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Ultraviolet Photodetection of Flexible ZnO Nanowire Sheets in Polydimethylsiloxane polymer

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Abstract

ZnO nanowires are normally exposed to an oxygen atmosphere to gain high performance in UV photodetection. In this work we present results on a UV photodetector fabricated using a flexible ZnO nanowire sheet embedded in polydimethylsiloxane (PDMS), a gas-permeable polymer, showing reproducible UV photoresponse and enhanced photoconduction. PDMS coating results in reduced response speed compared to that of a ZnO nanowire film in air. The rising speed is slightly slowed, while the decay time is about four times prolonged. We conclude that oxygen molecules diffusing in PDMS are responsible for the UV photoresponse.

Keywords

ZnO nanowires; UV photodetection; Polydimethylsiloxane; Permeable polymer; Photoresponse
Introduction

ZnO is a wide band-direct-gap semiconductor with a 3.37 eV gap and a high exciton binding energy of 60 meV at room temperature, promising for optoelectronic applications including light-emitting diodes, laser diodes, and photodetectors for the ultraviolet (UV) spectral range [1,2]. ZnO nanostructures are particularly interesting as they bear superior properties compared to the bulk crystal. UV light detection is one of the major applications of ZnO; Various UV photodetectors based on ZnO films or nanocrystals have been reported. It has been demonstrated that ZnO nanowires have high internal photoconduction gain and much stronger responsivity under UV light illumination compared to the bulk film [3]. The UV photoresponse mechanism of ZnO nanowires is dominated by the adsorption and desorption of oxygen molecules [4]. In vacuum, ZnO nanowires show prolonged UV photoresponse time and lowered responsivity [5]. So far many UV photosensors have been made using ZnO one-dimensional nanostructures with various configurations for sensing elements, such as single-nanowire devices [6], nanowire bridges [7], nanobelt network films [8], vertical nanowire arrays [9], and flexible nanowire sheets [10]. These nanostructures are normally exposed to air for measurement, neglecting the possible surface contamination caused by absorbance of other molecules from ambient atmosphere that can degrade the UV sensing performance of ZnO. For example, it has been demonstrated that the UV photoconduction of ZnO nanowires is degraded by the adsorption of water molecules in humid air [11,12]. Water molecules together with CO₂ in moist atmosphere bring out a slow chemical reaction with ZnO to form amorphous Zn(CO)₃ which roughens the nanowires surfaces [13]. Therefore, for practical application the device encapsulation is essential to ensure a clean oxygen atmosphere around the nanowires. In this work we investigate the performance of
polymer-embedded ZnO nanowires for UV photodetection, to avoid the necessity of a gas cavity to load the nanowires, simplifying the device fabrication.

Recent experiments demonstrated that coating ZnO nanobelts with UV-sensitive polymers enhances the UV photoconduction [14,15]. A thin layer (<100 nm) of such a polymer over ZnO nanocrystal produces excited states upon UV illumination. The excited states of polymer molecules act as transition states facilitating electron hopping from the valence band of ZnO into the conduction band. However, coating of such a polymer over ZnO nanowires cannot be thick because it would shade UV light. Moreover, carbon-based polymers undergo oxidation with aging, and the degradation could be expedited by absorbing UV light.

In this paper, we report results on UV photodetectors based on a thin sheet of ZnO nanowires embedded in polydimethylsiloxane (PDMS), an optically clear UV-transparent silicone polymer which has been used in contact lens, microfluidic devices, and stretchable displays. Due to its excellent gas permeability this polymer has been applied as membrane for gas separation [16]. In our work, we combine free-standing thin-sheets of ZnO nanowires with the elastic PDMS, suitable for developing bendable devices which are of interest in current research.

