first time.

2. Experimental

2.1. Materials and sample preparation

The P(VDF-TrFE) (70/30) copolymer was obtained from Piezotech France in the form of white pellets. N,N-dimethylformamide (DMF) and acetone were purchased from Guangzhou Chemical Reagent Company and used directly without any further treatment.

The P(VDF-TrFE) copolymer was dissolved in a mixed solvent of 8:2 DMF and acetone by magnetic stirring at 80 °C for 3 h. The polymer solutions were then put into a plastic syringe and the distance from the tip of the syringe to the metal collector was fixed at 15 cm. The applied direct current voltage was 15 kV. Two samples with different morphology (with or without beads) were obtained from different solution concentration (12 and 18 wt %). Another two samples with low porosity were obtained by hot pressing of the above two electrospun membranes. For the purpose of comparison, P(VDF-TrFE) solid film was also prepared by a hot pressing method. The detailed description and codes of the samples are listed in Table 1. All the samples were subjected to poling to make them piezoelectric (see Fig. 1). Aluminum electrodes were deposited on both sides of the samples before poling. Poling experiments were carried out in a silicon bath to prevent electric breakdown with a maximum field of 80 kV/mm at 70 °C.

2.2. Characterization

Fourier-transform infrared (FTIR) spectra were recorded on a Perkin Elmer spectrometer. A JSM-6490 scanning

Table 1
Sample code and Sample description.

Sample code	Sample description
Sample 1	P(VDF-TrFE) electrospun membrane without beads before hot-pressing
Sample 2	P(VDF-TrFE) electrospun membrane with beads before hot-pressing
Sample 3	P(VDF-TrFE) electrospun membrane without beads after hot-pressing
Sample 4	P(VDF-TrFE) electrospun membrane with beads after hot-pressing
Sample 5	P(VDF-TrFE) solid film

electron microscope (SEM) was employed to observe the microscopic structure of electrospun P(VDF-TrFE) membranes before and after hot-pressing. Thermal analysis was carried out with a Perkin Elmer DSC-7 differential scanning calorimeter ((DSC) under a nitrogen atmosphere. Samples were heated to 200 °C at a rate of 20 °C/min. Wide-angle X-ray diffraction (WAXD) analyses were performed using a high solution X-ray diffractometer system (Bruker D8 Discover). Dynamic mechanical analysis (DMA) was carried out by a Perkin Elmer diamond DMA lab system at a frequency of 1 Hz in a nitrogen atmosphere, with a temperature range from –80 to 120 °C at a scan rate of 5 °C/min. The relative permittivity of the sample was studied as a function of frequency using a HP 4194A impedance analyzer. The apparent piezoelectric strain coefficient, d_{33} value, was obtained using a ZJ-3A piezo d_{33} meter, and the results were the average of five measurements.

3. Results and discussion

3.1. Morphology and porosity of the electrospun membrane

It has been reported that the electrospun nanofiber morphology can be effectively controlled by adjusting the polymer concentration used during the electrospinning process [15]. In this study, we prepared two electrospun membranes from two different P(VDF-TrFE) concentrations of 12 and 18 wt% in 8:2 DMF/acetone mixed solvent. Fig. 2a shows a SEM image of sample 1 obtained from the lower polymer concentration of 12 wt%. There were a large number of polymeric beads in the electrospun membrane due to the low viscosity of the P(VDF-TrFE) solution, which made the electrospinning process unstable. For sample 2 with the concentration increased to 18 wt%, as shown in

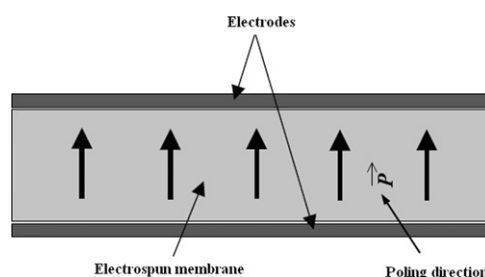


Fig. 1. Schematic illustration of the electrospun P(VDF-TrFE) copolymer membrane for poling.

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