

Energy Harvesting Based on PZT Nanofibers

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Abstract Energy harvesting technologies that are engineered to miniature sizes, while increasing the power delivered to wireless electronics [1, 2], portable devices, stretchable electronics [3] and implantable bio-sensors [4, 5] are strongly desired. Piezoelectric nanowire- and fiber-based generators have potential uses for powering such devices through conversion of mechanical energy into electrical energy [6]. However, the piezoelectric voltage constants of the semiconductor piezoelectric nanowires of the reported nanogenerators [7–12] are low. Here we introduce a piezoelectric nanogenerator based on lead zirconate titanate (PZT) nanofibers [13]. The PZT nanofibers, with diameters and lengths of approximately 60 nm and 500 μm , respectively, were aligned on interdigitated electrodes of platinum fine wires and packaged using a soft polymer on a silicon substrate. The measured output voltage and power under periodic stress applications to the soft polymer were 1.63 V and 0.03 μW , respectively.

1 Introduction

Recently, the piezoelectric properties of several nanowires, nanofibers and nanorods from zinc oxide [9], lead zirconate titanate (PZT) [14], cadmium sulphide [15], barium titanate [16] and gallium nitride [17], have been demonstrated. These

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one-dimensional piezoelectric nanostructures convert mechanical energy into electrical energy. As examples, various nanogenerators based on ZnO nanowires [9–12] and fine fibers [7, 8] proposed by Wang et al. have been demonstrated for potential applications in converting low frequency vibration and biomechanical energy into electrical energy.

However, the piezoelectric voltage constant of the piezoelectric nanomaterials, output voltage and output power of the nanogenerators still need to be improved further for practical applications. Furthermore, the fabrication method of the semiconductor piezoelectric nanomaterials may pose some drawbacks that can affect the performance of the nanogenerator. It is difficult to grow single crystal nanowires longer than 50 μm with diameters below 100 nm. The nanogenerator fabrication method and the output voltage of the nanogenerator could be significantly restricted by the short length of nanowires.

In order to overcome some of the drawbacks of the existing devices and demonstrate the possibility of energy harvesting using PZT nanomaterials, a highly efficient nanogenerator based on laterally aligned PZT nanofibers on interdigitated electrodes was created, and is reported herein. PZT is a widely used piezoelectric ceramic material with a high piezoelectric voltage constant and dielectric constant, which are ideal properties of the active materials for mechanical to electrical energy conversion. For a given volume under the same energy input, PZT can generate much higher voltage and power output than other semiconductor-types of piezoelectric materials.

As a ceramic material, bulk and thin film PZT structures are extremely fragile especially when subjected to alternating loads. Matters are made worse since thin film and micro fiber [18] structures are typically sensitive to high frequency vibration. However, unlike bulk, thin films or micro fibers, PZT nanofibers prepared by electrospinning process exhibit an extremely high piezoelectric voltage constant, high bending flexibility and high mechanical strength, which have been demonstrated in [14]. Therefore, utilizing PZT nanofibers in energy harvesting technology could provide a new way to make a portable, flexible, highly efficient device with low frequency vibration nature, since the nanofibers could be woven into fabrics and made into composites.

2 PZT Nanofiber Synthesis

PZT nanofibers were prepared by electrospinning [19]. The starting materials, PZT (52/48) sol-gel and poly vinyl pyrrolidone (PVP, Aldrich) were obtained from commercial sources. Alcohol was used as the solvent for PVP, while acetic acid was added to stabilize the solution and to control the hydrolysis reaction of the sol-gel precursor [20]. After being stirred vigorously for two hours at room temperature, the mixture was fed into a microscale metallic tube through a syringe pump. A droplet of the modified sol-gel solution was held at the orifice of the metallic tube due to the high surface tension. A high DC voltage (10 kV) between

