

Lead Zirconate Titanate Nanowire Textile Nanogenerator for Wearable Energy-Harvesting and Self-Powered Devices

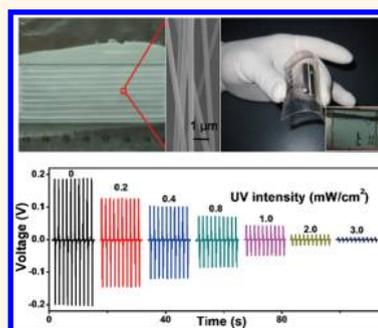
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Nowadays, miniaturization, multifunctionality, flexibility, and low energy consumption have become the development trend of electronic devices.^{1–6} High-quality power source matching in size is essential for these devices to form self-powered nano/microsystems. The power consumption of nano/microdevices is usually in the range of micro- to milliwatt.⁷ Harvesting energy from the environment to power a nano/microdevice is feasible and of vital importance to self-powered nano/microsystems. Recently, various forms of mechanical energy around us have been demonstrated successfully to generate electricity, such as air blowing/vibration,⁸ ultrasonic waves,⁹ body movement,^{10,11} pressure,^{12,13} and small friction.¹⁴ Many piezoelectric materials, such as ZnO,^{15,16} InN,¹⁷ ZnS,¹⁸ PZT,^{12,19–21} BaTiO₃,²² and PVDF,^{8,13,23} have been used for fabricating nanogenerators. In regard to ZnO-based nanogenerators, the low piezoelectric coefficient and electromechanical conversion efficiency is the major hindrance for increasing output voltage and current.^{24–29} On the other hand, high piezoelectric coefficient ceramics such as PZT³⁰ and BaTiO₃³¹ are brittle and difficult to manipulate for the fabrication of nanogenerators.

Portable, personal, and wearable self-powered nano/microsystems require the piezoelectric materials to be flexible and lightweight.¹² Piezoelectric nanowire films composed of random nanowires have been fabricated by the electrospinning method.^{12,13} However, there are still many challenges to meet the requirements of a self-powered nano/microsystem. First, for most piezoelectric materials, the as-electrospun nanowires need to be calcined to form a ferroelectric phase.^{32–34} Flexible substrates cannot endure

ABSTRACT



Wearable nanogenerators are of vital importance to portable energy-harvesting and personal electronics. Here we report a method to synthesize a lead zirconate titanate textile in which nanowires are parallel with each other and a procedure to make it into flexible and wearable nanogenerators. The nanogenerator can generate 6 V output voltage and 45 nA output current, which are large enough to power a liquid crystal display and a UV sensor.

KEYWORDS: nanogenerator · energy harvesting · wearable power source · self-powered nanodevice · electrospinning

high calcining temperature,⁴ which makes the high piezoelectric coefficient nanomaterials difficult to fabricate into flexible nanogenerators.¹² Besides this, shrinkage is a very common phenomenon in the calcining process,³⁵ which always leads to wrapping in the electrospinning nanowire film. When the piezoelectric film is wrapped or cannot attach to the flexible substrate very well, the performance of the nanogenerator will be greatly reduced. So these challenges limit the performance of flexible nanogenerators. Direct-writing is another effective way to fabricate a piezoelectric nanowire array, but the high expense of the facility and low efficiency hinder its application.²³ At the same time, the flexible substrate written

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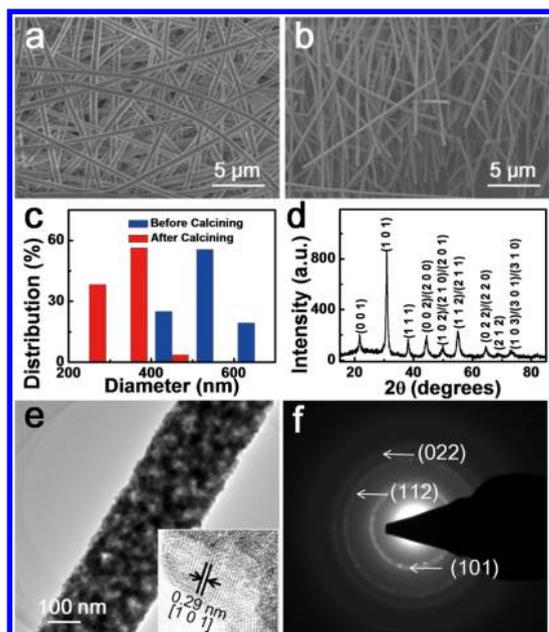


