High-performance flexible ultraviolet photoconductors based on solution-processed ultrathin ZnO/Au nanoparticle composite films

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Transparent ultraviolet (UV) ZnO thin film photoconductors are expected to have great applications in environmental monitoring, large-area displays, and optical communications, and they have drawn enormous interests in recent years. However, at present their performances are not satisfactory: the responsivity $R$ (a parameter characterizing the sensitivity of the device to light) is not high ($<1.0 \times 10^3$ AW$^{-1}$), and the transparency $T$ is not high either ($<80\%$). Realizing high $R$ and high $T$ remains a big challenge today. In this paper, by employing solution-processed ultrathin ZnO/Au nanoparticle composite films, $R$ as high as $1.51 \times 10^5$ AW$^{-1}$ and $T$ of over 90% are achieved. High values for detectivity $D^*$ and linear dynamic range LDR are also obtained, which are $2.05 \times 10^{15}$ Jones and 60 dB, respectively. Moreover, such high-performance devices can be fabricated on flexible PET (polyethylene terephthalate) substrates.

Fully-visible transparent ultraviolet (UV) thin film photoconductors have great application potential in environmental monitoring, large-area displays, and optical communications, and they are of great importance in both civilian and military fields. For an ideal visibly-blind UV thin film photoconductor, it should display high light response capability in the UV region, good linearity of the photocurrent versus incident optical power and good visible rejection (i.e., high spectral selectivity). Structural simplicity, low-cost fabrication and room-temperature operation are also crucial for real applications. In material selection, nitride compounds and metal oxides, which are wide-bandgap (larger than 3 eV) semiconductors, are considered ideal ones for high-performance UV photoconductors.

ZnO, which is environment-friendly and has a room-temperature bandgap of 3.2 eV, is emerging as an important material for the next generation UV photoconductors. In the early days, Liang et al. presented their ZnO film based photoconductors, which exhibited a $R$ value of $114$. Such small $R$ is resulted from both the low electron mobility (induced by the poor quality of the ZnO film) and insufficient depletion layer (induced by the small surface-to-volume ratio of the ZnO film). Later, most work concentrated on enhancing the surface-to-volume of ZnO (to enlarge the depletion area) by using nanostructured ZnO film, and the performance was indeed improved. For instance, Jin et al. have reported the ZnO nanoparticle based photoconductor, which showed a $R$ value of $61$ AW$^{-1}$, and recently Zhu et al.

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reported their electron-spun ZnO-nanofiber based photoconductor with a high R value of 790 AW \(^{-1}\). However, the unavoidable nanoscale networks formed in the nanostructured ZnO film would seriously weaken the carrier mobility (because the ZnO nanostuctures will not only increase the distance carrier travels between the two electrodes, but also will trap carriers at boundaries between them), which would severely limit the improvement of the R value. For example, it was reported that ZnO nanoparticle film showed an electron mobility of only \(5 \times 10^{-4} \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}\). Additionally, the nanostreams may produce strong scattering for incident light and affect the light transmission in the visible light range\(^{3,16}\), and hence reduce the device transparency. For example, the electron-spun ZnO-nanofiber based film exhibits only about 75% optical transmittance (T) in the visible light range\(^{3,16}\). Therefore, realizing high T and high R ZnO based thin film photoconductor still remains a challenge today.

