Ultraviolet photodetector based on vertical $\beta$-Ga$_2$O$_3$ nanowire array on GaN substrate

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Abstract

Driven by the requirement to ultraviolet detection, $\beta$-Ga$_2$O$_3$ UV photodetectors have attracted great attention. Using a metal organic chemical vapor deposition (MOCVD) reactor, we grew $\beta$-Ga$_2$O$_3$ nanowires array on a GaN substrate using Ga as catalyst. The density of the nanowires was optimized employing the substrate patterning technology. A UV detector based on the graphene/$\beta$-Ga$_2$O$_3$-nanowire-array was realized by micro-fabrication techniques. The device has a wide range of UV response covering UVC-UVA band and the peak response reaches 30.82 mA W$^{-1}$ at 258 nm corresponding to the band gap of $\beta$-Ga$_2$O$_3$. The rapid response speed ($<1$ s) is comparable to that of most reported Ga$_2$O$_3$ nanowire ultraviolet photodetectors.

1. Introduction

In recent years, Ga$_2$O$_3$, an oxide semiconductor with an ultrawide bandgap, has attracted much attention due to its excellent material properties. Among its five polymorphs ($\alpha$-, $\beta$-, $\gamma$-, $\delta$- and $\epsilon$-Ga$_2$O$_3$), monoclinic $\beta$-Ga$_2$O$_3$ is the most stable phase [1]. Its long wavelength limit of intrinsic absorption corresponding to its wide energy gap of $\sim$4.9 eV is about 254 nm in the solar-blind ultraviolet band. This benefit extends the applications of $\beta$-Ga$_2$O$_3$ to many fields, such as missile tracking and fire alarm prediction [2, 3].

There are three kinds of UV detectors based on $\beta$-Ga$_2$O$_3$, that is, the detectors made of $\beta$-Ga$_2$O$_3$ thin films, nanopores and nanowires, respectively [4–6]. As with other semiconductor materials, epitaxy methods of $\beta$-Ga$_2$O$_3$ thin film include molecular beam epitaxy (MBE), atomic layer deposition (ALD), metal organic chemical vapor deposition (MOCVD) and so on [7–10]. Nonetheless, the quality of the film is still a great challenge due to the existence of miscible phase and ‘six-fold domain’ torsion, etc. The nanopore structure is mainly obtained by reactive ion etching Ga$_2$O$_3$ thin film to form a nanopore array [11], and the top-down etching and oxidation of other nanowire materials are widely used to prepare Ga$_2$O$_3$ nanowire array [12]. A self-powered UV detector adopting a vertical $\alpha$/$\beta$-Ga$_2$O$_3$ junction nanorod array and a graphene-silver nanowire top electrode has been reported, and a responsivity of 0.26 mA W$^{-1}$ at 0 V was achieved at 254 nm. However, the nanowire crystal prepared by hydrothermal method is vulnerable to pollution and has high impurity content, which goes against the device performance [13]. He et al reported that a uniform Ga$_2$O$_3$ nanowire array can be achieved by oxidizing GaN nanowires. Because the oxidation process may damage the nanowire lattice and make a single nanowire be polycrystalline, the performance of the detector based on the array is limited [14]. In contrast, the Ga metal self-catalytic vapor-liquid-solid (VLS) method has special advantages in the radial heteroepitaxy of nanowires. The surface of the nanowires obtained by this method is smooth, and the crystal quality is good. At the same time, this process can avoid the problem of impurity residue in the hetero metal epitaxy of Ga$_2$O$_3$ [15]. Here, based on VLS growth principle, we grew $\beta$-Ga$_2$O$_3$ nanowire arrays on GaN substrates by Ga-catalyzed MOCVD. The self-catalyzed procedure can prevent unintentional impurity doping. The density of the nanowires was optimized by controlling the growth conditions and employing the substrate...
Patterning technology. We fabricated and tested an ultraviolet detector based on the $\beta$-Ga$_2$O$_3$ nanowire array with graphene as a transparent electrode. Results demonstrate that the device has a wide spectral response and a peak responsivity of 30.82 mA W$^{-1}$ at 258 nm.

