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Synthesis of ZnO comb-like nanostructures for high sensitivity H_2S gas sensor fabrication at room temperature

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Abstract. Zinc oxide (ZnO) comb-like nanostructures were successfully synthesized on the silicon substrate without a catalyst via chemical vapour deposition. The morphology and crystal structure of the product were characterized by scanning electron microscope and X-ray diffractometer. In this research, a simple gas sensor was fabricated based on the principle of change in resistivity due to oxygen vacancies, which makes its surface chemically and electrically active. The fabricated ZnO nanostructures proved to be quite sensitive to low concentration of H_2S gas at room temperature. The sensitivity and response time were measured as a function of gas concentrations. Small response time (48–22 s) and long recovery time (540 s) were found at H_2S gas concentrations of 0.1–4 ppm, respectively. ZnO comb-like structures are considered as the most suitable materials for gas sensor fabrication due to their high sensing properties. These nanostructures growth and H_2S gas sensing mechanism were also discussed.

Keywords. H_2S gas sensor; ZnO comb-like nanostructures; chemical vapour deposition; vapour–solid growth; growth mechanism.

1. Introduction

Zinc oxide (ZnO) nanostructures have attracted considerable attention due to their wide band gap of 3.37 eV at room temperature and a high exciton binding energy of 60 meV. A variety of ZnO nanostructures, nanowires [1,2], nanorods [3,4], nanoribbons [5], nanorings [6], nanohelices [7], nanotetrapods [8], nanoflowers [9] and nanocombs [10,11] have been discovered. Among them, ZnO nanocombs consisting of a ribbon and an array of parallel nanorods perpendicular to the ribbon are of interest for many applications such as the optical polarizer and grating [12], dye-sensitized solar cells [13], biosensors for glucose detection [14] and gas sensors [15]. Gas sensors based on metal oxide semiconductor nanostructures have received much attention due to their improved sensitivity compared with conventional thin film structures [16,17]. The nanostructures with large surface area to volume are relevant for optimized gas sensor performance towards H_2S gas. ZnO nanostructures for H_2S gas sensing were reported with different morphologies such as nanorods [18], nanoparticles [19], flower-like [20], nanospheres [21] and nanowires [22].

According to the mechanism differences of the nanostructures' formation, the extensively used vapour transport process can be categorized into the catalyst-free vapour–solid (VS) and catalyst-assisted vapour–liquid–solid processes. The VS is the most common and easy process used. So far, ZnO nanocombs have been mainly synthesized by thermal evaporation using Zn or ZnO usually mixed with graphite powder as precursors in a wide temperature range (440–1350°C) [23–25]. In most cases, Au was also used as a catalyst in the growth.

In the present work, a catalyst-free chemical vapour deposition (CVD) process using a precursor of Zn powder was conducted. Experiments were carried out to grow ZnO comb-like nanostructures on silicon using double zone tube furnace. The structure and morphology of ZnO comb-like structures were investigated. The growth mechanism of comb-like nanostructures was also discussed. Room temperature gas sensor was fabricated for H_2S gas at various concentrations. Their properties were also calculated and discussed. This sensor is provided to have very high sensitivity to low H_2S gas concentration (100 ppb).

2. Experimental

2.1 *Growth of ZnO comb-like nanostructures with CVD system*

In atypical synthesis of ZnO comb-like nanostructures (single teeth) on catalyst-free silicon (100) using CVD method was investigated. The synthesis was conducted in three zones tube furnace. The experimental setup is shown in figure 1.

A ZnO comb-like nanostructure was prepared by using CVD. One gram of Zn powders (99.995% purity) was loaded into a ceramic boat and placed in the centre of a horizontal quartz tube (about 3.5 cm diameter and 120 cm long) located inside three zones tube furnace, where the maximum temperature was fixed at 900°C (shown in figure 1). Si substrates were loaded in the right zone at 20 cm downstream from the source. Before the deposition, the air in the tube was removed by backfilling argon gas and then pumping the argon gas out until the pressure in the tube regained about 1.10^{-2} Torr. This procedure was repeated twice to remove air absorbed onto the tube furnace wall. Argon gas at a flow rate of 200 sccm was controlled using mass flow metre under continuous pumping. The furnace was programmed to 900°C (source material) and 550°C (substrate) at the central and the right zone, respectively. Later, the argon gas was fixed at a flow rate of 185 sccm. Oxygen gas was introduced into the tube at a flow rate of 15 sccm, when the central temperature reaches up to 420°C. These parameters were maintained during the whole growth process for 90 min. At the end of the process, the system was left to cool down to room temperature. The final product was a white layer deposited on the Si substrate.