**Results**

In this paper, a composite film consisting of ultrathin ZnO film (\(\approx 5 \text{ nm}\)) and Au-DT nanoparticles (Au NPs) is applied to realize photoconductors with high R and high T (seen in Figure 1a). The continuous ZnO film exhibits high electron mobility of over 1.0 cmV\(^{-1}\)s\(^{-1}\). Moreover, the surface-to-volume ratio of the ultrathin ZnO film is quite large: The O\(_2\) related depletion layer on ZnO surface is estimated to be about 1–3 nm\(^{14}\), which means that the depletion region is already quite sufficient for the 5 nm ultrathin ZnO film. The Au NP put on the surface of the ZnO film leads to the formation of a localized Schottky junction, which creates a ZnO depletion region in the vicinity of the Au NP. This will further enhance the already existing O\(_2\) related depletion layer. Thus, the Au NPs can play important roles in reducing the dark current, enhancing the sensitivity and improving the response speed of the ZnO based thin film photoconductors\(^{12,20}\). The very small amount of the Au NPs with small sizes (2–4 nm in diameter) on the ZnO film have negligible absorption, reflection/scattering for the incident visible light. A result, the ultrathin ZnO film itself shows a high T exceeding 96% while the final device shows a high T of over 90% (in the visible light range). R value as high as 1.51 \(\times 10^5\) AW\(^{-1}\) is achieved in the UV region.

Figure 1b presents the cross-sectional scanning electron microscopy (SEM) of our solution-processed ZnO film (the white line) and Figure 1c (PET substrate) and Figure 1d (glass substrate). The optical microscope photograph of the patterned ITO electrodes used in our photoconductors is given in Figure 1e (the light blue lines).

The surfaces of the individual ZnO films deposited 1, 2, 3 and 8 times (will be called 1T, 2T 3T, 8T ZnO later (seen in Methods part)) were studied by atomic force microscopy (AFM) and the morphologies are given in Figure 2a–d. It is seen that the deposition cycle affects the film morphology, the surface roughness is reduced from 0.38 nm for the 1T ZnO film to 0.21 nm for the 2T ZnO film, and then it almost remains unchanged for the 3T and the 8T ZnO films (Figure S1). In order to gain more information about the films, SEM experiments were conducted. The SEM images of the 3T ZnO film are given in Figure 2e and S2. Combining AFM and SEM image, it can be concluded that the surface of the ZnO film is consisted of small grains, which can result in a large surface-to-volume ratio. The compositions of these grains can be identified by the energy dispersive spectrometer (EDS) and the X-ray photoelectron spectroscopy (XPS) measurements. The EDS spectra (Figure 2f) and core level XPS spectra (Figure 2g and Figure S3) are both overwhelmingly dominated by the Zn and O peaks (the strong Si peak is from the SiO\(_2\) substrate). Figure S3, which is an enlarged figure of Figure 2g, provides detailed information about the Zn 2p and O 1s. The Zn 2p\(_{3/2}\) peak is located at 1021.27 eV and the Zn 2p\(_{1/2}\) peak is located at 1044.47 eV. The first O 1s peak is located at 530.37 eV, which corresponds to O atoms in ZnO. The second O 1s peak is located at 532.22 eV, which is attributed to oxygen-deficient component (for example, zinc hydroxide, Zn(OH)\(_2\)). The results are in well agreement with the XPS spectra of ZnO obtained by other research groups\(^{14,22}\).

The synthesized Au NPs are studied by transmission electron microscopy (TEM), absorption and X-ray Diffraction (XRD), shown in Figure 3a, b and c, respectively. These results clearly demonstrate that the purity of the Au NPs is very high (Figure 3c), and they are very small and uniform (Figure 3a and b), with diameters in a very narrow range of 2–4 nm (inset in Figure 3a). We also performed AFM measurement on the Au NPs spin-coated on the ultrathin 3T ZnO film, the height image and phase image are given in Figure 3d and Figure S4a, respectively. From the two images it is clearly seen that the Au NPs are sparsely distributed on the ZnO film, and they only cover a very small fraction of the whole surface of the ZnO film.

The optical transmission properties the ZnO films (1T to 8T) on quartz substrates without and with the Au NPs on top are investigated.

