Laser Modified ZnO/CdSSe Core-Shell Nanowire Arrays for Micro-Steganography and Improved Photoconduction

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Arrays of ZnO/CdSSe core/shell nanowires with shells of tunable band gaps represent a class of interesting hybrid nanomaterials with unique optical and photoelectrical properties due to their type II heterojunctions and chemical compositions. In this work, we demonstrate that direct focused laser beam irradiation is able to achieve localized modification of the hybrid structure and chemical composition of the nanowire arrays. As a result, the photoresponsivity of the laser modified hybrid is improved by a factor of ~3. A 3D photodetector with improved performance is demonstrated using laser modified nanowire arrays overlaid with monolayer graphene as the top electrode. Finally, by controlling the power of the scanning focused laser beam, micropatterns with different fluorescence emissions are created on a substrate covered with nanowire arrays. Such a pattern is not apparent when imaged under normal optical microscopy but the pattern becomes readily revealed under fluorescence microscopy i.e. a form of Micro-Steganography is achieved.
the fast rastering speed and high energy intensity of lasers, laser modification is a rapid and quenching process as compared with other slower processes.

To the best of our knowledge, this is a first report on the modification of hybrid ZnO/CdSSe core-shell nanowire arrays utilizing a scanning focused laser beam. By controlling the power of the scanning focused laser beam, micropatterns with different fluorescence emissions are created on a substrate covered with these hybrid nanowire arrays. If the pattern is created using low laser power, such a pattern is not apparent when imaged under normal optical microscopy but the pattern becomes readily revealed under fluorescence microscopy i.e. we have achieved a form of Micro-Steganography. In addition to the modification of the optical properties of the hybrid nanowires, the focused laser modification could also be simultaneously utilized to modify the electrical properties of these hybrid nanowires. In doing so, we strive to achieve a good balance in the control of the optical and electrical properties of the type II core/shell nanostructures en route to functional optoelectronic devices. Based on this, a high performance 3D photodetector is fabricated and demonstrated using laser pruned ZnO/CdSSe nanowire arrays with monolayer graphene as transparent top electrode. The construction of micropatterns with functionality/fluorescence control within the nanowire arrays would provide greater building blocks for optoelectronic applications.

**Results**

The nanowire arrays studied in this work consist of vertically aligned hybrid ZnO/CdSSe core/shell nanowires grown epitaxially on quartz substrates with pre-deposited 200 nm ZnO film using a sequential 2-step vapor-solid growth12,13 (see Methods). Notably, vertically aligned nanowires with high uniformity covering an area of more than 1 cm² are fabricated17. The composition and thickness of the shells is accurately controlled18. Figure 1a shows SEM images of the nanowire arrays before and after CdSSe shell deposition. These images clearly show the quasi-vertical alignment of the nanowires. Other crystalline structural characterizations are demonstrated by the TEM and HRTEM images. The stoichiometric uniformity is determined by the EDX mapping and line scans along an individual nanowire. Details of these characterizations are shown in Supplementary Section (S1).

Direct modification of the hybrid core-shell nanowires is carried out through a focused laser beam setup. Figure 1b shows the schematic diagram of the experimental setup. In this setup, a diode laser is employed as the source of the laser beam. In separate experiments, we have made use of two different types of diode laser in this work. Namely, one of the laser sources emits laser beam with a wavelength of 660 nm at a maximum power of 70 mW at source while the other laser source emits laser beam with a wavelength of 532 nm at a maximum laser power of 200 mW at source. The emitted laser beam is guided into an optical microscope through the reflection of two mirrors. Inside the microscope, the laser beam is directed toward an objective lens by a beam splitter. We have different types of objective lenses in this setup and we can select one with a magnification of 50× or 100× for our experiments. After passing through the objective lens, the laser beam is tightly focused onto the surface of a sample. The sample is mounted on a motorized stage which is flexibly moveable in X-Y direction. A CCD camera is used to monitor the process of laser modification and examine the quality of the pattern. When the laser beam is focused upon the nanowire arrays, the laser energy absorbed by the CdSSe shell is rapidly converted to local heat. The intense heat raises the temperature of irradiated nanowire tops and causes localized modification of the core/shell structure. This modification technique is akin to lawn pruning and thus we denote it as the laser pruning method. By controlling the stage movement in a programmable manner, micropatterns are constructed on the nanowire arrays. Figure 1c shows a SEM image of a micro-pattern fabricated

