



Energy harvesting performance of BaTiO₃/poly(vinylidene fluoride–trifluoroethylene) spin coated nanocomposites



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ABSTRACT

The energy harvesting efficiency of poly(vinylidene fluoride–trifluoroethylene) spin coated films and its nanocomposites with piezoelectric BaTiO₃ have been investigated as a function of ceramic filler size and content. It is found that the best energy harvesting performance of $\sim 0.28 \mu\text{W}$ is obtained for the nanocomposite samples with 20% filler content of 10 nm size particles and for 5% filler content for the 100 and 500 nm size fillers. For the larger filler average sizes, the power decreases for filler contents above 5% due to increase of the mechanical stiffness of the samples. Due to the similar dielectric characteristics of the samples, the performance is mainly governed by the mechanical response. The obtained power values, easy processing and the low cost and robustness of the polymer, allow the implementation of the material for micro and nanogenerator applications.

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1. Introduction

Energy harvesting from mechanical vibrations has received increasing attention over the last years due to the lower power requirement of small electronic components. Powering small electronics using the vibrational energy from their environment could reduce the requirement of external power sources, like batteries, and thus avoiding the associated maintenance costs [1–3].

There are three basic vibration-to-electrical conversion mechanisms: electromagnetic, electrostatic and piezoelectric [1,4]. The last one, has received the largest attention, mainly due to its large power density, easy application in the micro and macro scales due to well established production techniques and lack of needed external electrical input as the output voltage is obtained directly from the piezoelectric material itself [1], being the energy conversion in piezoelectric material based on variation in the dipolar moment network by applying a strain which results in a potential difference that can be used to power devices [2].

Strong efforts are being developed in order to improve the efficiency of piezoelectric power harvesting systems mainly based on

ceramics such as lead zirconate titanate (PZT) or barium titanate (BaTiO₃) [2]. Nevertheless, piezoceramics can undergo fatigue crack when submitted to high frequency cyclic loading, which encourages the search for alternative materials such as poly(vinylidene fluoride) (PVDF), a highly flexible and mechanically robust piezoelectric polymer [5,6].

PVDF and vinylidene fluoride–trifluoroethylene (VDF–TrFE) copolymers have attracted scientific and technological interest due to their chemical resistance, good mechanical properties and excellent piezoelectric, pyroelectric and ferroelectric properties, among polymers. (VDF–TrFE) has the ferroelectric – paraelectric (FE–PE) phase transition at a transition temperature, T_c , below the melting temperature, T_m , and has large electromechanical coupling coefficient [7,8].

PVDF and VDF copolymers crystalline structure in the ferroelectric phase is composed by packed polymer chains in an “all-trans”, *TT*, planar zigzag conformation [8,9]. The FE–PE transition temperature is highly dependent upon VDF content, heat treatments, electrical poling and processing history [10–12].

Nanoscale properties investigation of these materials are pointing out to their viability for the development of energy harvesting devices [13,14]. In this sense, initial works have been focused on the increasing the dielectric constant by the fabrication of thin

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films of PVDF with BaTiO₃ [15], as this positively influences the power output. Dielectric constants up to 32 were obtained for BaTiO₃/PVDF nanocomposites with 30%vol of ceramic.

Extensive research has been performed in single materials such PZT [16,17], BaTiO₃ [18], ZnO [13,19,20], CdSe [21] and PVDF [6,22] for energy harvesting applications, but a lack of work still exists for polymer based piezoelectric materials nanocomposites taking advantage both on the highly flexible electroactive polymer matrix and the larger dielectric and piezoelectric signal of the piezoelectric ceramics. Thus, this work focus on the production and characterization of spin-coated composite films of P(VDF-TrFE) with BaTiO₃ in order to study the influence of the ceramic filler size and content for energy harvesting applications. This work is relevant as, to our knowledge, there is a lack of studies concerning the preparation and systematic study of the influence of the processing conditions of BaTiO₃/P(VDF-TrFE) spin-coated nanocomposites on their performance in energy harvesting applications.

2. Experimental details

Poly(vinylidene fluoride trifluoroethylene) (P(VDF-TrFE)) (70/30) was purchased from Solvay. BaTiO₃ nanoparticles with an average size of 10, 100 and 500 nm were acquired from Nanoamor [23]. The structural characteristics of the nanoparticles are presented in Table 1. It is important to notice that just the particles with 500 nm size show tetragonal structure and therefore piezoelectric response.

