Nano Energy (2015) 15, 598–606

Available online at www.sciencedirect.com

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journal homepage: www.elsevier.com/locate/nanoenergy

RAPID COMMUNICATION

Wireless, power-free and implantable nanosystem for resistance-based biodetection

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Received 3 April 2015; received in revised form 5 May 2015; accepted 8 May 2015
Available online 19 May 2015

KEYWORDS
Nanogenerator;
Energy harvesting;
Power-free nanosystem;
In-vivo biodetection;
Wireless transmission technology

Abstract
In-vivo devices and systems are extensively used in medical field to real-time detect and adjust the physiological status of human being, but supplying energy *in-vivo* for these devices and systems is still a great challenge. In this work, we first developed a new kind of wireless nanogenerator (WLNG) based on biocompatible BZT-BCT nanowires (NWs). It works through compressing and releasing BZT-BCT NWs/PDMS nanocomposite by a changing magnetic field in wireless non-contact mode. The maximum output voltage reaches 3.9 V, and the maximum output current is 1.17 μA, which are 21.9% larger than the reported maximum output voltage 3.2 V and 23.4 times of the reported maximum 50 nA of non-contact nanogenerator. And we further integrated it with a new kind of transmitter into a wireless, power-free and implantable nanosystem for *in-vivo* biodetection. This nanosystem does not need any electrical power. An *in-vitro* changing magnetic field can be used to drive it to detect the variation of resistance *in-vivo* and wirelessly transmit the signal to the equipments *in-vitro*.

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http://dx.doi.org/10.1016/j.nanoen.2015.05.003
2211-2855/

Introduction

Nowadays, *in-vivo* devices and systems are extensively used in medical field, such as detecting and adjusting physiological function of human *in-vivo* or substituting a lesion organ [1-4]. Till now, nearly all the *in-vivo* devices and systems rely on a battery for operation, but the capacities of a battery is still limited. Therefore, surgical procedures to replace the depleted batteries are inevitable, which bring many health risks to the patients [5]. So *in-vivo* powering these devices and systems is still a huge challenge which restricts the application of these technologies. Transporting energy wirelessly from *in-vitro* to *in-vivo* should be an effective way to solve this problem. In previous works, scientists have developed a technology on the basis of electro-magnetic induction [6] and ultrasonic wave [7]. But, the high frequency electromagnetic field or sound wave used in these technologies is harmful for the body and their penetration depth is limited [8-10]. So by now, it is still a great challenge to search a way safe to the body to power *in-vivo* devices.

Nanogenerator (NG) is a technology which could convert low frequency, weak mechanical energy into electrical energy based on the piezoelectric effect [11,12]. After increasing the output voltage to more than 1 V, [13] many devices and systems as UV sensors, [13-15] chemical sensors [16] and biosensors [17] have been powered by the NG. In principle, the output power of NG is large enough to power many *in-vivo* devices, which makes NG a good candidate as an *in-vivo* power source. But powering an *in-vivo* biodetection system by a NG is still infeasible for the following reason. First, the energy export by human movements is unstable. Second, harvesting these movements may influence the normal work of human organ. Third, the energy generated by an *in-vivo* NG is still too low to directly power a wireless transmitter without energy storage. So it is almost impossible for NG to power the medical devices at present stage. In this work, we developed a power-free nanosystem for all time, wireless and *in-vivo* biodetection. In this nanosystem, a high performance wireless NG driven by a changing magnetic field was used for power supplying. As magnetic field could cross over human body without any hindrance and act on any materials with ferromagnetic property, this NG could be driven by a changing magnetic field applied *in-vitro* and provide energy for the nanosystem. In this way, the output is stable and influences less on the normal work of human organ. Its maximum output voltage reaches 3.9 V, and the maximum output current is 1.17 μA, which are 21.9% larger than the reported maximum output voltage 3.2 V and 23.4 times of the reported maximum 50 nA of non-contact nanogenerator [18]. Then, a new wireless transmitter with low energy consumption was integrated with the WLNG into a nanosystem, which could work *in-vivo*, send the *in-vivo* resistance's response to the *in-vitro* equipment. This power-free nanosystem makes it possible to all time, wirelessly and *in-vivo* detect the physiological parameters that can influence the nanodevice's resistance.