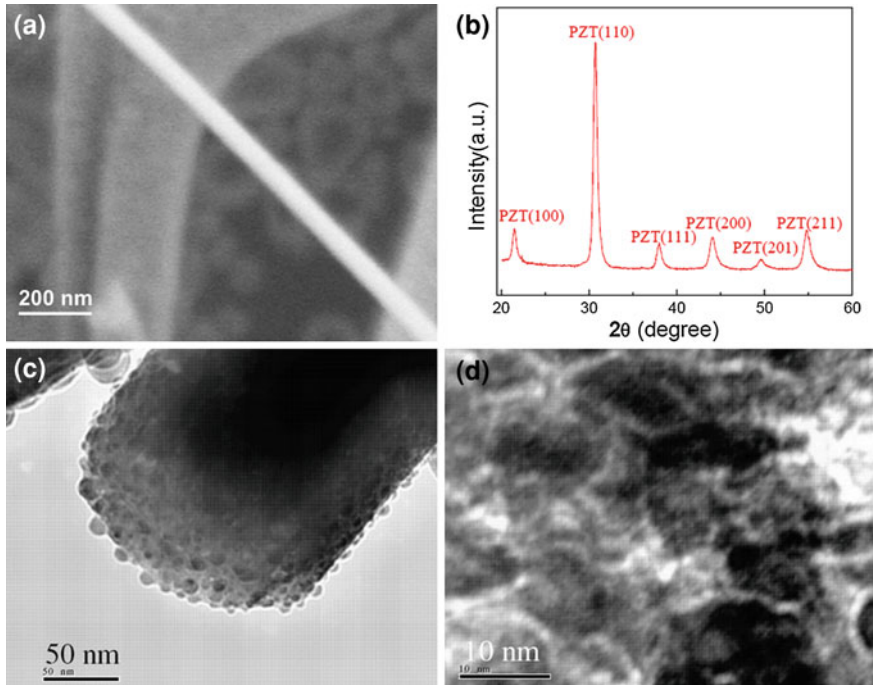


Fig. 1 **a** SEM image of single PZT nanofiber. **b** X-ray diffraction pattern for the PZT nanofibers. **c–d** TEM images of a PZT nanofiber. Reprinted with permission from [14]. Copyright 2009 American Institute of Physics

the tip of the micro metallic tube and the collecting substrate was applied to overcome the surface tension and generate an electrically charged jet of the modified solution. After electrospinning, the as-spun nanofibers were annealed at 650°C to obtain PZT nanofibers in the perovskite phase.

Figure 1 shows the morphology and X-ray diffraction pattern of the annealed PZT nanofibers. Figure 1a is a scanning electron microscopy (SEM) image of a selected nanofiber with a diameter of 70 nm. By varying the concentration of the polymer in the precursor, the average diameters of the nanofibers can be tuned from 52 ± 8 to 150 ± 12 nm. The X-ray diffraction (XRD) pattern of the annealed PZT nanofibers shown in Fig. 1b indicates that the PZT nanofibers annealed at 650°C were in the pure perovskite phase. The annealing temperature was lower than that used by most other methods. Transmission electron microscopy (TEM) images of the cross-section and surface of PZT nanofibers are shown in Fig. 1c–d. The grain size of the PZT nanofibers was about 10 nm. The structure of the nanofiber seemed porous, probably because of the polymer precursor solution of the sol-gel process [21]. By modifying the precursor and controlling the electrospinning parameters, a denser nanostructure can be obtained.

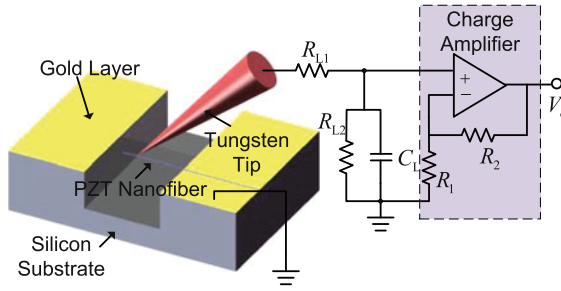


Fig. 2 Schematic diagram of the measurement system: R_{L1} , R_{L2} and C_L were load resistances and circuit capacitance respectively while V_o was the output voltage readout from the Keithley SPA. Reprinted with permission from [14]. Copyright 2009 American Institute of Physics

3 Piezoelectric Voltage Constant of a Single PZT Nanofiber

Piezoelectric response was measured by using a nanomanipulator under SEM. Two probes with tungsten tips were used for the measurements. The bending moment was applied to the nanofiber through one tungsten tip, while the other tip was used to form the ground by contacting the Au electrode directly near the PZT nanofiber. The output voltage from the PZT nanofiber was recorded in real time by a Keithley semiconductor parameter analyzer (SPA) with a charge amplifier; the equivalent circuit used for the measurement is shown in Fig. 2. To reduce the contact resistance between the tungsten tips and the PZT nanofiber, high currents were applied to burn the organic layer absorbed on the tungsten tips before the measurement. The resistance between the two tungsten tips, which were $1\ \mu\text{m}$ apart, was around $30\ \Omega$ after the burning process. The current in the circuit shown in Fig. 2 was set to be at a picoampere level for signal readout using the software of the Keithley measurement system.