2. Material growth and device fabrication

2.1. Growth of nanowire array by self-catalysis

In a MOCVD reactor (TNSC SR4000) and based on the VLS mechanism, a $\beta$-Ga$_2$O$_3$ nanowire array was heteroepitaxially grown by Ga metal self-catalysis on a (0001) GaN substrate. In this process, triethylgallium (TEGa) was used as the deposition source of Ga droplet catalyst and the gallium source for growth of $\beta$-Ga$_2$O$_3$ nanowire, and high-purity N$_2$O gas was used as the oxygen source. High-purity N$_2$ was used as the carrying gas for TEGa. The pressure in the reaction chamber was controlled at 20 kPa during whole procedure.

Firstly, the GaN substrate was cleaned with N$_2$O at 720 °C to prevent the introduction of impurities. Meanwhile, an oxide mask was formed on the surface of the substrate, which can inhibit the lodging growth of the nanowires.

Secondly, Ga catalyst was pre-deposited. The deposition temperature of the Ga droplets was set at 450 °C, and the deposition time was 16 min. After pre-deposition, the in situ annealing procedure was implemented at 650 °C and lasted for 1.5 min. The pre-deposition time and the annealing temperatures determined the diameters and densities of the nanowires.

Thirdly, TEGa and N$_2$O gases were introduced at the same time and Ga$_2$O$_3$ nanowires began to grow at 485 °C. The growth process of the nanowires can be described as follows: O atoms provided by N$_2$O are adsorbed on the surface of the Ga droplets, these absorbed O atoms then diffuse to the VLS three-phase junction along the surface of the droplets and form the core of the Ga$_2$O$_3$ nanowires, and subsequently the nanowires grow from bottom to top for 5 min.

The top and the cross-section morphologies of the Ga$_2$O$_3$ nanowire array were characterized by scanning electron microscope (SEM), as shown in figures 1(a) and (b). It can be seen that the nanowires are in the shape of a tree trunk, and there is a spherical Ga droplet atop each of them. Their heights are almost uniform, and their diameters vary from 100 to 200 nm. As we can see, with pretreating the substrate and the appropriate growth temperature, the verticality of the nanowires has been achieved greater than 90%. To illustrate the crystal quality of the nanowires, the TEM image of the vertical cross-section slice of the vertical nanowires and the substrate is provided in figure 1(c). The Ga droplets were squeezed during focused ion beam (FIB) sampling, therefore they became mushroom shaped. A high-resolution transmission electron microscopy (HR-TEM) image of a single nanowire was taken and demonstrated in figure 1(d). The single nanowire has a perfect single crystal structure, and the lattice is neat, which imply almost perfect crystal quality. The longitudinal lattice constant is 0.47 nm consistent with that of $\beta$-Ga$_2$O$_3$ (201) and the reported epitaxial relationship when $\beta$-Ga$_2$O$_3$ nanowires were grown on GaN [16].

Fourthly, the Ga droplets on the nanowires were etched with a 20:1 HF and NH$_4$F mixture (BOE). The SEM profile of the etched nanowires is shown in figure 1(e), and their heights are estimated as about 350 nm. The powder x-ray diffraction (XRD) image is illustrated in figure 1(f). There is the diffraction peak of $\beta$-Ga$_2$O$_3$ (201) at 18.95°. In the inset, the peak of GaN is very obvious, and that of $\beta$-Ga$_2$O$_3$ (201) is also visible. This indicates that the nanowire array grown under above process conditions has the preferred orientation of $\beta$ (201) [17]. The normally oriented $\beta$-Ga$_2$O$_3$ (201) nanowires are just the vertical wires in the SEM images, which has been confirmed by the XRD results.