Results and Discussion

ZnO nanowires were synthesized on a large scale by a modified carbothermal reduction method. Vaporized zinc and oxygen react at atmospheric pressure and result in a cotton-like product consisting of ZnO nanowires. The nanowires were processed into free-standing thin sheets which can be cut by a blade into any shape [17]. The optical photograph in Figure 1(a) shows a strap-shaped nanowire sheet held by a tweezer. Scanning electron microscopy (SEM) observation reveals that
these nanowires interlap with each other to form a felty film (Figure 1(b)). Transmission electron microscopy (TEM) images of the nanowires are shown in Figures 1(c) and 1(d). The high-resolution TEM image in Figure 1(d) reveals that the nanowires grow along [0001] direction, as the distance between (0001) planes is 0.52 nm. The nanowires are 20-60 nm in thickness and tens of microns in length. The high surface-volume ratio ensures large surface adsorbance of gaseous molecules.

![Figure 1. (a) An optical photograph of a strap-shaped ZnO nanowire film. (b) SEM image of the nanowire film. (c & d) TEM images these nanowires.](image)

Figure 2 shows the room-temperature photoluminescence (PL) spectrum of the ZnO nanowires, with the excitation wavelength of 325 nm. The insert is a PL excitation (PLE) spectrum taken for the emission at 570 nm. The PLE spectrum indicates that these nanowires absorb UV light with wavelength below 378 nm whereas they are transparent to visible lights. In the PL spectrum the sharp peak at 379 nm is ascribed to the near band-edge emission of ZnO. The broad emission
band in visible range is related to some crystal defects as luminescent centre. In our method the nanowires were grown quickly via vapour-phase reaction at low temperature, below 200 °C, and crystal defects are expected. Oxygen vacancies and antisite oxygen give contribution to green PL emission around 520 nm; Zinc vacancies and interstitial oxygen contribute to blue PL emission (450-470 nm) [18].

Figure 2. Room-temperature PL spectrum of the ZnO nanowire film. The insert is a PLE spectrum.

We made devices based on the strap-shaped nanowire sheets (width 2 mm). To make electrode contacts, silver paste made by mixing Ag nanopowder and the viscous PDMS liquid was painted onto a nanowire film strap at two ends, leaving a 10-mm-gap between the two electrodes. The whole device with a Ag-ZnO-Ag structure is embedded in PDMS for measurement. The PDMS liquid wets ZnO nanowires well so that after curing the interspaces between nanowires were filled with PDMS polymer, making the paper-like nanowire sheet more translucent. For comparison, we made two devices without embedment in PDMS. For these two devices we deposited metal electrodes (Ti or Ag) onto the strap-shaped ZnO
nanowire films (2 mm width) by e-beam evaporation through a shadow mask, in order to rule out the influence of PDMS on the UV-detection performance. The gap between two opposite electrodes is 10 mm, same as that of the device in PDMS. If applying Ag-PDMS paste onto the ZnO nanowire film, the liquid PDMS would slowly spread out of the agglomeration of Ag nanoparticles and infiltrate the nanowire film due to capillarity. Figure 3(a) shows the current-voltage (I-V) curves of the device in PDMS, measured under UV light illumination (312 nm, 30 mWcm\(^{-2}\)) and in dark. The I-V curves of two other devices measured in air are plotted in Figure 3(b). ZnO is naturally n-type with an electron affinity of 4.35 eV; The work functions of Ag and Ti are 4.26 eV and 4.33 eV, respectively. Under UV illumination Ti shows good ohmic contact on ZnO due to the extremely low barrier at Ti/ZnO junction. The vacuum deposited Ag also shows nearly ohmic contact on ZnO (Figure 3(b)). However, for the contact of Ag paste on ZnO, the I-V curves measured under UV illumination and in dark show rectifying features. Presumably, the Ag nanoparticles that we used to make Ag-PDMS paste were spontaneously oxidized due to aging. The surface Ag\(_2\)O layer, a narrow bandgap semiconductor, added an extra barrier for electrons flowing between Ag nanoparticles and ZnO nanowires, resulting in the nonlinear I-V curves in Figure 3(a). The I-V curve of the device in PDMS measured in dark, plotted in the insert in Figure 3(a), shows that at forward bias of 8 V the current is only 0.11 µA, whereas under UV light illumination the current reaches to 405 µA at 8 V, about 3650 times larger. However, for the two devices measured in air, the current values at 8V are around 85 µA for the device with Ti electrodes, and 78 µA for the device with Ag electrodes. Previously, we studied the UV photoconduction of ZnO nanowire films with different PL properties, i.e., defects contents. The UV photocurrent of those ZnO nanowire films (2 mm-wide and 10 mm-long) with vacuum deposited Ag
electrodes was in the range of 13-90 μA at 8V when measured in air [10]. In this work, the UV photocurrent of the device with Ag paste electrodes and PDMS embedment exceeds 400 μA at 8V. Even at reverse bias of -8V, the current is 160 μA, much higher than that of the device without PDMS. In dark, the current values of the device in PDMS are 110 nA at 8V and 18 nA at -8V. However, the dark current of the ZnO nanowire films exposed to air are extremely low, about 0.1 nA at 8V (see the insert in Figure 3(b)), indicating considerable adsorbance of oxygen in the nanowires surfaces. Hence, both the UV photoconduction and dark conduction of the nanowire film can be enhanced by PDMS coating. The responsivity, defined as photocurrent per unit of incident optical power, of the device is determined by the UV photoconductivity of ZnO nanowires. From the I-V curves we can deduce that PDMS coating over ZnO nanowires results in a ~5-times enhancement in responsivity of the device.