The PZT nanofiber across the trench was ruptured at one end using a tungsten tip to form a nanofiber cantilever, while the other end of the nanofiber remained anchored to the Au electrode. The tip of the nanofiber cantilever was firmly attached to the tungsten tip by electrostatic force when the two approached each other. The electrostatic potential, V_{nw} , of the nanofiber could be considered as an offset measured from the ground of the Keithley system when no force was applied on the nanofiber (Fig. 3a). When a concentrated load was applied to the tip of the nanofiber cantilever through the tungsten probe tip, the nanofiber was deformed, as shown in Fig. 3b. The measured voltage output is shown in Fig. 3c. The magnitude of the output voltage, V_o , generated from the single PZT nanofiber was $0.4\ \text{mV}$, while the discharging process due to the load resistor, R_L , of the measurement system lasted about $40\ \text{s}$. The higher noise level in the right part of the curve could be from the charge amplifier during the discharging process when the current in Keithley system was set to $0\ \text{nA}$. The results were reproducible and each voltage impulse lasted for the same period of time, as shown in Fig. 3d, when the nanofiber was bent repeatedly, as shown in Fig. 3a–b.

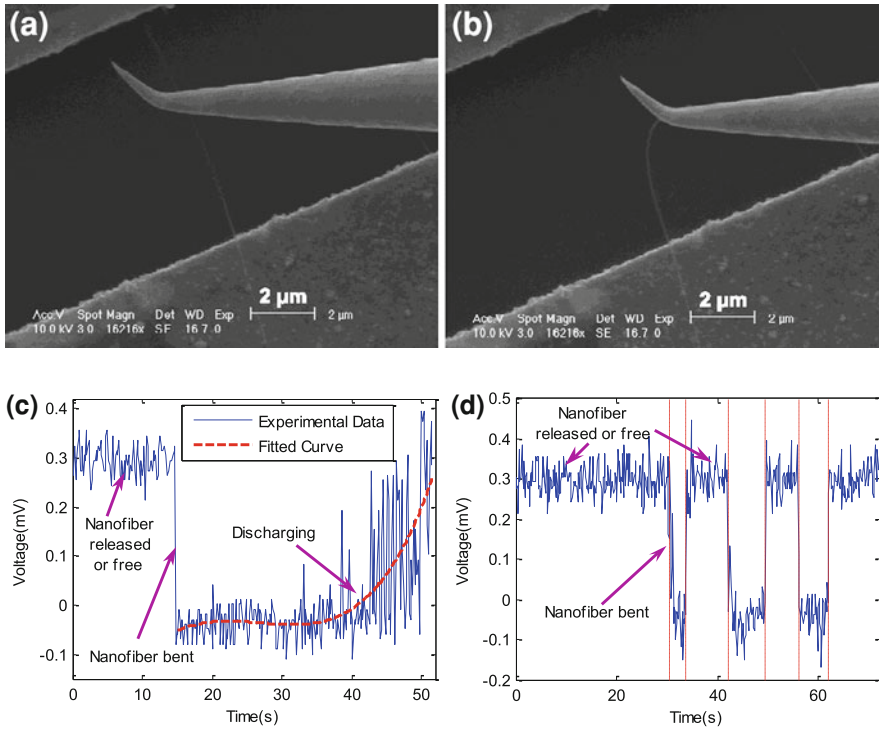


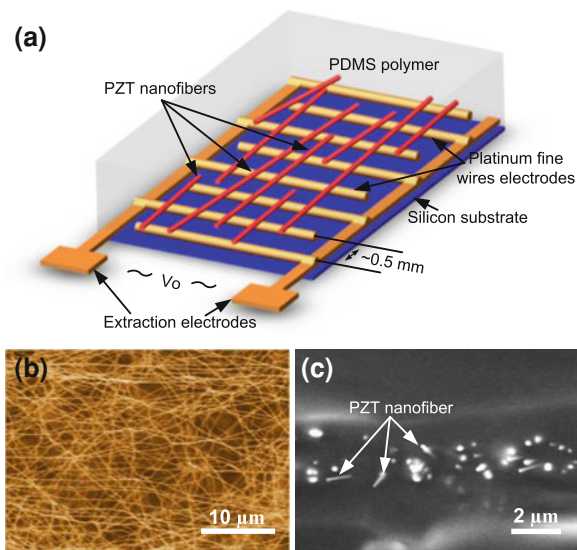
Fig. 3 a–b SEM images showing the free and bent single PZT nanofiber. c Voltage output and discharging from the single PZT nanofiber during the bending test. d Voltage output during the cycling tests. Reprinted with permission from [14]. Copyright 2009 American Institute of Physics

