Carbon doped tungsten oxide nanorods NO₂ sensor prepared by glancing angle RF sputtering

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Metal oxide semiconductor nanostructures offer potential advantages in sensing applications due to their large surface to volume ratio, lower electron recombination rate, and high stability. However, most methods produce nanostructures with random sizes, distribution and orientations, which are not reliable for practical applications because of poor reproducibility. In this work, homogeneous carbon-doped WO₃ nanorods are developed based on the glancing angle deposition (GLAD) technique using radio-frequency magnetron sputtering and investigated for NO₂ gas sensing application. The carbon doping is achieved by using acetylene gas as a carbon source. Characterization based on scanning electron microscopy, Auger electron spectroscopy and X-ray diffraction confirm the formation of uniform carbon-doped crystalline WO₃ nanorods. The gas-sensing results reveal that carbon-doped WO₃ nanorods sensor exhibits not only high response and selectivity to NO₂ (0.5–5 ppm) but also at low operating temperature (150 °C) compared with the undoped ones. The observed gas-sensing enhancement may be attributed to the increase of specific surface area and the decrease of activation energy by carbon doping.

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1. Introduction

Nanostructures such as nanowires, nanorods, nanotubes of metal oxide semiconductor (MOS) materials have recently attracted monumental interest in gas-sensing applications because of their excellent performances owing to their large surface to volume ratio, lower electron recombination rate and high stability. Among various MOS materials, tungsten trioxide (WO₃) is a highly promising candidate due to its fast response with high sensitivity toward NOₓ [1–4], H₂S [5–8], C₂H₂OH [8,9], CO [10], NH₃ [10–14], O₃ [15] and H₂ [16]. Moreover, addition of some foreign atoms such as Pt, Pd, or Au [17–21] to WO₃ usually improves the sensitivity and selectivity to some particular gases. However, these additives are mostly expensive noble metals, which are not practical for low-cost applications. Recently, carbon based dopants have been reported as promising low-cost alternatives. For instance, carbon nanotubes (CNTs) incorporation has been shown to be effective for improving sensitivity and selectivity toward H₂ of WO₃ thin film [22]. In another research, carbon doping provides considerable enhancement of toluene vapor sensitivity of WO₃ microtubes at low operating temperature [23].

WO₃ nanostructures have been fabricated based on a number of methods including thermal oxidation [24–26], spray pyrolysis [27], and chemical vapor deposition [28,29]. However, most methods produce nanostructures with random sizes, distribution and orientations, which are not reliable for practical applications because of poor reproducibility. Recently, some processes including e-beam lithography, nanolithography and template growth techniques have been developed to realize well-ordered nanostructures. Nevertheless, they require either expensive instrumentation or complicated fabrication procedures. Simple and low cost methods for well-ordered nanostructure construction are more preferred in gas-sensing and other commercial applications.

Glancing angle deposition (GLAD) technique is a relatively new method for fabrication of well-ordered and sophisticated nanostructures i.e. nanorods, nanoblade and zigzag nanocolumns [30–33]. It is a modified physical vapor deposition process, in which substrate surface is rotated and tilted to an angle greater than 80° with respect to the normal of substrate surface or less than 10°.
with respect to the direction of vapor flux. The vapor molecules directed to the substrate will experience shadowing and limited surface diffusion condition leading to the formation of isolated nanostructures. With this technique, the shape, size and density of nanostructures can be well controlled by deposition parameters such as deposition angle, operating pressure, substrate temperature, deposition power, and so on. There have been few reports of WO₃ nanorods fabricated by the GLAD technique using DC magnetron sputtering [34–36]. However, there has been no report of carbon-doped WO₃ nanorods prepared by the GLAD technique using radio-frequency (RF) magnetron sputtering and its application in gas sensing. In this work, carbon-doped WO₃ nanorods is fabricated by GLAD technique using RF magnetron sputtering and investigated for NO₂ gas sensing application. In addition, its performances are comparatively studied with that of the undoped ones.