2.2 *Characterization techniques*

The surface morphologies of the ZnO films were observed by using the scanning electron microscope (SEM/JEOL–JSM 5140) operated at 20 kV and equipped with energy-dispersive X-ray spectrometer (EDS). The crystal structures of ZnO comb-like nanostructures were characterized by X-ray diffractometer (XRD/PANalytical, X'Pert) with Cu Kα(λ = 0.154056 nm) radiation.

3. Results and discussion

3.1 *SEM and EDS analysis of ZnO comb-like nanostructures*

With the arrangement of the material source and substrates shown in figure 1, we obtained a cotton-like white layer of nanocombs deposited on a silicon substrate, where the temperature growth was 550°C. Typical SEM image of nanocombs recorded is shown in figure 2a. The morphological investigation confirmed that the as-grown nanocombs structures are made with a narrow ribbon-like stem and aligned nanorod arrays are attached uniformly along one side of the ribbon-like structure as shown in figure 2b. One type of single uniform teethed nanocombs with10–20 μm long and 3–5 μm width can be observed. The diameter of the nanorods is about 150 nm. Figure 2c is an EDS measured on the comb-like structures and it reveals that only Zn and O can be detected. This indicates that a pure ZnO product without any other impurities was found.

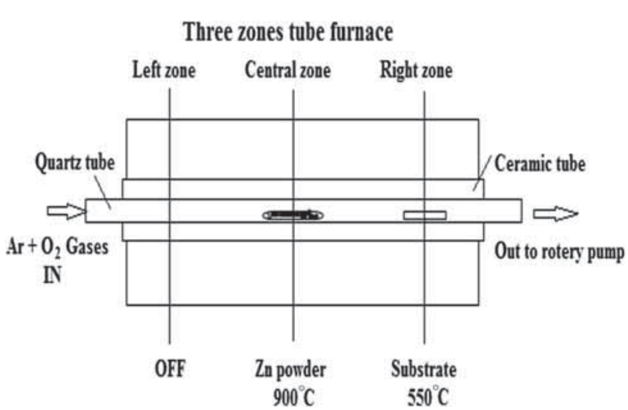

Figure 1. Schematic diagram of CVD system setup used for the synthesis of ZnO comb-like nanostructure.

3.2 *XRD analysis*

According to XRD results shown in figure 3 for as-deposited ZnO comb-like film exhibited a hexagonal wurtzite structure with (002) preferential orientation. The XRD pattern consists of a single (002) dominated peak, which occurs due to ZnO crystal and grows along the *c*-axis.

The lattice constants *a* and *c* of the ZnO wurtzite structure can be calculated using Bragg's law [26] (equations 1 and 2):

$$a = (1/3)^{1/2}\lambda/\sin\theta \tag{1}$$

$$c = \lambda/\sin\theta \tag{2}$$

where λ is the X-ray wavelength of the incident Cu Kα radiation (1.54056 Å). The lattice constants *a* and *c* were determined as *a* = 3.2498 Å and *c* = 5.2066 Å for pure bulk ZnO, respectively [27]. The lattice constants *a* and *c* for the reflection planes (100) and (002) are calculated using the relations 1 and 2, respectively. The calculated lattice constants of ZnO comb-like as prepared are: *a* (100) = 3.218 Å ($2\theta = 32.09°$) and *c* (002) = 5.158 Å ($2\theta = 34.75°$). These values are consistent with the values obtained for hexagonal ZnO wurtzite structure [28,29]. The lattice parameter variation as a function of temperature is discussed elsewhere [30,31].