The measured voltage output due to the bending of the single PZT nanofiber was significantly higher than the noise level of the whole testing and measurement system, which was less than ~ 0.1 mV. When the electron beam of the SEM was switched between the on and off states, the differences in the measured voltage output results was less than ~ 0.05 mV. During the cycling test process, the nanofiber tip was attached to the tungsten probe perfectly during the test, which proves that the measured signal was not from the initial surface charges of the PZT nanofiber. The piezoelectric nanofiber also exhibited extremely high flexibility and strength during the test, which was very attractive in developing active micro and nanodevices or nanoenergy scavenging devices.

The voltage generation mechanism can be explained by a model similar to the one suggested in [22]. The voltage change on the PZT nanofiber surface during the bending test can be determined by:

$$\Delta V = \int_0^r g_{33} \sigma(r) dr \tag{1}$$

Fig. 4 Concept of the PZT nanofiber generator. **a** Schematic view of the PZT nanofiber generator. **b** SEM image of the PZT nanofiber mat across the interdigitated electrodes. **c** Cross-sectional SEM image of the PZT nanofibers in PDMS matrix. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society



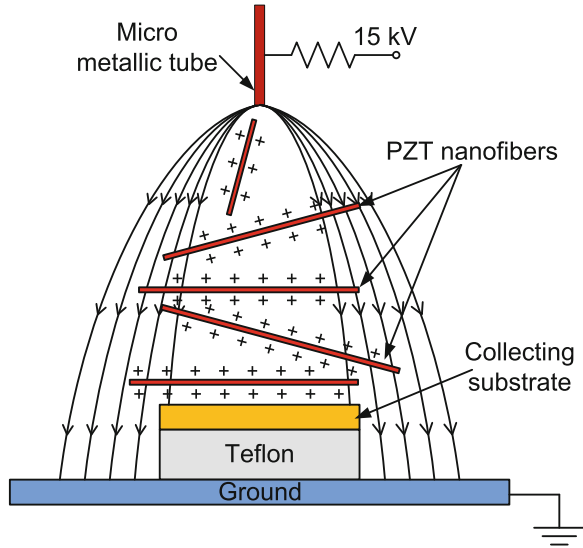
where r is the radius of nanofiber, $\sigma(r)$ is the stress function along the radius direction on the cross-section of the nanofiber; and g_{33} is the piezoelectric voltage constant. The stress function can be obtained using a FEM method based on the displacement measured from the SEM image. According to Eq. 1, using a Young's modulus of 42.99 GPa for the PZT nanofiber, as reported previously [19] and a voltage change of 0.403 mV, as measured in the test, the piezoelectric voltage constant, g_{33} , was determined to be about 0.079 Vm/N, which was larger than that found in bulk PZT. This was in agreement with the piezoelectric voltage constant measured from a bundle of PZT nanofibers during a dynamic bending test using a dynamic mechanical analyzer (DMA).

4 Fabrication Process for Nanogenerator

The nanogenerator device fabrication began with electrospinning the PZT nanofibers and depositing them on the pre-prepared interdigitated electrodes of platinum fine wire (diameter of 50 μm) arrays, which were assembled on a silicon substrate (Fig. 4a). The diameters of the PZT nanofibers were made to be around 60 nm (Fig. 4b) by varying the concentration of PVP in the modified sol-gel solution. The PZT nanofibers obtained were continuous while the distance between two adjacent electrodes was 500 μm as designed.

The pure perovskite phase was obtained by annealing at 650°C for about 25 min. Subsequently, a soft polymer (Polydimethylsiloxane, PDMS) was applied on top of the PZT nanofibers (Fig. 4c). The interdigitated electrodes of fine platinum wire were connected by extraction electrodes to transport harvested electrons to the external circuit. Finally, the PZT nanofibers were aligned by applying an electric

Fig. 5 Schematic of nanofiber alignment by controlling the electric field during the electrospinning process. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society



field of $4 \text{ V}/\mu\text{m}$ across the electrodes at a temperature of above 140°C for about 24 h. The nanogenerator can be released from the silicon substrate or prepared on flexible substrates, depending on the requirements of the applications for energy harvesting.