![Figure 3. I-V curves of ZnO nanowire films under UV illumination and in dark. (a) The device in PDMS with Ag-PDMS paste electrodes; (b) Two devices in air, with vacuum deposited metal electrodes (Ti or Ag).](image)

Figure 4 shows the UV photoresponse characteristics of two devices based on ZnO nanowire sheets with the same dimension. The device with vacuum-deposited Ti electrodes was exposed to air, and the other one with Ag paste electrodes is
embedded in PDMS. Both were measured at 8 V with the UV light switched on and off periodically. Figures 4(a) and 4(b) show the UV photoresponse characteristics of the devices in air and in PDMS, respectively. The current-time curves depicting the UV photoresponse are further plotted in Figures 4(c) and 4(d) on a logarithmic scale. The maximum photocurrent of the device in air is 86 μA, while that of the device in PDMS reaches to 476 μA. The PDMS coating over ZnO nanowires leads to enhancement of UV photosensitivity and prolongs the decay time. The photocurrent rising and dark current decay of the two devices are compared in Figures 4(e) and 4(f), respectively, with the current maximum normalized. Upon switching on the UV light the current of the nanowire film in PDMS rises fast with a speed equivalent with that in air. Within one second the current reaches to half of the maximum upon UV illumination. We define the dark current decay time as the time for the current to decay to 10% of the peak value. For the nanowire film in air the decay time is ~7s as shown in Figure 4(f), whereas for that in PDMS the decay time is 29s, about 4 times prolonged. Previous reports on ZnO nanowires, which have gone through a polymer-involved lithography process for making electrodes [19-23], demonstrate slow UV photoresponse compared to those never contacted with polymers [7,9,10]. Surface contaminations by photoresist or electron-beam resist polymers are probably responsible for the reduced response speed. The photoresponse speed of our ZnO nanowire film in PDMS is equivalent or even faster than those contaminated by polymers from lithography process, suggesting that ZnO nanowires in PDMS may be adequate for applications that do not require particularly high speeds.
Figure 4. (a and b) UV photoresponse characteristics of ZnO nanowire sheets in air and in PDMS, respectively. The electrode of the device in air is vacuum-deposited Ti, and that of the device is PDMS is Ag-PDMS paste. The bias is at 8V for both devices. (c and d) UV Photoresponse characteristics of the two devices with the current on a logarithm scale. (e and f) Comparison for UV photocurrent rising and dark current decay of the nanowire films in air and PDMS, respectively, with the current maximum normalized.