2.2. Improvement of nanowire density by substrate patterning

The density of the nanowires obtained in the above subsection is not high enough to support the graphene electrode to be applied. In the experiment, we found that growing nanowires on a patterned substrate can improve the array density. Here we will show the density enhancement effect and analyze the reasons. As shown in figure 2(a), the GaN substrate was covered with SiO$_2$ in some areas. Regions A and B are active regions, i.e., the bare GaN substrate, and region C is covered by SiO$_2$. Region A is near the boundary of the active region, and region B is away from it. The density and the growth mode of the nanowires grown on different materials or in different regions are different. Figures 2(b)–(d) demonstrate that the density of the nanowires in region A is higher than that in region B. The reason for that is the influence of step height difference, i.e. Ga on the SiO$_2$ dielectric layer at the boundary of the active region will fall into the active region during annealing within a square of a side length of about 30 μm. Then higher-density nanowires will be achieved in this square region due to more Ga droplets there. In figure 2(d), it can also be observed that some nanowires were also deposited in region C on the mask, but they are in the shape of ‘octopus’, which is completely different from the morphology of those grown directly on the substrate. The possible reason for this is that the SiO$_2$ mask is amorphous and is
Figure 1. Images and XRD spectrum of the β-Ga2O3 nanowire array: (a) and (b) Top and cross-section SEM images of the nanowire array with gallium droplets; (c) TEM image; (d) Local high resolution TEM image of a single nanowire; (e) Cross-section SEM image of the nanowire array without gallium droplets; (f) XRD spectrum of β-Ga2O3 (2θ); the inset shows the XRD pattern of GaN substrate with β-Ga2O3 nanowire array.

Figure 2. SEM images of β-Ga2O3 nanowires in different regions.
sparser than the GaN substrate. Therefore, by building a silicon oxide grid, the active region is gridded and the density of the nanowires in this region is increased.

2.3. Fabrication of UV detector

The fabrication process of the detector is shown in figure 3. Before the growth of the nanowires, the substrate was patterned. As shown in figures 3(a)–(c), first, a 350 nm-thick SiN mask was grown on the substrate by low pressure chemical vapor deposition (LPCVD). The SiN mask grown by this method is compact, and it is not easy to be corroded by the acid solution to be used later and will be the support of the electrode. Then, a 50 nm-thick SiO2 mask was deposited on SiN mask as the sacrificial layer. Next, after spin coating photoresist, photolithography was carried out by contact lithography equipment to reveal the active region. The opening window was designed as an array of squares with side length of 40 \( \mu \)m. The SiN and the SiO2 masks were etched by reactive ion etching and complete the patterning of the substrate for nanowire growth.

Then, the GaN substrate with mask layers was placed into the MOCVD reaction chamber for nanowire array growth, as shown in figure 3(d). After growth, the sample was put into BOE to remove the SiO2 sacrificial layer and the Ga catalyst on the top of the \( \beta \)-Ga2O3 nanowires (figure 3(e)). The next step is to fabricate the upper and lower electrodes of the device as shown in figures 3(f)–(h). Ti/Al/Ni/Au (20/130/50/150 nm) were grown on the GaN substrate surface and annealed to form ohmic contact. Graphene was transferred onto the top of the \( \beta \)-Ga2O3 nanowires as a transparent electrode. Finally, Ti/Au (20/150 nm) was evaporated on the graphene for lead electrode.

3. Measurement and results

As the graphene is stucked onto the top of the \( \beta \)-Ga2O3 nanowire array, a graphene/\( \beta \)-Ga2O3/GaN heterojunction is formed. The energy band structure of the device is shown in figure 4. Graphene is a weak p-type material and the unintentionally doped \( \beta \)-Ga2O3 nanowires are generally n-type [18]. Graphene and \( \beta \)-Ga2O3 combine into a p-n junction, and a depletion layer appears at their interface. The electron affinities of Ga2O3 and GaN are about 4.1 eV and 4.0 eV, respectively. The conduction band difference between them is about 0.1 eV. Their band gaps are 4.9 eV and 3.4 eV, respectively, which leads to a valence band difference of about 1.5 eV. This means that at the interface of \( \beta \)-Ga2O3/GaN, electrons can move almost freely in the conduction band, but it is difficult for holes due to the huge valence band difference between Ga2O3 and GaN. Under ultraviolet illumination, the light near 254 nm will be absorbed by \( \beta \)-Ga2O3, that near 360 nm will be absorbed by GaN material, and photogenerated carrier pairs will be produced along with these two absorption processes. Under a reverse bias, the built-in electric field in the depletion regions of the graphene/\( \beta \)-Ga2O3 and