By controlling the electric field distribution during the electrospinning process (Fig. 5), PZT nanofibers were laterally aligned on the interdigitated electrodes. PZT nanofibers, which were positively charged, were spun on the collection substrate under the electric field. PZT nanofibers were stretched by the controlled electric field in two directions and deposited across the substrate.

5 Working Principle

5.1 Power Generation Mechanism

The nanogenerator device and power generation mechanism are illustrated in Fig. 6a–b, where PZT nanofibers were operating in the longitudinal mode (an alternating pressure applied on the top surface of the nanogenerator). The applied pressure was transferred to the PZT nanofibers through the PDMS matrix and resulted in charge generation due to the combined tensile and bending stresses in the PZT nanofibers. A voltage difference between the two adjacent electrodes was thereby induced by this separation of charge. The interdigitated electrodes enhanced the power output of the nanogenerator. The piezoelectric nanofibers between each pair of adjacent electrodes served as unit cells and each cell was connected in parallel.

Electrons generated in the PZT nanofibers could transfer through the electrodes when the PZT nanofibers were subjected to external stresses. Compliant PDMS

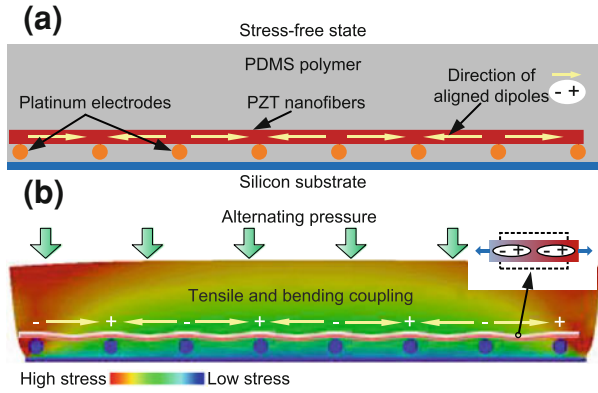


Fig. 6 Power generation mechanism of the PZT nanofiber generator. **a** Cross-sectional view of the PZT nanofiber in the generator. **b** Schematic view explaining the power output mechanism of the PZT nanofibers working in the longitudinal mode. The *color* presents the stress level in PDMS due to the application of pressure on the top surface. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society

covered the entire PZT nanofiber/electrode structure, due to the placement of the PZT nanofibers in a levitated position above the silicon substrate. The stress in the longitudinal direction, caused by Poisson's ratio of the composites, could be directly transferred to the PZT nanofibers when a stress was applied to the polymer matrix in the vertical direction. To avoid excessive stresses on the PZT nanofibers and to minimize the risk of damaging the electric connection of the electrodes, the silicon substrate was packaged with the nanogenerator as a rigid mechanical backing. This support could potentially be replaced by a flexible plastic backing for different applications. The final cured thickness of the PDMS polymer matrix was about 2 mm.

5.2 Mathematical Model

The potential generated from the PZT nanofibers between the interdigitated electrodes is given by:

$$\Delta V = \int_0^l g_{33} \sigma(l) dl \quad (2)$$

where l is the length of the nanofibers across two adjacent electrodes, $\sigma(l)$ is the stress function along the axial direction of the nanofiber and g_{33} is the piezoelectric voltage constant. Assuming perfect bonding between PZT nanofibers and PDMS polymer matrix, we have:

$$\varepsilon_p = \varepsilon_m = \varepsilon_c \quad (3)$$

where ε_m and ε_c are the longitudinal strains in the PDMS matrix and the composite, respectively. Assuming both the PZT nanofibers and PDMS matrix are elastic, the nanogenerator can be considered as unidirectional, continuous fiber lamina, and we have [23]:

$$E_{11} = E_p v_p + E_m(1 - v_p) \quad (4)$$

$$v_p = A_p/A_c \quad (5)$$

where E_{11} is the longitudinal modulus, E_p is the PZT nanofiber modulus, E_m is the PDMS modulus, A_p is net cross-sectional area for the fibers and A_c is the cross-sectional area for the composite. The major Poisson's ratio can be calculated as:

$$v_{12} = v_{13} = v_p v_p + v_m(1 - v_p) \quad (6)$$

where v_p and v_m are the Poisson's ratios of the PZT nanofibers and PDMS, respectively. The longitudinal strain can be given by:

$$\varepsilon_{xx} = \frac{\sigma_{xx}}{E_{11}} - \frac{\sigma_{yy}}{E_{11}} v_{12} - \frac{\sigma_{zz}}{E_{11}} v_{13} \quad (7)$$

where σ_{xx} , σ_{yy} , σ_{zz} are the stresses in three directions. By considering only the stress in the longitudinal direction:

$$\sigma(l) = E_p \cdot (\sigma_{xx}/E_{11} - \sigma_{yy}/E_{11} \cdot v - \sigma_{zz}/E_{11} \cdot v) \quad (8)$$

