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High-performance flexible photodetectors based on single-crystalline

Sb₂Se₃ nanowires

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Antimony selenide (Sb₂Se₃) has many potential applications in photoelectric devices, thermoelectric cooling devices and electrochemical devices *etc*. It also has many special properties due to its layered structure. A simple hydrothermal method was used to synthesize Sb₂Se₃ nanowires of high crystalline quality. (Sb₄Se₆)_n layers are parallel to the growth direction of Sb₂Se₃ nanowire. A single Sb₂Se₃ nanowire demonstrated a remarkable response to 635 nm light at 10 V with the responsivity and external quantum efficiency of 360 A/W and 7.0×10^4 %, respectively. The rise/fall time was 0.4/1.3 s. Flexible photodetectors were fabricated by dispersing a large number of Sb₂Se₃ nanowires onto the Au interdigitated electrodes on PET substrates, which showed fast response speed with the rise/fall time as low as 13/20 ms and excellent flexibility. The high-performance of photodetectors may be partially attributed to the layered structure. Generally, high-yield Sb₂Se₃ nanowires synthesized by the hydrothermal method are promising candidates for high-performance flexible photodetectors.

Introduction

Photodetectors are widely used in military and civil fields, such as binary switches, optical communications, flame sensors and missile detectors,^{1,2} In the last decade, one-dimensional (1D) semiconductor nanostructures have demonstrated their potential as the functional parts in photodetectors due to large surface-to-volume ratio and high crystalline quality. Wide band-gap nanomaterials, such as ZnS nanowires, ZnO nanorods, WO₃ nanowires, TiO₂ nanotubes, In₂Ge₂O₇ nanowires and Zn₂GeO₄ nanowires, have been investigated for UV-light detection.³⁻⁸ Ge nanowires, In₂Se₃ nanowires, CdSe nanowires, ZrS₂ nanobelts, Zn₃P₂ nanowires, ZrSe₃ nanobelts, HfSe₃ nanobelts, InP nanowires, GaP nanowires etc. have exhibited their promise in fabricating visible-light photodetectors.⁹⁻¹⁵ These reports confirm that photodetectors made from 1D nanostrucutres usually possess desirable performances, including high responsivity (R_{λ}) , fast response speed and excellent stability. Recently, flexible electronics have attracted more attention because of the need for portable devices.¹⁶⁻¹⁹ Compared to thin film and bulk materials, 1D nanostructures can be assembled easily into flexible electronics with high bending stability due to their large bending strain compliance.²⁰⁻²³

Sb₂Se₃ as an important member of group V-VI binary semiconductors can be used to fabricate electric, photoelectric, thermoelectric cooling and electrochemical devices, which is attributed to its direct and narrow band-gap, high environmental stability and Seebeck coefficient.²⁴⁻²⁷ It is a highly anisotropic semiconductor with the layered structure parallel to the [001] (c-axis) direction.^{28,29} Sb₂Se₃ have demonstrated

promise in solar cells due to its layered structure.³⁰ Thus, it can be believed that single-crystalline Sb₂Se₃ nanowires have many special properties owing to the carrier and photon confinement in the layered structure.²⁹ Sb₂Se₃ nanowires have been synthesized hydrothermal/solvothermal microwave-assisted by routes or methods.^{25,28,31} However, the studies on the application of Sb₂Se₃ nanowires are limited. Zhai et al. utilized individual Sb₂Se₃ nanowire to fabricate a photodetector with a marked response to 600 nm visible-light.²⁹ Choi et al. also studied the response of single Sb₂Se₃ nanowire to 655 nm visible-light.²⁷ The photodetectors based on Sb₂Se₃ nanowire films displayed a remarkable response to white light.³¹ The results above indicate that Sb₂Se₃ nanowires have bright prospects for visible-light photodetectors. However, these Sb₂Se₃ nanowire based photodetectors were fabricated on the rigid SiO₂/Si substrates, which do not meet the need for portable devices. To the best of our knowledge, the application of Sb₂Se₃ nanowires in flexible electronics has not been explored. Thus, it is necessary to further investigate the electrical transport and photoelectric properties of single Sb₂Se₃ nanowire, assemble a large number of nanowires into the flexible photodetector, and finally characterize its performances. It is desirable to achieve high-performance flexible photodetectors via a simple method.