Figure 1. Morphology and structure of electrospinning random PZT nanowires. (a and b) SEM images of as-electrospun nanowires and calcined nanowires, respectively. (c) Diameter distribution of nanowires before and after calcining. (d) X-ray diffraction pattern of a PZT nanowire after calcining. (e) TEM image of a PZT nanowire. The inset is a HRTEM image. (f) SAED pattern of a PZT nanowire.

with nanowires also cannot endure the high temperatures of the calcining process. Thus, exploring a general, high-efficiency, simple, and low-cost method to fabricate aligned high piezoelectric coefficient nanowires is necessary and very important for flexible and wearable nanogenerators.

In this study we developed a suspending sintering technique of electrospinning nanowires to fabricate a flexible, dense, and tough lead zirconate titanate (PZT) textile composed of aligned parallel nanowires. This textile was transferred onto a thick polyethylene terephthalate (PET) film and chemical fabric to make flexible and wearable nanogenerators. The maximum output voltage and current of the nanogenerator reached 6 V and 45 nA, respectively. This kind of nanogenerator was demonstrated to light a commercial LCD and power a ZnO nanowire UV sensor to detect UV light quantitatively.

RESULTS AND DISCUSSION

PZT Nanowire Textile. The detailed preparation process of PZT nanowires is depicted in the Experimental Section. Figure 1a is a scanning electron microscope (SEM) image of as-spun random-oriented PZT nanowires. Before calcining, these nanowires are a mixture of PVP and organometallic compounds which contain elements Pb, Ti, and Zr. Their diameter is about 530 nm. After calcining, the diameter decreases to about 370 nm, as shown in Figure 1b. From the diameter distribution of the above two kinds of nanowires (Figure 1c), the nanowires shrink obviously after burning off the polymer

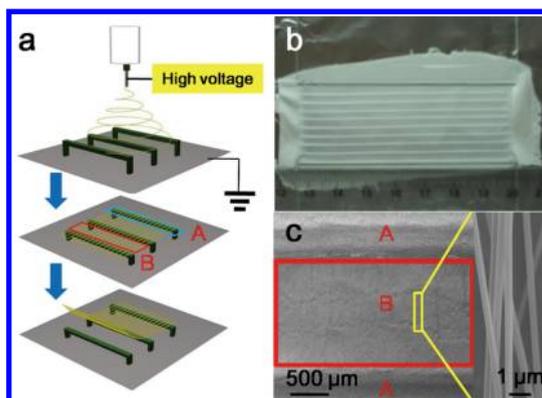


Figure 2. Fabrication and morphology of a PZT textile with aligned parallel nanowires. (a) Schematic diagram of the process of fabricating a PZT textile. (b and c) Photograph and SEM images of the PZT textile, respectively.

and crystallization of PZT. Figure 1d is the X-ray diffraction (XRD) spectrum of sintered nanowires. No peaks related to the pyrochlore phase are found in the XRD pattern, and all peaks can be indexed with perovskite PZT phase. It is evident that the nanowires have perovskite structure with good crystallinity. At the same time, the small crystal size of the PZT nanowires leads to the broadening of their diffraction peaks. Figure 1e is the bright-field image of a single PZT nanowire. Its inset is a high-resolution transmission electron microscopy (HRTEM) image, which shows that the main lattice plane is a (1 0 1) plane with lattice spacings of about 0.29 nm. Figure 1f is the selected area electron diffraction (SAED) of the PZT nanowires. It indicates that the nanowires have a well-crystallized polycrystal perovskite structure, which is consistent with XRD results.