Thus, the output voltage can be written as:

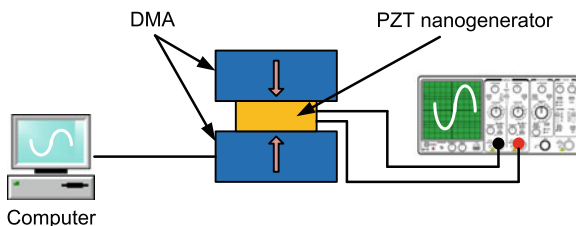
$$\Delta V = \int_0^l g_{33} \cdot E_p \cdot (\sigma_{xx}/E_{11} - \sigma_{yy}/E_{11} \cdot v - \sigma_{zz}/E_{11} \cdot v) dl \quad (9)$$

For a given applied load or impact energy, the maximum output voltage is primarily determined by the piezoelectric voltage constant. From our previous study, the piezoelectric voltage constant of PZT nanofiber is roughly 0.079 Vm/N, which is much higher than that of bulk PZT (0.025 Vm/N) or the PZT microfiber composite (0.059 Vm/N) [24]. By inspection, the significantly larger g_{33} and l/A ratio of the nanofiber generator should result in much higher voltage output compared to that of the PZT microfibers [18] under the same loading condition. For the same reason, PZT nanofibers can also be used as an ultra-high sensitivity force/vibration sensor.

6 Characterization of Nanogenerator

The characteristics of the nanogenerator as a potential power supply were investigated by measuring generated voltage versus strain in the polymer matrix, under varying dynamic load frequencies, and power output versus load resistance, both using a DMA. The experimental setup is shown in Fig. 7.

Fig. 7 The schematic of the experiment setup for the nanogenerator characterization test using the DMA. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society



The voltage generated by applying a harmonic force at a frequency of 250 rad/s (~ 39.8 Hz) and a specified maximum strain of 12% applied on the polymer matrix is shown in Fig. 8a. The positive and negative voltages were generated due to the sinusoidal load oscillations applied by the DMA (Fig. 8a). The peak to peak open circuit voltage, V_{p-p} , increased as the maximum strain applied increased. The maximum V_{p-p} was 1420 mV under a maximum applied strain along the PZT nanofibers of $\sim 7.5 \times 10^{-5}\%$ (established from mathematical and FEM models) at 250 rad/s, as shown in Fig. 8b.

The V_{p-p} versus various excitation frequencies under a maximum applied strain on the PDMS surface of 2.25% is illustrated in Fig. 8c. The highest output voltage of 62 mV occurred at a frequency of 220 rad/s (~ 35 Hz), corresponding to the lowest resonant frequency of the entire architecture. Voltage outputs were also recorded when varying the load resistances from 0.1 to 10 M Ω under a maximum specified strain of 10% applied to the PDMS surface and a harmonic load frequency of 250 rad/s (see Fig. 8d).

The power delivered to the load can be estimated from:

$$P_L = \frac{1}{T} \int \frac{V_o(t)^2}{R_L} dt \quad (10)$$

where $V_o(t)$ is the real-time voltage, R_L is the load resistance and T is the period of load application. The maximum measured output power reached 0.03 μ W with a load resistance of 6 M Ω , as shown in Fig. 8d.

7 Applications

Two applications of this nanogenerator are demonstrated. In the first application, the output voltage from the PZT nanofiber generator was measured when it underwent an impulsive loading, applied by tapping the top of the generator with a small Teflon[®] stack. As shown in Fig. 9a, the generated voltage, which was induced by piezopotential driven transient flow of electrons under the external load [25], reached 600 mV when a larger impact was applied on the nanogenerator by the periodic knocking. The higher the impact energy applied on the surface, the higher the output voltage generated by the device. The dampening effect of the soft