3.3 *Growth mechanisms of ZnO comb-like nanostructures*

The growth of ZnO comb-like nanostructures on the silicon substrate was conducted by CVD without metal catalyst. This was confirmed by the SEM and XRD observations. Therefore, this growth phenomenon confirmed the formation of comb-like ZnO nanostructures. The growth reaction in the present work follows the VS mechanism. Experimentally, by increasing the furnace temperature up to 900°C, the metallic zinc powder (melting point = 419.6°C) was melted and the generated zinc vapours were transferred to the silicon substrate through the argon carrier gas. In this process, the evaporated zinc vapours diffused and immediately reacted with oxygen to form the ZnO nuclei and deposited onto the silicon substrate at a lower temperature (550°C). The oxidation reaction was as follows (equation 3):

$$2Zn + O_2 \rightarrow 2ZnO \tag{3}$$

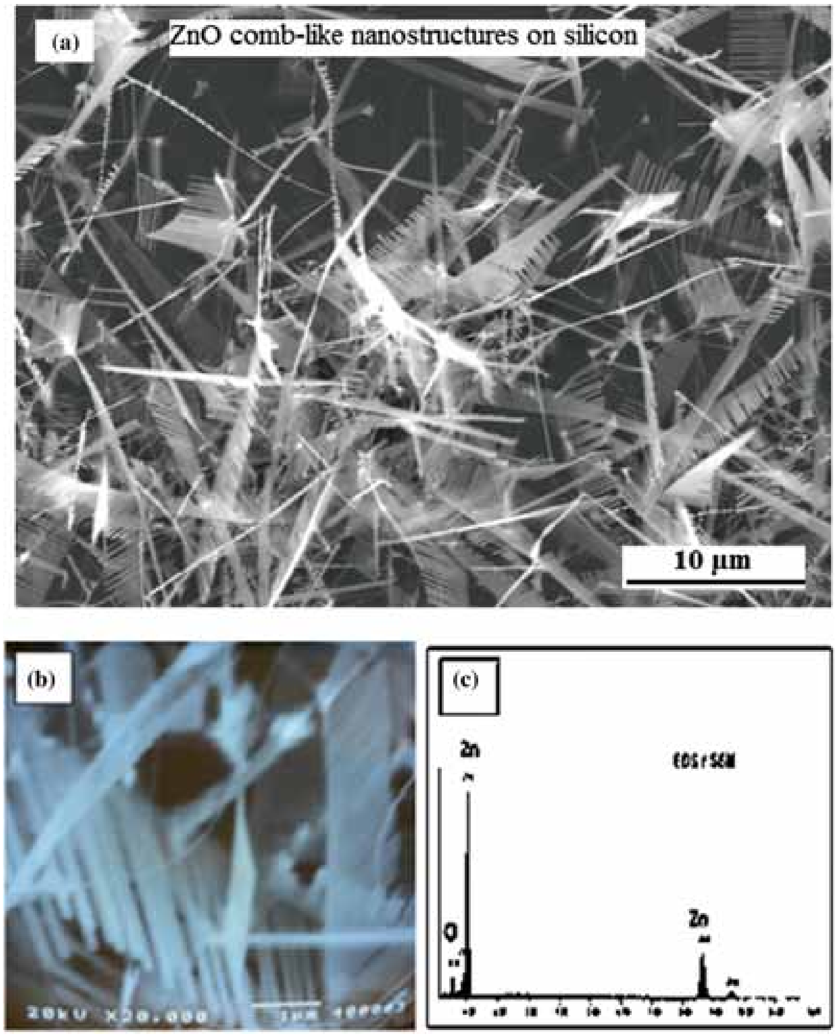

Figure 2. SEM images of ZnO comb-like nanostructures at (**a** and **b**) low and high magnifications; (**c**) energy-dispersive X-ray spectra recorded from a comb-like structure.