In this work, a simple hydrothermal method was used to synthesize single-crystalline Sb₂Se₃ nanowires in high yields. Single Sb₂Se₃ nanowire field-effect transistors (FETs) demonstrated the p-type transport characteristic of single nanowire. They were also utilized to investigate the photoelectric properties of single Sb₂Se₃

nanowire. A single Sb₂Se₃ nanowire showed a remarkable response to 635 nm light at 10 V with high responsivity (360 A/W) and short response time (0.4/1.3 s). Flexible photodetectors with fast response speed (13/20 ms) and excellent flexibility were made by dispersing a large number of Sb₂Se₃ nanowires onto the Au interdigitated electrodes on PET substrates, revealing the potential application of Sb₂Se₃ nanowires in flexible photodetectors.

Experimental section

Synthesis and characterization of Sb₂Se₃ nanowires

Sb₂Se₃ nanowires were synthesized by a template-free hydrothermal route.²⁸ 0.01 g Sb(AcO)₃, 0.0085 g Na₂SeO₃ and 0.3 ml hydrazine hydrate (80 wt%) were dissolved to 25 ml distilled water and the reaction solution was transferred into a 30 ml autoclave. Then, the autoclave was kept at 120 °C for 24 h. The synthesized product was characterized by scanning electron microscopy (SEM, QF400) equipped with an energy-dispersive X-ray (EDX) spectroscopy and high-resolution transmission electron microscopy (HRTEM, JEM 2100F).

Electrical and photoelectric measurements of single Sb₂Se₃ nanowire

To investigate the electrical and photoelectric properties of single Sb_2Se_3 nanowire, single nanowire based FETs were fabricated. Nanowires were dispersed into ethanol and then transferred onto p⁺-Si substrates covered with a 150 nm SiO₂ layer. The source and drain electrodes (Cr/Au 20/100 nm) were attached onto two terminals of a single Sb₂Se₃ nanowire by e-beam lithography, resist development, metal deposition

and liftoff processes. The thin Cr layer serves as the adhesion layer to improve the adhesion of electrodes. Metal Al as the gate electrode was deposited on the back of Si substrate. FETs were characterized by using an electrometer (Keithley 6517A) to record I_{DS} - V_{DS} curves at different gate voltages. To investigate the responses of a single nanowire to light, I_{DS} - V_{DS} curves were measured under the illumination of 635 nm light with different power densities without a gate voltage. Its time responses were measured by the electrometer *via* periodically turning 635 nm light on-off.

Fabrication and characterization of flexible photodetectors

Flexible photodetectors based on a large number of Sb₂Se₃ nanowires were fabricated by dispersing nanowires onto the Au interdigitated electrodes on flexible PET substrates. The adhesion between the Au film and the PET substrate is good. The adhesion layer is not required. Thus, only the Au film was deposited as the electrodes. The interdigitated electrodes were made by Au deposition, photolithography, resist development, etching. Each electrode is composed of fifteen fingers with a width of 5 μ m, a length of 500 μ m and an interspacing of 5 μ m. The responses of photodetectors to visible-light were measured by a sourcemeter (Keithley 2635B).

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Results and discussion



Fig. 1 (a) SEM image of Sb₂Se₃ nanowires. (b) Crystalline structure of Sb₂Se₃.
(Sb₄Se₆)_n layers are parallel to the growth direction of Sb₂Se₃ nanowire.

Fig. 1a shows an SEM image of the synthesized product, revealing that the product is composed of a large number of ultralong Sb₂Se₃ nanowires. Their length is up to tens of microns. Sb₂Se₃ nanowires have a diameter of about 100 nm and a smooth surface. An EDX spectrum was taken from the synthesized product (Fig. S1), showing that the product includes elements Sb and Se with an atomic ratio of 2:3. The morphology and crystalline structure of Sb₂Se₃ nanowires were further characterized by HRTEM. Fig. S2(a) demonstrates a typical Sb₂Se₃ nanowire with a uniform diameter of 98 nm along the growth direction and a smooth surface. Fig. S2(b) shows the HRTEM image and corresponding selected area electron diffraction (SAED) pattern of the nanowire, confirming that the Sb₂Se₃ nanowire is of the orthorhombic structure and it is a single crystal, free from dislocations and grows along the [001] direction. The (Sb₄Se₆)_n layer parallel to the [001] direction is the basic unit of Sb₂Se₃.

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growth direction of the nanowires, as shown in Fig. 1b. Carriers can be confined in the layered structure and transport efficiently along nanowires.³⁰



Fig. 2 I_{DS} - V_{DS} characteristics of a single Sb₂Se₃ nanowire FET at different V_G . Insets are a schematic illustration and an SEM image of the FET, respectively.