2. Experimental details

2.1. Fabrication of WO₃ nanorod-based gas sensors

WO₃ nanorod-based gas sensors were fabricated by the direct deposition of WO₃ nanostructures onto prefabricated interdigitated electrodes on alumina substrates. Interdigitated electrodes were used to reduce the resistance of WO₃ material. These electrodes were prepared by sputtering Cr (~50 nm) and Au (~300 nm) on alumina substrates with pre-patterned photoresist and lift-off process. The interdigitated Au electrodes have a sensing area of 2 mm × 5 mm and the interdigit spacing of 100 μm. In addition, WO₃ nanorods were also deposited on bare silicon substrates for structural characterization. Prior to deposition, silicon wafers and alumina substrates were pre-cleaned successively with acetone and isopropanol by ultrasonication, dried in nitrogen atmosphere and then loaded into the deposition chamber.

Carbon-doped and undoped WO₃ nanorods were deposited by a variable angle RF magnetron sputtering system as schematically illustrated in Fig. 1. A 3-inch tungsten disc with 99.995% purity (K.J. Lesker) was used as the sputtering target. The distance from the target to substrate center and the substrate rotation were fixed at 7 cm and 60 rpm, respectively. The chamber was evacuated by mechanical and turbomolecular pumps (Pfeiffer Inc.) while the vacuum pressure was continuously monitored with Pirani and Penning pressure gauges. For undoped WO₃ nanorod deposition, W target was reactively sputtered in a mixture of 99.999% argon and 99.999% oxygen after a base pressure of 5.6 × 10⁻⁶ mbar. The flow rates of Ar and O₂ were controlled with a mass flow controllers (MKS Inc.) at 9.6 and 11.0 sccm, respectively. The plasma discharge was generated at a constant RF power of 200 W at a sputtering pressure of 5.0 × 10⁻³ mbar for 3 h. The substrate normal was positioned at an angle of 85° (α = 85°) with the respect to the vapor incident flux (the vertical axis). For carbon doping, 99.99% acetylene (C₂H₂) as a carbon source was added at a flow rate of 1.1 sccm into the gas mixture and RF plasma was induced at a sputtering pressure of 6.0 × 10⁻³ mbar. The undoped and carbon-doped WO₃ nanorods were then annealed in-air for 3 h at 400 °C to stabilize the crystalline structures. Photograph of the fabricated sensor is displayed in the inset of Fig. 1.

2.2. Characterization

The morphology, chemical composition and crystallographic structure of carbon-doped and undoped WO₃ nanorods were characterized using a field emission scanning electron microscopy (FE-SEM, Hitachi S-4700), Auger electron spectroscopy (AES, EMSL 680), and X-ray diffraction (XRD, Rigaku Trax III). SEM and AES characterizations were conducted at an electron acceleration voltage of 10 kV. Prior to AES measurement, the surface of WO₃ nanorods was sputtered to clean surface contamination by a 2 keV Argon ion beam for 1 min. Grazing-incidence XRD measurement was operated at 50 kV/300 mA with Cu-Kα1 radiation (λ = 1.54056 Å). The detector scanning speed was set at a rate of 1°/min and the measurement was run from 20 to 60° incident angles in steps of 0.02°.

2.3. Gas sensing measurements

The gas sensing properties of the undoped and carbon-doped WO₃ nanorods were measured using a dynamic flow system with gas concentrations controlled by using a mass flow controllers (Brook Instrument Inc.). A set of heaters made of Ni–Cr coils were used to supply a well-controlled operating temperature in the range of 100–300 °C. The sensors were tested by applying a constant voltage (10 V) and measuring electrical current using a 8846 A Fluke multimeter with 6.5 digit resolution. The resistance of gas sensors can be calculated from the simple Ohm’s law. The target gases including nitrogen dioxide (NO₂), hydrogen sulfide (H₂S), hydrogen (H₂), ammonia (NH₃), carbon monoxide (CO), and ethanol (EtOH) vapor were introduced at various concentrations to assess the response and selectivity of the sensors. To avoid the humidity effects, the zero-grade dry air was used as the carrier gas. The times for response and recovery period were selected from the times to achieve at least 90% resistance change of the saturated value. The data were recorded every second using LabVIEW via a USB DAQ device for subsequent analyses.