The process of initial nucleation includes the diffusion–collision of atoms and the reaction between zinc and oxygen vapours. When the supersaturation increases to a level at which the nuclei were formed, the produced ZnO nuclei grow larger than the critical size. The formed larger ZnO nuclei are providing the energetically favoured sites for the further adsorption of incoming species as the reaction proceeds. Basically, the growth process of comb-like structures reported here could be divided into three steps: (1) the formation of individual nanowire, (2) belt/ribbon-like stem, (3) growth of nanowire branches (teeth of combs) at the side surfaces of the belt/ribbon-like stems as shown in figure 4a. As illustrated in figure 4b, ZnO nanowires may grow first along the [0001] direction (blue-1); then ultrafine nanoteeth grow epitaxially from the original nanowire along the direction (red-2). For group II–VI semiconductors with a wurtzite crystal structure, the polar surface, that is, cation or anion-terminated atomic planes can induce asymmetric growth, leading to the formation of unique nanostructures, such as nanocombs and nanocantilevers [32].

3.4 *Gas sensors fabrication*

The room temperature gas sensing properties of ZnO comb-like nanostructures for H_2S gas at various concentrations were investigated in a static gas sensing setup as shown in figure 5. The chamber with 300 cm^3 was supplied with an inlet rubber plug for gas injection and an outlet for gas exhausts. The sensor films were mounted and fixed through copper wires inside the test chamber. A nylon bag (1 litre in volume) was filled with H_2S gas from a large H_2S gas bottle. The desired concentration of the H_2S gas was achieved by injecting the measured quantity of gas into the chamber. The current, resistance and conductance of the film were monitored and acquired as

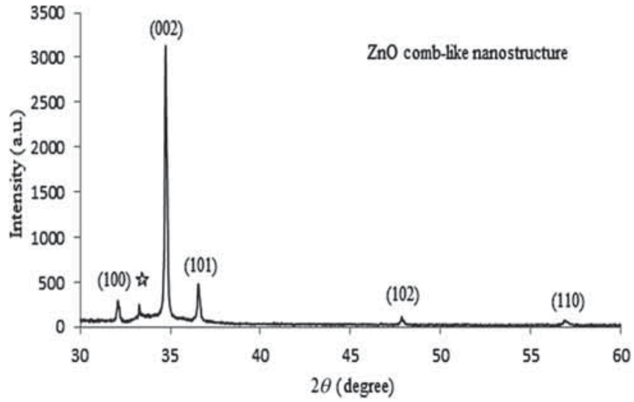

Figure 3. XRD pattern of as-prepared ZnO comb-like nanostructures are grown on silicon substrate. Peak from the Si substrate is marked with an asterisk.

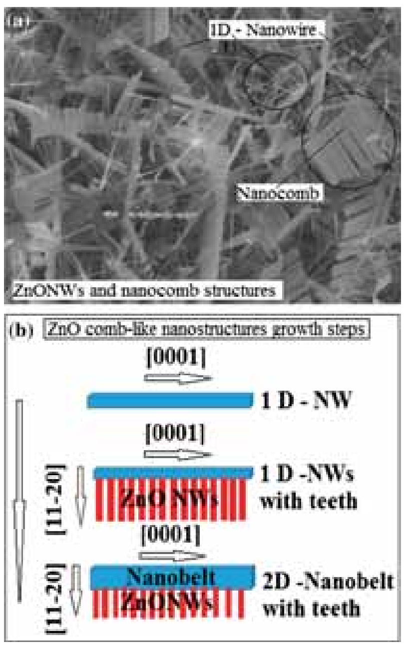

Figure 4. (**a**) SEM images of ZnO nanocomb structures. (**b**) Possible growth mechanisms of ZnO nanocomb.

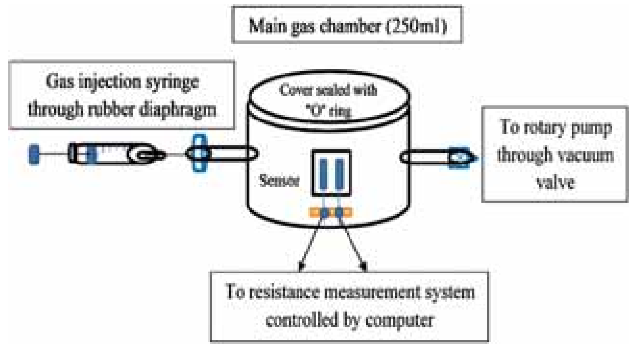

Figure 5. Schematic diagram of static gas sensing setup.