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**Figure 1** (a) SEM images of as-grown ZnO nanowire arrays (upper image) and ZnO/CdSSe core/shell nanowire arrays (lower image). Scale bars are equivalent to 1 μm. (b) Schematic of focused laser beam set-up. (c) – (e) SEM, bright field optical, and FM images respectively of a micro-pattern created. Scale bars are equivalent to 200 μm.
using this method and Figures 1d and e show the same pattern observed using a bright field microscope and fluorescence microscope, respectively. Note that in this case, the laser source that emits laser beam with a wavelength of 660 nm at a power of 50 mW is utilized. The SEM image does not reveal a striking contrast from the created microstructure. This is because the length of the core/shell nanowires is not significantly affected. However, the reflectivity of the pruned regions becomes different from the pristine regions. As a result, the bright field optical image provides a clearer contrast. The bright orange background is reminiscent of the optical properties of the pristine nanowires. Prominently, the laser modified region exhibits a different reflective color which is bright yellow. Therefore, the micropattern becomes more visible. Perhaps the most significant change in the nanowire array is displayed by the image captured by the fluorescence microscope. The weak reddish-orange background originates from the pristine core/shell nanowires. CdSSe nano-structures possess high yield luminescence. Nevertheless, the formation of type II core/shell architecture impedes the irradiative carrier recombination. Therefore, only weak fluorescence emission is achieved upon UV excitation. Remarkably, laser treated nanowire array exhibits a different fluorescence behavior. The laser pruned region emits a dazzling quasi-white color and the difference in this color from the pristine nanowire array results in the best contrast among the three types of images. Clearly, the laser modified nanowires have been innovated into another type of material which shows different fluorescence property. This is a surprising result considering the destructive attribute of a high energy focused laser beam.

Quantitative investigations on the fluorescence behavior are carried out by the micro-photoluminescence spectroscopy. Figure 2a shows the photoluminescence spectra for pristine and laser pruned regions, respectively. The laser pruning is carried out with the diode laser with center wavelength at 660 nm and the laser power is maintained at 50 mW. Evidently, the pristine region shows a narrower photoluminescence spectrum and consequently facilitate the quasi-white light emission. As shown in Supplementary Section S3c, the HRTEM indicates that the nanoparticles are crystalline with the size smaller than 10 nm. It is possibly formatted by alloying among ZnO and CdSSe to facilitate the formation of quaternary compounds. Furthermore additional defects are introduced into the ZnO and CdSSe due to the laser irradiation and these defects can contribute their corresponding defect peaks in photoluminescence study. All these factors contribute to the broadening of the photoluminescence spectrum and consequently facilitate the quaswhite light emission.

As observed, the nanoparticle cluster is only constructed on the tip of the nanowires. This is caused by the heat energy accumulation at
the top of the nanowires. When the laser beam is focused upon the
top of the nanowires, the absorbed laser energy is rapidly converted
to local heat. Due to the ultralow thermal conductivity of the CdSSe
alloys (as measured in Supplementary Section S4, the calculated
thermal conductivity coefficient $k < 1 \frac{W}{mK}$), the heat energy cannot
be transmitted efficiently to the rest of the nanowires and the sub-
strate. Therefore, most of the heat energy is retained at the tip of the
nanowires. The gathering of the high temperature at such small
location causes the nanowire tips to “explode” to nanoparticle clus-
ters. We carry out additional investigation on the laser induced heat
distribution in these nanowires array by the finite element method
(FEM) simulation. A model with 100 core/shell nanowires is built.
The laser beam is rastered over the surface of the nanowire array at a
speed of 100 $\mu$m/s. Figure 2g shows the 3-dimensional temperature
distribution in the nanowires array after laser scanning of 0.01 s and
0.05 s, respectively. Evidently, most of the heating energy is found
localized at the top part of the nanowires. The dynamic process of the
temperature distribution is shown in the Supplementary Section (.gif
file).