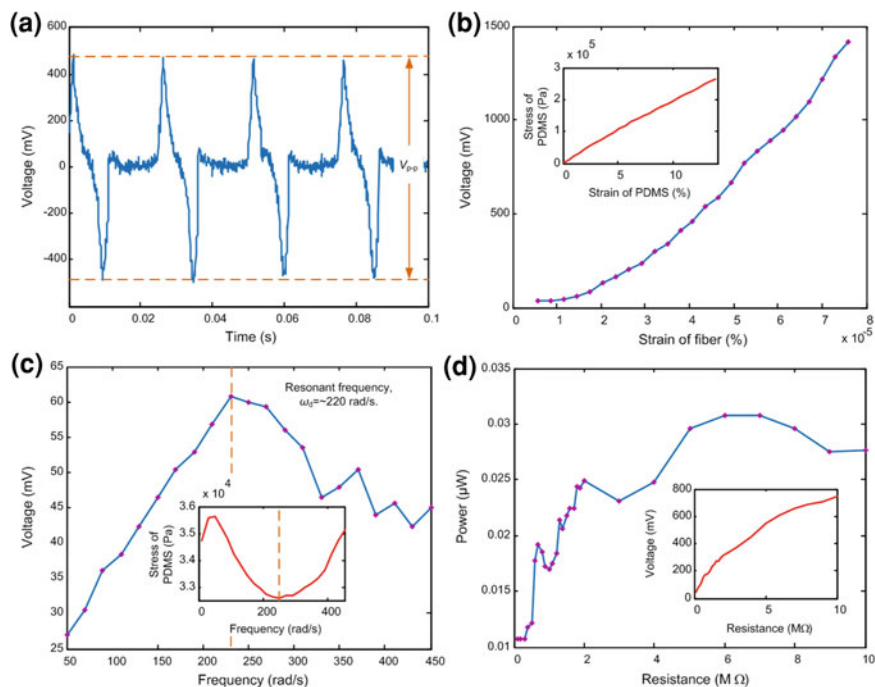


Fig. 8 The voltage generation properties of PZT nanofiber generator tested via DMA. **a** Voltage output when a harmonic force at the frequency of 250 rad/s (~ 39.8 Hz) and a maximum strain of 12% were applied on the PDMS surface. **b** The open circuit peak to peak voltage output versus strain of PZT nanofiber at the frequency of 250 rad/s (~ 39.8 Hz). The inset in **b** shows the stress of composites versus strain applied between the top and bottom PDMS surface. **c** The open circuit peak to peak voltage output versus frequencies of the harmonic forces at the maximum strain of 2.25% applied on PDMS. The inset in **c** shows the stress of composites versus frequency applied on the nanogenerator. **d** The power delivered to the load resistors versus the load resistance. The inset in **d** shows the voltage output versus the load resistance. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society

polymer matrix on the resonant frequency was also observed during the energy harvesting process.

In the second application, fingers were used to apply a periodic dynamic load to the top of the nanogenerator, during which positive and negative voltage outputs were observed (Fig. 9b). The negative voltage distribution was generated due to the carriers flowing in reverse when the external load was removed and the piezopotential vanished. The highest output voltage recorded during the test was 1.63 V. The amplitudes of the voltage outputs depended on how much pressure was applied on the nanogenerator surface.

In order to eliminate the influence of the bioelectric field of the human body and the electromagnetic interference from the testing equipment, a free vibration test using the PZT nanogenerator as a damper was conducted (Fig. 10a). The output voltage from the nanogenerator was measured when a Teflon[®] cantilever was

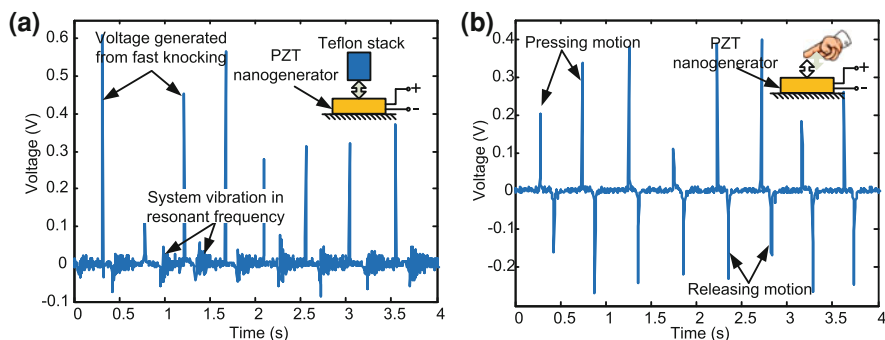


Fig. 9 Measurements of output voltage from PZT nanofiber generator. **a** Voltage output measured when a small Teflon[®] stack was used to impart an impulsive load on the top of the PZT nanofiber generator. The inset in **a** shows the schematic of a Teflon[®] stack tapping on the nanogenerator. **b** Voltage output measured when using a finger to apply a dynamic load on the top of the generator. The inset shows the schematic of a finger applying the dynamic load. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society