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**Figure 1** | (a) Schematic illustration, (b) cross-sectional SEM image, (c) device flexibility, (d) device transparency and (e) patterned ITO electrodes on the substrate of the fabricated UV photoconductor (L = 50, 100, 150, and 200 \(\mu\)m, W = 5000 \(\mu\)m).
by the UV-vis transmission measurements. The transmission properties of the quartz substrate \((T_{\text{quartz}})\) itself is also measured to extract the pure transmission properties of the ZnO films \((T_{\text{ZnO}} = T_{\text{ZnO/Quartz}} - T_{\text{quartz}})\). \(T_{\text{ZnO}}\) and \(T_{\text{ZnO/Quartz}}\) are given in Figure S4c and Figure S4d, respectively \((T_{\text{quartz}}\) is provided in Figure S4d). It is seen from these figures that for all the samples, the transparency is very high with \(T\) value larger than 96% in the visible range \((400–800\, \text{nm})\), and \(T\) value larger than 90% at low wavelength regions \((<400\, \text{nm})\). The high \(T\) is obviously originated from the ultrathin nature of the films. Although the surface plasmon in the Au NPs can couple with incident light, producing strong scattering and absorption of incident light\(^{23–25}\), the Au NPs have negligible influence in the \(T\) value. This can be clearly seen by comparing Figure S4d and Figure 3f, in which transmission properties of the ZnO films with Au NPs are given with almost the same transparency of \(>96\%\) in the visible range \((400–800\, \text{nm})\) and of \(>90\%\) in the lower wavelength range \((<400\, \text{nm})\). The high transparencies of the ZnO films with Au NPs are also clearly demonstrated in Figure 3e, which is an optical photograph of the 1T, 2T, 3T, 8T ZnO/Au NPs films on a underlying logo.
We performed XRD measurements on the four ZnO films with Au NPs (Figure S5a) aiming to relate the possible crystal nature to their electronic properties, however no XRD peak shows up in the spectra except the one from the quartz substrate. The absence of XRD peak should be that the films are too thin to give any detectable XRD signals. Field effect transistors (FETs) are fabricated for the four ZnO/Au NPs films, and their transfer curves are given in Figure S5b. It is seen that 1T ZnO/Au NPs film does not show any field effect. This could be induced by the discontinuous nature of the 1T ZnO film (seen AFM image in Figure 2a), 2T, 3T and 8T ZnO/Au NPs films all show good field effect performances, which should be induced by the high-quality continuous nature of the three ZnO films (see AFM images in Figure 2b, c and d). We also fabricated FETs with the 1T, 2T, 3T, 8T ZnO films without Au NPs on top, and their performance are very similar to that of the corresponding devices with the ZnO/Au NPs films. The 3T ZnO/Au NPs film exhibits FET performance better than the 2T ZnO/Au NPs film, but very close to that of the 8T ZnO/Au NPs film. Thereby in later study we only perform investigation on devices based on the 3T ZnO/NPs film. The transfer curve and output curve of the FET based on the 3T ZnO/Au NPs film are shown in Figure 4a and b, respectively. The device has an electron mobility of 1.78 cm² V⁻¹s⁻¹ and an on/off ratio of 6 × 10⁸. The mobility was calculated based on the following equation:

\[ I_{DS} = C_i \mu \frac{W}{L} (V_{GS} - V_T)^2 \]  

Where \( I_{DS} \) is the drain current, \( C_i \) is the capacitance per unit area of the gate dielectric layer, \( \mu \) is the carrier saturation mobility, \( W \) and \( L \) are the channel width and channel length (here they are 2000 and 50 μm, respectively), \( V_{GS} \) and \( V_T \) are the gate voltage and threshold voltage respectively.

We studied the light response capability of the 3T ZnO/Au NPs FET by illuminating the device with white light, and the results are given in Figure 4c. It is seen that the drain current increases dramatically when the device is under a very weak light illumination (8.3 μW cm⁻²), and the current increases with the light intensity (transfer curve moves upwards). For a photodetector device, R and signal to noise ratio (P) are two key parameters to characterize its performance. R is defined by the following equation:

\[ R = \frac{I_{Light} - I_{Dark}}{P_{ill}} \]  

Where \( I_{Light} \) is drain current under the illumination, \( I_{Dark} \) is drain current in dark, \( P_{ill} \) is the incident illumination power on the effective area (channel area). P is expressed by the following equation:

\[ P = \frac{I_{Light} - I_{Dark}}{I_{Dark}} \]  