![Figure 3. Preparation process of the \( \beta \)-Ga2O3 nanowire UV detector based on a double-mask layout. (a) GaN substrate; (b) SiN and SiO2 mask grown on GaN substrate; (c) Selectively etching of SiN and SiO2 masks; (d) \( \beta \)-Ga2O3 nanowire growth; (e) Removal of the SiO2 sacrificial layer and the Ga catalyst; (f) Fabrication of the lower electrodes; (g) Graphene was transferred on the \( \beta \)-Ga2O3 nanowires; (h) Fabrication of the upper electrodes.](image-url)
the β-Ga2O3/GaN heterojunctions can enhance the separation of the photogenerated electron-hole pairs, which will be collected by the electrodes as the photocurrent.

The rectifying ratio (1 V/−1 V) of the device exceeds 400 in dark and under room temperature as shown in figure 5(a). The rectification characteristic in the current-voltage (I–V) characteristics is derived from graphene/β-Ga2O3 heterojunction. For heterojunction barrier $\varphi_B$ and ideal factor $n$, we can get the function relation between $\ln(I)$ and $qV/kT$ by the following formula:

$$I = I_s \left( \frac{qV}{nkT} \right) \exp \left( \frac{q\varphi_B}{kT} \right)$$

where $I_s$ is the saturation current,

$$I_s = A^*T^2 \exp \left( -\frac{q\varphi_B}{kT} \right)$$

$q$ is the elementary charge, $T$ is temperature in Kelvin, $k$ is the Boltzmann constant, $n$ is the ideality factor, $V_D$ is the diode net voltage, $A$ is the effective diode area, $A^*$ is the Richardson constant ($A^* = 41$ A cm$^{-2}$ K$^{-2}$ at room temperature), and $\varphi_B$ is the effective barrier height without bias. The above equation can be rearranged to extract $\varphi_B$ and $n$ under forward bias. As shown in the inset of figure 5(a), $\varphi_B$ and $n$ are estimated as 0.67 eV and 1.69, respectively. These values are close to reported results in the literature for graphene/β-Ga2O3 heterojunction [19]. In addition, we compared the I–V characteristics of the device with and without illumination, as shown in figure 5(b). Under a −5-V bias, the dark current of the detector is 105 nA, and under 254 nm illumination, the photocurrent is 249 nA. The effective area of the device is 0.16 mm$^2$, and the intensity of the 254 nm illumination is $3.10 \times 10^{-5}$ W mm$^{-2}$. Therefore, it is proved that our device does respond in the solar-blind UV band.

In order to quantitatively evaluate the response characteristics of the detector to ultraviolet light, the response spectrum of the fabricated photodetector was measured by a self-built system [20] when the bias voltage was −5 V, as shown in figure 6(a). The starting wavelength was 200 nm, and the responsivity of the UV

![Figure 4. Energy band diagram of the device under illumination.](image)

