Selective growth of ZnO nanorods and its gas sensor application

Shao-Lin Zhang, Jeong-Ok Lim, Jeung-Soo Huh, and Wooyoung Lee

***Abstract*—Facile sonochemical method has been used to selectively grow ZnO nanorods sensing layer on alumina substrate directly. The growth region of ZnO nanorods array was precisely controlled by pre-coated Pt/Zn seed layer on alumina substrate. No growth of ZnO nanostructure is found on the substrate without seed layer. The grown ZnO nanorods were well aligned, densely packed, with wurtzite crystal structure and flat hexagonal tip. The sensing properties of the obtained ZnO nanorods sensing layer to three kinds of toxic gases (NH_3, CO and CH_4) have been investigated. The sensing observation revealed that the selectively grown ZnO nanorods demonstrated good response, repeatability and excellent linearity towards toxic gases. This result indicates that selective growth of ZnO nanorods directly on sensor substrate is an accessible and advantageous method for sensor fabrication.**

***Index Terms*—ZnO nanorods, selective growth, sonochemical, gas sensor**

I. Introduction

SEMICONDUCTOR metal oxides as gas-sensing materials have attracted great attention for a long time due to their advantageous features, such as small dimensions, low cost and simplicity in fabrication [1-6]. Among them, ZnO is one of the earliest discovered and most widely applied metal oxide materials for gas sensing due to its high mobility of conduction electrons, good chemical, physical and thermal stability under operating conditions [7]. However, traditional gas sensors were prepared from a bulk material or thick film. Thus the detection limit was quite high for developing environmental pollutant sensors. Recently, 1-D nanostructured ZnO material has been investigated to fabricate new semiconductor gas sensors due to its high density of surface reaction site and surface-to-volume ratio [8, 9].

Various methods have been developed to synthesize 1-D nanostructured ZnO, such as thermal evaporation deposition [10-12], chemical vapor deposition (CVD) [13-16] and aqueous chemical growth [17-24]. However, previous two methods had employed high temperature process in synthesis of nanostructured ZnO. The high growth temperature is un-compatible with microelectronic process. Recently, aqueous chemical growth method has been widely investigated for producing ZnO nanorods in low temperature. However, the obtained nanorods were randomly dispersive. For further application to gas sensor, these dispersive nanorods should be transferred to sensor substrate by screen printing method, followed by a high temperature (around 600-800^{o}C) annealing treatment [25, 26]. The high processing temperature inhibits its compatibility with microelectronic process. It is necessary to develop a relatively low temperature and selective growth method for growing desired nanorods sensing layer on the sensor substrate directly.

In this work, we report the facile sonochemical synthesis of well aligned ZnO nanorods on alumina substrate at room temperature. Selective growth of nanorods array was obtained by controlling the dimension of metallic Zn seed layer. We also investigated the structural and gas sensing properties of the sensing layer. The gas sensor from the nanorods showed very high sensitivity and stability. The results indicated that selectively grown ZnO nanorods are potential candidate for developing high-performance gas sensor. It also suggested that direct growth of sensing layer on sensor substrate is promising method for gas sensor fabrication.

II. Experimental

A. Synthesis

An alumina plate comprising interdigitated electrodes and back heater was used as sensor substrate. As shown in Fig. 1 (a), the dimension of the substrate was 10 mm × 8 mm, and the distance between two electrodes was 0.33 mm. The resistance of the back heater was about 10 Ω. The alumina substrate was washed in acetone, ethanol and deionized water sequentially for several times and dried in vacuum. A high-temperature resistance tape was first covered on part of the substrate as a mask patterning the following deposition. Pt layer with 10 nm thickness was then coated on the alumina substrate by ion-coater. The Pt layer was used as a catalyst and a bonding

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agent because the Zn is not adhesive with alumina substrate. Zn thin film with 100 nm thickness as seed layer was subsequently deposited on the substrate by the thermal evaporation method as shown in Fig. 1(b). Then the tape mask was removed and the substrate was immersed in a solution which zinc nitrate hydrate [$Zn(NO_3)_2 \cdot 6H_2O$, 0.1 M] and hexamethylene tetramine (HMT) [$(CH_2)_6N_4$, 0.1 M] were dissolved in deionized water. And the solution was irradiated with high intensity ultrasound radiation (100 W, 20 kHz) for 2 hours under ambient condition. Finally, the substrate was slightly washed by deionized water and ethanol for several times.

