Full paper

A flexible photo-thermoelectric nanogenerator based on MoS$_2$/PU photothermal layer for infrared light harvesting

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A B S T R A C T

Recently, ambient energy harvesting has attracted a great deal of attention to develop clean and renewable energy technologies. In this work, a flexible photo-thermoelectric nanogenerator (PTENG) based on MoS$_2$/PU photothermal film and Te/PEDOT thermoelectric layer has been demonstrated for harvesting environmental infrared (IR) light. The MoS$_2$/PU photothermal film is carefully designed to exhibit excellent flexibility, transferability, and photothermal property. By integrating the photothermal layer with a Te/PEDOT thermoelectric device, the PTENG can produce electrical output without a spatial temperature gradient which is necessary for conventional thermoelectric device. Under IR light illumination, a high temperature difference can be generated across the device, and hence a potential difference established between two electrodes based on a coupling of photothermal effect and Seebeck effect. This type of PTENG exhibits numerous advantages, such as flexible, shape-adaptive, light-weight, and simple-fabrication, which may have a great potential of application in photo-thermoelectric energy harvesting for wearable electronics and implantable electronics.

1. Introduction

With the rapid growth of economy, the fossil fuel consumption has been sharply increasing to satisfy the energy demand, which results in global warming and environmental pollution [1]. It is crucial to develop clean and renewable energy technology for the sustainable development of human society [2]. In this regard, energy harvesting technologies which convert ambient energy into electricity have attracted great attention and become prevalent [3]. Nanogenerator (NG) [4,5], originated from Maxwell's displacement current [6], has been widely recognized as a promising approach for mechanical energy harvesting with huge potential in blue energy [7], self-powered sensors [8], and implanted systems [9]. In addition to mechanical energy, thermal energy is greatly abundant and universally existing in our living environment, but it has been accustomed to waste. Targeting at scavenging thermal energy, both pyroelectric effect and thermoelectric effect have been utilized to develop thermal energy harvesting technology. The pyroelectric effect is defined as the change in spontaneous polarization in certain anisotropic solids which is caused by the temperature change [10]. The electricity will be generated due to the asymmetric potential at two polarized surfaces. Additionally, the electricity can also be generated by a temperature gradient across a material/device when the thermoelectric effect is referred to a phenomenon [11]. Compared to the pyroelectric energy harvester which has little energy output, thermoelectric devices have been widely recognized as more effective and efficient technologies which has been utilized in practical applications [12]. However, how to harvest thermal energy by using thermoelectrics when the environmental temperature is spatially uniform without any gradients is still a crucial issue and needs to be conquered [10]. The key challenge is to create a significant temperature difference ($ΔT$) across the device which can act as driving force of thermoelectric generators [13].

In our living environment, in addition to the direct heat source, light source (such as the infrared light) can also provide thermal energy through the photothermal effect [14]. Based on the photothermal effect and Seebeck effect, photo-thermoelectric generator has been rapidly developed for converting photo energy into electricity without a spatial temperature gradient in the environment [13–15]. To generate a
necessary $\Delta T$, the conventional approach is utilizing various bulky components such as a vacuum enclosure, condenser lens, and heat sink [13]. Nevertheless, these extra modules will not only increase the weight and size of thermoelectric generators but also are unfavorable for the flexibility of the whole devices which is of great importance for wearable electronics. Recently, Jung et al. reported a wearable and portable photo-thermoelectric generator with superlattice structures to absorb solar light and generate a high $\Delta T$ in the lateral direction [16]. However, the solar absorber requires sophisticated design process and its toxicity may also be a big concern. Therefore, developing novel photothermal materials and device structure is essential for the photo-thermoelectric nanogenerator (PTENG).

Two-dimensional (2D) materials, such as graphene and transition metal dichalcogenides (TMDC), have attracted tremendous attention in the fields of electronics, catalysis, energy storage, and optical device [17,18]. Among them, molybdenum disulfide ($\text{MoS}_2$) is an important representative due to its excellent electronic behavior and mechanical properties [19,20]. Recently, $\text{MoS}_2$ has been demonstrated as an excellent photothermal material with higher absorbance in IR region than graphene oxide and gold nanorods [21,22]. Until now, most reported works only focus on biomedical applications, such as cancer therapy or drug release. There are rarely researches concerning about the application of energy harvesting technology, especially for photo-thermo-electric generator.

