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UV-assisted room temperature-gas sensing of Ga₂O₃-core/ZnO-shell nanowires

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Ga₂O₃-based gas sensors show very poor performance at room temperature despite being able to detect a range of gases efficiently at high temperatures of 600-1,000 °C, which limits their practical use. Ga₂O₃-core/ZnO-shell nanowires were synthesized by the thermal evaporation of GaN powders followed by the atomic layer deposition of ZnO and then multiple networked Ga₂O₃-core/ZnO-shell nanowire gas sensors were fabricated. The morphology, crystal structure, and sensing properties of pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires to NO₂ gas at room temperature under ultraviolet (UV) illumination were examined. Pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires showed responses ranging from ~249 to ~703% and from ~557 to ~2,110%, respectively, to 1-5 ppm NO₂ under UV (365 nm, 1.2 mW/cm²) illumination, corresponding to 2.2-3.0 fold increases, by encapsulation of ZnO nanowires with ZnO. On the other hand, the responses of both types of nanowires were almost 0 at room temperature in the NO₂ concentration range of 1-5 ppm. The underlying mechanism for the enhanced gas sensing properties of Ga₂O₃-core/ZnO-shell nanowires toward NO₂ gas under UV illumination is discussed in detail.

Key words: Ga₂O₃, Core-shell nanowire, Gas sensor, NO₂, UV.

Introduction

Monoclinic gallium oxide (β -Ga₂O₃) is an important wide band gap semiconductor material with potential applications in high temperature gas sensors [1-4] as well as transparent conducting electrodes [5], phosphors [6] and dielectric gates [7]. Recent studies on Ga₂O₃ thin film-based gas sensors reported that they detected O₂, H₂, CO, and CH₄ gases efficiently at high temperatures of 600-1000 °C [8, 9]. On the other hand, they showed very poor performance at room temperature, which limits the practical use of Ga₂O₃-based gas sensors.

ZnO is a functional material that is sensitive to toxic and combustible gases [10]. ZnO gas sensors with various structures, such as powders [11], thin films [12], heterostructures [13], nanoparticles [14], and onedimensional nanostructure [15] have all been demonstrated. Among these nanostructures, one-dimensional (1D) ZnO nanostructures are particularly useful due to their large length-to-diameter ratio and large surface-to-volume ratio. Studies of the gas sensing properties of ZnO 1D nanostructures focused mainly on CO, H2S, HCHO, NH₃, H₂, ethanol and humidity [16]. Despite the enhanced gas sensing properties of 1D nanostructures compared to thin films, enhancing their sensing performance and detection limit is still a challenge. A range of techniques such as doping of noble metals [17-19], surface functionalization with noble metals [20-22], core-shell structure formation [23-26] and UV illumination [27-32] have been developed to enhance the sensitivity and stability of the 1D nanostructure-based sensors.

We reported ultrasensitive multiple networked Ga_2O_3 -core/ZnO-shell nanorod gas sensors previously [33]. However, the sensing properties of the gas sensors were measured at 300 °C in the earlier study. There is no doubt that a sensor with high sensitivity at room temperature is desirable. This study examined the nitrous oxide (NO₂) gas sensing properties of the multiple networked Ga_2O_3 -core/ZnO-shell nanowire sensors at room temperature under UV illumination. In particular, the effects of the UV illumination intensity on the response of Ga_2O_3 -core/ZnO-shell nanowire sensors to NO₂ gas were examined.

Experimental

Synthesis of Ga₂O₃-core/ZnO-shell nanowires

Ga₂O₃-core/ZnO-shell nanowires were synthesized by thermal evaporation of GaN powders followed by atomic layer deposition (ALD) of ZnO. First, Aucoated Sapphire wafer was used as a substrate for the synthesis of 1D Ga₂O₃ structures. Au was deposited on a c-plane sapphire substrate by direct current (dc) sputtering. A quartz tube was mounted horizontally inside a tube furnace. 99.99% pure GaN powders were placed on the lower holder at the center of the quartz tube. An Au-coated Sapphire substrate was placed on the upper holder, approximately 5 mm apart from the GaN powders. The furnace was heated to 1050 °C and maintained at that temperature for 1 h in a N₂/3 mol%-O₂ atmosphere with constant flow rates of oxygen (O₂) (15 sccm) and N₂ (485 sccm). The total pressure was