Based on the Equation (2) and (3), we calculated R and P of the phototransistor device under white light illumination with an intensity of 242.3 μW cm⁻², and they are shown in Figure 4d. For a practical photodetector, the device should keep both high P and R. Thus, the region around the crossing point of two curves can be chosen as the useful workable region. In our ZnO FETs based phototransistor, the crossing point is close to the \( V_G = 0 \) V (with high R value of around 440 AW⁻¹ and high P value of 6 × 10⁴), which is very good for practical application because only a very small applied voltage is needed.

We fabricated 3T-ZnO based photoconductors on commercial PET and Glass substrates with patterned 120 nm-thick ITO electrodes, which are shown in Figure 1c and d, respectively (The ITO electrodes are given in Figure 1e). The transparencies of the final devices based on the 3T ZnO/Au NPs films are demonstrated in
Figure 5. (a) Optical transmittances of the photoconductors. (b) Spectral R of the photoconductor based on the 3T ZnO/Au NPs film (L = 50 μm, V = 50 V), the inset shows the response of I-V curve to illumination light wavelength (10.6 μW/cm²). (c) I-V curves of the photoconductors (with and without Au NPs) in dark and under 350 nm UV light illumination with an intensity of 10.6 μW/cm² (L = 50 μm); (d) I-V curves of the photoconductors (with Au NPs) with different channel lengths under 350 nm UV light illumination (10.6 μW/cm²).

For a photoconductor, R and G are two important parameters, and R can be expressed by the following equation:

\[
R = \frac{\text{EQE}^2 q G}{h \nu} \approx (1 - T) \frac{\text{EQE}^2 q G}{h \nu}
\]  

(4)

Where EQE is the external quantum efficiency, \(\lambda\) is the wavelength of interest, \(q\) is the electron charge, \(h\) is the Planck constant and \(c\) is the speed of light. G is defined as the ratio between the number of electrons collected per unit time and the number of absorbed photons per unit time, and can be expressed by the following equation:

\[
G = \frac{\mu_n + \mu_p}{L} \frac{\tau E}{c} = \tau \left( \frac{1}{t_n} + \frac{1}{t_p} \right)
\]  

(5)

Where \(\mu_n\) is the electron mobility, \(\mu_p\) is the hole mobility, \(\tau\) is the photo carrier lifetime, \(E\) is the electrical field, \(L\) is the device channel length, \(t_n\) is the electron transit time, \(t_p\) is the hole transit time. From the Equation (4) and (5), it is seen that light response can be improved by enhancing the photo carrier lifetime \(\tau\) and reducing the carrier transit time \(t_n/t_p\). In our photoconductor, the electron mobility \(\mu_n\) is high (and hence electron transit time \(t_n\) will be small), and the photo generated holes will migrate into the depletion region induced by O₂ and the Au NPs on the ZnO surface, and are then trapped there, largely preventing them from recombining with photo generated electrons. The much suppressed recombination will greatly enhance the carrier lifetime \(\tau\). Based on equation (5), the high electron mobility \(\mu_n\) and long carrier lifetime \(\tau\) would result in high \(G\), and hence high \(R\) (equation (4)), although the light absorption of...
our device (<4% at a wavelength of 350 nm) is quite poor due to its ultrathin nature. This is proved by the experiment results. High G and high R are indeed obtained.