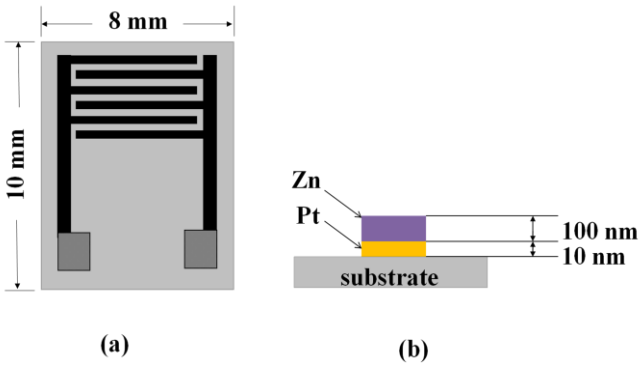

Fig. 1 Schematic diagram of (a) alumina substrate with interdigitated electrodes and (b) substrate after coating.

B. *Characterization*

The obtained sample was characterized by X-ray diffraction (XRD, Cu Kα) to observe the structural nature of ZnO nanorods. XRD pattern was recorded from 20 to 80˚ (2θ) with a scanning step of 0.02˚. The size distribution and morphology of the samples were analyzed by field emission scanning electron microscopy (FE-SEM) with a Hitachi S-4300 field-emission scanning electron microscope operated at 15 keV.

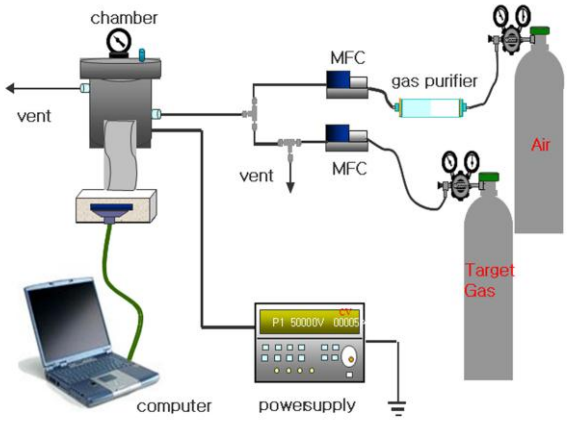

Fig. 2 Schematic diagram of the measuring system.

C. *Gas Sensitivity Test*

The gas sensing properties of the sensor sample was tested with various kinds of toxic gases (NH_3, CO and CH_4). The schematic diagram of measuring system is shown in Fig. 2. The volume of the testing chamber was about 1000 ml. The flow-through technique was used to test the detection capability of the gas sensor. The gas sensitivity was measured in static state. The working temperature of sensors was adjusted via heating voltage. The desired concentration levels of target gases were obtained from certified bottles and a mixing system with mass flow controllers (MFC). Sensitivity is defined as shown in Equation (1),

$$S(\%) = \left[\frac{R_g - R_a}{R_a}\right] \times 100\% \quad (1)$$

where R_a is the sample resistance measured at ambient environment while R_g is that under the test gas. The response

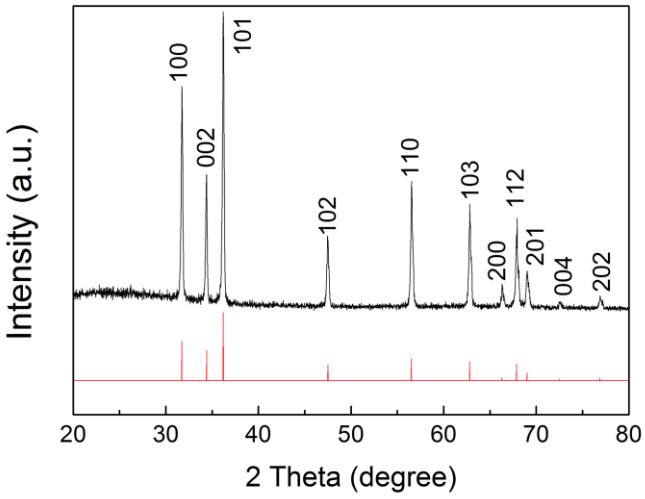

Fig. 3 XRD pattern of the ZnO nanorods.