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set to 1.5 Torr. For the second stage, the as-synthesized Ga_2O_3 nanowires were transferred to an ALD chamber and coated with ZnO by ALD for 50 cycles. Diethylzinc (DEZn) and H₂O were kept in bubblers at 0 and 10 °C, respectively. The source gases were fed alternatively into the chamber through separate inlet lines and nozzles. The typical ALD pulse lengths were 0.15 s for DEZn (0 °C), 0.2 s for H₂O (10 °C) and 3 s for purging the reactants. The substrate temperature and pressure in the chamber were 150 °C and 0.1 Torr, respectively.

Characterization

The collected pristine Ga₂O₃ and Ga₂O₃-core/ZnOshell nanowire samples were characterized by scanning electron microscopy (SEM, Hitachi S-4200), transmission electron microscopy (TEM, Philips CM-200), and X-ray diffraction (XRD, Philips X'pert MRD diffractometer). The crystallographic structure was determined by glancing angle XRD using Cu K_α radiation (0.1541 nm) at a scan rate of 4 °/min. The sample was arranged geometrically at a 0.5 ° glancing angle with a rotating detector.

Measurement of gas sensing properties

Multiple networked Ga₂O₃-core/ZnO-shell nanowire gas sensors were fabricated by pouring a few drops of nanowire-suspended 2-propanol onto SiO₂ (300 nm)/Si substrates equipped with a pair of interdigital electrodes (IDEs) with a gap of 20 µm. The sensor was tested for its response to NO2 using a home-built computer-controlled characterization system. The chamber was connected to the gas inlet line coming from the mass flow controllers. Two mass flow controllers were used in combination with a controller to control the NO₂ gas flow rate. A gas exhaust tube was located at the opposite side of the chamber. The flow-through technique was used to test the gas sensing properties of the Ga₂O₃-core/ZnO-shell nanowires. All the measurements were executed in a temperature-stabilized sealed chamber at room temperature under controlled humidity. The test gas was mixed with synthetic air to achieve the desired concentration, and the flow rate was maintained at 200 cm³/min using mass flow controllers. National Instruments LabviewTM program was used to control the mass flow controllers, sourcemeter and record the NO₂ concentration. A Keithley sourcemeter-2612 was used to acquire the resistance data. The sourcemeter was hooked to a computer via a universal serial bus (USB) connector and this data was also acquired using the LabViewTM software. The electrical resistance of gas sensors was determined under UV $(\lambda = 365 \text{ nm})$ illumination at 1.2 mW/cm² by measuring the electric current that flowed at room temperature when a potential difference of 1 V was applied between the Ti (~ 10 nm)/Au (~ 50 nm) IDEs. The response R is defined as $R(\%) = R_g/R_a \times 100$, where R_g is the resistance of the sensors in NO_2 gas, and R_a is the resistance of the sensors in air ambience, respectively.

