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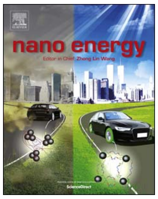

Communication

All-organic microelectromechanical systems integrating electrostrictive nanocomposite for mechanical energy harvesting

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ABSTRACT

Recent advances in the field of microelectromechanical systems (MEMS) have generated great interest in the substitution of inorganic microcantilevers by organic ones, due to their low cost, high flexibility and a simplified fabrication by means of printing methods. Here, we present the integration of electrostrictive nanocomposites into organic microcantilever resonators specifically designed for mechanical energy harvesting from ambient vibrations. Strain sensitive nanocomposite materials composed of reduced graphene oxide (rGO) dispersed in polydimethylsiloxane (PDMS) are integrated into all-organic MEMS by means of an innovative low-cost and environment friendly process by combining printing techniques and xurography. Static tests of the electrostrictive nanocomposite with 3.7 wt% rGO show good performances with variations of capacitance that exceeds 4% for strain values lower than 0.55% as the microcantilever is bent. The results in dynamic mode suggest that the organic MEMS meet the requirements for vibration energy harvesting. With an applied sinusoidal acceleration (amplitude 0.5 g, frequency 15 Hz) a power density of 6 μW/cm^3 is achieved using a primitive circuit.

1. Introduction

Over the last twenty years, increasing attention has been paid to energy harvesting from ambient energy sources such as light, heat and mechanical vibrations. Alternatively to the commercially successful photovoltaic area, mechanical energy harvesting is also of particular interest since a wide variety of vibration sources surround us (human beings, washing machines, cars, etc.). In most of cases, mechanical energy can be converted into electrical power by means of three types of mechano-electrical transducers: electrostatic, electromagnetic and piezoelectric. Numerous studies use the piezoelectric elements for small-scale energy harvesting because these materials have a relatively high energy density and a strong intrinsic electromechanical coupling. However, these materials (especially piezoelectric ceramics) are restrictively stiff and slightly deformable in response to the vibrations. In this context, a recent trend goes towards the substitution of inorganic materials by soft and highly deformable polymer-based materials. Piezoelectric and electrostrictive polymers or nanocomposite materials are very attractive, in particular for ensuring good mechanical adaptation, ease of implementation and low-cost manufacturing. While piezoelectric polymers such as the poly(vinylidenefluoride-*co*-trifluoroethylene) (P(VDF-TrFE)) copolymer are popular for mechanical energy harvesting, an alternative concerns the introduction of electrostrictive materials in this field as they require low operating voltage combined to interesting electromechanical properties [1]. For instance, Jaaoh *et al.* studied the electrostrictive behavior and the performance for vibration energy harvesting of conductive polyaniline (PANI) formulated in polyurethane (PU) at low percolation threshold [2]. In this case, a power density of 1.5 μW/cm^3 was generated. In another work, the use of P(VDF-TrFE-CTFE) electrostrictive terpolymer showed the possibility of obtaining a relatively high power density (7.2 μW/cm^3) under a polarization field of 5 V/μm [3].

In fact, once a strain is applied to an electrostrictive material, changes in the dielectric permittivity occur, inducing variations in the capacitance of an electrical capacitor composed of the electrostrictive material sandwiched between two electrodes. For proper application of electrostrictive materials for mechanical energy harvesting, they must be integrated into functional and autonomous devices. Microelectro mechanical systems (MEMS) are particularly suitable for this application as they permit efficient mechano-electrical conversion once implemented with a dedicated layer. In the case of electrostriction, the capacitance variation will depend on the MEMS's strain operating in dynamic mode, while the detection of electrostrictive effects at the MEMS resonance has not been achieved in the literature yet.

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Microcantilevers are powerful structures for MEMS-based energy harvesters [4,5], where triangular shaped microcantilevers incorporating a seismic mass at their end are of particular interest for their relatively low resonant frequency and high uniform strain [6]. However, most of the mechanical microgenerators proposed to date are made of the inevitably expensive and rigid silicon-based material and associate micromachining techniques [7]. Recent advances in organic MEMS have aroused interest in the field, due to their low cost, high mechanical deformation capability and ease of processing, in particular by the use of printing methods [8,9]. But the integration at low-cost of soft transduction materials into operational organic MEMS while maintaining their functionality remains particularly challenging. Here, we report the fabrication and application of autonomous mechanical energy harvesters based on electrostriction and integrated in a specifically designed, all-organic MEMS resonator.

2. Materials and methods

2.1. MEMS fabrication

After cutting square pieces of polyethylene naphthalate (PEN, Goodfellow) substrates, they are cleaned in an isopropanol bath for 10 min under ultrasound. The next step consists in the deposition and patterning of the PEDOT:PSS (768650 ALDRICH) bottom electrode by screen-printing. After deposition through a 400-mesh screen coated with 15 µm of emulsion, the printed layer is baked in an oven for 20 min at 120 °C. Final thickness of the dried film is approximately 1 µm. Thereafter, the electrostrictive film is spin-coated over the entire surface of the substrate and annealed at 200 °C for 3 h. The formulation of the rGO/PDMS electrostrictive nanocomposite is described elsewhere [10]. The average thickness of the deposited layer is approximately 50 µm. After the crosslinking of the electrostrictive material, the top electrode made of PEDOT:PSS is printed by doctor blade through a plastic stencil, before a baking step of 20 min at 120 °C. To finish the fabrication, the shape of the microcantilever is simply defined by xurography (Graftec Craft ROBO Pro). Then, the microcantilever is glued to a glass slide, which will serve as a support. Finally, the electrical contact is established by bonding conductive wires to both electrodes using an epoxy paste loaded with Ag particles (ESL1901).