Another two figures of merits for evaluating the photoconductor performance are the detectivity (D*, in units of Jones to characterize the sensitivity) and the linear dynamic range (LDR, typically quoted in dB to measure the photosensitivity linearity). The D* can be expressed by the following equation:

\[ D^* = R \sqrt{\frac{S}{2qC}} \]  

(6)

Where \( S \) is the effective area under illumination, \( q \) is the electron charge, \( I_{\text{dark}} \) is the dark current. LDR is given by the following equation:

\[ \text{LDR} = 20 \log \left( \frac{I_{\text{Light}}}{I_{\text{Dark}}^2} \right) \]  

(7)

Where \( I_{\text{Light}} \) is the photocurrent with a light intensity of 1 mW/cm\(^2\), \( I_{\text{Dark}} \) is the dark current. Based on equation (2) and the experiment results shown in Figure 5, we calculated R value of the 3T ZnO/Au NPs device, and then calculated G value of the device based on equation (4). The calculated R and G as a function of voltage are shown in Figure 6a. Based on equation (6), \( D^* \) values are calculated, and they are shown in Figure 6b. G, R and \( D^* \) for the photoconductor under a bias of 50 V (\( \lambda = 350 \text{ nm} \)) are 1.77 \( \times \) 10\(^5\), 1.51 \( \times \) 10\(^5\) AW\(^{-1}\) and 2.05 \( \times \) 10\(^{15}\) Jones, respectively. Such performance is much better than that of previously reported ZnO based thin film UV photoconductors, whose performances are already described in the introduction part of this paper. Figure 6c shows the photocurrent versus the white light intensity for the device, from which LDR can be calculated based on equation (7), the calculated LDR is 60 dB of intensity linear dynamic range, a value typically required for most imaging applications.

Figure 6d shows the photocurrent as a function of time under white light illumination with different intensities for the 3T ZnO/Au NPs photoconductor (under a bias of 50 V). It can be clearly seen that the photocurrent responses sensitively to the illumination light intensity. In Figure 6e, the on-off switching properties of the device is shown under light illumination with 350 nm wavelength (under a bias of 50 V and illumination intensity of 10.6 \( \mu \text{W/cm}^2 \)) with both on and off time are 200 s, it is seen that the device exhibits good on-off switching performance. We also show the time-resolved photocurrent response of the device in Figure 6f. Upon illumination, the current of the device rises within 2 s. The rapid photocurrent rise is followed by a slower component, in which the photocurrent keeps increasing for about 40 s before saturation. After the light is turned off, the photocurrent drops rapidly in the first 3 s, followed by a slow decay process of more than 56 s before it returns to its initial current in dark.

Such rise and decay responses can be described and fitted by equations (8) and (9), respectively:

\[ I_{\text{Light}} = I_{\text{Dark}} + A \left[ \exp \left( -\frac{t}{\tau_1} \right) \right] + B \left[ \exp \left( -\frac{t}{\tau_2} \right) \right] \]  

(8)

\[ I_{\text{Light}} = I_{\text{Dark}} + A \left[ \exp \left( \frac{1}{\tau_1} \right) \right] + B \left[ \exp \left( \frac{1}{\tau_2} \right) \right] \]  

(9)

Where \( I_{\text{Dark}} \) is the dark current, \( A \) and \( B \) the scaling constants, \( t \) is the time when the light was turned on or off, and \( \tau_1 \) and \( \tau_2 \) are the time constants. The two equations are widely used to analyze the photocurrent rise and decay processes of a photoconductor device\(^{30-32}\). By fitting the experiment data with the two equations, the time constants for rise and decay can be estimated. Here for our device, the estimated time constants for the rise process are: \( \tau_1 = 10.3 \text{ s}, \tau_2 = 70.6 \text{ s} \). And the time constants for the decay process are: \( \tau_1 = 14.2 \text{ s}, \tau_2 = 72.3 \text{ s} \). \( \tau_1 \) (in both rise and decay processes) should be related to the carrier generation and recombination processes in the ZnO thin film, and \( \tau_2 \) (in both the rise and decay processes) should be mainly related to hole trapping and release processes in the depletion layer on the surface of the ZnO film.