Results and Discussion

Fig. 1(a) shows a SEM image of the Ga₂O₃-core/ZnOshell nanowires prepared by thermal evaporation. It shows that the synthesis scheme adopted in this study can grow Ga2O3-core/ZnO-shell nanowires with diameters of 20-60 nm and lengths up to a few tens of micrometers. The nanowires had a fiber-like morphology with a relatively uniform diameter. The XRD patterns of Ga₂O₃core/ZnO-shell nanowires and pristine Ga₂O₃ nanowires are presented in Fig. 1(b). The main diffraction peaks in the pattern of the core-shell nanowires were indexed to the reflections from the lattice planes of monoclinicstructured crystalline Ga₂O₃, indicating that the nanomaterial is Ga₂O₃. In addition to the tall reflection peaks, several small broad peaks of wurtzite-structured crystalline ZnO were observed. The low-magnification TEM image (Fig. 1(c)) shows a typical Ga₂O₃-core/ZnOshell nanowire with a mean ZnO shell layer thickness of ~ 10 nm. A fringe pattern was observed in the HRTEM image of a Ga₂O₃-core/ZnO-shell nanowire (Fig. 1(d)). The resolved spacings between two neighboring parallel fringes were approximately 0.28 and 0.26 nm which were in good agreement with the interplanar spacings of the $\{002\}$ and $\{111\}$ planes of Ga_2O_3 of monoclinic-structured bulk Ga₂O₃ crystals (JCPDS No. 87-1901, $\alpha = 1.221$ nm, b = 0.3037, c = 0.5798 nm, $\beta = 103.83^{\circ}$ and the {101} plane of wurtzite-structured bulk ZnO crystals (JCPDS No. 89-1397, $\alpha = 0.3253$ nm, c = 0.5213 nm), respectively. The corresponding selected area electron diffraction (SAED) pattern recorded perpendicular to the long axis, was indexed for the [110] zone axis of Ga₂O₃. The reflection spots in the corresponding selected area electron diffraction (SAED) pattern (Fig. 1(e)) were identified as the (111) and (002) reflections of monoclinic-structured Ga₂O₃. On the other hand, a close examination of the SAED pattern



Fig. 1. (a) SEM image and (b) XRD patterns of Ga2O3-core/ZnOshell nanowires. (c) Low-magnification TEM image, (d) highresolution TEM image, and (e) selected area electron diffraction pattern of Ga2O3-core/ZnO-shell nanowires.

revealed that the pattern contained dim concentric rings assigned to the (002) and (101) reflections of wurtzitestructured ZnO as well as the strong spots for Ga_2O_3 , suggesting that the ZnO shell layer was polycrystalline.

Fig. 2(a) shows the dynamic responses of pristine Ga2O3 nanowires and Ga2O3-core/ZnO-shell nanowires to NO₂ gas at room temperature under UV illumination. On the other hand, the dynamic responses of pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires to NO₂ gas at room temperature are not shown in Fig. 2(a) because both types of nanowires did not nearly respond to 1-5 ppm NO₂ gas at room temperature. In other words, the responses of both types of nanowires were almost 0 at room temperature in the NO₂ concentration range of 1-5 ppm. The resistance increased to a maximum resistance instantaneously upon exposure to NO2 and recovered completely to the initial value after the removal of NO2. The increase in resistance was enhanced with increasing NO₂ concentration. The resistance showed a good reversibility during the cycles of introduction and exhaust of NO2. The difference in response between pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires to NO₂ in the dark was negligible, whereas the difference in response between pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires to NO₂ gas under UV illumination was significant and the response difference was larger for higher UV illumination intensity. Pristine Ga2O3 nanowires and Ga2O3-core/ZnO-shell nanowires showed responses ranging from ~ 249 to $\sim 703\%$ and from ~ 557 to ~ 2,110% , respectively, to 1-5 ppm NO_2 under UV (365 nm) illumination.

Fig. 2(e) shows the responses of pristine Ga_2O_3 nanowires and Ga₂O₃-core/ZnO-shell nanowires to NO₂ gas at room temperature under UV illumination as a function of the NO₂ concentration. The response of a semiconductor is commonly expressed as $R = A [C]^n + B$, where A and B are constants, n is an exponent and [C] is target gas concentration [34]. Data fitting provided the following equations: R = 111.7 [C] + 163.2 and 376.4 [C] + 45.6 for pristine Ga₂O₃ nanowires and Ga₂O₃-core/ ZnO-shell nanowires, respectively, under UV (365 nm) illumination. The response of Ga2O3-core/ZnO-shell nanowires tended to increase more rapidly than that of pristine Ga₂O₃ nanowires as the NO₂ gas concentration was increased, suggesting that the response of the former would be far higher than that of the latter at high NO₂ gas concentrations such as a few hundreds ppm of NO_2 , even though the response of NO_2 nanowires were examined only in a NO2 concentration range of 1-5 ppm. These plots show a synergistic effect of a combination of UV irradiation and core-shell nanostructures formation on the gas sensing properties of Ga₂O₃ nanowires more clearly.