Material and methods

Preparation of BZT-BCT nanowires

The BZT-BCT NWs are fabricated by the electrospinning method shown in previous works [21]. First, tetrabutyl titanate (2.4750 g) is mixing with ethanol (3 g), acetylacetone (1.5 g), acetic acid (9.75 g) and stirring until homogenous. After that, calcium hydroxide (0.0900 g), barium hydroxide octahydrate (2.1717 g), zirconium acetylacetonate (0.3939 g) and polyvinylpyrrolidone (0.53 g) are added into the solution in order, each composition is added after the previous one dissolved totally, the precursor solution is prepared after stirring homogenously. The solution is

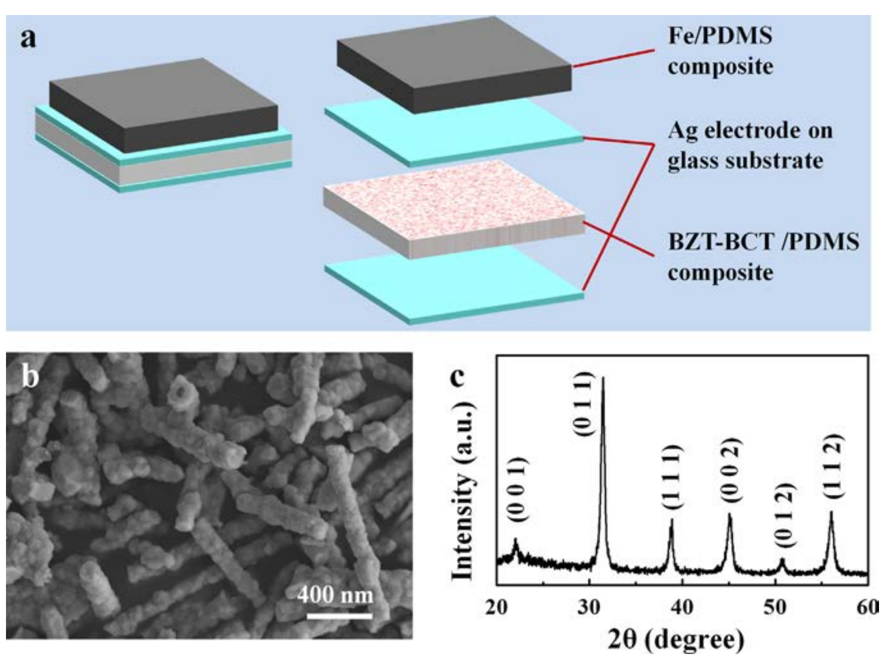

Figure 1 Structure of the WLNG. (a) Schematic image showing the structure of the WLNG. (b) SEM image of the BZT-BCT NWs after grinded. (c) XRD spectrum of the BZT-BCT NWs.

electrospun with 23 kV voltage and 20 cm distance between the needle and the collector. The NWs collected by the collector are annealed (850 °C) for three hours at a ramping rate of 2 °C/min.

Preparation of the magnetic film

Iron powder is mixed with as prepared PDMS (mass ratio of the monomer and the cross-linker is 10:1) in a 3:1 ratio (w/w), then the mixture is placed in a plastic culture dish. After it is solidified, the film is taken out from the culture dish and incised into needed size, the thickness of the film is two to three millimeter.

Fabrication of the WLNG

The as prepared BZT-BCT NWs are first grinded in a mortar. The BZT-BCT powder is then mixed with as prepared PDMS in a 1:9 ratio (w/w). Then, the mixture is spin-coated on a cleaning cover glass substrate (width and length are both 24 mm) with Ti/Ag electrode on one surface at 500 rpm for 30 s, a wire is fixed on one edge of the glass substrate by carbon paste. The glass substrate with the mixture is standed for more than one hour after spin-coating and then heated in an oven at 80 °C for one hour. After the mixture solidified, another cover glass substrate (width and length are 18 mm) with Ti/Ag electrode covered on its whole surface is fixed on the surface of the mixture by the natural stickiness of PDMS, a wire and the magnetism film are fixed on the top surface by carbon paste and PDMS, respectively. The device is coated by a thin PDMS film at last to make it insulation *in-vivo*. At last, the WLNG is polarized by applying an electric field of 2 V/μm at 150 °C.

Measurment of the WLNG

The WLNG was put in the magnetic field generated by an electromagnet, the magnetic field intensity was changes by adding different voltage on the electromagnet, and the magnetic field intensity was measured with a gauss meter. The output voltage and current of the WLNG was measured by Stanford Research Systems (low-noise preamplifier SR560 and low-noise current preamplifier SR570).