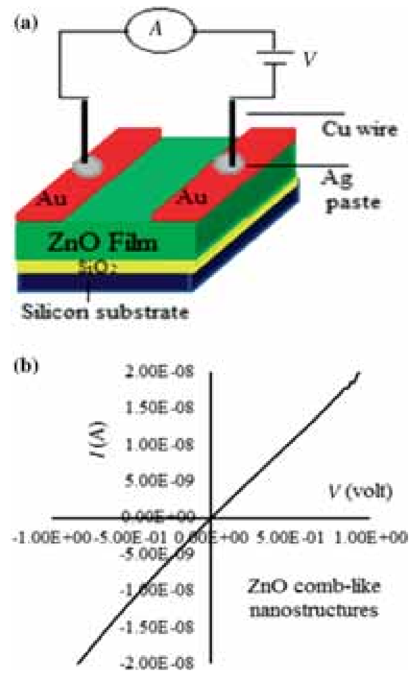

Figure 6. (**a**) Device structure based on silicon substrate; (**b**) $I-V$ characteristic of ZnO comb-like nanostructures in the air and at room temperature.

a function of time using a personal computer equipped with Lab view software. Recovery of the sensors was achieved by the roughing process through the small vacuum pump.

3.4a *Current–voltage (I–V) characteristics*: Sensor measurements were conducted using the gas sensor setup shown in figure 6. The device structure based on silicon covered with silicon dioxide is shown in figure 6a. Two gold electrodes with a thickness of 50 nm was deposited on the surface of the sensing films by using the magnetron sputtering system (Cressington 308R/USA). Then, a very fine copper wire for electrical measurements was connected to the gold electrodes by a silver paste. *I–V* characteristic was measured by using the Keithley 6221 DC and AC current source. Figure 6b shows (*I–V*) a characteristic plot of the as-synthesized ZnO comb-like nanostructures, which were measured in air and at room temperature. The linear behaviour, in this case, under low input power implies ohmic contact.

3.4b *Current vs. time*: The current *vs*. time was measured using Keithley 2182A at constant 1 volt DC. Figure 7a shows current *vs*. time for the ZnO base as a gas sensor tested for H_2S gas at room temperature. The tested data were in the range of 0.1–4 ppm for H_2S concentrations. The sensor can detect H_2S gas at a lower concentration of 100 ppb. As

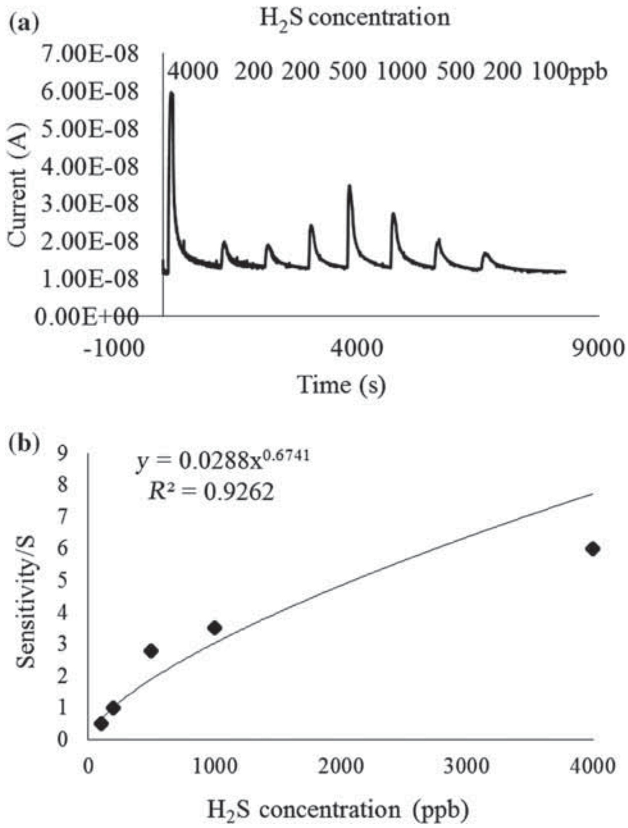

Figure 7. (**a**) Response curves of ZnO comb-like nanostructures towards different concentrations of H_2S at room temperature. (**b**) Corresponding concentration dependence of sensor response.