The improvement in the response of the Ga_2O_3 nanowires to NO_2 gas by encapsulating them with ZnO can be explained by the modulation of electron transfer by the n- Ga_2O_3 -n-ZnO junction [35]. The surface



Fig. 2. (a) Dynamic responses of Ga2O3-core/ZnO-shell nanowires to 1-5 ppm NO2 gas at room temperature under UV illumination. (b) Enlarged part of the response curve of pristine Ga2O3 nanowires and (c) enlarged part of the response curve of Ga2O3-core/ZnO-shell nanowires to 5 ppm NO2 under UV (365 nm, 1.2 mW/cm2) illumination, (d) Responses of pristine Ga2O3 nanowires and Ga2O3-core/ZnO-shell nanowires at room temperature under UV illumination as a function of the NO2 gas concentration.



Fig. 3. Energy band diagram of the Ga2O3-ZnO system showing an energy barrier at the Ga2O3-ZnO junction [41].

states and/or defects on single crystalline nanowires can induce trap energy levels at the surfaces and interfaces, resulting in band bending due to Fermi level pinning [36-38]. The trapping of carrier electrons in the trap states can cause electron depletion in the surface and interface regions of the nanowires. On the other hand, the ZnO shell layer thickness (~10 nm) is smaller than the width of the surface depletion layer in ZnO, which is the order of the Debye length of ZnO, 30 nm [39, 40] (Fig. 3). Consequently, the depletion region in the vicinity of the core-shell interface (Ga₂O₃-ZnO junction) overlaps the surface depletion region of the ZnO shell. In addition to the depletion regions, an energy barrier exists at the n-Ga₂O₃-n-ZnO junction due to electron trapping at the Ga2O3-core/ZnO-shell interface as shown in Fig. 3. The energy barrier at the n-n junction should also be considered when explaining the sensing mechanism of the core-shell nanowire sensors because electron transport is modulated by the junction. The conductivity, σ , can be expressed as

follows [35]:

$$\sigma = \sigma_0 \exp\left(-\Phi_{\rm eff}/kT\right),\tag{1}$$

where σ_0 is a constant, Φ_{eff} is the effective energy barrier at the n-n junction, k is a Boltzmann constant and T is the absolute temperature. Upon exposure to NO_2 gas, Φ_{eff} will increase because NO_2 gas is adsorbed by the core-shell nanowire and electrons are attracted to the adsorbed NO₂ molecules. Consequently, the electrical resistivity increases. On the other hand, after stopping the NO₂ gas supply, the electrons trapped by the adsorbed NO₂ molecules will be released and trapped not only by the ZnO shell layer but also by the Ga_2O_3 core and Ga_2O_3 -ZnO junction. Φ_{eff} will decrease because the trapped electrons will return to the conduction band of ZnO. Consequently, the conductivity of the core-shell nanowire will increase or the resistivity will decrease. Therefore, electron transport is modulated by the n-n junction with an adjustable energy barrier height.

Fig. 3 presents the proposed mechanism for the enhanced NO₂ response of the Ga₂O₃-core/ZnO-shell nanowire sensor under UV illumination [41]. When the core-shell nanowire is exposed to air, it interacts with oxygen by transferring electrons from the conduction band to the adsorbed oxygen atoms, forming ionic species, such as O, O² and O₂, according to the following reactions [38, 42]:

$$O_2(g) \to O_2(ads), \tag{2}$$

$$O_2(ads) + e \to O_2(ads), \tag{3}$$
$$O_2^{-}(ads) + e^{-} \to 2O^{-}(ads) \tag{4}$$

$$O^{-}(ads) + e^{-} \rightarrow O^{2-}(ads).$$
 (1)

A depletion layer was created in the surface region of the core-shell nanowire due to the consumption of electrons in the surface region of the core-shell nanowire. Electron-hole pairs will be generated in the ZnO shell upon exposure to UV light with a photon energy larger than the band gap of ZnO. During migration to the surface of the ZnO shell, some of the photo-generated electrons and holes will recombine with each other and many of the remaining photo-

Table 1. Relative responses measured at different NO_2 concentrations, for Ga_2O_3 nanowires and Ga_2O_3 -core/ZnO-shell nanowire sensors.