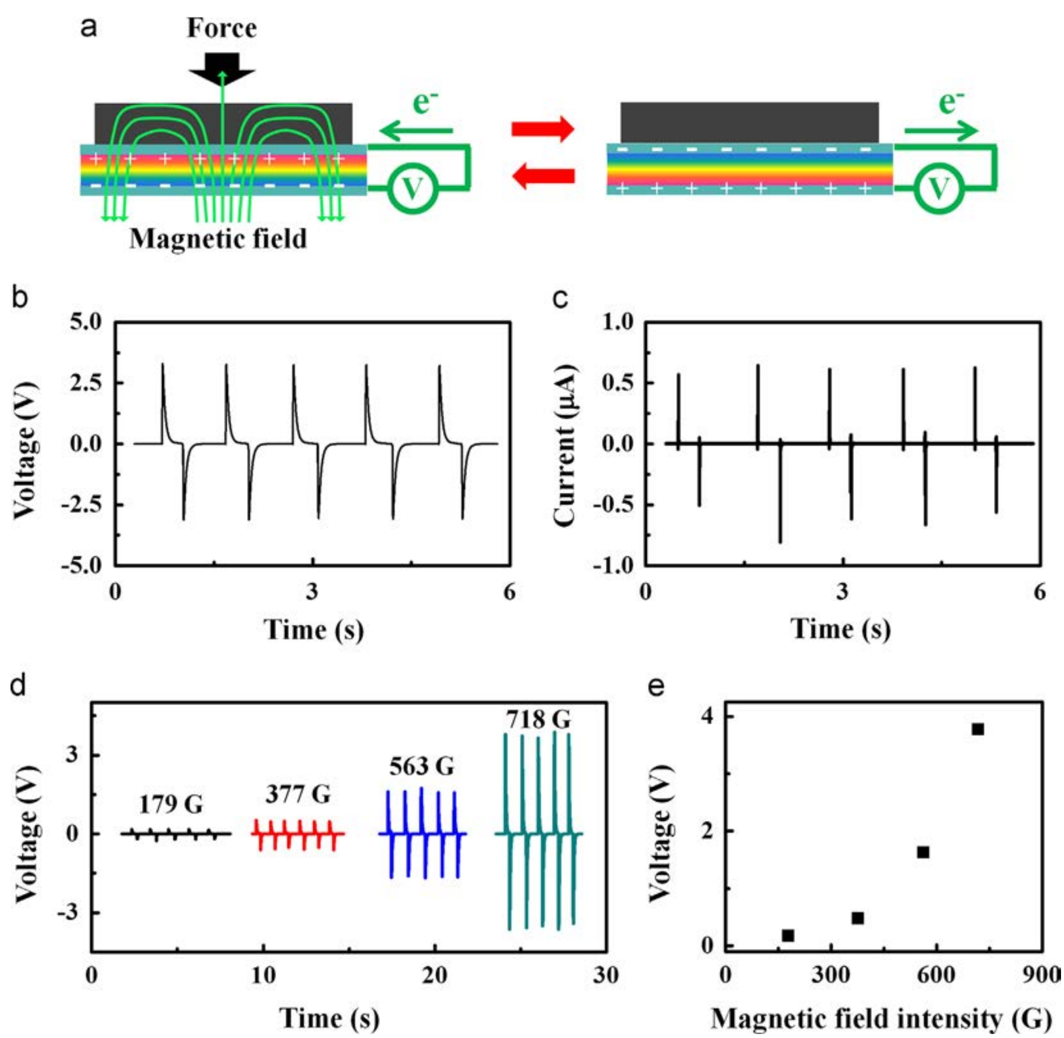

Figure 2 Output of the WLNG. (a) Working mechanism's schematic of the WLNG. (b) Output voltage of the WLNG. (c) Output current of the WLNG. (d) The voltage–time curve under the magnetic field intensity marked in the image. (e) The plot of peak output voltage with the magnetic field intensity.

Results and discussion

The wireless nanogenerator

For wireless *in-vivo* biodetection, three units are needed, a power supplying unit, a sensing unit and a wireless data transferring unit. Here, we integrate the power supplying unit and the wireless data transferring unit into a wireless nanosystem in which a WLNG is used as the power unit.