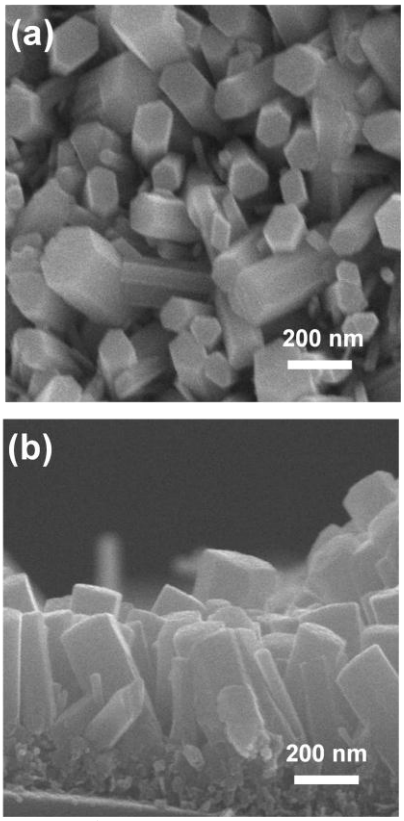

Fig. 4 FE-SEM images of ZnO nanorods obtained on Zn coated alumina substrates: (a) top-view, (b) cross-section view.

time in our experiment is defined as the time required for the sensitivity variation to reach 90% of the equilibrium value, while the recovery time is defined as the time needed to return to 10% below its original state.

III. Results and Discussion

A. *Crystalline structure*

The XRD pattern of the ZnO nanorods grown on substrate is plotted in Fig. 3. All the diffraction peaks could be indexed to hexagonal wurtzite ZnO (JCPDS Card No. 36-1451, a=0.325 nm, c=0.521 nm). The strong diffraction peaks present highly crystallized ZnO. No characteristic peak is observed for other impurities. The pre-coated Pt does not arise in the pattern due to the small quantity. The disappearance of the peak of metallic Zn suggests the complete reaction of Zn seed layer during the ultrasonic treatment. In other reports dealing with the vertically aligned ZnO nanorods, typically the peak (002) in XRD pattern is much more intense than other peaks [27]. The intense peak of (002) suggests the preferential growth of ZnO nanorod along c axis. However, due to the irregularity of the polycrystalline alumina substrate, the ZnO array in our sample is polydirectional and the peak (002) is not outstanding comparing to other peaks.

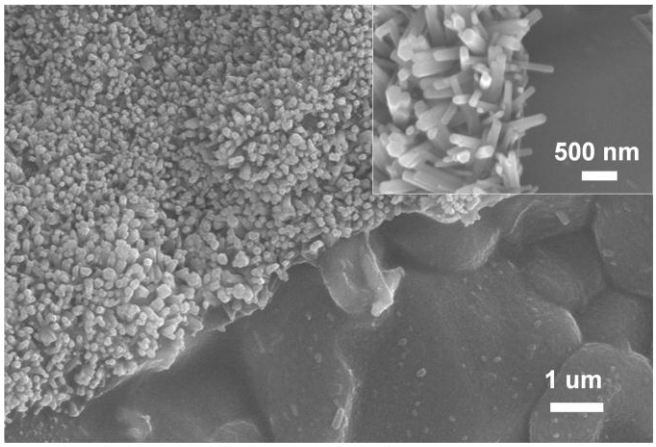

Fig. 5 FE-SEM image of the boundary area of a selective grown ZnO nanorods array. The inset is the enlarge magnification image of the nanorods grown on the boundary of the Zn seed layer.