In this work, we report on a flexible photo-thermoelectric nanogenerator (PTENG) by hybridizing $\text{MoS}_2$/PU photothermal layer with tellurium (Te) nanowire based thermoelectric device. The $\text{MoS}_2$/PU film which is flexible and transferable exhibits excellent photothermal characteristics due to exceptional surface-area-to-mass ratio of $\text{MoS}_2$ nanoclusters [21]. Te nanowire is chosen for the thermoelectric nanogenerator because of its reported outstanding thermoelectric properties, such as low thermal conductivity and a wide temperature range [23]. By integrating the photothermal layer with thermoelectric device, the PTENG can absorb infrared light to form a temperature difference across the device. With this, a potential difference between two electrodes can be established and used for electrical energy generation. Therefore, the PTENG can generate electricity without a spatial temperature gradient. Furthermore, the PTENG which is flexible and shape-adaptive can demonstrate great practical application of photo-thermo-electric energy harvesting for wearable electronics and implantable electronics.

2. Experimental work

2.1. Synthesis of $\text{MoS}_2$ nanoclusters

$\text{MoS}_2$ nanoclusters were synthesized by a hydrothermal method [24]. 0.608 g ammonium molybdate and 1.109 g thiourea were dissolved completely in 20 ml double-distilled water. After that, 200 μl 3-mercaptopropionic acid was added to get the precursor solution. Then, the above solution was transferred into a Teflon-lined stainless steel autoclave and loaded into a furnace, which was heated to 220°C. The temperature was maintained at 220°C for 18 h. After finishing the reaction, the autoclave was cooled down to room temperature. Subsequently, the final products were centrifuged and washed with double-distilled water for several times and finally dried at 65°C to obtain pristine $\text{MoS}_2$ nanoclusters.

2.2. Preparation of tellurium nanowires

Tellurium (Te) nanowires were prepared through a hydrothermal reaction process [25]. Firstly, 1 g polyvinylpyrrolidone and 0.1 g sodium tellurite were dissolved in 33 ml double-distilled water. Then, 1.67 ml hydrazine monohydrate and 3.35 ml ammonia were added into the above solution. The obtained precursor solution was transferred to a Teflon-lined stainless steel autoclave and maintained at 180°C for 3 h in a furnace. After the autoclave was cooled to room temperature, the samples were centrifuged and washed with deionized water for several times and finally dried at 65°C to obtain pristine Te nanowires.

2.3. Fabrication of flexible and transferrable $\text{MoS}_2$/polyurethane film

Polyurethane (PU) and double-distilled water were mixed at a volume ratio of 3:1 to make a solution. Subsequently, different quantities of $\text{MoS}_2$ nanoclusters were added into the above solution with rigorous vortexing to get different weight percentages of $\text{MoS}_2$ (0, 0.5, 1, 2, 3 wt %) in the mixture. After that, about 100 μl of the mixture solution was coated onto PET substrates with size of $1 \times 1 \text{cm}^2$. Finally, the samples were dried at 65°C for 2 h to obtain flexible and transferrable $\text{MoS}_2$/PU films.

2.4. Preparation of flexible Te/PEDOT film

Isopropanol (IPA) and poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) were mixed at a volume ratio of 1:1, and then 0.0144 g Te nanowires were added with rigorous vortexing. To obtain flexible Te/PEDOT films ($1 \times 5 \text{cm}^2$), 250 μl of the above mixture was coated onto the PET substrates and dried at room temperature.

2.5. Fabrication of flexible photo-thermoelectric nanogenerators

The above achieved flexible Te/PEDOT nanocomposite films were chosen as the thermoelectric layer. Next, thermoelectric nanogenerator (TENG) was fabricated by depositing a pair of electrodes made of silver paste onto the top of Te/PEDOT films with size of $1 \times 1 \text{cm}^2$ and left-to-right spacing of 3 cm. Subsequently, the photo-thermoelectric nanogenerators (PTENG) were achieved by transferring $\text{MoS}_2$/PU films onto one of the electrodes to act as photothermal layers to generate a temperature difference ($\Delta T$) between the two electrodes.