shown in figure 7b, the sensor films exhibited good sensitivity $[S = (I_a - I_g)/I_a]$ towards H_2S gas at room temperature. The sensitivity increases as a function of gas concentrations. It exhibits a power law dependence on concentration given as (equation 4):

$$S = aC^b, \tag{4}$$

where S is the sensitivity, a is constant, C the gas concentration (ppb) and b is the power law fitting constant. From the fitting curve of figure 7b, the value of $b = 0.6741$ was found. It has been discussed that the power law exponent (b) plays an important role on the characteristics of the surface reaction and determine the dominating species of oxygen O_2^-, O^- and O^{2-}) adsorbates. Moreover, the power law exponent (b) takes the values of 1, 0.5 and 0.25, respectively [33]. Our experimental results in figure 7b were used to calculate the value of the exponent (b), which is around 1. This implies that the dominate oxygen species is considered as O_2^- at room temperature. It is quite consistent with the reported results at temperature $<100°C$, where a single oxygen molecule absorbs only one electron and forms a molecular ionic oxygen species, O_2^- [34].

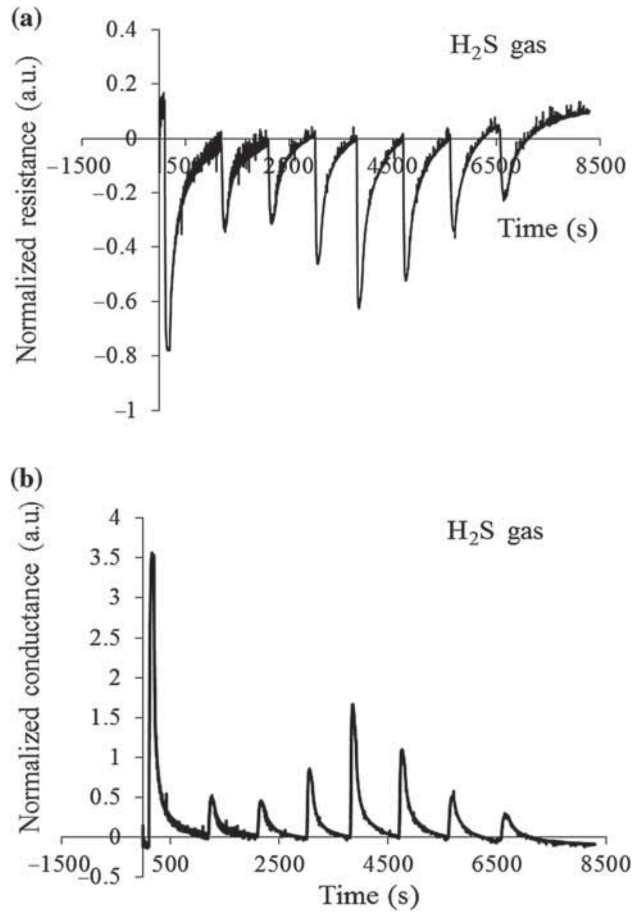

Figure 8. (**a**) Normalized resistance and (**b**) conductance *vs.* time for ZnO comb-like nanostructure with H_2S at RT.

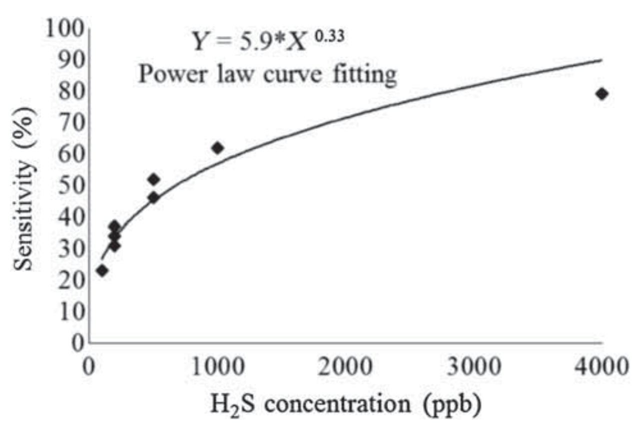

Figure 9. Sensitivity curve for ZnO comb-like nanostructure *vs.* H_2S gas concentrations at room temperature.