Figure 2a is the schematic of fabricating a PZT textile composed of parallel nanowires. The electrospinning setup contains a jet, a collector, and a high-voltage source. When a high voltage is applied at the jet, the droplet of PZT precursor is elongated into nanowires. Simultaneously, multipairs of parallel electrodes are used to collect aligned nanowires during the process.³³ In this electrospinning process, Coulomb force plays a very important role to obtain parallel nanowires between parallel electrodes. In narrow electrode area "A", the positively charged nanowires discharge immediately when they deposit on the electrodes randomly. So Coulomb force does little effect to the alignment of the PZT nanowires. In area "B" between electrodes, the as-spun nanowires can retain their charges. Thus, Coulomb force makes them parallel with each other. After the electrospinning process, the textile made up of nanowires is peeled off from the collector and laid on a flat alumina ceramic substrate. After slowly evaporating the organic solvent, a 20 cm² textile consisting of parallel nanowires is obtained (Figure 2b), which looks like a transparent and soft silk. Finally, the as-spun PZT nanowire textile is sintered at 650 °C in a muffle furnace for 3 h. Figure 2c shows PZT nanowires at different areas. The

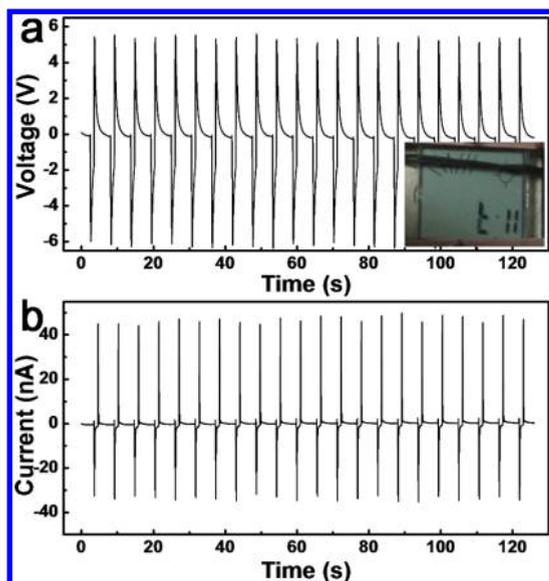


Figure 3. Output open-circuit voltage (a) and short-circuit current (b) of a FTNG. The inset is a photograph of lighting a LCD with a FTNG.

well-aligned textile with nanowires parallel with each other at area "B" between electrodes is a good candidate for fabricating laterally integrated nanogenerators.⁶

Fabrication and Performance of a Flexible Nanogenerator Based on a PZT Textile (FTNG). PET film is chosen to fabricate a FTNG. First, an ultrathin layer of poly(dimethylsiloxane) (PDMS) is spin-coated on a piece of clean PET film with a rotation speed of 5000 rpm, and it is precured for 60 min at 60 °C. Then a single row of parallel-aligned PZT nanowire textile is cut away from the whole electrospinning textile, placed on the PET film covered with PDMS, and slightly pressed with a glass slide to make it tightly attach to the PDMS layer. After that, Ag electrodes are deposited at two ends of the nanowire textile using a shadow mask and magnetron sputtering. Silver paste is used to connect copper wires with the electrodes. Finally, this device is packaged with PDMS and polarized with a 4 kV/mm electric field at 130 °C for 15 min to form a FTNG.

Open-circuit voltage and short-circuit current are measured using an electrochemical workstation (RST5000, Zhengzhou Shiruisi Technology Co. Ltd., China) to characterize the performance of the nanogenerator. The thick PET film is periodically bent and released by a linear motor. When the PET film is bent, the PZT nanowires suffer a tensile stress and have a strain along their length direction. A piezoelectric potential difference appears between the two ends of the PZT nanowires,²⁰ which drives a free electron flow from the low-potential end to the high-potential end through an outer load in the external circuit. Because of the insulating property of the PZT, the electrons cannot flow through the PZT nanowires and accumulate at the interface between the electrode and the nanowires. This process generates output

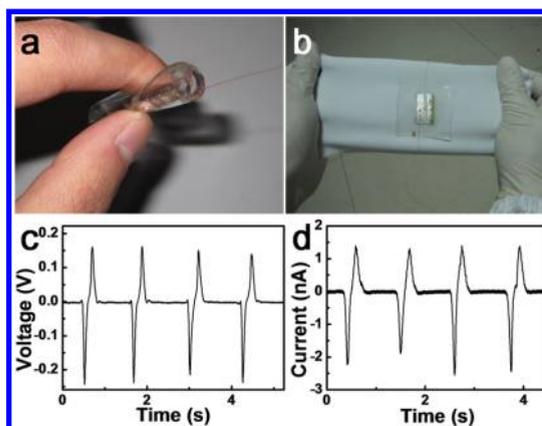


Figure 4. (a) Photograph of a tube bent with a substrate-free PZT textile nanogenerator, which shows its flexibility. (b) Working mode of a wearable nanogenerator; the stretching and releasing of the chemical fabric can drive the nanogenerator. (c and d) Output open-circuit voltage and short-circuit current of a wearable nanogenerator, respectively.