As sketched in Figure 1a, the WLNG has a stratified structure containing two parts: a PDMS layer mixed with biocompatible $0.5Ba(Zr_{0.2}Ti_{0.8})O_3$-$0.5(Ba_{0.7}Ca_{0.3})TiO_3$ (BZT-BCT) [19] sandwiched by two glass substrates coated with Ag electrodes and a PDMS layer mixed with iron powder attached on the top of the device. When putting the device in an inhomogeneous magnetic field, the film with iron powder squeezes the device, and the film with BZT-BCT NWs converts mechanical energy into electronic energy. Details of the fabrication process are described in the part of methods. Figure 1b and c shows the scanning electron microscope (SEM) image and X-ray diffraction (XRD) spectrum of BZT-BCT NWs, indicating that the diameter and length of the NWs are 100–150 nm and 1–2 μm, respectively, and the XRD spectrum of the NWs corresponds with that shown in previous works [20,21].

Figure 2a shows the working process of a WLNG. When applying an inhomogeneous magnetic field on the device, the iron powder is attracted by the magnet, compressing the film with BZT-BCT NWs and generating positive and negative charge on the top and bottom surface of the film, respectively. The potential difference between the electrodes leads to electrons flowing from the bottom electrode to the top electrode and accumulating at the top electrode, which makes the current flowing reversely from the top electrode to the bottom electrode. When the magnetic field is removed, the charge generated by piezoelectric effect disappears, leading to the accumulated electrons flow back

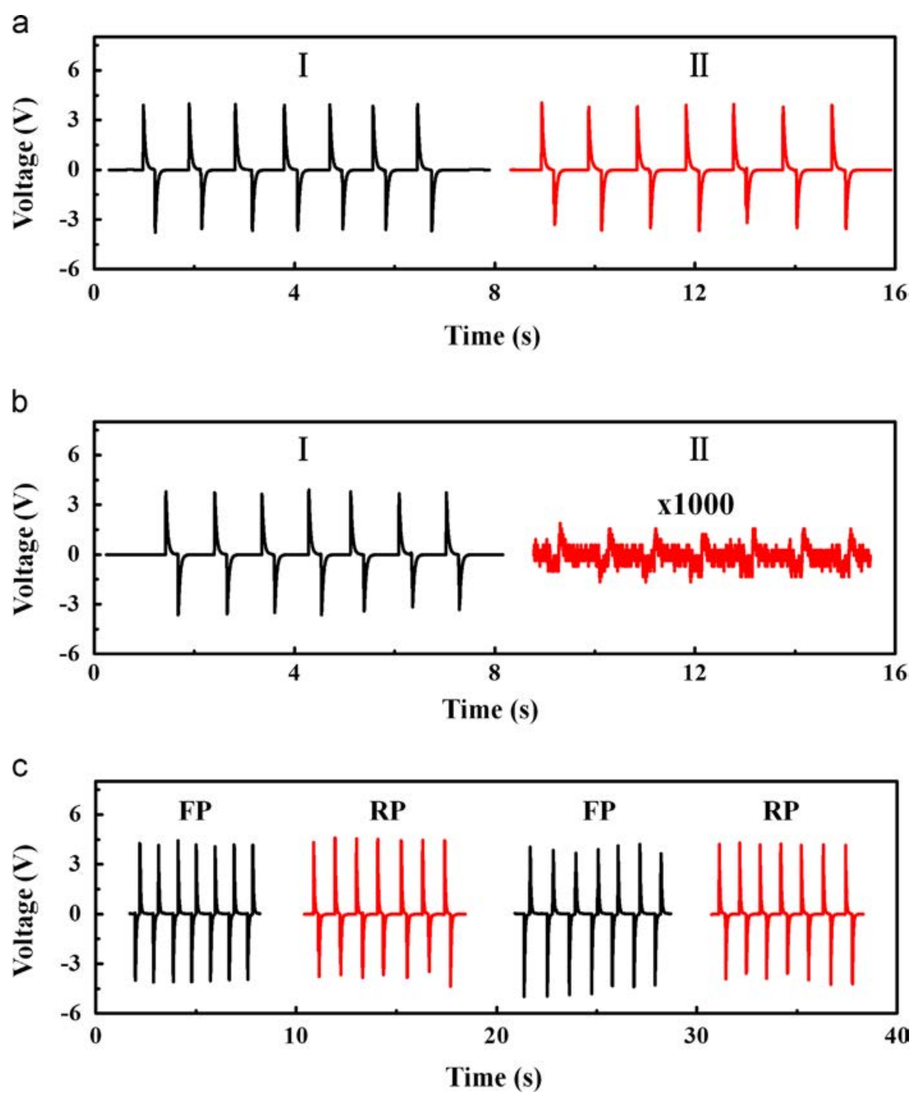

Figure 3 Verification of the output signal. (a) Output voltages of the WLNG before (I) and after (II) reversing the direction of current flowing in the electromagnet. (b) Output voltages of the WLNG before (I) and after (II) removing the magnetism film of the device, the right curve shown in this image is 1000 times of its actual value. (c) Output voltages of the WLNG with reversed polarization directions, FP and RP marked in this image indicate the polarization directions, respectively.

and the current flows from the bottom electrode to the top electrode.

