An integrated flexible multifunctional sensing system for simultaneous monitoring of environment signals

Haoran Chen^{1†}, Zheng Lou^{1,2†} and Guozhen Shen^{1*}

ABSTRACT With the rapid development of portable devices and internet of things, the requirement of system wearability and integration accelerates the investigation of flexible multifunctional sensors. In this study, we developed an integrated flexible sensing system with four nanowire-based sensors and a Ni microwire-based temperature sensor. The four nanowire-based sensors are three kinds of photodetectors responding to lights with different wavelengths and a gas sensor. Due to the large surface volume ratio and considerable sub wavelength effect, all the nanowire-based sensors show good sensing response and excellent linear relationship between sensitivity and temperature. The as-fabricated flexible sensing system can simultaneously detect environmental parameters, including temperature change, light intensities from UV-Visible to near infrared regions, and harmful gas concentration. Our flexible multifunctional sensing system therefore opens up a new way for the emerging portable and wearable electronics.

Keywords: flexible device, nanowire, multifunctional sensing system, gas sensor, photodetector

INTRODUCTION

Tracking environmental conditions, including air particles, harmful gas concentration, humidity level, ambient temperature and ultraviolet (UV) radiation, offers valuable information for the prediction, treatment and management of chronic diseases [1–5]. Wearable devices are considered as one of the most feasible and effective ways for daily environmental monitoring [6,7]. Although the state-of-the-art developed wearable devices show superior performance, they usually have a single function to the target and can only work under specific conditions. Sometimes it cannot meet the requirements of miniaturization and the planar integration of the system. In this

context, multifunctional sensing devices are more and more concerned by both industry and academia [8–10]. In addition to good multifunction, wearable sensors should also have high sensitivity, low power consumption, low cost, and good flexibility [11–14]. To achieve these characteristics, nanowires (NWs), which have excellent electrical properties, large surface area and compatibility with low-cost manufacturing process, are widely used to construct flexible devices [15–20]. For example, Fang *et al.* [21] recently reported a flexible photodetector based on InAs NWs, which showed great performance with high external quantum efficiency, responsivity, and detectivity. Kwon *et al.* [22] investigated a flexible gas sensor based on ZnO NWs with excellent gas sensing performance and good mechanical flexibility. The device can still work normally without obvious degradation even after 10,000 dynamic bending tests with a bending radius of 5 mm. Despite the impressive achievements of NW-based wearable sensors, multifunctional perception of various stimuli is still the ideal choice to expand its practical application.

Herein, we proposed a multifunctional system integrated with five kinds of flexible sensors based on different NWs and Ni microwires. This coplanar system has good flexibility, can respond to a variety of stimuli (such as gas, light and temperature) under outdoor conditions, and has high linearity, high resolution and no time delay. Our work represents a good example to integrate different types of flexible sensors to achieve highly specific and sensitive environmental signal detection, which could meet the wearable electronic requirements for real-time understanding of environmental conditions under different stimuli. It exploits a new strategy for the development of the next generation robust systems based on

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multifunctional wearable sensors.

EXPERIMENTAL SECTION

Synthesis of CdS NWs

CdS NWs were synthesized *via* a facile hydrothermal method. Firstly, 2 mmol of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ was dissolved in ethylenediamine (EDA, 30 mL), and 4 mmol of $\text{CH}_4\text{N}_2\text{S}$ was added and stirred at room temperature for 30 min. Then, the mixed solution was transferred to a 50-mL Teflon-lined stainless steel autoclave, and heated at 200°C for 24 h. After the autoclave was naturally cooled down to room temperature, the product was taken out and washed with ultra-pure water and ethanol for several times.

Synthesis of ZnO NWs

ZnO NWs were prepared *via* the electrospinning process. Typically, 0.1 g of zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) was dissolved in 50 mL of *N,N*-dimethylformamide and 50 mL of ethanol. Subsequently, 1 g of polyvinylpyrrolidone (PVP, molecular weight=1,300,000) was added under stirring for 1 h to achieve a transparent solution. In order to achieve an electrospinning process, the resulting solution was loaded into a syringe using an 8-gauge needle. The feeding rate was 0.2 mL h^{-1} , and an electrical field was applied as high as 1 kV cm^{-1} between the tip of the syringe and the collector. Then, the nanofibers were collected on the surface of the collector and calcined in a muffle furnace at a temperature of 650°C for 4 h, and then were ground into powder.