2.6. Characterizations

A field emission scanning electron microscope (FESEM, JSM-7600F) was used to characterize the morphology of the prepared $\text{MoS}_2$ and Te nanomaterials. In addition, Raman spectrometer (JASCO, RFT-6000) was applied to investigate the structure properties of the two kinds of nanomaterials in this study. Infrared (IR) camera with infrared thermal imager was adopted to get the thermal image of $\text{MoS}_2$/PU film illuminated by an infrared (IR) diode laser (Jiahuang technology, 808 nm, 1 W). Photograph of $\text{MoS}_2$/PU films were obtained by metallographic microscope (BA210Met). The optical power measurement was realized by a premier single channel laser power and energy meter (Edmund Optics, 89313/89306). An electrochemical analyzer (CHI621E) was employed to test the electrical performance of thermoelectric devices.

3. Results and discussion

The structure of flexible TENG was carefully designed and depicted in Fig. 1(a). As can be seen from the figure, there are one flexible PET substrate, one layer of Te/PEDOT nanocomposites served as the thermoelectric materials, a pair of Ag electrodes with left-to-right spacing of 3 cm, and a layer of $\text{MoS}_2$/PU film located on top of one electrode acted as photothermal agent.

Both the $\text{MoS}_2$ and Te nanomaterials were synthesized via one-step hydrothermal processes. The surface morphology and structure properties of the as-prepared samples were characterized by FESEM and Raman spectroscopy, respectively. As illustrated in Fig. 1(b), the $\text{MoS}_2$ nanoclusters exhibit a nanoflower-like structure with an average diameter of 2.5 μm and are composed of well-dispersed nanosheets, which are similar to the reported 3D $\text{MoS}_2$ nanomaterials [26]. Fig. 1(d) reveals the Raman spectroscopy of $\text{MoS}_2$ nanoclusters, which are composed of two characteristic Raman peaks at 376 cm$^{-1}$ and 407 cm$^{-1}$.
corresponding to the $E_{2g}^1$ and $A_{1g}$ modes of 2H-MoS$_2$, respectively [27]. Here, the in-plane $E_{2g}^1$ mode is arisen from an opposite vibration between Mo atom and two S atoms. And the $A_{1g}$ mode is attributed to an out-of-plane vibration of only S atoms along the opposite directions [26]. To further examine the structural characteristics of the product, X-ray diffraction (XRD) was carried out. As shown in Fig. S2, all the diffraction peaks can be readily indexed to hexagonal 2H-MoS$_2$ (JCPDS No. 73-1508) [26], which is in good agreement with the Raman analysis. Fig. 1(c) and Fig. S1 indicate the FESEM image of Te nanowires. The nanowires have an average width of 50 nm and average length of 1.5 µm. The one-dimension nanostructure (such as nanowire) can be utilized to modify thermoelectric materials owing to the stronger phonon scattering at the surface of the nanostructures and hence the lower thermal conductivity [12]. Fig. 1(e) shows the Raman spectroscopy of as-prepared MoS$_2$ nanoclusters. The strongest Raman peak is located at 112 cm$^{-1}$ which corresponds to the $A_1$ mode. This mode is related to chain expansion mode in which each atom moves in the basal plane. Moreover, there are two smaller peaks beside the $A_1$ peak with $E_1$ mode at 85 cm$^{-1}$ and $E_2$ mode at 131 cm$^{-1}$, which are caused by bond-bending and bond-stretching types with larger admixture in Te, respectively [28,29]. Fig. S3 shows the XRD pattern of the as-prepared sample. It can be observed that the product is a single phase of well-crystallized elemental Te with the hexagonal structure (JCPDS No. 36-1452) [25].