**Figure 6** | The 3T ZnO/Au NPs photoconductor (\( L = 50 \mu \text{m} \)): (a) Calculated photogain and R and (b) calculated \( D^* \) under 350 nm UV light illumination (10.6 \( \mu \text{W/cm}^2 \)). (c) LDR measured under white light. (d) Current versus time under various white light illumination intensities under a bias of 50 V. (e) On-off switching properties measured under 350 nm UV light illumination (10.6 \( \mu \text{W/cm}^2 \)) under a bias of 50 V. (f) Time resolved photocurrent: rise and decay (inset) curves.
Discussion

In conclusion, by employing solution-processed ZnO ultrathin film and solution-processed Au NPs, high-performance UV photodetectors are fabricated. Due to the high electron mobility (over 1.0 cm²V⁻¹s⁻¹) exhibited by the ZnO film and the depletion layer deepening effect induced by the Au NPs on top of the ZnO film, these ZnO-based UV photodetectors exhibit R values as high as 1.51 × 10⁸ AW⁻¹ and D* values as high as 2.05 × 10⁹ Jones in the UV region. The ultrathin nature of the ZnO film (~5 nm) and the ultrasmall sizes of the Au NPs (2–4 nm in diameter) render the ZnO/Au NPs films and the final devices (On PET and Glass substrates) highly transparent. The films and the photodetectors show extremely high T (transmittance) values of exceeding 96% and 90%, respectively. The devices fabricated on PET substrates display high flexibility, which are very important in real applications. We also demonstrate that by shortening the channel length, the device performance could be further improved. Our results indicate that the approach we used here is very useful in fabricating high-performance ZnO-based thin film UV photodetectors.

Methods

Material preparation. All of the materials were purchased from Sigma-Aldrich and were used as received without further purification. The polyethylene terephthalate (PET), Corning Eagle XG glasses and N-doped silicon with 300 nm silicon dioxide were used as the substrates. It should be noted that the ITO electrodes of 50, 100, 150 and 200 μm in channel length and 5000 μm in channel width were patterned on the PET and Glass substrate.

Au-DT core-shell nanoparticles preparation. The Au NPs were prepared by the two-phase arrested growth method²⁸. We dissolved 0.62 g HAuCl₄·H₂O in 50 ml water and mixed it with 3 g tetracyanoethylene bromide dissolved in 160 ml p-xylene solution. The organic phase was isolated, and 0.8 ml was added. The formation of the Au NPs were completed by adding 50 ml aqueous solution of 0.76 g NaBH₄ drop wise into the organic solution under vigorous stirring. The Au NPs were purified by washing with 300 ml methanol at least three times and result in a narrow size distribution (2–4 nm in diameter).

Solution ratio and concentration. Zn ammonium complex solution was prepared by dissolving ZnO hydrate in ammonium hydroxide (28–30%) to molar concentration according to equation (1) (2–4 nm in diameter).

\[\text{ZnO} + \text{NH}_4\text{OH} \rightarrow \text{Zn}^{2+} + 2\text{NH}_3 + \text{H}_2\text{O}\]

Measurement. All electrical characterizations were recorded on a Keithley 4200 and a Micromanipulator 6150 probe station at room temperature in air. A white-light halogen-tungsten lamp with power intensity adjustable was used for illumination. The monochromatic light is from a Newport Oriel 200TM, and the intensity is 10.6 Wcm⁻². Tungsten lamp with power intensity adjustable was used for illumination. The spectral response and the light intensity were calibrated prior to the use of the light, the spectral response and the light intensity were calibrated using a photodetector. The XPS measurements were performed in a Kratos Ultra Spectrometer (a base pressure of 1 × 10⁻¹⁰ Torr) using monochromatized Al Kα X-ray photons (hν = 1846.6 eV) discharge lamp. UV-vis spectra were recorded using JASCO V-570 spectrophotometer. The phase identification was determined by using a Rikaku D/MAX-2004 XRD with Cu Kα radiation (λ = 1.54178 Å) operating at 40 kV and 60 mA.

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Z.J., L.G. and Q.Z. performed the experimental work. Z.J. performed the data analysis and experimental planning. The project was conceived, planned and supervised by Z.J. and J.W. The manuscript was written by Z.J. and J.W. All authors reviewed the manuscript.

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