Synthesis of SnS NWs

To synthesize SnS NWs *via* the physical vapor deposition (PVD) method, an alumina boat which contained 0.15 g of high-purity SnS powder (99.5%, Alfa) was placed at the center of a small quartz tube in a tube furnace. A Si wafer coated with 10-nm-thick Au catalysts was placed 16 cm downstream away from the SnS powder to collect SnS NWs. After the tube furnace was flushed with Ar gas for 30 min, the tube furnace temperature was raised to 700°C in 35 min and then kept at 700°C for 30 min. During the growth process, the Ar flow rate was maintained at a rate of 100 sccm. When the deposition process was ended, the tube furnace was naturally cooled down to room temperature under Ar flow.

Synthesis of SrGe_4O_9 NWs

SrGe_4O_9 NWs were prepared by the hydrothermal method. First, 2 mmol of $\text{Sr}(\text{CH}_3\text{COO})_2 \cdot 1/2\text{H}_2\text{O}$ and

8 mmol of GeO_2 were added to deionized water (30 mL). After stirring for 60 min, the mixture was transferred to a Teflon-lined autoclave, and then heated to 180°C for 24 h, followed by natural cooling to room temperature. After being washed thrice and dried at 80°C for 12 h, the SrGe_4O_9 NWs were obtained.

Fabrication of flexible multifunctional sensing system

Multi-mask photolithography was utilized to fabricate the integrated system. First, Ni electrode layer (100 nm) was patterned on a polyimide (PI) substrate after a photolithography, thermal evaporation and lift-off process to build the temperature sensor. With a similar process, the source and drain electrodes (Cr/Au 10/50 nm) of photodetectors and gas sensors were then deposited on the flexible substrates. After that, four kinds of NWs were spin-coated on the corresponding electrodes. A wearable multifunctional integrated sensing system was thus successfully fabricated on the flexible PI substrate.

Characterizations

Scanning electron microscopy (SEM, Zeiss Supra55 (VP)) and powder X-ray diffraction (XRD, Rigaku D/Max-2550, $\lambda = 1.5418 \text{ \AA}$) analysis were employed to measure the morphologies and crystallinities of the four NWs, respectively. A probe station supported with a semiconductor characterization system (Keithley 4200-SCS) was utilized to measure the electronic characteristics of the four sensors. The light illumination source was a power-adjustable light source capable of outputting homogeneous light.

RESULTS AND DISCUSSION

Fig. 1a shows the schematic illustration of the designed flexible multifunctional sensing system for environmental monitoring. The entire system consists of a temperature sensor, three kinds of photodetectors and a gas sensor. All these components are integrated on a PI film *via* a conventional photolithography process. Fig. 1b, c show the photographs of the fabricated device, which exhibits good flexibility and could be attached to human body.

SEM images of the NWs used as sensing elements for different types of sensors are depicted in Fig. 1d. For all the four kinds of semiconducting NWs, they possess obvious one-dimensional (1D) nanostructures with smooth and clean structures. Typical diameters of the NWs range from ~50 to 200 nm, depending on the techniques used to synthesize the NWs. XRD was used to check the purity and phase structure of the NWs and the corresponding results are depicted in Fig. 1e. For all the

Figure 1 (a) A schematic of five pixels of a multimode wearable sensor capable of mapping five separate stimuli. PD: photodetector. (b) A photograph of the multifunctional device on a PI substrate with good flexibility. (c) A photograph of the matrix attached to the back of the hand. (d) SEM images and (e) XRD patterns of the four different sensing materials, including ZnO, CdS, SnS, SrGe₄O₉. Schematic device structure of the three different kinds of sensors, (f) temperature sensor, (g) photodetector and (h) gas sensor.

samples, XRD data reveal that they are pure phases and can be indexed into wurtzite ZnO (JCPDS Card No. 36-1451), wurtzite CdS (JCPDS Card No. 41-1049), orthorhombic SnS (JCPDS Card No. 39-0354) and hexagonal SrGe₄O₉ (JCPDS Card No. 14-0029), respectively. Fig. 1f–h demonstrate the device structures of the temperature sensor, the photodetectors, and the gas sensor. The resistance-based temperature sensor was realized by fabricating waved Ni metal microwires as the sensing materials for detecting environmental temperature signals (Fig. 1f). The photodetectors based on three different kinds of NWs (ZnO NWs, CdS NWs and SnS NWs) could achieve different light spectrum detection from ultraviolet, visible to near-infrared light (Fig. 1g, respec-

tively). SrGe₄O₉ NW-based gas sensor could adsorb ammonia molecules to realize real-time monitoring of toxic and harmful gases in the environment (Fig. 1h).