Figure 2b and c shows the output of WLNG driven by changing the magnetic field through an electromagnet. The output voltage reaches 3.3 V, and the current reaches 810 nA. A "switching polarity" testing method [11,22] was employed to rule out the system artifacts. The voltage and current reversed their signs when the connection manner changed from forward connection (FC) to reverse connection (RC), indicating they are not noises coming from the measurement equipments (shown in Supplementary Figure S1). The WLNG's output could be controlled easily by changing the magnetic field intensity, Figure 2d and e shows the output voltage under different magnetic field intensity. Because the force is related with both of the magnetic field intensity's gradient and the magnetization intensity of the iron powder, the voltage shows nonlinear relationship with the magnetic field intensity.

As an electromagnet is used in the measurement, it is necessary to clarify that the signals are not produced by electromagnetic induction. Two experiments are made to exclude this possibility. First, if the signal is produced by electromagnetic induction, the signal should change with the direction of the current flowing in the electromagnet, on the contrary, the sign of the output produced by piezoelectric effect will not change with the current flowing in the electromagnet. On the basis of this deduction, we tested the output voltages of a WLNG under the same condition except for reversing current in the electromagnet. The experimental result (shown in Figure 3a) shows equal output voltage under these two conditions, indicating that the output is not produced by electromagnetic induction. Second, because the magnetic force acted on the film with iron powder, if this film is removed, the signal produced by piezoelectric effect will disappear, but the noise coming from the electromagnetic induction will not. We measured the output voltages of a WLNG before and after removing the film with iron powder. As Figure 3b shows, the output voltage of the WLNG before removing the film with iron powder is 3.8 V, and the output voltage of the WLNG after removing the film with iron powder is about 1.7 mV. This result further indicated that the magnetic field only supply a mechanical force on the device in the WLNG, and the effect of electromagnetic induction is ignorable. Additionally, the WLNG is reversely polarized to exclude the noise produced by static electricity existing in the device. As Figure 3c shows, the output voltage of the WLNG changes its sign after reversing the polarization direction but the value of the peak voltages change little after twice polarization at each direction, which proves again that the output voltage and current are true signals from piezoelectric effect.

The results shown above indicate the WLNG could be driven by an inhomogeneous magnetic field and generate electronic energy through piezoelectric effect. As magnetic field could pass through anything without ferromagnetism, the WLNG could be used inside human body to power the implanted medical devices or be applied in other conditions where mechanical movements are not available.

To check the work status of WLNG inside body, as shown in Figure 4a-c, a WLNG is implanted into the subcutaneous part of a rabbit and driven by an electromagnet placed *in-vitro*. Both the output voltage and current of the WLNG are measured before and after implanting, respectively (Figure 4d-e and Supplementary Figure S2). For this WLNG, before being implanted *in-vivo*, the output voltage is 3.6 V and current is 1170 nA. After being implanted *in-vivo*, the voltage decreases to 1.2 V and current decreases to 510 nA. The output decreases owing to the following two reasons. The increase of distance ,between the device and the electromagnet, leads to the decrease of magnetic field intensity, and the tissue covering the device acts as the buffer layer and decreases the deformation rate of the piezoelectric layer. The combined effect decreases the WLNG's output to about one third. Although comparing with