Single Sb₂Se₃ nanowire based FETs were fabricated to investigate the electrical transport properties of single nanowire. The upper inset in Fig. 2 is a schematic illustration of the FET. The lower inset in Fig. 2 is an SEM image of the FET, showing that two Cr/Au electrodes are attached onto a single Sb₂Se₃ nanowire. The FET's channel is about 6.4 μ m in length and 135 nm in width. Fig. 2 displays I_{DS} versus V_{DS} curves at a different back-gated voltage (V_G) varying from -30 V to 30 V. The nonlinear I_{DS} - V_{DS} curves indicate Schottky contacts between the nanowire and Cr/Au electrodes. The conductance of the Sb₂Se₃ nanowire decreases with the increasing of positive V_G , proving that the nanowire is p-type. Compared to bulk materials, nanomaterials could have different electrical transport properties due to

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large surface-to-volume ratio. Sb_2Se_3 in bulk is well-known p-type semiconductor.²⁵ Here, Sb_2Se_3 nanowires have the same conductivity type as the bulk.



Fig. 3 (a) *I-V* curves of the Sb₂Se₃ nanowire in the dark and under the 635 nm light illumination. Inset is a schematic illustration of measurement. (b) The relationships between photocurrent and power density at different biases. (c,d) The time responses to 635 nm light with the powder density of 1.52 mW/cm² at 1 V, 5 V and 10 V, respectively.

In the previous report,²⁵ the band-gap energy of Sb₂Se₃ nanowires have been examined by UV-vis absorption spectra and derived to range from 1.16 to 1.17 eV, indicating that Sb₂Se₃ nanowires can detect all visible-light in principle. The photoelectric properties of the Sb₂Se₃ nanowire, shown in the lower inset of Fig. 2, were investigated. The inset of Fig. 3a is a schematic illustration of photoelectric measurement. Fig. 3a demonstrates *I-V* curves of the nanowire in the dark and under

the 635 nm light illumination of different power densities. The photocurrent (I_{light} - I_{dark}) increases with the increasing of the power density of incident light at a fixed bias. It is clear that the nanowire is sensitive to 635 nm light. The energy of incident photons is 1.95 eV, which is larger than the band-gap energy of Sb₂Se₃. Under the illumination of 635 nm light, electron-hole pairs are generated in the Sb₂Se₃ nanowire, which leads to the increase of the conductivity of the nanowire.

Responsivity $(R_{\lambda}=I_{ph}/(PS))$, where I_{ph} is photocurrent, P is the power density of incident light and S is the effective illuminated area) and external quantum efficiency $(EQE=hcR_{\lambda}/(e\lambda))$, where λ is the wavelength, h is Planck's constant, e is the electronic charge and c is the speed of light) are important parameters representing the sensitivity of photodetector.³² The single Sb₂Se₃ nanowire photodetector has R_{λ} and EQE as high as 360 A/W and 7.0×10^4 % respectively at a bias of 10 V (The calculation is shown in the supporting information). The R_{λ} is higher than that (R_{λ} =8.0 A/W) of the single Sb_2Se_3 nanowire photodetector reported by Zhai *et al.* and comparable to that (R_{λ} =560 A/W) reported by Choi *et al.*^{27,29} It can be concluded that Sb_2Se_3 nanowires synthesized by this hydrothermal method have a potential application in visible-light detection. The higher R_{λ} and EQE are attributed to excellent crystalline quality of Sb₂Se₃ nanowires.¹⁴ Fig. 3b shows the dependences of the photocurrent on the power density of incident light at 1 V, 5 V and 10 V, respectively. Usually, the relationship can be described by a power law $(I_{ph}=A \times P^{C}, I_{ph})$ is photocurrent, P is power density, A and C are constants). The solid lines are the best fitting results. The C values correspond to 0.82, 0.59 and 0.57, respectively. They are

smaller than 1, indicating that multiple trap states exist in a single Sb₂Se₃ nanowire and lead to a complex process of electron-hole generation, trapping and recombination under the illumination.^{1,3} The time responses of the single Sb₂Se₃ nanowire were recorded by periodically turning 635 nm light on and off, as shown in Fig. 3c.d. It is noted that the photocurrents are stable and reversible. The rise time (fall time) is defined as the time interval for the response to rise (decay) from 10-90 % (90-10 %) of photocurrent. The single Sb₂Se₃ nanowire photodetector has the rise/fall time of 2.2/3.7 s, 0.6/3.1 s and 0.4/1.3 s at a bias of 1 V, 5 V and 10 V, respectively. These response times are comparable to those of Sb₂Se₃ nanowire photodetectors and other nanowire photodetectors.^{29,33,34} There are rare dangling bonds on the surface of Sb₂Se₃ nanowires due to the layered structure.³⁰ Less dangling bonds indicate less surface states in the surface region. It has been known that a long response time is due to the existence of a large amount of surface states, and vice versa.³⁵ Thus, the fast response to 635 nm light should be attributed to the layered structure of Sb₂Se₃. It is also observed that the response time can be shorted by increasing the bias. Increasing the bias can enhance the strength of the electric field in the nanowire, which could accelerate the trapping and releasing process of carriers and finally lead to a fast response. In addition, the fall time is longer than the rise time at a fixed bias.