Because of the small size and large specific surface area, photodetectors based on 1D NWs have better performance compared with their thin film or bulk counterparts [23,24]. Surface carrier trapping has an important effect on the transport and photoconductivity of NWs. The schematic of the photodetection mechanism of NWs is shown in Fig. 2a. The process of oxygen adsorption and desorption in the dark and under light illumination is generally considered to be related to the free carriers produced. When exposed to air, the surface of NWs adsorbs oxygen molecules, which capture free electrons. A

Figure 2 (a) Sensing mechanism of the NW-based photodetector. (b) I - V curves of the CdS NW-based photodetector in dark and under 480 nm light illumination, respectively. (c) The dependence of photocurrent on light intensity ($I \sim P^{1.01}$). (d) Transient photoresponse properties of the photodetector with the light intensity of $6.75 \mu\text{W cm}^{-2}$. (e) Enlarged part of the middle cycle of (d).

depletion layer is then formed on the surface of the NWs, resulting in low conductivity. As the device is irradiated by the incident light, the electron-hole pair is generated, and the unpaired electrons leave because the holes are easily trapped on the surface [25]. Under the action of an external electric field, the conductivity of the NWs increases, resulting in the decrease of depletion barrier thickness and the increase of free carrier concentration.

The photoresponse characteristics of CdS NW-based photodetector were studied in detail, and the corresponding results are shown in Fig. 2b–e. Fig. 2b shows the I - V curves of the device in the dark and under 480 nm light illumination, respectively. Obvious response to the visible light was observed for the device. Under the bias

voltage of 2 V, the current increases from ~ 10 fA to $2 \mu\text{A}$ (light intensity of $6.75 \mu\text{W cm}^{-2}$). A power law of the photocurrent *versus* light intensity is described in Fig. 2c as $I = AP^\theta$, where I , A , P , θ correspond to the photocurrent, a constant, the light intensity and the empirical value, respectively. From Fig. 2c, it can be seen that the light intensity and the photocurrent are highly dependent, resulting in the value of θ at 2 V bias voltage of 1.01 ($I \sim P^{1.01}$), which proves that CdS NWs possess excellent photocurrent capability to visible light.

Reproducibility and response speed are critical factors to evaluate the performance of the photodetector, especially when used in optical communication, imaging and so on. By periodically turning the light on and off, we

measured the transient photoresponse of the CdS-based photodetector and the corresponding result was shown in Fig. 2d. For the given six light on/off periods, the photocurrent does not show obvious change, indicating that the device has good repeatability and stability under light irradiation. The photoresponse speed and recovery speed of the device can be deduced from a single light on/off curve, as demonstrated in Fig. 2e. The rise and fall times were thus measured to be about 1.6 and 0.3 s, respectively, comparable to the previous reported data [16].

In addition to the visible light, our system can also monitor UV light in real-time through the integrated ZnO NW-based photodetector. The photoresponse measurements of the ZnO-based devices under 350 nm UV light were systematically investigated, as demonstrated in Fig. 3. Fig. 3a shows the typical I - V curves of the ZnO-based devices under UV illumination with the power intensity of $4.46 \mu\text{W cm}^{-2}$ and in the dark, respectively. The good linearity of the I - V curve indicates that the ZnO NWs channel materials contact well with the metal electrode. When the light was exposed on the device, a significant increase in current was observed.

With the increase in light intensity, the photocurrent also increased accordingly (Fig. 3b). As shown in Fig. 3b, the dependence of photocurrent on the incident light intensity (P) at the $V_{\text{bias}} = 2 \text{ V}$ can be accurately fitted by the power-law relationship of $I \sim P^{0.71}$. The result shows the nonlinear power dependence of the net photocurrent on the power density, and reveals that there are complex processes of electron hole generation, trapping and recombination in the NW channel. In order to study the ability of the ZnO NW photodetector to track the changed light signal, the time-resolved photocurrent measurement was carried out by periodically modulating the 360 nm UV light on and off. Obviously, Fig. 3c depicts the photocurrent trace of the device at a bias voltage of 2 V with a light intensity of $4.46 \mu\text{W cm}^{-2}$. The effective switching behavior indicates that the ZnO NW device has good periodicity and stability with a rise time of 35.69 s and a fall time of 29.93 s (Fig. 3d). The relatively slow response of the ZnO NW device was often observed for metal oxide NWs and had been extensively reported [26].