B. *SEM morphology*

The SEM images of a typical ZnO nanorods array grown on Pt/Zn coated alumina substrate are shown in Fig. 4. Using the simple sonochemical method from aqueous solution, a well aligned, densely packed and large scale array of ZnO nanorods was successfully obtained. On the basis of SEM images we determine the value for nanorods densities and diameters. It is found that the nanorods densities are of 10^7 to 10^8 mm^{-2} and diameters are of 50 to 150 nm, respectively. Furthermore, the average height of the nanorods array measured with a profiler is about 500 nm, as shown in Fig. 4(b).

Moreover, for further applications it is necessary to selectively grow the ZnO nanorods on the desired area. The SEM images of the boundary area of the Zn seed layer are shown in Fig. 5. No growth is found on the bare surface of the alumina substrate. On the other hand, there is a densely packed ZnO nanorods array on Pt/Zn metalized surface of the substrate. The boundary of the growth is well-defined and very clean suggesting that one can grow more complicated nanorods pattern if the seed layer is previously defined well. It is also observed that the ZnO nanorods are polydirectional, as shown in the inset of Fig. 5. This observation corroborates the results obtained by XRD analysis. The polydirectional nanorods intercross each other and form lots of contacts. It is believed that the large number of contacts between nanorods will enhance the sensing properties of the material.

The growth mechanism of ZnO nanorods can be explained as follows. The ultrasound radiation used in our experiments caused high energy chemistry via the process of acoustic cavitation: the formation, growth and implosive collapse of bubbles in a liquid. During cavitational collapse, intense local heating (5000 K) and pressure (1000 atm) of the bubbles occurs for a very short time (few microseonds) resulting in high-velocity interparticle collisions [28]. With this extreme condition, the growth process is greatly promoted. Typically, the growth process of ZnO nanorods can be divided into two parts: nucleation and growth. During the initial-stage of the reaction, the aqueous solution of zinc nitrate hydrate converted into a colloidal-gel of zinc hydroxide. The zinc hydroxide then combined with the ammonia and resulted in soluble complex. This transformation can be represented by the following chemical reactions [29, 30]:

$$(CH_2)_6N_4 + 10H_2O \rightarrow 6HCHO + 4NH_3 \cdot H_2O \quad (2)$$

$$Zn(NO_3)_2 \cdot 6H_2O + 2NH_3 \cdot H_2O \rightarrow Zn(OH)_2 + 2NH_4NO_3 + 6H_2O \quad (3)$$

$$Zn(OH)_2 + 2H_2O \rightarrow Zn(OH)_4^{2-} + 2H^+ \quad (4)$$

Then, due to the sonication energy, the zinc hydroxide complex was split into ZnO and OH^- anion according to reaction (5).

$$Zn(OH)_4^{2-} \rightarrow ZnO + H_2O + 2OH^- \quad (5)$$

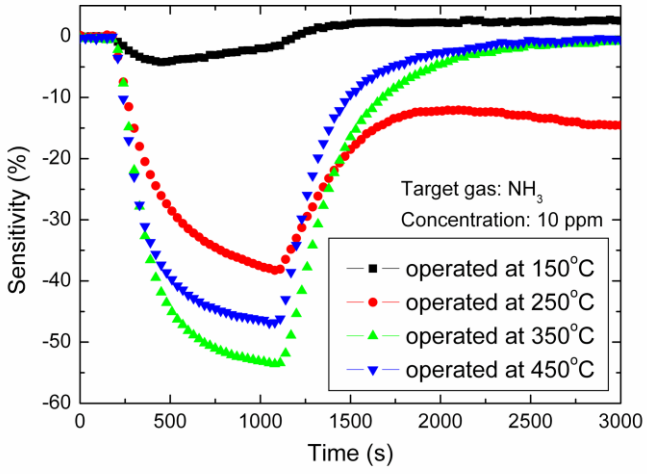

Fig. 6 Sensing response of ZnO nanorods-based sensor towards 10 ppm NH_3 at various operating temperatures.

In our experiment, alumina coated with thin Zn metal layer was used as substrate. We believe that these crystalline structures of Zn layer have an effect on the nucleation of the ZnO. Heterogenous nucleation took place on the substrate and the patterning Zn layer acted as seeds for nucleation. It is found

that there are no nanorods grown on the area without seeds. This provides us a possible approach to fabricate patterning sensing layer directly on the substrate. It is also observed that the diameter of the nanorods in our result is relatively inhomogenous. This is attributed to the inhomogenous size of the seeds particles as well as the polycrystalline property of the substrate.