Discussion

Systematic studies on the detailed change in the optical property
caused by the laser modification are carried out. Low temperature
and time-resolved photoluminescence spectra are measured with the
excitation of a pulsed diode laser operating at 405 nm. In order to
gain greater insight into the effects of laser pruning on the carrier
localization, the photoluminescence spectra of pristine and pruned
regions at a series of temperatures from 5 K to 300 K are carried out
and shown in Supplementary Section S5. As the temperature
decreases, the main peak of both regions increases remarkably in
intensity, whilst the peak position gradually blue shifts with decreas-
ting temperature. This blue-shift with temperature indicates the
expansion of band gap due to the suppression of both exciton-pho-
non interaction and thermal expansion$^{19-21}$. However, in contrast to
the room temperature photoluminescence, the spectrum of pristine
region exhibits much higher intensity than that of laser modified
region at 5 K (Figure 3a). This is because low temperature restrains
the thermal energy loss and carriers transport, which will in turn
suppress the separation of electron-hole pair at the type II core/shell
interface. Therefore, excitons are restricted inside the CdSSe shell
and thus irradiative recombination is enhanced. By applying peak
separation fitting on the spectrum (Supplementary Section S5), all
the peaks can be attributed to the recombination of free exciton A
(FXA), free exciton B (FXB), spatially localized donor-acceptor pairs
(DAP) and a series of longitudinal photon (LO) replicas of DAP in
CdSSe$^{22,23}$. Relatively, due to the small particle size and high density
of defects of the laser modified region, the temperature effects on the
carrier localization and decay are less distinct than that shown by
pristine region. Further evidence can be revealed from the time-
resolved photoluminescence measurements which will provide
details of the exciton dynamics. Figure 3b shows the photolumines-
cence decay traces of pristine region, laser pruned region and pure
CdSSe nanostructures (CdSSe nanowires without ZnO core) mea-
sured at 300 K. Evidently, both the laser pruned region and the
pristine region exhibit much faster decay than pure CdSSe nanos-
structure. By applying exponential function fitting on the experi-
mental measurements, the laser pruned sample is found to exhibit

![Figure 3](https://www.nature.com/scientificreports)

Figure 3 | (a) PL spectra of pristine (black curve) and laser pruned (red curve) regions measured at 5 K. Insert shows the bright field image of the pristine and pruned regions (created by 660 nm diode laser at 50 mW) used for low-temperature and time-resolved PL measurements. Scale bar is 50 $\mu$m. (b) PL decay traces of pristine region, laser pruned region and pure CdSSe nanostructures at 300 K. (c) Temperature depend PL decay traces of laser pruned sample. (d) PL decay traces of pristine region, laser pruned region and pure CdSSe nanostructures at 5 K.
the shortest photoluminescence lifetime, as listed in Supplementary Section S5. Inside the laser pruned nanostructures, a number of processes such as defect or boundary related trapping, thermal energy induced carrier delocalization, exciton dissociation, and non-radiative recombination can affect the carrier recombination. So the presence of many recombination paths results in the fast decay of the photoluminescence in laser pruned nanostructures. The ultrashort photoluminescence lifetime opens the possibility for implementation of laser modified nanostructures in ultrafast switching devices. In our previous study, the characteristic timescale for photoluminescence decay of the pure CdSSe nanostructures did not show monotonic variation with temperature. In contrast to previous result, the photoluminescence lifetime of the laser pruned nanostructure (Figure 3c) increases monotonically with decreasing temperature. This result is in contrast to the behavior observed in most conventional semiconductors whose photoluminescence lifetime decreases with decreasing temperature\(^{24,25}\). The increase of the lifetime can be attributed to the decrease in the number of additional recombination paths for the excitons as the temperature decreases. As a result, the photoluminescence lifetime of these two samples are increased when compared with the pure CdSSe nanostructure at 5 K, as shown in Figure 3d. Due to the large number of the intrinsic recombination paths, the laser pruned complex nanostructure still exhibit the shortest lifetime among these three samples.