3. Results and discussion

3.1. Characterization of structure and morphology

The surface morphologies of the as-grown products are displayed in Fig. 2. It can be seen that the glancing-angle RF magnetron sputtering deposition technique produces well-aligned WO₃ vertical nanorods with highly uniform distribution over a large area (Fig. 2a). The nanorod structures result in a rougher surface and larger specific surface area compared to undoped ones. The average diameter (Fig. 2b) and length of carbon-doped WO₃ nanorods are
estimated to be ∼174 nm and ∼1.1 μm, respectively, corresponding to an aspect ratio of ∼6.3 while the undoped WO₃ nanorods have an average diameter of ∼74 nm (Fig. 2c) and length of ∼235 nm, leading to an aspect ratio of ∼3.2. It should be recalled that the time of deposition for both structures were three hours. Thus, acetylene gas plays an important role in accelerating the growth of WO₃ nanorods, resulting in nanorod structure with higher aspect ratio. This phenomenon may be attributed to the enhancement of local combusive oxidation and temperature gradients and more details in the carbon-assisted growth mechanism can be found in Ref. [37]. From top-view SEM images at medium and low magnifications (Fig. 2b and c), nanorods seem to be isolated and connected together only at their base. However, more detailed examination as illustrated in Fig. 2d and e reveals that nanorods are partially connected along parts of their walls and the connection occurs continually throughout the nanorod array.

The growth mechanism of nanorods can be schematically illustrated in Fig. 3. The vertically well-aligned WO₃ nanorods are formed due to the dominant of strong atomic self-shadowing under conditions of limited adatom mobility [38–40]. The growth process may be divided into two stages including (I) initial nucleation to form shadowing centers and (II) formation of columnar structures due to the shadowing effect. Initially, deposited atoms are randomly distributed on a substrate forming initial nucleation sites. The initial nucleated islands behave as the shadowing centers that hide unoccupied sites and smaller islands from the incident vapor flux arriving at a glancing angle (α = 85°). Thus, atoms from vapor flux only accumulate on taller islands, which are grown into columns. For materials with the limited adatom mobility such as WO₃, voids are not filled due to very slow surface diffusion [38]. Therefore, the layer consisting of nanorods is formed with separation of narrow pores. It should be noted that vapor flux can be divided into vertical and lateral components with respect to substrate. However, the average lateral component becomes zero because of the cancelation of lateral component at opposite directions under a constant-speed substrate rotation, leading to vertical growth [39].

Auger electron spectra of carbon-doped and undoped WO₃ nanorods are demonstrated in Fig. 4. The spectra contain only three existing elements including carbon, tungsten and oxygen.
The spectrum of carbon doped sample shows apparently higher carbon peak than that of the undoped one and the carbon contents of carbon-doped and undoped are estimated to be 20.1 and 6.4 at% respectively. Thus, the result confirms that carbon is incorporated into WO₃ nanorods and the produced nanorods have high purity without contamination from other elements. Fig. 5 shows XRD patterns of carbon-doped and undoped WO₃ nanorods. Both carbon-doped and undoped WO₃ nanorods can be well indexed to the standard pattern of monoclinic WO₃ with lattice constants of \( a = 7.297 \text{ Å}, b = 7.539 \text{ Å}, \) and \( c = 7.688 \text{ Å} \) (JCPDS No. 43-1035). No peak of impurities is found in these XRD patterns. By using Scherrer equation, the average grain size based on the peak from 21.5° to 25.5° can be calculated to be 23 nm and 26 nm for the undoped WO₃ nanorods and the carbon-doped WO₃, respectively. Therefore, carbon doping causes a small increase of grain size but does not change the crystallinity of the WO₃ nanorods, which is in agreement with another report of C doping in WO₃ films [41].