Herein, it is worthy to highlight the advantages of this process for the fabrication of gas sensor. The processing temperature throughout the experiment was below 60°C, and no high pressure or vacuum was required. Furthermore, this process was free of surfactant, catalyst and template decreasing the risk of insufficient purification and improving the reliability. Thus the proposed method is an accessible and reliable method for ZnO nanorods array fabrication.

C. Gas sensitivity

In general, the sensitivity of gas sensors is affected by the operating temperature. Fig. 6 plots the response of the fabricated ZnO nanorods sensor to 10 ppm NH_3 gas, measured at various temperatures. Based on the Fig. 6, the measured responses were approximately -4, -39, -54 and -47% when the gas sensor was operated at 150, 250, 350 and 450°C, respectively. The maximum response was obtained at 350°C. It was also found that sensor operating at 350°C recovered almost 100% after removing the NH_3 gas, indicating the optimal operating temperature for our sensor is 350°C.

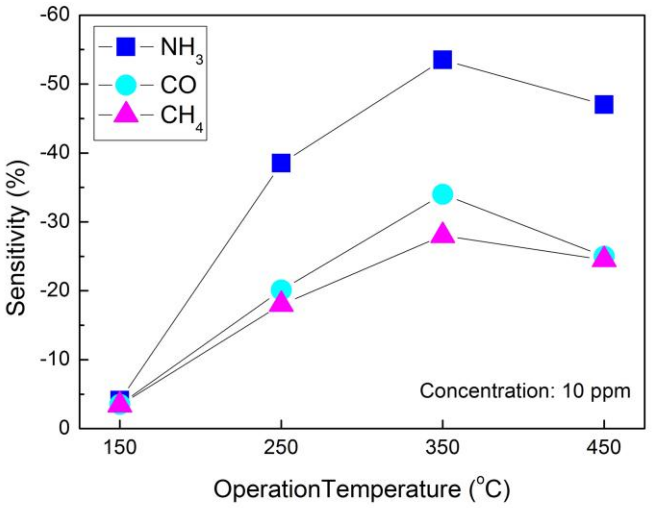

Fig. 7 Sensing response of ZnO nanorods-based sensor to 10 ppm NH_3, CO and CH_4 at various operating temperatures.

The response and recovery time of the gas sensor is important for the practical application. The response and recovery time of our sensor towards to 10 ppm of NH_3 gas at 350°C were estimated to 400 s and 800 s, respectively. The further investigation revealed that the response and recovery time changed slightly upon the variation of gas species and gas concentration. In several previous reports, the response and recovery time of ZnO nanomaterial based gas sensor were tens min [31-33]. The response and recovery time of our sensor are comparable to these reports.

Fig. 7 shows the sensing response of the ZnO nanorods sensor to 10 ppm NH_3, CO and CH_4 as a function of operating temperatures. The response of nanorods sensor was found to increase with the increasing operating temperature, which attained the maximum at 350°C towards all three target gases, and then decreased with a further rise of the operating temperature.

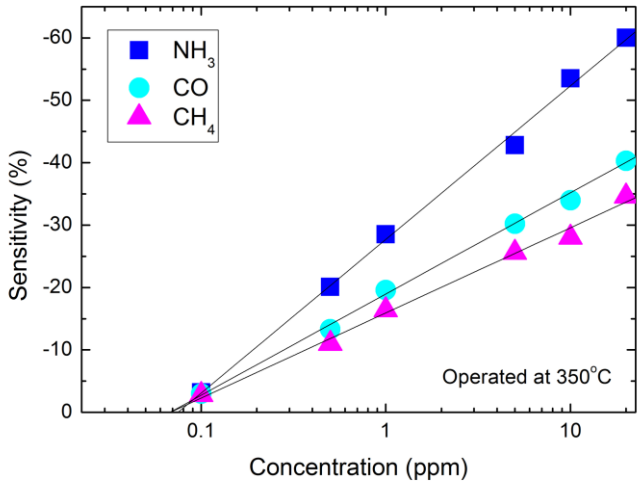

Fig. 8 Sensitivities of ZnO nanorod-based sensor for various kinds of toxic gases with concentration ranging from 0.1 to 20 ppm at 350°C.