Infrared photodetector represents another important optoelectronic device and has attracted considerable re-

Figure 3 (a) I - V characteristics of the ZnO NW-based photodetector under UV light illumination. (b) The relationship between photocurrent and light intensity ($I \sim P^{0.71}$). (c) The transient photoresponse curves of the ZnO NW-based photodetector under UV light illumination. (d) The single response and recovery curve of the ZnO-based photodetector.

search attention in recent years. To endow our integrated system with the ability to detect infrared light, we integrated SnS NW-based devices into the flexible system, as SnS was an ideal candidate for mid near infrared (NIR) photodetectors because of its mid NIR direct band gap of 1.3 eV and high absorption coefficient larger than 10^4 cm^{-1} . Fig. 4a shows the optical image of the measurement of the SnS-based device in the fabricated flexible integrating system. As shown in Fig. 4b, under constant source drain bias, the output current of the device obviously increases with the irradiation of incident infrared light (808 nm). The time-resolved photoresponse measurements were performed by periodically modulating the light illumination (Fig. 4c, d). The effective switching characteristics show that the SnS NW detector has good periodicity and stability. The rise and fall times can be determined from a single light on/off curve, as shown in Fig. 4d. The rise time and the decay time are 0.93 and 2.91 s, respectively, indicating the fast response of the SnS-based photodetector.

As a basic physical parameter, temperature changes in time and space can be used to monitor physiological

activities, especially the heat transfer between the environment and the biological tissues [27]. Wearable temperature detection requires the sensor to have enough sensitivity and accuracy, which can directly attach to the non planar skin surface with different posture and motion, and continuously measure the minimum user perception. To measure the temperature in real-time and to eliminate the temperature effect on other sensors, we integrated a Ni microwires-based temperature sensor on the fabricated integrated sensing system (Fig. 5a). The temperature sensor was fabricated *via* the photolithograph process, followed with the deposition of Ni on the patterned structure to form the waved Ni microwires. Fig. 5b shows the change of current with changed temperature from 25 to 100°C. With increased temperature, an increase in current was observed at a bias voltage of 1 V. It indicated an obvious negative temperature coefficient (NTC) feature [28]. Due to the well-defined linear normalized resistance *vs.* temperature relationship, the sensitivity of the temperature sensor was estimated to be about $3.4 \Omega/^\circ\text{C}$.

In order to eliminate the effect of temperature on other

Figure 4 (a) Optical image of the measurement of the SnS-based device in the fabricated flexible integrating system. (b) *I*-*V* curves of the photodetector measured under NIR light illumination and in the dark, respectively. Inset is the enlarged part from 0.9–2.1 V. (c) The photoreponse curve of the SnS-based photodetector under 808 nm light with 2 V bias voltage. (d) Response and recover time of the device.

Figure 5 (a) Photographs and optical image of the flexible temperature sensor. (b) The relationship between resistance and temperature of the Ni microwires-based sensor. (c) Power dependence of the CdS NW photodetector, (d) dynamic photoresponse curves of the CdS NW photodetector, (e) power dependence of the ZnO NW photodetector, (f) dynamic photoresponse of the ZnO NW photodetector under different environment temperatures (25 to 75 $^{\circ}\text{C}$).

type sensors, the relationship between temperature and photodetectors was then studied by measuring the photoresponse of the photodetectors at different temperatures, as shown in Fig. 5c–f. The dependence of photocurrent on the incident light intensity under different temperatures at the bias voltage of 2 V is depicted in Fig. 5c. A linear relationship between the photocurrent and the changed light intensity was observed, which was also confirmed by the result in Fig. 5d. Moreover, the response and recovery time of the device did not show obvious change with the changed temperature. Similar

results were also obtained on the ZnO NW-based photodetector, as shown in Fig. 5e, f. Briefly, temperature has a linear effect on the photocurrent of the ZnO devices. With the increase of temperature, the photocurrent of the device increases at different light intensities. Based on these results, it is possible to compensate the effect of temperature on other sensors by circuit design for practical applications.