voltage and current (the negative pulses in Figure 3). When the PET film is released, the PZT nanowires are released, and their strain disappears. The piezoelectric potential difference between their two ends disappears too. The accumulated electrons flow back through the outer load in the external circuit. The FTNG generates output electricity again (the positive pulses in Figure 3). From Figure 3a, the open-circuit voltage reaches about 6 V, which is sufficient to light a LCD (inset of Figure 3a and supplementary video). Figure 3b indicates that the output current of the FTNG is about 45 nA. The nanogenerator's power delivered to the load could be estimate from the following equation:¹²

$$P_1 = \frac{1}{T} \int \frac{U^2(t)}{R_l} dt \quad (1)$$

where $U(t)$ is the real-time voltage, R_l is the load resistance, and T is the period of load application. Considering the 100 M Ω load resistance of the measurement system, the measured output power of the FTNG reaches 0.12 μ W. The length, width, and thickness of an effective PZT textile in a nanogenerator are 1.5 cm, 0.8 mm, and 5 μ m, respectively. It can be calculated that the volume of the textile is about 6 $\times 10^{-4}$ cm³. Thus, the power density is 200 μ W/cm³.

Fabrication and Performance of a Wearable Nanogenerator.

Taking advantage of the flexible and soft properties of the PZT textile, a wearable nanogenerator is fabricated by combining the textile with a chemical fabric. First, a piece of PET film substrate is cleaned with acetone, alcohol, and deionized water sequentially. Second, a layer of AZ P1350 photoresist is spin-coated on it at a rotation speed of 5000 rpm. After that, a PZT textile nanogenerator is fabricated on this photoresist layer using the same procedure as described above. Then, the packaged textile nanogenerator is immersed into alcohol to dissolve the photoresist. After removing the

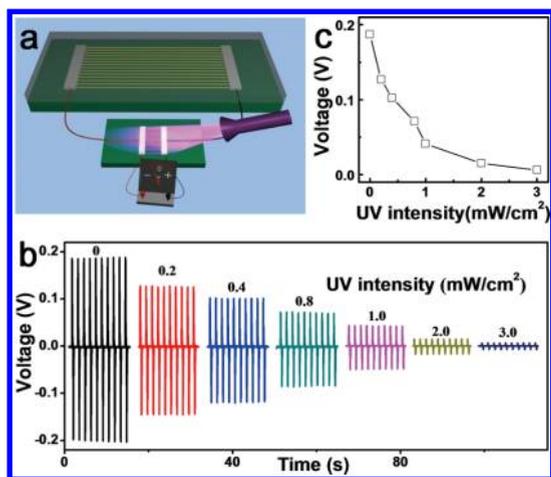


Figure 5. (a) Schematic of a self-powered nanosystem composed of a UV sensor and FTNG. (b) UV photoresponse of a ZnO nanowire UV sensor powered with a FTNG. (c) Plot of the voltage drop on a UV sensor versus UV light intensity.

photoresist, the substrate-free generator detached from its PET substrate. This nanogenerator is very flexible and even can be rolled into a tube, as shown in Figure 4a. Lastly, the substrate-free generator is firmly attached on the surface of a chemical fabric with PDMS. After baking at 70 °C for 1 h, the wearable nanogenerator is obtained. It is worth noting that the thickness and hardness of PDMS should be carefully controlled. If PDMS is too thick or hard to deform, only a very small stress can be transferred into the PZT nanowires, which leads to a weak output performance of the wearable nanogenerator.