Due to the different absorption responses to different wavelength lasers, the nanowire arrays can also be pruned by focused green laser beam (\(\lambda = 532 \text{ nm}\)). Remarkably, the pruned nanowires show significantly different morphology as compared with those modified by red laser (\(\lambda = 660 \text{ nm}\)) (Figure 2a and b). As shown in the SEM image (Figure 4a), matchstick-like nanostructures are created by the green laser pruning. Besides the morphology difference, photoluminescence emission is also widened and enhanced (Figure 4b). However, the increase in the intensity of the photoluminescence is lower than sample modified by laser with a wavelength of 660 nm. By careful control of the laser powers, different fluorescence emission can be observed. Figures 4c and d show optical and FM images for four micro-boxes created with the focused laser beam using different laser powers (3 mW, 5 mW, 16 mW, and 18 mW for I, II, III, and IV, respectively). Evidently, different emission colors are observed under blue light excitation. Systematic studies on the dependence of such laser modification on the power of the laser beam are carried out. A set of patterned micro-squares are created using laser beam with different laser powers. The corresponding characterizations of FM images, optical microscope images, SEM images and PL spectra are carried out and shown in Supplementary Section S6. Once we have established control over the optical properties of the as-grown nanowire arrays and the laser pruned arrays, we can engineer functional components with interesting applications. As an example, we can construct hidden image (micro-steganography) onto a substrate with aligned nanowire arrays. Figures 4e and f show an example of such a hidden image. We create a micro-square on the nanowire array using a low laser power (~2 mW). Under bright field optical microscopy, nothing can be observed as shown in Figure 4e1. The laser power used is so low that the laser modified nanowires do not show any apparent difference from the as-grown nanowires so the micro-square is not distinguishable. However, where the same feature is imaged under fluorescence microscopy as shown in Figure 4eii (UV light excitation) and eiii (blue light excitation), the hidden micro-square is revealed readily. More complex micro-steganography is demonstrated in Figure 4f. Firstly, we use a high laser power of 18 mW to create the boundary of a box so that the box is visible under bright field optical microscopy imaging as illustrated in Figure 4f1. Then we use a lower laser power (~2 mW) to create a micro-dragon within the box. The clear micro-encryption effect is demonstrated under fluorescence microscopy. The colored micro-dragon is obviously displayed under both UV (Figure 4fii) and blue light (Figure 4fiii) excitation. Thus this achieves the goal of hidden message creation and demonstrates the protocol for effective read-out.

Besides the modification in the optical properties, the laser pruned nanostructure system can be further functionalized to increase its potential applications. In our previous study, CdSSe nanostructure has been demonstrated to be a potential photoelectronic material with high photoconductivity\(^{26,27}\). Laser pruned CdSSe has been shown to exhibit superior light trapping ability in comparison to the pristine counterpart\(^{24}\). In recent research progress, graphene has been demonstrated to be a good transparent electrode due to its high degree of transparency\(^{29,30}\). We seek to combine the unique characteristics of the different components and create a hybrid system that shows potential in optoelectronic applications. A 3D device architecture is designed based on the assembly of monolayer graphene onto laser pruned (or pristine) ZnO/CdSSe nanowire arrays en route to unique functional 3D microstructures. To assemble the designed device architecture, the steps illustrated in Figure 5a are carried out. The graphene electrode is transferred from a CVD grown graphene on copper substrate through a standard transfer process as described previously\(^{31,32}\). Figure 5b shows the SEM images of the assembled hybrid structures consist of graphene on a typical nanowire array and laser pruned nanowire array, respectively. Note that the red laser beam is utilized in this case. Evidently, the monolayer graphene sheet is observed to overlay across individual nanowires on top of the pristine hybrid nanowire array and across the densified nanostructures on laser pruned array. A top-down, two probe technique is implemented for electrical transport measurements of the 3D structure. Figure 5c shows the typical I-V characteristics of the 3D devices based on pristine nanowire array (D1) and laser pruned nanowire array (D2). Clearly, the pristine nanowire arrays are poor conductors. We measure only a small current of \(1.4 \times 10^{-10} \text{ A}\) through D1 at an applied bias of 4 V. However, D2 shows a significant increase in conductivity. We measure a current of \(2.3 \times 10^{-9} \text{ A}\) at an applied voltage of 4 V. Such an improvement can be attributed to the obvious increase of the contact area (see from the SEM images Figure 5bii and iv) between the graphene electrode and the nanowire arrays. As shown by the SEM images, the expanded tip of the laser pruned nanowires gives rise to an increase in surface area at the top of the nanowire array and this helps to ensure better contact with the graphene electrode. Whilst, the focused laser induced compositional modification to the nanowire tops might possibly alter the electrical transport of the device. Besides a clear difference in conductivity, D1 and D2 also exhibited marked difference in photo-detection performance. Figure 5d shows the photoresponse characteristic of both photodetectors under applied bias at 4 V with broad beam laser illumination (4 mW/cm\(^2\), \(\lambda = 405 \text{ nm}\)). Both devices exhibit increased output current under illumination, indicating their obvious photoresponse to 405 nm laser light. However, D2 shows a higher photoresponsivity which can be calculated by\(^{34}\)