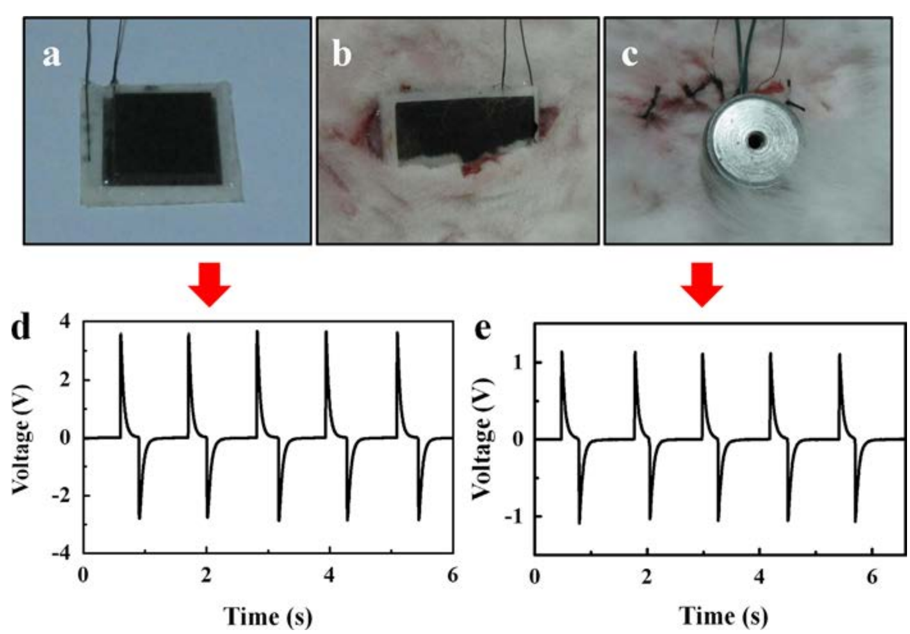

Figure 4 *In-vivo* application of the WLNG. (a-c) Optical images of the WLNG before, during the process and after being implanted into the subcutaneous part of a rabbit. (d,e) Output voltages of the WLNG working *in-vitro* (d) and *in-vivo* (e).

the WLNG measured *in-vitro*, the implanted NG has a relative small output, it is still large enough to drive some nanodevices.

New kind of wireless transmitter

Then, we designed a wireless transmitter to transfer data from *in-vivo* to *in-vitro*. As Figure 5a shows, this transmitter consists of two electrodes connected with the signal source implanted *in-vivo* and two electrodes connected with a voltmeter put *in-vitro*. Working principle of this transmitter is schematized in Figure 5b, when the signal source added a voltage on the electrodes, the electrodes will take positive and negative charge, respectively, and producing opposite charge on the other two electrodes, adding a corresponding voltage on the voltmeter. The voltage measured by the voltmeter depends on the input signal's waveform and amplitude and the input signal could be derived exactly by the signal obtained by the voltmeter. Figure 5c shows the equivalent circuit of this transmitter, here, R stands for voltmeter's internal resistance, and C stands for the capacitance between two electrodes. If we use V_i stands for the input signal and V_o stands for the received signal, they should satisfy the following equation:

$$V_o = \sum_{\omega} \frac{R}{\frac{2}{i\omega C} + R} V_i(\omega)$$

Here, $V_i(\omega)$ and ω stands for V_i's fourier decomposition result. Using this equation, the input signal and output signal could be calculated from each other. Then, we simulated the condition that the *in-vivo* WLNG act as the signal source by the C language, and the result was shown in Figure 5d. We can see that, the output signal have similar waveform with the WLNG, and the peak value was lower than the input signal because some energy was consumed in the transmitter.

The nanosystem for resistance-based biodetection

Using the WLNG and the new wireless transmitter, we fabricated a power-free nanosystem to connect with a series of resistance to demonstrate the wireless and all time *in-vivo* resistance-based biodetection. As the schematic image shown in Figure 6a, this system consists of two parts: the power supplying unit and the data transferring unit. The power supplying unit contains a WLNG (implanted *in-vivo*) and an *in-vitro* electromagnet that is used for driving WLNG to power other *in-vivo* devices. The abovementioned new wireless transmitter acts as the data transferring unit, two electrodes are implanted *in-vivo* and connected in parallel with the type of resistance-based sensor and the WLNG, the other two electrodes are set *in-vitro* close to the *in-vivo* electrodes and connected with the voltmeter *in-vitro*. For biodetection, this nanosystem can be connected in parallel with resistance-based biosensor whose resistance changes monotonously with the physiological parameter being detected. With the changing of surrounding physiological parameter, the bio-sensor changes its resistance to a certain value corresponding with the value of the detected parameter. As the output voltage