Fig. 4 (a) Schematic illustration and (b) SEM image of a flexible photodetector based on multiple Sb_2Se_3 nanowires. Inset shows three pairs of interdigitated electrodes on the flexible PET substrate. (c) *I-V* curves of the photodetector (D1) in the dark and under the illumination of 635 nm light. (d) The relationships between photocurrent and power density.

The fabrication process of single nanowire based photodetectors is complex, expensive and time consuming. Moreover, single nanowire's photocurrent is too small to be detected. Thus, single nanowire based photodetectors are hard to be used widely in daily life. Recently, flexible electronics have attracted more attention due to the demand for portable devices. In this work, a large number of Sb₂Se₃ nanowires were randomly dispersed onto the Au interdigitated electrodes on PET substrates to fabricate flexible photodetectors. Fig. 4a is a schematic illustration of flexible photodetector. Because the Au interdigitated electrodes are made on the flexible PET substrate, as shown in the inset of Fig. 4b, the photodetector could possess excellent

flexibility. A flexible photodetector was characterized by SEM (Fig. 4b). It is observed that many Sb₂Se₃ nanowires are on the interdigitated electrodes and connect adjacent electrodes. As the PET substrate is insulating, there are some bright areas between electrodes induced by the charging effect. Fig. 4c reveals *I-V* characteristics of a multiple Sb_2Se_3 nanowires based photodetector (D1) in the dark and under the illumination of 635 nm light with different power densities. The photocurrent increases by increasing the power density of incident light at a fixed bias, and their relationships are shown in Fig. 4d. Similarly, the relationships still can be described by the power law $(I_{ph}=A \times P^{C})$. C values are taken by fitting corresponding data and are 0.79, 0.72 and 0.80 at a bias of 1 V, 5 V and 10 V, respectively. These values are different from those of the single Sb₂Se₃ nanowire photodetector, which is attributed to the difference of electron-hole generation, trapping and recombination processes in two kinds of photodetectors. It is possible that interfaces among nanowires lead to this difference. It is desired to achieve photodetectors of a higher C value.³² Compared to the single Sb_2Se_3 nanowire based photodetector, the multiple nanowires based photodetector has higher C values at 5 V, 10 V and a similar value at 1 V.



Fig. 5 (a) *I-V* curves of another flexible photodetector (D2) in the dark and under the illumination of 31 mW/cm² 635 nm light. (b,c) The time responses to 31 mW/cm² 635 nm light at a bias of 10 V with 100 ms and 1 ms time resolutions respectively.

Fig. 5a shows *I-V* curves of another flexible Sb_2Se_3 nanowires based photodetector (D2) in the dark and under the illumination of 31 mW/cm² 635 nm light. Compared to D1, the D2 displays a tenfold increase in photocurrent and dark current under the same conditions due to the increasing of the number of nanowires on the interdigitated electrodes. The time responses of the D2 were recorded (Fig. 5b, c). With 635 nm light on and off, the D2 shows "on-state" and "off-state" repeatedly. It has a fast response to the light and a reproducible and stable photocurrent. Fig. 5c illustrates an on-off cycle of the D2 under the illumination of 31 mW/cm² 635 nm light at a bias of 10 V with 1 ms time resolution. According to this on-off cycle, it can

be deduced that the rise/fall time is 17/20 ms. In order to confirm the fast response of this kind of flexible photodetectors, the performances of other photodetectors also were characterized. One of them is shown in Fig. S3. The rise/fall time of the DS1 is 13/20 ms and close to 17/20 ms obtained above. Fig. S3c reveals the ability of the DS1 to detect 635 nm light with an on-off frequency up to 230 Hz. The response time is an important parameter determining the capability of photodetector to follow a fast changing light. The response speed of our flexible photodetectors is faster than those of many flexible photodetectors.^{15,20,22} Thus, our synthesized Sb₂Se₃ nanowires are promising to fabricate high-speed flexible photodetectors. Compared to the single nanowire photodetector, multiple Sb₂Se₃ nanowires photodetectors have a faster response speed. Fig. S4 shows that increasing the number of nanowires in the photodetector can improve response speed. The mechanism is still unknown and will be further studied. It is possible that the faster response speed of multiple nanowires photodetector is attributed to the rapid recombination of photogenerated electron-hole pairs at the interfaces between nanowires.¹¹ Except for 635 nm light, flexible Sb₂Se₃ nanowires photodetectors can be used to detect visible-light of other wavelength, such as 405 nm violet light. Fig. S5 illustrates the response of a flexible photodetector to 405 nm light, which is similar to the response to 635 nm light.