\[
R_{\text{res}} = \frac{I_{\text{ph}}}{P_{\text{opt}}} = \frac{I_{\text{illus}} - I_{\text{dark}}}{P_{\text{inc}}} S^{-1}
\]

where \(P_{\text{opt}}\) is light power, \(I_{\text{illus}}\) and \(I_{\text{dark}}\) are output current under illumination and in dark, respectively, \(S\) is effective area of nanowire device, and \(P_{\text{inc}}\) is power density of the incident light. It is very attractive that the responsivity of D2 can reach as high as 144.3 mA/W under 4 mW/cm\(^2\) illumination, whilst the responsivity of D1 is measured to be only 50 mA/W under same illumination condition. The higher responsivity supports the potential applications of D2 in optoelectronic devices such as retro-sensors, optoisolators and photoamplifiers\(^{35,36}\). Nevertheless, despite the lower responsivity, D1 also possesses its unique advantage. Namely, the photocurrent of D1 increases 146 times when compared to that measured at dark condition. This represents a high on/off ratio \((I_{\text{ph}} - I_{\text{dark}})/I_{\text{dark}}\) in the range of 102 under 4 mW/cm\(^2\) illumination. As comparison, the on/off ratio of D2 is measured to be less than
Meanwhile, D1 exhibits a rapid on/off switching behavior. Each photoresponse cycle consists of three distinct stages, a sharp rise, a steady state, and a sharp decay process to original state. The fast photocurrent rise and decay time is estimated from the photocurrent versus time plots and shown in Figure 5e. The rising response is determined to be as short as 28.5 ms which is even faster than the recovery time of 35 ms. However, D2 shows more complex response processes to the photo illumination. Besides a rapid photoresponse process, both the rising and recovery processes comprised of a slowly varying components. The slow processes of rising and decaying time (Figure 5f) can be well fitted by the following exponential functions, respectively.

Figure 4 | (a) SEM image of the green laser pruned nanowires. Scale bar is 2 μm. Insert shows the high magnification image. Scale bar is 100 μm. (b) PL spectra for pristine (black curve) and laser pruned (green curve) regions. (c) Bright field optical and (d) FM images of created micro-squares. (e) and (f) Encryption application demonstration. (ei) Optical images of a square created on the sample, FM images of the square excited by (eii) UV light and (eiii) blue light. (fi) Optical images of a “dragon” created on the sample, FM images of the “dragon” excited by (fii) UV light and (fiii) blue light.
$$I(t) = I_0 + Ae^{-\frac{(t-t_0)}{\tau}}$$

(2)

$$I(t) = I_0 + A\frac{e^{\left(-\frac{t-t_0}{\tau}\right)}}{\tau}$$

(3)

where $t_0$ and $t$ are the initial and final response time, $I_0$ is dark current, $A$ is current amplitude, and $\tau$ is the characteristic time constant, related to the slow photoresponse processes observed. The slow rising and recovering response times of D2 obtained from curve fitting are $\sim 2.6$ s and $\sim 3.3$ s, respectively. We attribute such a slow photoresponse time to a heightened thermal effect of the induced defects by laser pruning. Generally, D2 exhibits higher photoresponsivity while D1 exhibits faster response time.