Nanocomposites of P(VDF-TrFE) and BaTiO₃ were prepared by dispersing the ceramic powder in a solution of polymer with N,N-dimethylformamide (DMF, from Merck) in a fixed polymer plus ceramic concentration of 15% (w/w) of the total solution (Fig. 1). The polymer to ceramic relative concentration ranged from 0% to 20% ceramic weight content (w/w). The final solution was dissolved at room temperature with the help of a magnetic stirrer until complete polymer dissolution (Fig. 1).

Nanocomposites were deposited on clean glass substrates using a spin coater (Laurel WS-6505-6NPP/A1/AR2) and thermally annealed using a hot-plate (PrazithermPZ23-2) at 120 °C. The samples were produced at 500 rpm speed and an acceleration of 750 rpm/s² during 30 s (Fig. 1).

The thickness of the samples was measured using a profilometer (SloanDektakIIA) and digital micrometer (Fischer Dualscope 603-478). The average thickness of the samples was 12 μm.

Table 1
Structural properties of the used BaTiO₃ nanoparticles [23].

	$\phi = 10$ nm	$\phi = 100$ nm	$\phi = 500$ nm
Purity (%)	99.8	99.9	99.9
Crystallographic form	Cubic	Cubic	Tetragonal

The morphology of the samples was analyzed by Scanning Electron Microscopy (SEM) with an ESEM Quanta 650 FEM-FEI apparatus with an accelerating voltage of 10 kV. Previous to the measurements, the films were coated with a thin gold layer using a sputter coating Polaron, model SC502 sputter coater.

Electrical poling of the samples was performed at room temperature in a parallel plate configuration under an electrical field of 150 MV/cm for 60 min. The dielectric response was evaluated with a Quadtech 1920 in the frequency range of 500 Hz to 1 MHz at room temperature. For the dielectric measurements, a gold layer on the glass substrates, before spin coating procedure, and circular gold electrodes of 5 mm diameter on the top of the sample were previously evaporated by sputtering (model SC502 sputter coater).

The energy harvesting performance of the samples was evaluated by periodic bending tests in a electromechanical generator (Frederiksen 2185.00) excited from 1 Hz to 1 kHz using a signal generator (Circuitmate GF2) with an internal resistance of 1 M Ω. The voltage output was measured using an oscilloscope (Axi 4005) and the data collected with the Picoscope software. The maximum average power output was calculated from the first 100 ms of vibration.

3. Results and discussions

3.1. Nanocomposites morphology

The microfibrillar microstructure typical of P(VDF-TrFE) (Fig. 2a) is also observed in the BaTiO₃/P(VDF-TrFE) nanocomposites (Fig. 2). Further, the microstructure is maintained all along the sample thickness as observed in the cross section images (Fig. 2c, inset), which shows a random distribution of the ceramic particles within the polymer matrix. It was observed that the smaller particles (average diameter of 10 nm) have the tendency to form agglomerates whereas the particles with larger average diameter reach a better dispersion (Fig. 2c and d) and a more homogeneous distribution. As it will be shown later, filler agglomerates at the levels shown in Fig. 2 do not influence the electroactive response of the materials. The fact that even for the larger filler contents, 20% BaTiO₃ (w/w), and for all ceramic particle sizes the characteristic microfibrillar structure of the PVDF-TrFE is maintained, demonstrates that the ceramic filler does not influence polymer morphology and crystallization [24].

3.2. Electrical behavior

The dielectric constant influences the performance of a piezoelectric power generator, higher dielectric constants, in harvesters systems, lead to larger outputted powers [25]. The improvement of the efficiency of an electrostatic energy harvester systems can be therefore achieved by including high permittivity material layers [26].

Fig. 3 shows the room temperature frequency dependence of the real part of the dielectric permittivity, ϵ' , for the polymer matrix

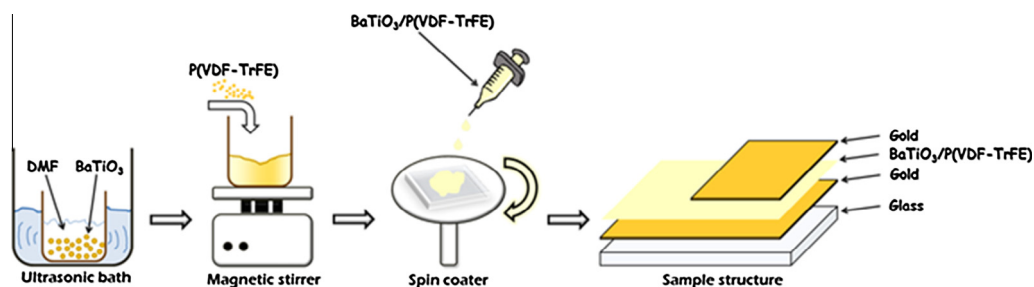


Fig. 1. Scheme of the procedure for sample preparation.

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