The photoresponse of ZnO in air is known to be governed by adsorption (in dark) and desorption (under UV illumination) of oxygen molecules. In dark, oxygen molecules adsorbed on the surface of ZnO nanowires capture free electrons of the n-type semiconductor: $O_2(g) + e^- \rightarrow O_2^-(ad)$. This produces a depletion layer near
the nanowire surface, resulting in upward band bending near the surface. Due to the large surface-to-volume ratio, the adsorption of O₂ significantly reduces the conductivity of the nanowires. UV light illumination with photon energy higher than E_g generates electron-hole pairs in ZnO. Holes migrate to the surface along the potential slope created by the band bending and recombine with O₂-trapped electrons, thus releasing oxygen from the surface: \( O_2^- (ad) + h^+ \rightarrow O_2(g) \). The heavily populated electrons in conduction band enhance the conductivity of ZnO nanowires.

The UV photoresponse of ZnO nanowires in PDMS can be explained with the help of Figure 5. After polymerization, the PDMS molecule chains of CH₃[Si(CH₃)₂O]ₙSi(CH₃)₃, where n is the number of repeating monomer [SiO(CH₃)₃] units, form a network polymer structure that is highly permeable. PDMS has high oxygen permeability due to the large free volume from the flexibility of the siloxane (-SiO-) linkages; The oxygen concentration in PDMS has been reported as 2 mM [24]. The diffusion coefficient of oxygen in PDMS is reported as \( 3.55 \times 10^{-5} \) cm²/s [25]. Therefore, entangled PDMS molecule chains and dissociated oxygen molecules are around ZnO nanowires as illustrated in Figure 5(a). The interaction between ZnO and cross-linked PDMS is believed to be of the Van der Waals type. Charge interaction between the two materials is unlikely to occur because the PL properties of ZnO nanowires in air and embedded in PDMS are very similar. In dark the current of the nanowire film in PDMS could reach 110 nA at 8 V as shown in Figure 3(a), whereas the dark current of such a nanowire film exposed to air was just 0.1 nA at 8V, as reported in our previous work [10]. This means that the nanowires in PDMS have a smaller surface available to absorb oxygen molecules. In fact if a ZnO nanowire is embedded in cross-linked PDMS, it is conceivable that only the nanowire surface areas not in contact with PDMS would adsorb oxygen molecules diffusing
through the rubbery polymer (Figure 5(b)). Upon UV illumination the holes in valence band combine with \(O_2\)-trapped electrons, releasing the surface \(O_2\) and enhancing the photoconduction (Figure 5(c)). The desorbed \(O_2\) diffuse into PDMS molecule networks, and will be slowly re-adsorbed to ZnO nanowires after switching off the UV light. PDMS molecule chains hamper the migration of oxygen molecules towards the ZnO nanowire surface, leading to the slow decay of dark current. It is worth mentioning that the UV photoresponse speed of ZnO nanowires in oxygen atmosphere is also dependent on the gas pressure, i.e., oxygen concentration. The lower the pressure, the slower the UV photoresponse speed [4]. A number of silicone and polysulphone polymers have been studied for gas separation membranes [26,27]. Hence we speculate that ZnO nanowires embedded in a gas permeable polymer with higher oxygen solubility would have faster UV photoresponse. The UV penetration depth in ZnO is less than 100 nm while the thickness of the nanowire film is several micrometers, indicating that the contribution to photoconduction of a nanowire sheet comes mostly from the top layers. With PDMS (refractive index \(\sim\)1.5) filling the interspaces between nanowires, the paper-like ZnO nanowire film becomes more translucent to naked eyes, facilitating UV light scattering to a deeper level in the nanowire sheet. Therefore more nanowires will receive UV photons causing the enhanced responsivity of the nanowire sheet in PDMS.
Figure 5. (a) Illustration of a ZnO nanowire in cross-linked PDMS. The blue lines represent PDMS molecule chains. (b) Illustration for a nanowire in PDMS without UV illumination, with oxygen adsorbed in the surface at partial areas. The colour gradient across the cross-section indicates a depletion layer near the surface. (c) Under UV illumination the oxygen molecules desorb from ZnO and diffuse into the molecule network.