In summary, we have presented a high throughput and inherently efficient technique to directly modify type II core/shell structures with multinary alloyed constituents, ZnO/CdSSe nanowire arrays. Well-defined microstructures are achievable via a simple focused laser beam. In addition, the fluorescence emission of the created micropatterns can be distinctly improved and precisely controlled with this effective technique. As a result, micro-steganography at a selected location can be facilitated on the nanowire array surface. Moreover, the laser modified nanowire arrays exhibit more superior photoconductivity. This facilitates a high performance 3D photodetector constituted by the laser modified ZnO/CdSSe nanowire arrays as the main building block and monolayer graphene as the transparent top electrode.

**Methods**

**ZnO nanowire arrays synthesis.** ZnO nanowire arrays were synthesized via a chemical vapor deposition approach (CVD) in a sealed horizontal tube furnace. A 0.3 g powder mixture of ZnO and graphite in a molar ratio of 1:1 was placed in the bottom of a one-end-closed quartz tube. A piece of silicon wafer predeposited with a 200 nm ZnO seed layer by RF sputtering served as substrate and was inserted nearer to the open end. The quartz tube was placed into a large alumina work tube. The system was initially evacuated to a base pressure of $2.0 \times 10^{-2}$ mbar before Ar gas mixed with 0.1% O$_2$ by volume of a total flow rate of 80 sccm was introduced. The pressure in the alumina work tube was raised to 2 mbar. The furnace was heated to 900 $^\circ$C and held at that temperature for 50 min. The growth temperature at the Si substrate was approximately 800 $^\circ$C.

**ZnO/CdSSe core/shell nanowire arrays synthesis.** To deposit the CdSSe outer-shells, the mixture of CdS and CdSe powder were placed into the reactor as the
precursor. A piece of freshly prepared ZnO nanowire arrays was vertically inserted as the substrate. After the furnace was first evacuated to the base pressure, high-purity He gas was flown in the rate of 50 sccm. The system was ramped to 600 °C and held for 10–30 min depending on the thickness of CdSSe shell required. The local temperature at the substrate during the growth process was approximately 500 °C. To avoid the temperature influence for the chemical stoichiometry, a specially designed substrate holder was vertically fixed to the substrate to obtain CdSSe shell with high-uniformity stoichiometry. The composition of the shells was controlled by adjusting the molar ratio of Cd and S and CdSe powder. After that, the Si wafers covered with uniform colors (from yellow to dark-brown corresponding to the stoichiometry from CdS to CdSe) were obtained for further analysis.

**Optical property measurement.** The micro-PL spectra were carried out by the Renishaw inVia system with the excitation of a He-Cd laser centered at 325 nm. The time-resolved PL spectra were measured using a time-correlated single-photon counting system (PicoHarp300, PicoQuant). The samples were excited by a pulsed laser diode operating at 405 nm. The PL signal was dispersed by a monochromator (Acton, SpectroPro 2300i) and detected with a liquid-nitrogen-cooled charge-coupled device detector.

**Transparent graphene electrode assembly.** Monolayer graphene was synthesized on both sides of copper foils by the CVD method. A layer of PMMA resist was spin-coated on one side of the copper foils. On the opposite side, the unprotected graphene was etched away with O₂ plasma by a RIE system. The exposed copper layer was etched using a solution of ammonium persulphate (7 g L⁻¹) and rinsed in deionized water. Finally, the resulting PMMA and graphene was transferred onto the desired hybrid nanowire arrays and acetone was utilized to remove the PMMA layer leaving behind a layer of graphene.

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