### 3.2. Gas sensing properties

The gas-sensing properties of the sensors are primarily characterized in terms of gas response. The gas response to reducing gases (i.e. H₂, NH₃, CO, H₂S, EtOH) and to oxidizing gases (i.e. NO₂) are defined as \( R_\text{air}/R_g \) and \( R_\text{air}/R_\text{red} \), respectively, where \( R_g \) is the resistance of sensor in pure air, \( R_\text{red} \) is the resistance of sensor in reducing gas, and \( R_\text{air} \) is the resistance of sensor in oxidizing gas. Fig. 6 shows the gas response of the C-doped and undoped WO₃ nanorods gas sensors to 3 ppm NO₂ at different operating temperatures. It can be seen that the gas responses of both gas sensors increase with increasing operating temperatures from 100 °C to 250 °C and then decrease as the operating temperature increases further. Thus, the optimum operating temperature of both nanorods gas sensors are around 250 °C. In general, the increase of operating temperature contributes to the increase of the adsorption, diffusion and surface reaction rates [22,42]. In other words, sufficient thermal energy helps the reactions involved overcome their respective activation energy barriers leading to increase sensor response with increasing operating temperatures from 100 °C to 250 °C. However, desorption process will dominate and the diffusion depth becomes lower when operating temperature becomes too high leading to the decrease of gas response [43]. This behavior can also be well explained by the diffusion theory [44].

Dynamic response of the carbon-doped and undoped WO₃ nanorods gas sensors toward NO₂ at the optimum operating temperature of 250 °C is illustrated in Fig. 7. It can be seen that both carbon-doped and undoped WO₃ nanorods behave as n-type semiconductors since the sensor’s resistances increase upon the introduction of oxidizing NO₂ gas. In addition, the resistance of carbon-doped WO₃ nanorods is more than an order of magnitude lower than that of undoped one, indicating a significant increase...
of charge carrier concentration due to carbon incorporation. Moreover, the sensors exhibit fast and stable response toward NO₂. The response and recovery times are increased with increasing gas concentrations. The response times of the carbon-doped and undoped WO₃ nanorods gas sensors to 0.1 ppm NO₂ are estimated to be 17 s and 21 s, respectively while the corresponding recovery times are 50 s and 69 s. The improved response rate by carbon doping may be attributed to increased adsorption and desorption rates of NO₂ molecules on the WO₃ surface due to the increase of charge carriers.

In order to investigate the response and selectivity of C-doped WO₃ nanorods gas sensor, the sensor was exposed to NO₂, H₂S, H₂, NH₃, CO, and EtOH at various concentrations. Various gas responses of carbon-doped WO₃ nanorod sensor at the optimum operating temperature of 250 °C are displayed in Fig. 8. It can be seen that carbon-doped WO₃ nanorod sensor exhibits high response to NO₂ but is almost insensitive to H₂, NH₃, CO, and EtOH at concentrations ranging from 1 ppm to 5 ppm. At the concentration of 5 ppm, the gas response of carbon-doped WO₃ nanorod sensor to NO₂ and H₂S are ~348 and ~40. Thus, the sensor clearly shows the selectivity to NO₂ at concentrations ranging from 1 ppm to 5 ppm. However, the gas response of sensor to NO₂ and H₂S are not significant different in the low concentration range (0.1 and 0.3 ppm, inset of Fig. 8). The detection limit of NO₂ for carbon-doped WO₃ nanorods sensor is estimated to be <0.5 ppm at the operating temperature of 250 °C.

From Fig. 6, it can also be noticed that carbon-doped WO₃ nanorods gas sensor exhibits relatively high NO₂ response as compared to the undoped one at low operating temperature of 150 °C. It is thus useful to evaluate its response at low operating temperature (150 °C) for low-power gas-sensing applications. Fig. 9 shows gas responses of carbon-doped and undoped WO₃ nanorods sensors to NO₂ at various gas concentrations. It is evident that the gas responses of carbon-doped WO₃ nanorods sensor are considerably higher than those of the undoped sensor at all NO₂ concentrations. At concentration of 5 ppm, the gas response of carbon-doped WO₃ nanorods sensor is ~114, which is ~2 times higher than that of undoped WO₃ nanorods sensor (~52). The enhancement of gas response at low operating temperature after carbon doping may be explained as follows. It was found that carbon doping results in the decrease of sensors resistance at 150 °C from 128 MΩ to 4.6 MΩ. This may imply the decrease of activation energy barrier for charge carrier generation. At low operating temperature, the undoped sensor exhibits low conductivity and low gas response because thermal energy is not enough to overcome the activation energy barrier. After carbon doping, the activation energy is reduced and charge carrier can be better thermally activated leading to higher gas response. In addition, the WO₃ nanorods sensor prepared by this method can detect NO₂ at relatively low operating temperature with high gas response compared to some other methods. For example, gas response to 1 ppm NO₂ of our carbon-doped WO₃ nanorods sensor is 18.2 at operating temperature of 150 °C while gas responses of WO₃ nanorods sensors prepared by thermal evaporation [25] and hydrothermal process [45] at their optimum operating temperature of 300 °C were reported to be 7 and 5.3, respectively.

