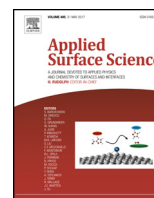




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Enhanced output performance of a lead-free nanocomposite generator using BaTiO₃ nanoparticles and nanowires filler

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ABSTRACT

Flexible nanocomposite generators based on piezoelectric nanoparticles (NPs)-polymeric matrix have been attracted attention as the energy harvesting device converted the electricity from the mechanical deformations. To enhance the piezo-potential difference introduced inside the piezoelectric nanocomposite, one-dimensional nanostructures such as CNTs, copper nanorods, and Ag nanowires (NWs) should be used inevitably as a dispersing agent for achieving well-distributed piezoelectric nanoparticles in an elastomer. These non-piezoelectric additives showed versatile roles; however, their toxicity to living organism has been an obstacle to realize the bio-eco-friendly flexible energy harvesters. Replacing them with piezoelectric NWs with non-toxic can be a challengeable approach to achieve not only the original purposes of additives but also the improvement of output performance. Here, we synthesized well-crystallized BaTiO₃ spherical and acicular NPs via a simple hydrothermal reaction and the two-step hydrothermal reactions, respectively and produced piezoelectric nanocomposite made of piezoelectric BaTiO₃ NPs and NWs without toxic dispersion enhancers. Output performance of the fabricated flexible energy harvesters with varying the composition of NPs and NWs were investigated by the well-optimized measurement system during the periodical bending and unbending. A nanocomposite-based energy harvester with 4:1 wt ratio generated the maximum open-circuit voltage and short-circuit current of 60 V and 1.1 μA, respectively.

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1. Introduction

An emerging technology often requires the development of materials and their processing techniques to be more advanced and matured form of technology. Once a new form of technology suggested, numerous studies have been concentrated to improve the performance by optimizing the parameters and components. Among many kinds of research fields, researches related with energy issues have received significant attention to suggest a novel method to offer the environment-friendly and sustainable energy more efficiently. Energy harvesting technologies have made a great breakthrough for the past few decades to improve the output performance to the power level of small electronic devices [1–5].

A piezoelectric energy harvester (PEH), as an independent and stable energy source, has attracted a great interest because it can harvest the electricity from diverse mechanical energy sources that are more available in almost everywhere [6–10]. Among many types of PEH, a flexible PEH can even convert from tiny pressure, deflection, vibration, and stretch from human or machinery movements into electrical energy [6–10]. Since Wang et al. firstly demonstrated ZnO nanowire (NW)-based PEH, the various types of flexible PEH have been investigated by many researchers [6]. They used the various piezoelectric materials such as wurtzite-structured ceramics (ZnO [6], ZnS [11], CdS [12], and GaN [9,13,14]), polymers [poly(vinylidene fluoride) (PVDF)] [15], and perovskite-structured ceramics [BaTiO₃ [16–19], PbZr_xTi_{1-x}O₃ (PZT) [5,20–22], and (1-x)Pb(Mg_{1/3}Nb_{2/3})O₃-xPbTiO₃ (PMN-PT) [23–26]] by employing various fabrication processes (e.g., electrospinning, direct growth, transfer technique, and spin-casting).

In 2012, Park et al. suggested the new type of PEH, named a nanocomposite generator (NCG), having the merits of low-cost,

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scalable, and high-output by using simple spin-coating of the piezoelectric nanocomposite (p-NC) that was made by dispersing the piezoelectric BaTiO₃ nanoparticles (NPs) inside a polydimethylsiloxane (PDMS) elastomer [18]. Several research groups have developed lead-free, high-output, and large-area NCG devices by introducing the (K, Na)NbO₃ [27,28], Li-doped (K, Na)NbO₃ [29,30], 0.5(Ba_{0.7}Ca_{0.3})TiO₃–0.5Ba(Zr_{0.2}Ti_{0.8})O₃–0.5Ba(Zr_{0.2}Ti_{0.8})O₃ [31,32], BaZr_xTi_{1-x}O₃ [33] NPs. To avoid the aggregation of piezoelectric nanoparticles (usually nearly spherical-shaped or atypical-shaped) inside a polymeric matrix for inducing the uniform piezo-potential distribution, one-dimensional structured materials such as CNT [18,24,34], Cu nanorods [29], and Ag NWs [31] are usually incorporated in piezoelectric nanocomposites. Although the non-piezoelectric additives can disperse piezoelectric nanomaterials and improve piezoelectric potential inside a device, it cannot contribute to the piezoelectric energy generation. To enhance the output performance generated from NCG devices, we should replacing these agents with biocompatible and piezoelectric one-dimensional structured nanowires [19,35].

In this study, we incorporated one-dimensional BaTiO₃ nanoparticles to the NCG device to achieve both homogeneous distribution of piezoelectric materials and enhanced performance. BaTiO₃ spherical nanoparticles (SPs) and BaTiO₃ nanowires (NWs) were synthesized by hydrothermal reaction. The various p-NC layers with different weight ratio of BT SP and NW were prepared to find the optimal ratio to maximize the output performance of NCG device. The output signals obtained from the NCG devices with various BT SP and NW weight ratios reached the highest output values ($V_{OC} \sim 60$ V and $I_{SC} \sim 1.1$ μ A) at a ratio of 4:1. The instantaneous power (40 μ W) was calculated from the output signals as a function of the external resistance.