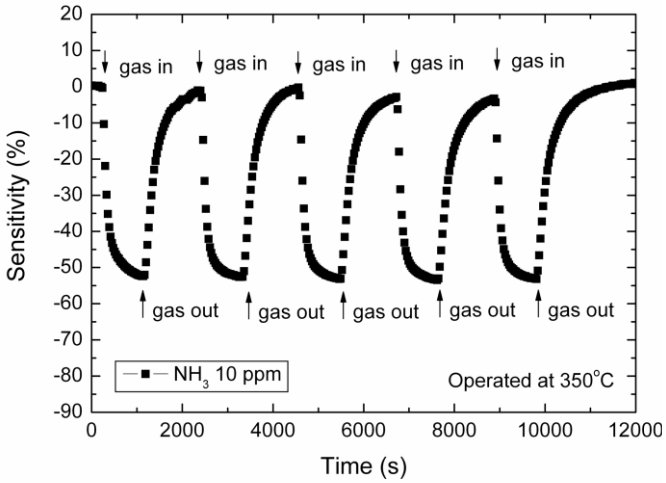

Fig. 9 The reproducibility of ZnO nanorod-based sensor in 10 ppm NH_3 at 350°C.

When ZnO nanorods are exposed into air, oxygen molecules will absorb on the ZnO surface to generate chemisorbed oxygen species (O_2^-, O^-, and O^{2-}). The absorption of oxygen seizes electrons from the conduction band of ZnO. This process decreases the conductivity of ZnO nanorods, and thus the sensing layer shows a high resistance. When reductive toxic gas flows to the sensor at an elevated temperature, gas molecular reacts with oxygen species on the ZnO nanorods surface. Thus the concentration of oxygen species on the nanorods surface decreases. The desorption of the oxygen species releases electrons into conduction band of ZnO, which increases the conductivity of ZnO nanorods array. At relatively low

operating temperature, the nanorods surface preferentially adsorbs O_2^- and O^-, and the sensitivity of the material is consequently very small. With the increase of operating temperature, the adsorption of O^{2-} is more reactive on the surface of ZnO nanorods and thus makes the material more sensitive to the presence of a reductive gas. If the temperature increases too much, desorption of all the oxygen ionic species adsorbed previously occurs. As a result, the sensitivity decreases [34-36].

We measured the response of ZnO nanorods sensor to NH_3, CO and CH_4 with the concentration ranging from 0.1 ppm to 20 ppm under a temperature of 350°C. The sensitivities to these reductive gases are shown in Fig. 8. It is found the sensing response increased with increasing concentration of target gases. The sensor showed good linearity to three toxic gases in full concentration range. The linear behavior shows a power law dependence of the response on the concentration. The well-known power law can be expresses as $S=\alpha c^{\beta}$, where α is a concentration independent factor, c is the concentration, and β is the exponent of power law related to sensor materials and target gas ambient [37, 38].

The reproducibility of the sensor was also measured. Fig. 9 shows the repeating response of ZnO nanorod-based sensor. The sensor was tested with 10 ppm NH_3 for more than 5 times at 350°C. No obvious variations on sensor sensitivity were observed. The sensor finally recovered fully after removing the NH_3 gas, indicating selectively grown ZnO nanorods sensor has excellent sensing property.

IV. Conclusions

In summary, we reported a simple sonochemical route to selectively grow well aligned ZnO nanorods directly on sensor substrate with mild condition. The as-grown ZnO nanorods were with wurtzite structure and hexagonal tip. SEM observation revealed that the placement of ZnO nanorods array can be controlled by the pre-coated Pt/Zn layer. The boundary of fabricated sensing layer was clean and well-defined. The gas sensing measurements showed that ZnO nanorods had excellent potential applications as gas sensors. The sensor showed good response, reproducibility and excellent linearity to NH_3, CO and CH_4. The result suggests the applicability of our sensor for quantitative detection of reducing toxic gases.

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