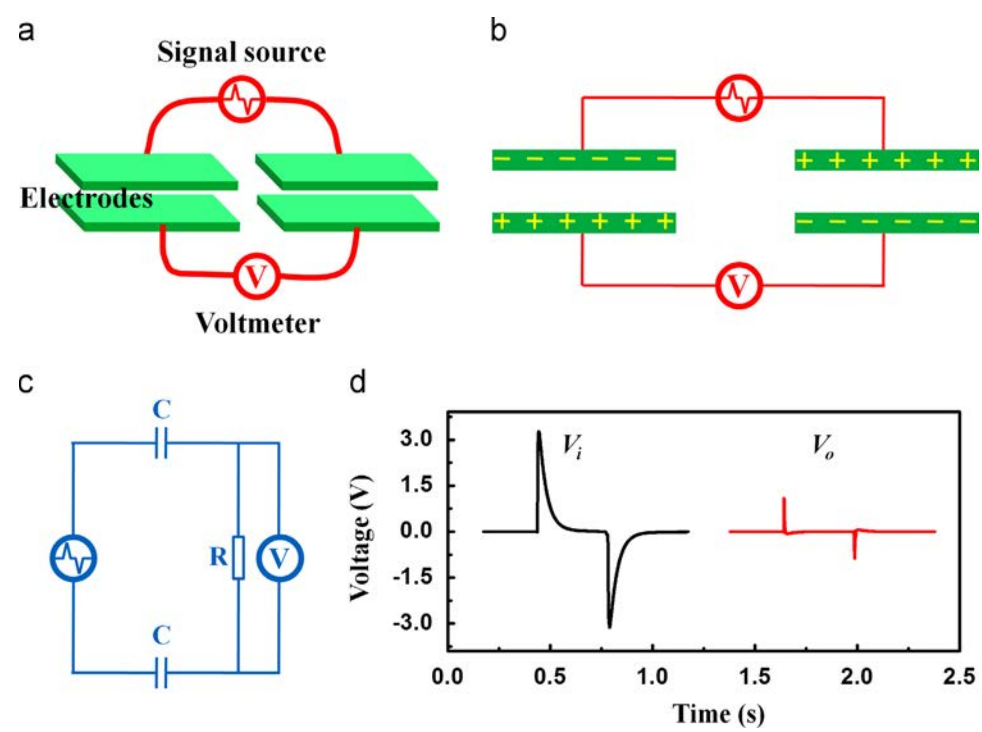

Figure 5 Structure and working principle of the wireless transmitter. (a) Schematic of the wireless transmitter, (b) working principle of this wireless transmitter, (c) equivalent circuit of the wireless transmitter and (d) calculated result of the voltmeter's signal when the a WLNG acted as the signal source, here V_i stands for the signal send to the transmitter and V_o stands for the signal measured by the voltmeter.