2. Experimental section

2.1. Synthesis and characterization of the spherical and acicular BaTiO₃ nanoparticles

For the preparation of the spherical BaTiO₃ nanoparticles, previously reported procedures were adopted [36–38]. Titanium butoxide (Ti[(CH₂)₃CH₃]₄, reagent grade 97%, Sigma-Aldrich)(2.5 mmol) was mixed in ethanol (10 mL) and heated to 80 °C under magnetic stirring in a water bath. The amorphous TiO₂ particles were precipitated by the addition of 4 mL of an ammonia solution (25% NH₃ in H₂O, Sigma-Aldrich) to the mixture. Barium hydroxide monohydrate (Ba(OH)₂ · H₂O, 98%, Sigma-Aldrich)(3.75 mmol) was dissolved in 10 mL of preheated deionized water. The barium hydroxide aqueous solution was added to the TiO₂ precipitates, and the resultant suspension was stirred for an hour. The mixed solution was transferred to a 100 mL Teflon container, and the container was then sealed in a stainless-steel autoclave. A hydrothermal reaction was conducted at 200 °C for 60 h under autogenous pressure to obtain larger BaTiO₃ nanoparticles than the reported particle size of approximately 100 nm. After the reaction, the autoclave was cooled to room temperature. The as-synthesized BaTiO₃ nanopowder was washed repeatedly using a mixture of high-purity ethanol and deionized water, after which it was dried at 80 °C for 24 h in air.

To obtain the acicular BaTiO₃ Nanoparticles, a two-step synthesis route of the type used in earlier studies was applied. First, Na₂Ti₃O₇ nanowires were prepared via a hydrothermal reaction of anatase TiO₂ particles in a strongly basic (10 M) sodium hydroxide solution at 200 °C for three days. The BaTiO₃ NWs were obtained via an ion-exchange process of NTO NWs in a barium hydroxide solution (0.12 M) under a mild hydrothermal reaction at 100 °C for

24 h. Detailed information pertaining to the synthesis parameters can be found in our previously reported paper [19].

The morphology of the synthesized spherical and acicular BaTiO₃ nanoparticles was observed via a field-emission scanning electron microscope. The particle sizes were measured using the *Image J* program, and the average particle sizes and size distributions were calculated based on the measured values. The crystal structures of the obtained BaTiO₃ particles were investigated via X-ray diffraction (XRD) (Rigaku D/Max-RB (12 KW), Tokyo, Japan) with Cu K α radiation ($\lambda = 1.5406$ Å) operating at 40 kV and 300 mA with a step size of 0.01° at 2° min⁻¹ in a 2-theta range of 20° to 80°. The vibration modes to reveal the degree of the displacement from the central Ti⁴⁺ ion in the synthesized BaTiO₃ particles at room temperature were confirmed from the Raman spectra range of 100–1000 cm⁻¹ using a high-resolution dispersive Raman microscope (514.5 nm line of an Ar⁺ laser, LabRAM HR UV/Vis/NIR, Horiba Jobin Yvon, Longjumeau, France).

2.2. Fabrication and evaluation of BaTiO₃ NPs-based nanocomposite generator

Several nanocomposite generators were prepared to confirm the enhancement of the piezoelectric performance upon the addition of the acicular BaTiO₃ nanoparticles. The weight ratio between the BT SPs and BT NWs was varied within the range of 20:1 to 2:1 to find the optimal amount of BaTiO₃ nanoparticles to add. The synthesized BT SPs were mixed with the designed amount of BT NWs by magnetic stirring with ethanol. The well-mixed nanomaterials were dried sufficiently and the mixture was sieved using a testing sieve (mesh #200) to obtain granulated particles. Subsequently, 13 wt% of the mixed powder was poured into a PDMS matrix (containing a curing agent at a ratio of 0.1) to produce the piezoelectric nanocomposite (p-NC) layer. A PDMS elastomer was spin-coated at 1500 r/min onto an ITO-coated plastic substrate, after which p-NC and PDMS layers were spin-coated sequentially. The thickness of the spin-casted p-NC layer is about 300 μ m. Intermittent curing processes were done between each coating step in a dry oven for 10 min at 80 °C to prevent any unwanted intermixing of the layers. The cured PDMS/p-NC/PDMS layers were diced into pieces 3.5 × 3.5 cm² in size. Each diced p-NC layer was peeled from the substrate and placed between two ITO-coated PET films with different thicknesses (5 mil. and 7 mil.). A device with a sandwiched structure was fully hardened for a day and Cu wires were connected to a conductive ITO with conductive epoxy (Chemtronics). Finally, the prepared device was polarized to align the dipoles in the piezoelectric materials in the p-NC layer by applying an electric field of 1 kV for 12 h at 120 °C.

3. Results and discussion

The simultaneous use of spherical nanoparticles and acicular nanoparticles with piezoelectricity can be beneficial to improve the piezopotential in a p-NC matrix by augmenting the actual amount of piezoelectrically active materials. A synthesis method to obtain both spherical- and acicular-shaped BT nanoparticles has been well established for a few decades. BT SPs and NWs were prepared via hydrothermal reaction. In our previous studies, well-crystallized BT SPs with a uniform size distribution were easily obtained by a burst reaction of hydrolyzed B-site alkoxide precursors with an aqueous A-site precursor [37,38]. BT NWs can also be synthesized from a two-step hydrothermal reaction that consists of the preparation of a one-dimensional sodium titanate nanostructure followed by an ion-exchange reaction with barium ions under hydrothermal condition [19].

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