_	Response (%)	
NO ₂ Conc.	Ga ₂ O ₃	Ga ₂ O ₃ /ZnO
1 ppm	248.68	557.14
2 ppm	402.10	781.43
3 ppm	526.88	985.71
4 ppm	610.75	1,439.43
5 ppm	702.80	2,109.96

generated holes will react with negatively charged oxygen ions adsorbed on the surface according to the following reactions [30]:

$$h^{+}(h\nu) + O^{2}(ads) \rightarrow O_{2}(g), \qquad (6)$$

$$h^+(h\nu)+ O^-(ads) \rightarrow 1/2O_2(h\nu),$$
 (7)
or

$$2h^{+}(hv) + O^{2}(ads) \rightarrow 1/2O_{2}(hv).$$
 (8)

The surface depletion layer was reduced in the ZnO shell of each core-shell nanowire due to these reactions.

Upon exposure to NO₂ gas, NO₂ gas adsorbs on the ZnO shells, the remaining photo-generated electrons are released from the shells, and are attracted to the adsorbed NO₂ molecules because an oxidizing gas, e.g. NO₂, acts as an electron acceptor, as shown in the following reactions [27]:

$$NO_2 (g) + e^{-}(hv) \rightarrow 2NO(hv) + O_2^{-}(hv).$$
 (9)

This reaction widens the surface depletion region in the ZnO shell, resulting in an increase in the resistance of the nanowire sensor. Therefore, the depletion layer width and the electrical resistance of the sensor increase with increasing NO2 concentration and UV illumination intensity due to an increase in the number of electrons participating in the above reactions. Electron transfer is modulated by the n- Ga₂O₃-n-ZnO junction under UV illumination as well as in the dark, because the surface depletion layer overlaps the interface depletion layer and $W_D(h\nu) > W_D(dark) >$ 10 nm (ZnO shell layer width) (Fig. 3), where $W_D(hi)$ and W_D(dark) represent the depletion layer width in the core-shell nanowire under UV illumination and in the dark, respectively. In other words, the Ga₂O₃-ZnO junction acts as a lever in electron transfer through which electron transfer is facilitated or restrained, resulting in enhanced sensing properties of the coreshell nanowire sensor. In particular, as shown in Fig. 3, the height of the energy barrier at the Ga₂O₃-ZnO junction (Φ_{eff} in eq. 1) is larger under UV illumination than in the dark and increases with increasing UV illumination intensity because more electrons are trapped by the Ga₂O₃-ZnO junction under stronger UV illumination. On the other hand, after stopping the NO₂ gas supply, the resistivity recovers completely to the initial value due to the release of electrons trapped by the adsorbed NO₂ molecules in the same manner as in the dark.

Overall, the substantial improvement in the response of the core-shell nanowires to NO_2 gas by UV irradiation might be attributed to the larger change in resistance caused by the increased number of electrons participating in the reactions with NO_2 molecules due to the photo-generated electron-hole pairs and the larger energy barrier height at the Ga₂O₃-ZnO junction due to a higher density of trapped carriers. Our results show that a synergistic effect on the gas sensing properties of nanostructure sensors can be obtained by a combination of two different techniques. We believe that this paper will contribute to the development of gas sensors with high performance that can be operated at room temperature

Conclusions

Pristine Ga₂O₃ nanowires and Ga₂O₃-core/ZnO-shell nanowires showed responses ranging from ~ 249 to ~ 703% and from ~ 557 to ~ 2,110%, respectively, to 1-5 ppm NO₂ under UV (365 nm) illumination. On the other hand, the responses of both types of nanowires were almost 0 at room temperature in the NO₂ concentration range of 1-5 ppm. The substantial improvement in the response of the core-shell nanowires to NO₂ gas by UV irradiation might be attributed to the larger change in resistance caused by the increased number of electrons participating in the reactions with NO₂ molecules due to the photo-generated electron-hole pairs and the larger energy barrier height at the Ga₂O₃-ZnO junction due to a higher density of trapped carriers.

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