This kind of wearable nanogenerator works through stretching and releasing the chemical fabric (Figure 4b). Because the substrate-free generator is tightly bonded with the fabric, the stress in the fabric can be transferred to the nanogenerator. There is a piezoelectric potential difference along the PZT nanowires of the wearable nanogenerator at the stretching state. After releasing, this piezoelectrical potential difference disappears. As a result, the periodic stretching and releasing of the fabric makes electrons flow back and forth continually. In this way, the wearable nanogenerator generates alternating current (ac) electricity. Figure 4c and d show the output signal of a wearable nanogenerator. The maximum output voltage and current are larger than 0.24 V and 2.5 nA, respectively.

EXPERIMENTS AND METHODS

Synthesis of the PZT Precursor. All the solvents and raw materials were analytically pure and used without any further purification. First, 2.4 g of ethanol, 1.0 g of acetylacetone, and 3.5 g of acetic acid are added into an Erlenmeyer flask and stirred for 5 min with magnetic stirring to form a homogeneous solvent. Then, 0.625 g of tetrabutyl titanate, 0.931 g of zirconium acetylacetonate, and 1.03 g of lead subacetate are added into the above solution in sequence. After stirring for more than 24 h, 0.3 g of PVP ($M_w = 1\,300\,000$) is dissolved into it to form a transparent precursor with a suitable viscosity.

Powering a UV Sensor with a FTNG. A portable, personal, and self-powered UV sensor is highly desirable. Here we use a FTNG to power a flexible integrated ZnO nanowire UV sensor, as shown in Figure 5a. A voltmeter is connected with a UV sensor to measure the voltage drop on it. When the UV light is off, the resistance of the UV sensor is very large and the corresponding voltage drop on the UV sensor is about 0.2 V (black curve in Figure 5b). When UV light is shining on the UV sensor, its resistance decreases because of the increasing carriers, and the voltage drop on the sensor decreases accordingly. With the increasing of UV light intensity, more carriers are generated in the ZnO nanowires, and the voltage drop decreases further. By monitoring the voltage drop on the UV sensor, UV light can be detected quantitatively. Figure 5b shows the UV response of a UV sensor powered by a FTNG. The voltage drop obviously decreases with an increase of UV light intensity. Their values are plotted in Figure 5c, which shows the one-to-one relation between them. Therefore, a FTNG is successfully used to power a UV sensor to form a flexible self-powered UV sensor, which can detect UV light quantitatively.

CONCLUSION

A simple and general method is developed to fabricate soft and flexible PZT textiles with nanowires parallel with each other. A PZT textile is made into two kinds of nanogenerators: a FTNG and a wearable nanogenerator. The FTNG has an output power density of $200\ \mu\text{W}/\text{cm}^3$, which is three times higher than that of a ZnO-based nanogenerator. Moreover, a FTNG is successfully demonstrated to power an integrated ZnO nanowire UV sensor to detect UV light quantitatively. This work has the following advantages. First, it gives an effective way to synthesize every kind of high piezoelectric coefficient ceramic nanowire, especially nanowires parallel with each other, which is very important for high-output nanogenerators. Second, the synthesized textile composed of parallel nanowires is very soft, flexible, and wearable, which benefits flexible nanogenerators. Third, this work demonstrates a wearable nanogenerator and flexible self-powered UV sensor, which is valuable for personal, portable, and self-powered nanodevices.

Preparation of Electrospinning PZT Nanowires and Textiles. The electrospin setup consists of a jet, a collector, and a high-voltage source. The precursor is added in a plastic needle tube. The distance between the jet and the collector is about 25 cm. The temperature and humidity are about 20 °C and 30%, respectively. The collector is made up of multipairs of parallel ion wires and grounded. When a high voltage of 25 kV is applied to the jet, the droplet of the PZT precursor is elongated to nanowires. After electrospinning, the PZT nanowire textile is calcined at 650 °C for 3 h in a box furnace (KSL-1100X, KJMT).

Fabrication of a UV Sensor Based on Electrospinning ZnO Nanowires. The precursor of ZnO is synthesized using a reported method.³⁶ Then, the as-spun ZnO nanowires are calcined at 450 °C for 3 h with a ramping rate of 2 °C per minute. After that, an integrated method is adopted to make a high-performance UV sensor.³⁷ During the fabrication process, a shadow mask and magnetron sputtering are used to fabricate the electrodes, and ZnO nanowires between the electrodes are exposed without any packaging.

Conflict of Interest: The authors declare no competing financial interest.

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Supporting Information Available: Movie of lighting a LCD using a nanogenerator. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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