2.2. Static characterization

The rectangular electrostrictive microcantilever is bent intermittently by applying a controlled force at its end using a microprobe (MiBot, Imina Technologies SA) while monitoring the evolution of capacitance with an impedance analyzer (HP-4194A). The maximum strain, located at the clamped part of the rectangular microcantilever due to an applied force at the microcantilever tip, is defined as:

$$\varepsilon = \frac{3hw}{2L^2} \tag{1}$$

With h and L being the thickness and length of the microcantilever respectively, and w the deflection at the tip of the microcantilever. The deflection w is obtained by image processing. The profile of the microcantilever is monitored using a horizontal camera (Navitar) as shown in Fig. S1. The recorded profile enables to determine the tip deflection by correlating pixel size with the profile.

2.3. Dynamic characterization

A shaker (Modal Shop-2075E) drives the devices into resonance for this purpose. As a reference, a laser vibrometer (Polytec MSA 500) is used to determine optically the resonance spectrum of the microcantilevers, while the electrical dynamic behavior is recorded with a gain-phase analyzer (Agilent E5061B). In this case, the electrostrictive microstructure is mounted in a Wheatstone half-bridge configuration with an adjustable resistance R_{comp} and capacitance C_{comp} to compensate parasitic, electrical and environmental effects (Fig. S2). This half bridge is powered by $E = \pm 20$ V DC voltage.

3. Results and discussion

Keeping the intended final application closely in mind, we aim to develop a simple and economic process for the fabrication of electrostrictive organic MEMS resonators to be able to preserve their attractiveness as low-cost, potentially mass-produced devices. The developed fabrication process is based on printing methods and does not require expensive equipment used for silicon technology, which considerably reduces the manufacturing cost of the MEMS energy harvesters. In particular, organic electrostrictive microcantilevers are manufactured in an all-organic approach thanks to printing techniques associated to xurography, an inexpensive method allowing rapid prototyping. The potential of this numerical cutting patterning method has already been demonstrated by the realization of various devices for physical and chemical detection applications, strain sensor, temperature sensor, etc [11,12]. Concretely, the fabrication of the microdevices starts with the cutting by xurography of a $2 \times 2\,\mathrm{cm}^2$ square made of polyethylene naphthalate (PEN) with a thickness of 125 µm for the rectangular cantilevers and 50 µm for the triangular ones (Fig. 1). This plastic substrate acts as supporting layer of the free-standing microcantilevers at the end of the process. The next step consists in screen-printing a thin layer of poly (3,4-ethylenedioxythiophene) doped with sodium poly (styrene sulfonate) (PEDOT:PSS) conductive polymer on the PEN substrate. This layer acts as bottom electrode. Thereafter, the electrostrictive film, made of graphene oxide (GO) dispersed in a PDMS-based matrix at different concentrations of GO, is spin-coated over the entire surface of the substrate. The insulating GO is subsequently thermally reduced into conductive rGO at 200 °C for 3 h. The thermal reduction of graphene oxide is carried out in the solid composites to increase their dielectric permittivity without changing the state of dispersion of the nanocomposite in the final material [10]. Increase of permittivity is here associated to the inclusion of conductive particles into an insulating matrix close to its percolation threshold [10]. After the thermal crosslinking of the electrostrictive material, the top electrode is patterned. Patterning an electrode on top of a PDMS-based material remains challenging. In the present work, different conducting materials have been tested, from metals to conductive polymers, including conductive inorganic inks. The PEDOT:PSS conductive polymer shows the best performances. This PEDOT:PSS top electrode is printed by doctor blade through a plastic stencil for its microstructuring. It is a few microns thick to avoid conductivity issues caused by the surface roughness of the electrostrictive layer. The advantage of patterning the top and bottom electrodes is to optimize their overlap in the vibrating part of the MEMS devices, to obtain large capacitance variations during testing. To complete the fabrication, the rectangular or triangular shape of the microcantilevers is simply defined by xurography. When necessary, a seismic mass can be added at the tip of the microcantilevers by depositing with a syringe an epoxy paste loaded with Ag particles. To finish the process, the resulting microdevices are transformed in free-standing cantilevers by gluing the substrates on a glass slide, while keeping the microcantilever part suspended beyond the latter. As shown in Fig. 1g, the electrostrictive MEMS are spatially resolved, with different shapes achievable by means of xurography. Here, three types of microstructures have been produced: rectangular microcantilevers, triangular microcantilevers, and triangular microcantilevers with seismic mass. Typical dimensions of the microcantilevers are in the millimeter range. The final dimensions are given in Table S1. In this process, one should note the all-organic nature of the electrostrictive MEMS associated with versatile printing methods, making them particularly suitable for future mass production at low-cost, in particular roll-to-roll printing processes.

Before applying the integrated electrostrictive MEMS to mechanical