Because of excellent photothermal properties and biocompatibility, MoS$_2$ is widely used in the biomedical field to absorb IR light with temperature change [21,22]. For a wide variety of applications (such as wearable electronics), flexible and transferrable MoS$_2$ photothermal layers are highly desirable and favorable. To fabricate flexible and transferrable MoS$_2$ film, PU/water solution was adopted as solvent for well-dispersed MoS$_2$ nanoclusters in this work. Fig. S4 shows the photos of the as-fabricated MoS$_2$/PU films. To examine the photothermal performance of the samples, an IR laser ($\lambda = 808$ nm) with output power density of 2.625 W/cm$^2$ was used to illuminate the surface of photothermal films with different weight percentages of MoS$_2$ for 200 s. Meanwhile, an IR camera was employed to record the sample temperature in situ. Fig. 2(a) displays the temperature profiles and thermal images obtained from the samples after 200 s irradiation from IR laser. For all of the samples, the area within the laser beam exhibits the highest heating temperature. Besides, the heat dissipates outwards along the edge of laser spot, which illustrates that the rise in the temperatures is caused by the irradiation of IR laser. Moreover, it is clearly observed that the samples with larger content of MoS$_2$ exhibits higher surface temperature. The change in the temperature of the illuminated area with respect to time is plotted and shown in Fig. 2(c). All of the samples were heated rapidly from the initial temperature (room temperature) within 30 s and finally remain at equilibrium temperatures. With the increase in the content of MoS$_2$ in the photothermal films, the equilibrium temperatures elevate dramatically to about 337, 339, 340, and 343 K, corresponding to 0.5, 1, 2, and 3 wt% samples, respectively. The results illustrate that MoS$_2$ can absorb IR light and convert it into heat efficiently. To further investigate the photothermal effect of MoS$_2$, two samples without MoS$_2$ were fabricated and exposed to the IR laser with the same condition for comparison. As shown in Fig. 2(c), the equilibrium temperatures for the PET with Ag electrode and pure PU film were about 310 K and 322 K, respectively. The above temperatures are much lower than that of the MoS$_2$ samples. Therefore, it can be concluded that MoS$_2$ is an excellent photothermal agent with high

Fig. 1. (a) Structure of photo-thermoelectric nanogenerator (PTENG) based on MoS$_2$/PU photothermal film and Te/PEDOT thermoelectric layer; (b) SEM image of as-prepared MoS$_2$ nanoclusters; and (c) Te nanowires; (d) Raman spectroscopy of as-prepared MoS$_2$ nanoclusters; and (e) Te nanowires.
Fig. 2. (a) Thermal images; (b) Optical microscope images of MoS$_2$/PU film with different weight percent (0, 0.5, 1, 2 and 3 wt%) of MoS$_2$; (c) Photo-thermal heating curves of Ag electrode (without MoS$_2$/PU film) and MoS$_2$/PU film with different weight percent (0, 0.5, 1, 2 and 3 wt%) of MoS$_2$.

Fig. 3. (a) Schematic diagram of thermoelectric nanogenerator (TENG) based on Te/PEDOT film heated by hotplate; (b) Open circuit voltage; (c) Short circuit current of the TENG at $\Delta T$ of 45 K; (d) Dependence of open circuit voltages on temperature differences caused by hotplate.
absorption efficiency of IR light. Here, the temperature elevation will be mainly originated from the photothermal effect of the MoS$_2$ nanoclusters which possess exceptional surface-area-to-mass ratio as shown in Fig. 1(b) [21].

Metallurgical microscope (BA210Met) was used to investigate the surface morphology of the MoS$_2$/PU films. As indicated in Fig. 2(b), MoS$_2$ nanoclusters distribute increasingly dense and uniform in the photothermal layer with the increasing weight percentage of MoS$_2$, corresponding to the enhanced photothermal performance. However, even though higher heating temperature can be achieved (as shown in Fig. 2(c)), breakages and cracks will occur and appear in the film when the content comes to 3 wt%. This drawback is unfavorable for the transfer process. Therefore, based on the above discussion, the photothermal film with MoS$_2$ content of 2 wt% was chosen for the following discussion. The reason for the occurrence of the breakages and cracks may be described as follows: When the weight percentage of MoS$_2$ comes to 3 wt%, it is difficult to disperse MoS$_2$ nanoclusters uniformly in the PU aqueous solution since the MoS$_2$ nanoclusters are easier to aggregate. Thus, when the MoS$_2$/PU mixture solution was deposited onto the PET substrate for film fabrication, the content of MoS$_2$ in some regions may be much higher than 3 wt%, while that in other regions may be much lower than 3 wt% or even 0 wt%. The regions with different MoS$_2$ concentration may have different thermal expansion coefficient. Therefore, during the heating and drying process of the wet film (at 65 °C for 2 h), the film was likely to be cracked due to the thermal stress.