![Figure 5. (a) I–V characteristic of the device measured in dark at room temperature. The inset shows the $\ln(I)$ versus $qV/kT$ curve for estimating $\varphi_B$ and $n$ at the graphene/Ga2O3 heterojunction; (b) The dark current and the photocurrent under illumination at 254 nm.](image)
The detector began to increase rapidly from 230 nm and reaches 30.82 mA W$^{-1}$ at 258 nm, which corresponds to the 4.9 eV bandgap of Ga$_2$O$_3$. Besides, because the substrate is GaN with a bandgap about 3.4 eV and a response peak near 360 nm, the response bandwidth of our device has been expanded. The responsivity exceeds 15 mA W$^{-1}$ in the 248–368 nm band spanning from UVC to UVA. The 370 nm cutoff wavelength corresponds to the bandgap of GaN. The responsivity of the device increases as the absolute value of the reverse bias voltage increases, as shown in Figure 6 (b). Because the nanowire array is still somewhat sparse, the overall responsivity is not very high. However, compared to thin film detectors, this responsivity is slightly higher at the same bias voltage and 258 nm \cite{11}. The main reason for this is that the crystal quality of our $\beta$-Ga$_2$O$_3$ nanowires is good, and the dark current is small. In addition, the nanowire array has a much larger surface/volume ratio and a larger absorption area than the film devices, and for some non-perpendicular incident light, the nanowire structure can make the light reflected several times in the cavities formed by the graphene and the nanowires and absorbed efficiently. Graphene is transparent and has larger transmission to ultraviolet light compared with ordinary electrodes. This benefits the detection ability of the detector. Table 1 lists several important device metrics of some nano-structured or hetero-structured Ga$_2$O$_3$ photodetectors. It can be seen that the responsivity ($R$), detectivity ($D^*$) and external quantum efficiency (EQE) of our device are close or better than $\beta$-Ga$_2$O$_3$ nanowires \cite{21}, indium zinc oxide (IZO)/$\beta$-Ga$_2$O$_3$/IZO M-S-M structure \cite{22} and Ga$_2$O$_3$/SnO$_2$:Ga core–shell detectors \cite{23}. The performance of our device could be improved if the density of the nanowires increases by such as secondary lateral epitaxy method.

At last, the transient response characteristics of our detector were measured by a frequency generator and an oscilloscope \cite{24}. Figures 7(a) and (b) display the transient response of the device to the 254 nm illumination at a –5-V bias, from which the rising time $\tau_{\text{on}}$ (the time for the current increasing from 10% to 90% of the peak value) of 0.38 s and the decay time $\tau_{\text{off}}$ (the time for the current dropping from 90% to 10% of the peak value) of 0.31 s were extracted. Even the sum of $\tau_{\text{on}}$ and $\tau_{\text{off}}$ is less than 1 s. Generally, in photoconductive detector, part of the photocurrent comes from the change of photo generated carrier concentration. Its response speed is fast, and the response time is short. The other part is caused by carrier capture and release by the defects in the material, and its speed is slow. For our device, because the crystal quality of the nanowires is good and there are few defects, the photo generated carriers can reach the positive and negative electrodes quickly, it can response quickly.

### 4. Conclusions

In this paper, the Ga$_2$O$_3$ nanowire array were fabricated on GaN substrate by VLS method using MOCVD equipment. The results of SEM, HRTEM and XRD show that the nanowires grown by this method are in $\beta$-
phase, and most of them are oriented in (201) direction with regular lattice arrangement and good crystal quality. The verticality of the nanowires can reach about 90% by substrate pretreatment and suitable growth temperature. At the same time, the patterned design of two masks with a net structure not only increases the growth density of the nanowires, but also provides a firm support framework for graphene transfer on the nanowires. High quality and relatively dense nanowires, as well as the net-structure of double-layer framework are critical foundation for the successful fabrication of the detector. Based on the $\beta$-Ga$_2$O$_3$ nanowire array and the GaN substrate as the active material and using graphene as a transparent electrode, an ultraviolet detector was fabricated. The device exhibits a broadband spectral response (248 nm – 368 nm) with a responsivity exceeding 15 mA W$^{-1}$ and a peak responsivity of 30.82 mA W$^{-1}$ at 258 nm at a −5-V bias. The response time of the device is less than 1 s. Compared to other Ga$_2$O$_3$ detectors, the response speed of our detector is superior. These results indicate that the growth method of vertical $\beta$-Ga$_2$O$_3$ nanowire array and the fabrication technique of UV detectors proposed and realized in this paper may inspire the development of broadband UV detectors in solar-blind band.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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