3.4c *Resistance and conductance vs. time*: Figure 8a and b shows the measured normalized resistance and conductance as a function of time for the ZnO nanocomb-based gas sensor at different concentrations of H_2S, respectively. When H_2S

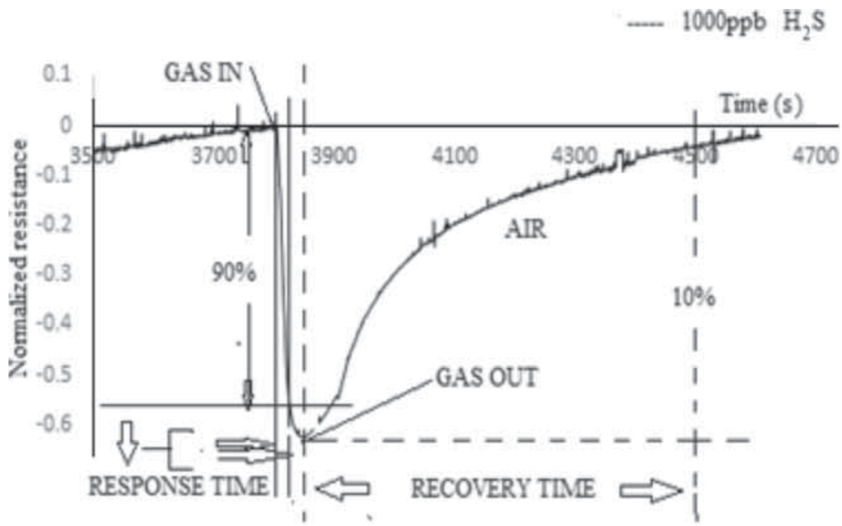

Figure 10. Typical response and recovery characteristics of ZnO comb-like sensor exposed to 1000 ppb of H_2S gas at room temperature.

gas is exposed to the sensor, it would react with the surface oxygen species, which decreased the surface concentration of oxygen ions and increased the electron concentration. This leads to a decrease in the resistance of the sensor and vice versa for the conductance.

3.4d *Sensitivity vs. gas concentration curves fitting*: The sensitivity ($S\%$) of conductometric sensors is defined as $[(R_a - R_g)/R_g \times 100]$, where R_a and R_g are the sensor resistance at air and H_2S gas exposure, respectively. Figure 9 shows the sensitivity curve for ZnO comb-like nanostructure *vs.* H_2S gas concentrations at room temperature. Power law curve fitting to the experimental data (represented by dots) presents an empirical relation between sensitivity and concentration for ZnO nanocomb gas sensor as follows:

$$S(\%) = 5.9C^{0.33}, \tag{5}$$

where $S(\%)$ is the sensitivity and C is the H_2S concentration in ppb. This is considered as an empirical formula for our ZnO nanocomb gas sensor. The sensitivity varied from 20 to 80% as H_2S gas concentrations varied from 100 to 4000 ppb, respectively.

3.4e *Response and recovery times*: Response and recovery times are quite important parameters for gas sensors' performance. The adsorption and desorption of gas molecules on the surface of metal oxides are both thermally activated processes, which cause the response and recovery time to be usually very slow at room temperature. In addition, the presence of water humidity at room temperature could affect the recovery time. Response (recovery) time is defined as the time period needed for the device to undergo resistance changing from 10 (90%) to 90% (10%) of the value in equilibrium upon exposure to an oxidizing (reducing) analyte. These parameters are explained in our typical experimental response curve at H_2S gas concentration of 1000 ppb as shown in figure 10. In the present work, low response time was compared with other nanostructures values shown in table 1. On the other hand, the high recovery time (540 s) compared with the value shown in table 1 for dendrite nanostructure is lower compared with nanorod and nanowire structures. The high recovery time value in our case could be attributed to the presence of humidity in the chamber. Breifly, ZnO comb-like exhibited much higher gas sensing response with rapid response–recovery time compared with ZnO dendrite, nanorod and nanowire structures, which is explained in table 1.