placed on top of the nanogenerator and subjected to free vibration, as shown in Fig. 10b. The damping ratio, ζ , and the natural frequency of this system were determined to be 0.064 and the 49.9 rad/s (~ 7.9 Hz), respectively. The output voltage from a dummy block without PZT nanofibers or any other active materials in it was also measured using the same setup. The measured result revealed that the amplitude of noise signal is only at about the 10 mV level. This confirmed that the power output from the PZT nanogenerator was in fact the energy harvested from mechanical vibration.

8 Conclusion

Energy harvesting technology that can increase operation time and decrease device size is urgently needed in wireless electronics, portable devices and implantable bio-sensors. This PZT nanofiber-based nanogenerator provides a promising solution for the challenges we are facing. This nanogenerator produces high voltage and power output, and has a high dielectric constant for a power source, which can be directly used for wireless sensors, personal electronics, implantable bio-sensors and bio-actuators. This low cost, flexible nanogenerator can also be packaged in bio-compatible polymers for embedding in shoes or clothes to harvest the kinetic energy of human body and charge batteries for devices such as iPods and cell phones on the go.

The peak output voltage from this nanogenerator was 1.63 V, and the output power was 0.03 μ W with a load resistance of 6 M Ω . The piezoelectric voltage constant and dielectric constant of PZT nanofibers were much higher than those of

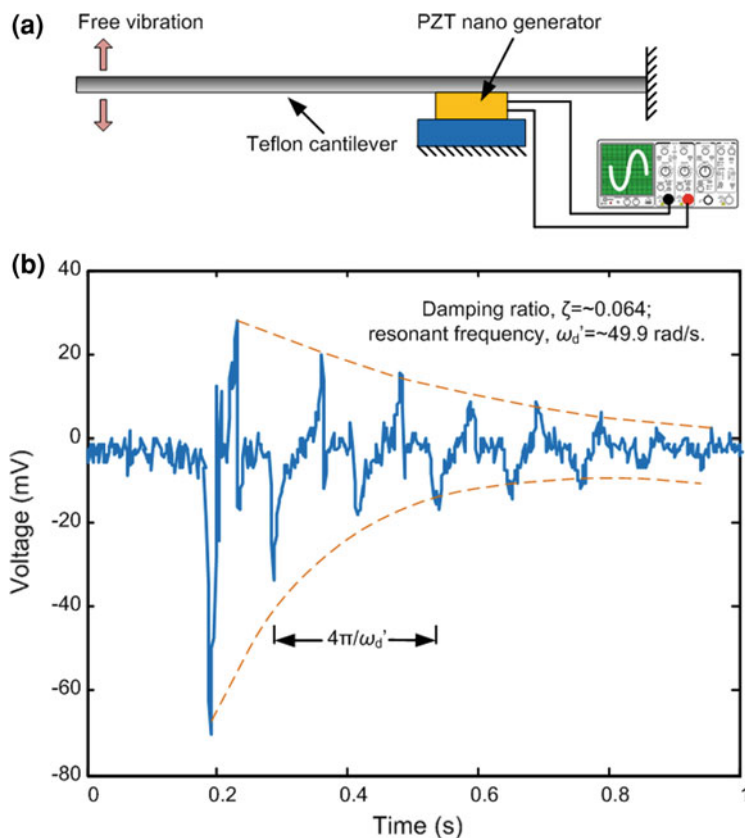


Fig. 10 Energy harvested from the free vibration of a Teflon[®] cantilever. **a** Schematic of the experimental setup. **b** The open circuit voltage output when the cantilever was under free vibration. Reprinted in part with permission from Ref. [13]. Copyright 2010 American Chemical Society

the semiconductor-type of piezoelectric nanowires and nanofibers, making this material ideal for nanogenerator or nanobattery applications. The flexible PZT nanofibers were embedded in soft PDMS polymer matrix, which helped to prevent the PZT nanofibers from being damaged, thereby extending the life cycle of the nanogenerator. The simple fabrication and assembly process would allow for the facile mass production of this type of nanogenerator.

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