Fig. 6 (a-d) Photographs show that a flexible photodetector was measured at different bending states. (e) *I-V* curves of the flexible photodetector under the white light illumination corresponding to the bending states a-d, respectively. (f) The influence of the bending curvature on the current of the flexible photodetector. (g) *I-V* curves measured without bending and after 1000 cycles of bending.

As multiple Sb_2Se_3 nanowires photodetectors were fabricated on PET substrates, they should possess a significant property, i.e. flexibility. It is necessary to know the influence of bending on the electrical properties of flexible photodetectors. Fig. 6a-d are photographs recording different bending states of a flexible photodetector during *I-V* tests. The corresponding *I-V* curves are displayed in Fig. 6e. It is noted that the bending curvature has not an obvious influence on *I-V* characteristic of the flexible photodetector. The relationships between the current through the photodetector and

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the bending curvature are shown in Fig. 6f. The current slightly decreases with the increasing of bending curvature, which is reasonable since the electrode interspacing should slightly increase with the bending curvature and finally has an influence on the current.¹ However, the currents of the photodetector with different bending curvatures keep at 6.0 ± 0.2 nA. The change of current is negligible and acceptable, indicating the excellent flexibility of this kind of photodetector. Fig. 6g demonstrates *I-V* curves of the photodetector without bending (blue solid line) and after 1000 cycles of bending (red circles). It can be observed that *I-V* characteristic of the photodetector is hardly affected even after 1000 cycles of bending. All results confirm that dispersing Sb₂Se₃ nanowires onto the Au interdigitated electrodes is suitable for making flexible photodetectors with high-performance. Since (Sb₄Se₆)_n layers are parallel to the growth direction of Sb₂Se₃ nanowire, the nanowires could have large bending strain compliance. Thus, excellent flexibility should be also related to the layered structure.

Photodetectors	Response time (s)	Responsivity (A/W)	EQE (%)	Flexibility	Reference
Single Sb ₂ Se ₃ nanowire		560	1.06×10 ⁵	No	27
Single Sb ₂ Se ₃ nanowire	<0.3 s	8.0	1650	No	29
Multiple Sb ₂ Se ₃ nanowires	0.2/1.2 s			No	31
Single Sb ₂ Se ₃ nanowire	0.4/1.3 s	360	7.0×10^4	No	This work
Multiple Sb ₂ Se ₃ nanowires	13/20 ms			Yes	This work

Table 1 Comparison of Sb₂Se₃ nanostructure based photodetectors

Our results are compared with other Sb_2Se_3 nanostructure based photodetectors reported so far (Table 1). In this work, the single Sb_2Se_3 nanowire shows an excellent

light-detection performance with high responsivity and fast time response. Sb_2Se_3 nanowires can be assembled into a flexible photodetector with faster time response and excellent flexibility by a simple method. This work first reveals that high-yield Sb_2Se_3 nanowires are promising candidates for flexible photodetectors.

Conclusions

High-quality single-crystalline Sb₂Se₃ nanowires have been fabricated by a simple hydrothermal method. The conductivity type of Sb₂Se₃ nanowires is confirmed as p-type, which is same as that of the bulk. The response of a single Sb₂Se₃ nanowire to 635 nm light has been investigated. Its responsivity and EQE are 360 A/W and 7.0×10^4 %, respectively. Its rise/fall time is 0.4/1.3 s. A large number of Sb₂Se₃ nanowires can be assembled easily into a flexible multiple nanowires based photodetector. This kind of flexible photodetectors has a fast response to 635 nm light with the rise/fall time as low as 13/20 ms. The current through flexible photodetectors remains nearly unchanged at different bending states and after 1000-cycles bending, confirming excellent flexibility. Our results illustrate the promising application of Sb₂Se₃ nanowires in high-performance flexible photodetectors.

Acknowledgements

This work was financially supported by the National Natural Science Foundation of China (Project No. 61204016 and 11104047) and the Program for Excellent Talents in University of Education Bureau of Liaoning Province (LJQ2014047).

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Graphical Abstract

Flexible visible-light photodetectors were fabricated by dispersing a large number of Sb₂Se₃ nanowires onto the Au interdigitated electrodes on PET substrates, which showed fast response speed and excellent flexibility.