As for the TENG part, Te nanowires were selected as the thermoelectric materials due to its excellent thermoelectric performance, such as low thermal conductivity, high power factor, and a wide range from 300 to 700 K [23]. In the fabrication process, aqueous PEDOT:PSS solution was used for the film formation to improve the electrical conductivity of the thermoelectric layer. Then, a pair of Ag electrodes was deposited on top of the Te/PEDOT film with left-to-right spacing of 3 cm. Two Cu wires were connected to the Ag electrodes as lead wires for the electrical measurement. To examine the output performance of the TENG, a hotplate was applied as a heat source to generate a $\Delta T$ of about 1.9 mV under a voltage ($\Delta V$) shown in Fig. 3(b). The peak output voltage can reach about 1.5 mV when the exposed time was increased from 30 to 200 s, the ISC sharply from 0.6 mV and 0.1 mA ($\Delta V$) achieved 0.8, 1.0, 1.3, 1.5, 1.6, and 1.9 mV when the different $\Delta T$ was at 20, 25, 32, 36, 40, and 45 K, respectively. The variation trend can be fitted in a good linear relationship, which indicates its potential application in self-powered temperature sensors.

To fabricate flexible TENG, the MoS$_2$/PU film (2 wt%) was peeled off from the PET substrate and transfer onto the left electrode of the Te/PEDOT TENG with Ag paste as adhesive. After using a hotplate to dry the Ag paste, the photothermal film was attached firmly to the top surface of the electrodes. The thicknesses of the MoS$_2$/PU film, Ag layer, and Te/PEDOT film are 90, 25, and 10 µm, respectively. The assembled device exhibits excellent flexibility and deformability as shown in Fig. 4(b), which makes it shape-adaptive and thus applicable to even curved surfaces for wide practical applications. To investigate the photo-thermoelectric performance of the TENG, an IR laser (λ = 808 nm) was adopted to illuminate the electrode with MoS$_2$/PU film as depicted in Fig. 4(a). Once the device was exposed to the IR laser, the photothermal layer can absorb IR light and induce an increase in temperature of its own. As a result, it can act as a heat source for the attached electrode. Since the temperature of other electrode still remain at initial room temperature, there will be a temperature difference between two electrodes, and hence a potential difference built between them based on the Seebeck effect. This voltage will drive charge carriers flow through an external circuit and generate an output current. During the illumination, two factors will affect the electrical output of the TENG: one is the illuminating laser power, and the other is the illuminated time. Fig. 4(c) and (d) illustrate the relationship between the electrical output of TENG and the laser power under the same illumination time of 200 s. When the IR laser was turned on, the VOC increased rapidly and reached saturation values of about 1.2, 0.7 and 0.5 mV corresponding to the various laser power densities of 2.625, 2.3, and 2 W/cm$^2$, respectively. The higher laser power was introduced, the larger voltage could be achieved. This reveals a strong dependence between laser-power and photothermal effect for MoS$_2$/PU film [33]. When the IR laser was turned off after irradiation for 200 s, the VOC declined gradually to zero owing to the thermal convection between the device and the surrounding atmosphere. As for $I_{SC}$ similar changing tendencies were observed with the highest output value of 0.18, 0.06 and 0.03 µA at various laser power densities (2.625, 2.3, and 2 W/cm$^2$). The observation indicates that the tunable output could be achieved for the TENG. Fig. 4(e) and (f) illustrate the response of PTENG’s electrical output with different illumination times (30, 50, 100, 150 and 200 s) under the same laser power density of 2.625 W/cm$^2$. When the exposed time was increased from 30 to 200 s, the VOC and the $I_{SC}$ first rose sharply from 0.6 mV and 0.1 mA to 1.1 mV and 0.15 mA (for 100 s) and then saturated at the maximum values of 1.2 mV and 0.18 mA (for 200 s), respectively. The curves of photo-thermoelectric characteristics are similar to that of the photothermal property (Fig. 2(c)). This indicate that the temperature difference between the two electrodes is the driving force for the electrical output and the primary working mechanism of PTENG is formed by coupling of the photothermal effect and Seebeck effect. It is worth noting that the electrical output shows no obvious decay after 200 s illumination. Actually, the PTENG can stably generate steady electrical output under the illumination of IR laser (2.65 W/cm$^2$) for even 10 min, as shown in Fig. S7, which is much longer than the previous reported result (~20 s) [16]. Zhu et al. have
reported a light-concentrated solar thermoelectric generator with a long-duration time of 10 min [34]. However, it requires a bulky condenser lens and heat sink to maintain the stable output.