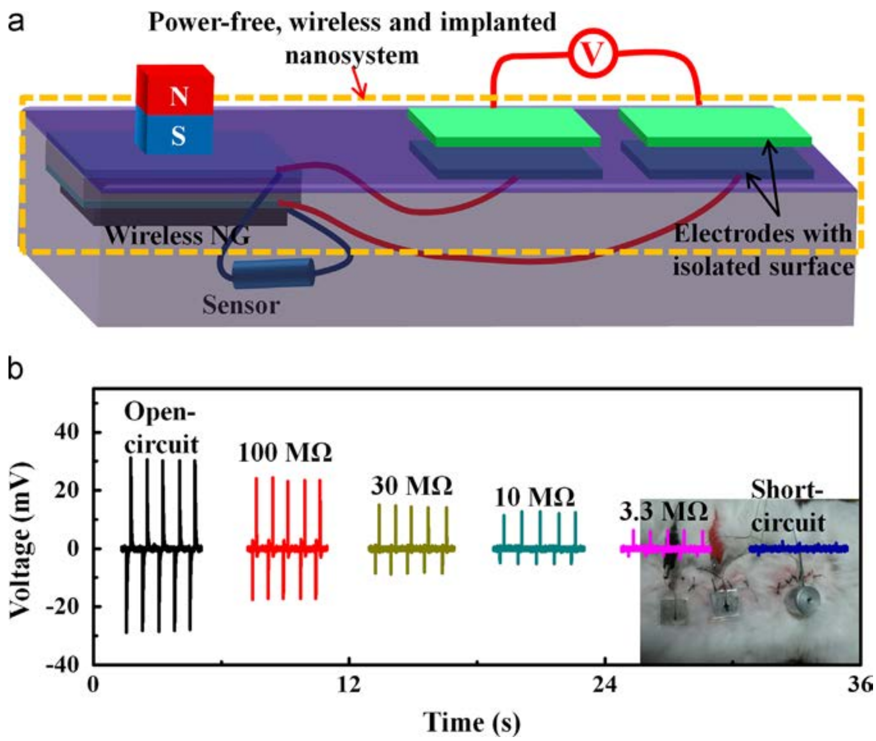

Figure 6 The wireless, power-free and implantable nanosystem. (a) Schematic image of the power-free and wireless nanosystem, the devices in the yellow box shows the power-free nanosystem, and the devices in the lavender area are implanted *in-vivo* into a rabbit, and the other devices are set *in-vitro*. (b) Voltage read from the *in-vitro* voltmeter at different *in-vivo* resistors replacing the resistance of a biosensor under different conditions, the words marked on each curve indicate the resistance of the resistors, and the insert shows an optical image of the system planted in the subcutaneous of a rabbit.

of a NG changes monotonously with the load resistance, the output voltage of the WLNG will change to a certain value corresponding with the detected parameter, this voltage is then sent to the wireless transmitter and the physiological parameter could be figured out *via* reading the voltage from the *in-vitro* voltmeter.

Because most of biosensors detect the bio-information through changing their resistance corresponding to different bio-environment, in the experiment, the bio-sensor is replaced by a group of resistors with different resistances to simulate the signal change of bio-sensor, and the system is implanted into the subcutaneous part of a rabbit. Figure 6b shows the voltage–time curve read from the voltmeter with different resistances, and the reverse connecting result shown in Supplementary Figure S3 indicates this is a true signal. This result corresponded well with the theory discussed above, and the signal shows same waveform with the calculated result. When the resistance decreases, the peak value of the voltage reading from the voltmeter decreases accordingly, and because of this one-to-one relationship between the resistance and the peak voltage, the bio-sensor's resistance could be figured out easily. As the energy consume in this system is transported wirelessly *in-vivo*, and the signal is read wirelessly *in-vitro*, the system's lifetime is no longer limited by the energy stored in the *in-vivo* system. Additionally, this system is compatible with most biosensors whose resistance changes with the detected physiological parameter, such as the biosensors based on the field effect [23,24].

Conclusions

In summary, we developed a power-free nanosystem for all time, wireless and *in-vivo* biodetection. This nanosystem contains a new kind of WLNG and a new wireless transmitter. The WLNG's performance is much better than that of previous non-contact nanogenerator, especially its output current is 23.4 times of the reported maximum 50 nA of non-contact nanogenerator. And energy cost of the new wireless transmitter is very low that it could be driven directly by a NG without energy storage. Using this nanosystem, we succesfully detected the *in-vivo* resistance's change and sent the information outside. This power-free nanosystem supplies a new general technical solution of wireless, power-free and all time mode for *in-vivo* resistance-based biodetections by connecting it with a resistance-based biosensor. It will contribute to the monitoring of *in-vivo* physiological information, the disease diagnosis and health monitoring.

Acknowledgments

Research was supported by NSFC (NO. 51322203 and 51472111), the Fundamental Research Funds for the Central Universities (No. lzujbky-2014-m02), and the Thousands Talents program for pioneer researcher and his innovation team, China.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.nanoen.2015.05.003.

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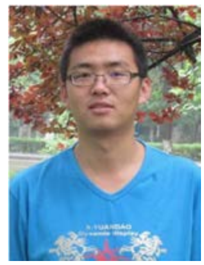

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Zhong Lin Wang received his Ph.D. from Arizona State University in physics. He now is the Hightower Chair in Materials Science and Engineering, Regents' Professor, Engineering Distinguished Professor and Director, Center for Nanostructure Characterization, at Georgia Tech. Dr. Wang has made original and innovative contributions to the synthesis, discovery, characterization and understanding of fundamental physical properties of oxide nanobelts and nanowires, as well as applications of nanowires in energy sciences, electronics, optoelectronics and biological science. His discovery and breakthroughs in developing nanogenerators established the principle and technological road map for harvesting mechanical energy from environment and biological systems for powering a personal electronics. His research on self-powered nanosystems has inspired the worldwide effort in academia and industry for studying energy for micro-nano-systems, which is now a distinct disciplinary in energy researchand future sensor networks. He coined and pioneered the field of piezotronics and piezophototronicsby introducing piezoelectric potential gated charge transport process infabricating new electronic and optoelectronic devices. Details can be found at: http://www.nanoscience.gatech.edu.