Conclusions

In summary, we demonstrated that ZnO nanowires embedded in PDMS polymer show great promise for UV detection. Our device based on a free-standing nanowire sheet in PDMS is bendable and facile to fabricate. The rubbery PDMS, which is gas permeable, not only provides protection to ZnO nanowires but also enables them to adsorb oxygen molecules diffusing in the polymer. Hence the UV photoresponse mechanism of the nanowires in PDMS is analogous to those exposed to an oxygen atmosphere. The PDMS coating over ZnO nanowires leads to an enhancement of responsivity, at the expenses of speed compared to the same uncoated nanowires. However, the response speed of the ZnO nanowire film in PDMS is still acceptable.
and in the range of previously reported ZnO nanowire films exposed to air. Our study opens a way for making robust UV photo-sensors using ZnO nanowires embedded in gas-permeable polymers.

**Experimental**

The ZnO nanowires were synthesized within a horizontal tube furnace (inner diameter 5 cm) at atmospheric pressure without using any catalyst. Mixtures of ZnO and graphite powders (2-3 g), a weight ratio of 1:1, were heated to 1100-1200 °C, and the vaporized growth species were transported by a gas flow that consisted of 1000-sccm N₂ and 30-sccm O₂. Cotton-like white products were deposited in low-temperature (between ~200 °C and room-temperature) region. We put an aluminium foil curved against the inner wall of the quartz tube to collect the nanowire product [18]. Thin sheets of ZnO nanowires were fabricated by a simple filtration method. First, a ZnO-nanowire suspension solution, concentration of 1 mg/ml, was prepared by ultrasonically dispersing the nanowires in isopropanol. After, the ZnO-nanowire suspension was vacuum-filtered through a porous anode aluminum oxide (AAO) membrane, diameter of 4.3 cm and pore size of 200 nm, purchased from Whatman Co. Then the network film of ZnO nanowires on an AAO membrane was dried in air at 100 °C for 1 h. Finally, the thin sheet of ZnO nanowires was detached off the membrane filter.

PDMS polymer was prepared by mixing the viscous PDMS liquid (Sylgard 184) and the cross-linking agent with a mass ratio of 10:1. The mixed PDMS was poured into a polystyrene petri dish and dried in vacuum for 5 hours to form a rubbery PDMS film. The PDMS liquid was also used for making silver paste by blending with Ag nanopowder (diameter ~100 nm). The device fabrication process is illustrated in Figure 6. First, a poly(ethylene terephthalate) (PET) film is placed onto a PDMS
layer. Onto the PET film a trap-shaped ZnO nanowire film is laid down and then silver paste is painted at two ends. At each end a thin copper wire is in contact with the silver paste. We wait for 5 hours until the PDMS blended with Ag nanoparticles is cured. Afterwards, we put the device into a plastic petri dish and pour PDMS liquid onto the nanowire film to thoroughly seal the device, followed by a vacuum drying process for cross-linking of the polymer. The thickness of PDMS layer above the ZnO nanowire film can be controlled by adjusting the volume of PDMS liquid in the petri dish. In this work the thickness of the PDMS layer above the ZnO nanowire film is about 2 mm.

![Illustration for the device fabrication process.](image)

Figure 6. Illustration for the device fabrication process.

For the devices to be measured in air, we deposited 100-nm-thick metal electrodes (Ti or Ag) onto the strap-shaped ZnO nanowire sheets (width 2 mm) by e-beam evaporation through a shadow mask. The electrode area is 3 mm-long and 2 mm-wide, as defined by the hollow pattern in the shadow mask. The gap between two opposite electrodes is 10 mm.

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