In the above experiment, the IR laser was adopted to irradiate the one electrode of PTENG. However, selective illumination by concentrated light sources is rare in our living environment. To explore the practical application, an infrared lamp (Philips BR125) was used to illuminate the PTENG which was attached onto a window as shown in Fig. 5(a) and (b). As discussed in Fig. 2(c), the MoS2/PU film and Ag electrode exhibit distinct photothermal properties which result in a higher temperature increment for the electrode with MoS2/PU film than the pure Ag electrode. Hence, a temperature difference between two electrodes will be formed. As a result, a potential difference will be generated based on Seebeck effect to drive electrons flow between two electrodes passing through the external circuit. From the Fig. 5(c), when the infrared lamp was turn on and lasting for 50 s, the $V_{OC}$ of PTENG increased rapidly and plateaued at 1.2 mV. To further demonstrate the shape-adaptive property and practical application of the PTENG, we connected ten PTENGs in series and attached them onto a rock with irregular surface, as shown in Fig. 5(d). By harvesting the photo-thermal energy of outdoor sunlight at an atmospheric temperature of 20 °C, the PTENGs can deliver an output voltage of 1.48 mV, which can be recorded directly by a digital multimeter. When the PTENG was reversed connected, the output voltage was reversed accordingly with the value of $-1.42$ mV (as shown in Fig. S9), which confirms the electrical signals are from the photo-thermoelectric output of the PTENG.

Compared to the previous reported photo-thermoelectric devices, the flexible PTENG possesses several unique advantages. First, the PTENG can stably generate electrical output for a long time (as long as 10 min) without any bulky cooling components such as a vacuum enclosure and heat sink. As a result, the device is light-weight and compact-size for wearable electronics. Secondly, the photo-thermal layer utilized in the PTENG is based on a simple MoS2/PU film which does not require sophisticated design process such as superlattice structures. In addition, the MoS2/PU photothermal layer is non-toxic and bio-compatible, which is essential for the wearable electronics. Consequently, considering the high flexibility of device, the PTENG has a great application potential in photo-thermoelectric energy harvesting for wearable electronics, such as solar photo-thermoelectric conversion.

4. Conclusion

In summary, a flexible PTENG based on MoS2/PU photothermal film and Te/PEDOT thermoelectric layer has been demonstrated and investigated systematically. The MoS2/PU layer is carefully designed to possess excellent flexibility, transferability and photothermal property by tuning the content of MoS2. By hybridizing the photothermal layer...
with a Te/PEDOT thermoelectric device, the PTENG can generate electrical output by harvesting environmental IR photo energy based on a coupling of photothermal effect and Seebeck effect. Further, the obtained PTENG exhibits numerous advantages, such as flexibility, shape-adaptiveness, light-weight, and simple-fabrication, which may have a great potential of application in photo-thermoelectric energy harvesting for wearable electronics and implantable electronics.

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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.2018.04.072.

References


Fig. 5. (a) Schematic diagram of the PTENG illuminated by IR lamp; (b) Photograph of the shape-adaptive PTENG attached on window; (c) Output voltages of the 10 PTENGs in series attached to a rock to harvest photothermal energy of outdoor sunlight at an atmospheric temperature of 20 °C.
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