Highly toxic gases including ammonia (NH_3) not only have a negative impact on the surrounding environment, but also are harmful to public health. By integrating

flexible gas sensors, our wearable multifunctional system could be used to successfully detect the NH_3 gas at room temperature [29]. The proposed sensing mechanism of the SrGe_4O_9 NW-based gas sensors in air and NH_3 is shown in Fig. 6a. When exposed to air, oxygen molecules were physically adsorbed on the surface of the SrGe_4O_9 NWs through gaining electrons in the trapping conduction band, forming O^- . As the depletion layer widened, the measured resistance of the sensor increased. When the sensor was exposed to NH_3 , the O^- reacted with NH_3 as follows[30]:

The narrow depletion layer resulted in the decrease of the measured resistance of the sensor.

The repeatability of the SrGe_4O_9 NW sensor toward 100 ppm NH_3 gas is shown in Fig. 6b. Four exposure/recovery cycles were performed under the same conditions. No significant changes in response amplitude and recovery time were observed, which indicated that our SrGe_4O_9 NW gas sensor had excellent stability. Fig. 6c shows the sensing performance of the SrGe_4O_9 NW to NH_3 gas at room temperature with various concentrations of 20–500 ppm, respectively. With increased NH_3 concentrations, the responsivity was also gradually improved. For instance, when exposed to 100 ppm NH_3 gases, the responsivity was 2.48, while it was about 8 with the NH_3 concentration of 500 ppm. For oxide gas sensor, temperature will affect the sensitivity of the device. As shown in Fig. 6d, increasing the temperature from 25 to

75°C, the responsivity of the device decreases. As a result, for the gas sensor, the effect of temperature on its performance can also be compensated by circuit design to accurately measure the NH_3 gas. The humidity effects on the sensing performance of the five devices were also studied. Fig. S1 shows the sensitive retention performance of the devices under different relative humidity (RH). The definition of sensitive retention is the ratio of sensitivity in atmosphere to the sensitivity with different RH. These results show that the sensitivity is maintained from 80%–95% of its original value of the five devices even under 95% RH (the ambient RH is about 40%). Therefore, for our sensing system, we can conclude that the effect of humidity on the sensor performance can be ignored. Furthermore, we also studied the mechanical stability after bending the devices for hundreds of cycles and the corresponding results are depicted in Fig. S2. After bending for 5000 cycles, there is slight change in the conductance of the flexible devices, revealing the outstanding long-term and mechanical stability of these devices.

CONCLUSION

In summary, we designed and constructed a flexible multifunctional sensing system by integrating three NW photodetectors, one NW gas sensor with one microwire temperature sensor. The integrated system can monitor the environmental signals, such as detecting the concentration of NH_3 gas through the gas sensing part, de-

Figure 6 (a) Schematic of the sensing mechanism of SrGe_4O_9 NW-based gas sensor. (b) Cyclic response curves of the SrGe_4O_9 NW sensor under 100 ppm of NH_3 at room temperature. (c) Response curves of the SrGe_4O_9 NW sensor toward NH_3 with different concentrations at room temperature. (d) Time-resolved response of the SrGe_4O_9 NW device towards 100 ppm NH_3 under different environment temperatures.

tecting the brightness of light through the photodetectors and monitoring the environmental temperature through the temperature sensing part. More importantly, the linear relationship between the sensors and temperature was observed, making it possible to compensate the effect of temperature for practical applications. Our work demonstrates the great potential of the integrated system in the next-generation wearable electronics for healthcare applications.

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一种用于监测多种环境信号的柔性多功能传感系统

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摘要 随着便携式设备和物联网技术的飞速发展, 对系统可穿戴性和集成性的要求加快了柔性多功能传感器的研究. 本文以半导体纳米线为传感材料, 制备了四种平面器件, 包括光电探测器和气体传感器. 由于纳米线具有大的表面体积比以及良好的亚波长效应, 所研制的基于纳米线的传感器都表现出优异的传感响应, 以及灵敏度与温度间良好的线性关系. 将这些传感器与基于Ni微米线的柔性温度传感器集成在一个柔性平台上, 构建了一种可穿戴的多功能传感系统. 该系统可以同时检测多种环境参数, 包括温度变化、从紫外可见光到近红外的光强变化、有害气体浓度等. 我们所设计的柔性多功能传感系统为新兴的便携式、可穿戴电子产品提供了一条新的思路.