3.3. Sensing mechanism

Based on the observation of FE-SEM (see Fig. 2d and e), nanorods connect together not only at their base but also along some parts of their sidewall. This allows current to flow from one electrode to another electrode with contribution of partial wall-wall nanorods connections as schematically illustrated in a simplified model (Fig. 10a). Thus, the sidewalls of WO₃ nanorods can also contribute to gas-sensing, leading to a large active gas-sensing area and high gas response. This is in agreement with the observed high NO₂ response (300–400), which should not be possible if only base of nanorods takes part in gas-sensing.

From the results, doping of carbon into WO₃ nanorods does not change the crystallinity but it tends to decrease activation energy and alter the thickness of the depletion layer. Sensing mechanism of carbon-doped WO₃ nanorods sensor may therefore be explained based on the model of general WO₃ nanorod sensor [43,46]. The sensing mechanism of NO₂ reactions on the surface of a single C-doped WO₃ nanorod is displayed in Fig. 10b. Initially, oxygen molecules from air adsorb on the surface of C-doped WO₃ nanorods and transform to chemisorbed oxygen species (O²⁻, O₂⁻, and O⁻) by trapping the electrons at the surface according to the following reactions [47]:

\[ \text{O}_2(g) \rightarrow \text{O}_2(\text{ads}) \]  
\[ \text{O}_2(\text{ads}) + e^- \rightarrow \text{O}_2^-(\text{ads}) \]  
\[ \text{O}_2^-(\text{ads}) + e^- \rightarrow 2\text{O}^-\text{ads}) \]  
\[ \text{O}^-\text{ads}) + e^- \rightarrow \text{O}_2^-\text{ads}) \]

At the operating temperature of 100–300 °C, the chemisorbed oxygen species, O⁻, is known to be dominant. When O⁻ species adsorb on surface, a depletion layer known as the space charge layer is formed at grain boundaries near the carbon-doped WO₃ nanorod surface. When a carbon-doped WO₃ nanorod is exposed to NO₂,
NO$_2$ molecules directly adsorb on the surface of nanorods because NO$_2$ molecules own very high electron affinity (2.28 eV) compared with oxygen’s value of 0.43 eV [48,49]. Moreover, the products from the reaction between NO$_2$ and e$^-$ on the surface also react with the O$^-$ species, resulting in the continuously decreasing concentration of electrons on the surface. The process can be described by the following reactions [50]:

$$\text{NO}_2(g) + e^- \rightarrow \text{NO}_2^-(ads)$$ (5)

$$\text{NO}_2^-(ads) + 2O^-(ads) \rightarrow 2O^2-(ads) + \text{NO}_2(g)$$ (6)

The above reactions decrease the electrons from carbon-doped WO$_3$ nanorods and increase the thickness of the depletion layer, leading to the increase in resistance of carbon-doped WO$_3$ nanorods sensor.

4. Conclusion

Carbon-doped and undoped WO$_3$ nanorods gas sensors have successfully been fabricated by the GLAD technique with RF magnetron sputtering. By this technique, the vertically well-aligned homogeneous WO$_3$ nanorods with very low defect were achieved over a large area. It was found that carbon doping does not change any crystalline structure but increases the grain size and accelerates the nanorods growth leading to relatively high aspect ratios as compared to the undoped one. The fabricated carbon-doped WO$_3$ nanorods sensor exhibits high response and selectivity to NO$_2$ at the concentration range of 0.5–5 ppm at an optimum operating temperature of 250°C. In addition, the carbon-doped sensor still works well for NO$_2$ detection at lower operating temperature of 150°C due to the decrease of activation energy and alteration of the depletion layer. Therefore, carbon-doped well-ordered WO$_3$ nanorods with improved NO$_2$ gas-sensing performances in terms of response, response time, selectivity and operating temperature has been realized. Moreover, our technique offers distinct advantages over other methods such as high productivity, simplicity and low cost for well-ordered nanostructure construction.

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