3.4f *H_2S sensing mechanism based on ZnO comb-like vs. dendrite, nanorod and nanowire structures*: Sensing mechanism of n-type metal oxide such as ZnO comb-like based sensors was proposed to be the adsorption and desorption of the gas molecules on the surface of the sensing material. When this structure is exposed to air atmospheric ambient, oxygen molecules adsorb onto the surface of the nanostructures and form a chemisorbed oxygen species such as O^-, O^{2-} and O_2^-. These species act as acceptors by trapping electrons from the ZnO conduction band and creating depletion region on the surface of the comb-like surface according to the following equations [35]:

$$O_2(\text{gas}) \rightarrow O_2(\text{ads}) \tag{6}$$

$$O_2(\text{phys}) + e^- \rightarrow O_2^-(\text{ads}) \tag{7}$$

$$O_2^-(\text{ads}) + e^- \rightarrow 2O^-(\text{ads}) \tag{8}$$

$$O^-(\text{ads}) + e^- \rightarrow O^{2-}(\text{ads}) \tag{9}$$

Table 1. Summarized data for H_2S gas sensor based on ZnO comb-like nanostructure compared with other references at room temperature and 10 ppm only.

Present work		References data			
ZnO comb-like nanostructures		ZnO nanostructures	$[S = R_a/R_g]$/concentration (ppm)	Response/recovery time (s)	Ref. no.
$[S = R_a/R_g]$/concentration (ppm)	Response/recovery time (s)				
$[S = 6]$/4	22/540	ZnO dendrites	$[S = 3.3]$/10	15–20/30–50	[38]
		ZnO nanorods	$[S = 1.28]$/10	150/5000	[39]
		ZnO nanowires	$[S < 1]$/10	250/700	[22]

As previously mentioned, the favourably chemisorbed oxygen species on the surface at room temperature is $[O^-_{2\,(ads)}]$ [36,37]. This adsorbed oxygen species on the ZnO comb-like structures can play a crucial role in sensing H_2S gas. This comb-like structure, having a combination of two different structures (nanowires and nanorods), exhibits unique morphologies. Usually the surface area of a gas sensor has an important influence on the gas response of the sensor and a large surface area typically results in higher gas response. Therefore, owing to these properties of the particular comb-like structure, more oxygen species can adsorb and form much more depletion region through the surface. Thus, more efficient charge transfer takes place on the surface and excellent gas sensing properties can be expected compared with dendrite, nanorod and nanowire structures. In brief, this structure could have specific large surface area and thus increase interaction with gases and finally will expect to possess higher sensitivity compared with dendrites, nanorod and nanowire structures presented in table 1. The evaluation parameters for H_2S gas sensor based on ZnO comb-like nanostructure at room temperature and 4 ppm of H_2S gas concentration are summarized in table 1. The present data are compared with other workers data at room temperatures and 10 ppm of H_2S concentration. The results of this work are quite encouraging when compared with the data reported for dendrite structure [36,38] and also are lower than that reported for nanorod [37,39] and nanowire [22] structures even at higher concentration of H_2S gas.

4. Conclusions

We have successfully synthesized ZnO comb-like nanostructures on the silicon substrate without catalyst via CVD method. The growth mechanism of the ZnO nanostructures is controlled by the VS growth mechanism and attributed to the diffusion-limited process. This facial and direct way for growing pure ZnO comb-like nanostructures on silicon will facilitate the broad applications of these nanostructures. High density and large surface area of ZnO comb-like nanostructures were produced. A gas sensor based on ZnO comb-like nanostructures was successfully fabricated, which exhibited a high response for detection of H_2S at room temperature within a very short response time. The ZnO comb-like nanostructures could be potentially useful for nanodevice due to the uniform structure and perfect geometrical shape.

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