

# Large-scale fabrication of highly sensitive SnO<sub>2</sub> nanowire network gas sensors by single step vapor phase growth

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## ABSTRACT

The SnO<sub>2</sub> nanowire networks were grown on a laser-scribed Al<sub>2</sub>O<sub>3</sub> substrate containing 16 sensor elements with Au electrodes and Pt heaters via a single step vapor phase reaction, and their gas sensing characteristics were studied. The sensor showed a high response to 5 ppm NO<sub>2</sub> ( $R_g/R_a = 1909$ ,  $R_g$ : resistance in gas,  $R_a$ : resistance in air) at the sensor temperature of 141 °C and high and selective responses to 100 ppm C<sub>2</sub>H<sub>5</sub>OH ( $R_a/R_g = 301.8$ – $326.4$ ) at 240 and 296 °C, which demonstrated that high performance SnO<sub>2</sub> nanowire network gas sensors can be fabricated on a large scale through a facile and cost-effective route.

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## 1. Introduction

Recent progress on the synthesis of 1-dimensional (1-D) oxide nanowires (NWs) has triggered a wide range of researches on their functional applications, such as Li-ion batteries, catalyst, optoelectronic devices, and gas sensors [1,2]. From the viewpoint of chemo-resistive gas sensors, oxide semiconductor NW networks are very promising as devices that could deliver high gas response, fast response speed, and excellent thermal stability on account of their high surface area/volume ratio, the chemo-resistive contact between NWs, the low level of agglomeration in the network configuration, and the high crystallinity of NWs [3–9].

The 1-D oxide NW networks can be fabricated into gas sensor devices via various routes. Deposition of a slurry containing crystalline NWs onto the electrode array and subsequent heat treatment is a facile method to fabricate gas sensors through low-temperature processing [10,11]. However, the reproducible deposition of NW networks onto a defined area is relatively difficult to achieve using this method due to the spreading of the slurry solution [6], although alignment of NWs on the electrode

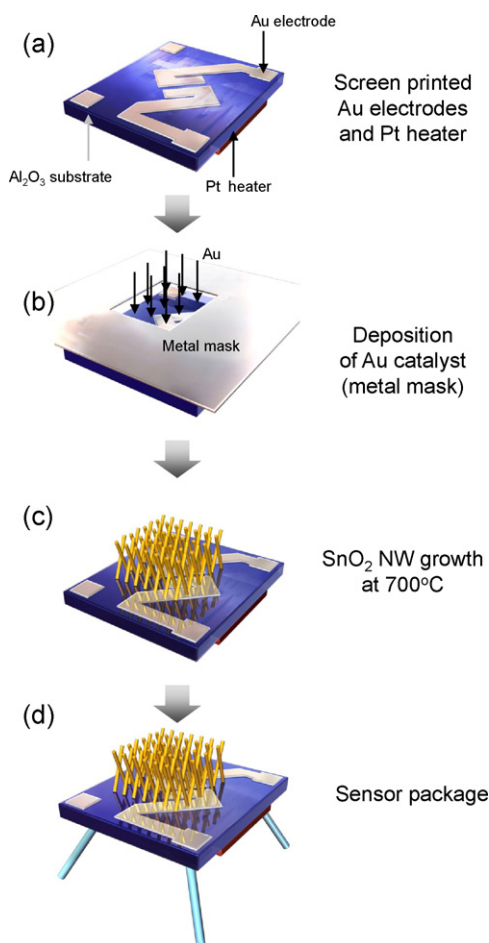
via dielectrophoresis can be employed [12,13]. In particular, the well-defined deposition of slurry becomes more challenging when the sub-millimeter-scale or micrometer-scale deposition area is required to provide a highly integrated sensor or sensor array. Moreover, the sensor stability against vibrational environments can be deteriorated due to the weak adhesion between the highly crystalline NWs and the electrodes.

The direct growth of oxide NWs on metal or metal oxide electrodes can be an alternative method to fabricate well-defined oxide NW gas sensors [14–18]. The strong adhesion between the NWs and electrodes significantly improves not only the electrical contacts between the NWs and electrodes but also the operational stability against mechanical vibration. In addition, highly miniaturized sensor devices can be fabricated by the selective growth of NWs within a defined area. Although gas sensor devices have always been fabricated for research purposes by the direct growth of NWs [19,20], a reliable processing route for the fabrication of SnO<sub>2</sub>-based NW network sensors on a large scale for commercial purposes has been barely explored.

In this contribution, single crystalline SnO<sub>2</sub> NW network sensors are fabricated by the direct and simultaneous growth of NWs on a laser-scribed Al<sub>2</sub>O<sub>3</sub> substrate containing 16 sensor elements with Au electrodes and Pt heaters and the sensor elements are packaged into sensors using a stainless steel holder. The sensors showed highly sensitive and selective detection of NO<sub>2</sub> and C<sub>2</sub>H<sub>5</sub>OH at low and high sensing temperatures, respectively. The large-scale sensor

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**Fig. 1.** Experimental procedure to fabricate  $\text{SnO}_2$  nanowire network gas sensors: (a)  $\text{Al}_2\text{O}_3$  substrate with 2 Au electrodes and Pt heater pattern; (b) deposition of Au catalyst using metal mask onto defined area; (c) direct growth of  $\text{SnO}_2$  nanowires via vapor phase reaction; (d) packaging of sensor element into stainless steel holder. ((c) and (d) show the schematic diagrams of nanowire networks grown on  $\text{Al}_2\text{O}_3$  substrate. Actual images of  $\text{SnO}_2$  nanowire networks are shown in Fig. 4).

fabrication route suggested in the present study can be employed for the commercialization of NW network gas sensors.

## 2. Experimental

The fabrication procedures of in situ grown  $\text{SnO}_2$  NW network gas sensors are shown in Figs. 1 and 2. The 16 sensors (size:  $5\text{ mm} \times 5\text{ mm}$ , thickness:  $0.25\text{ mm}$ ) were fabricated by the growth of  $\text{SnO}_2$  NWs on  $\text{Al}_2\text{O}_3$  substrates (size:  $20\text{ mm} \times 20\text{ mm}$ ,

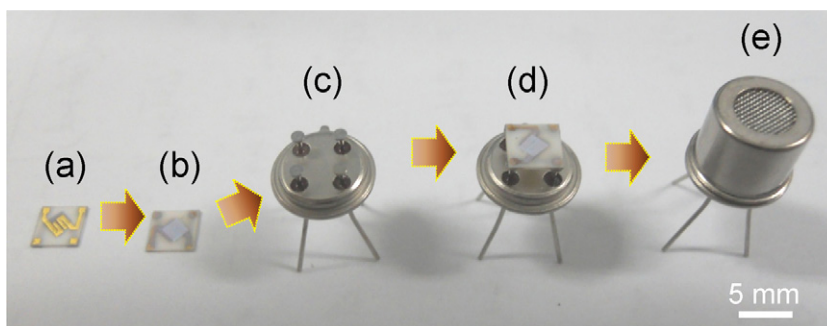
thickness:  $0.25\text{ mm}$ ) with laser scribing. The Au electrodes and Pt heater patterns were formed on the top and bottom sides of the  $\text{Al}_2\text{O}_3$  substrate by screen printing and subsequent heat treatment (Figs. 1a and 2a). The square-shaped Au catalyst layers (thickness:  $3\text{ nm}$ ) were deposited on the regions near the Au electrodes using thermal evaporation (Fig. 1b). The  $\text{SnO}_2$  NWs were grown on Au electrodes via the thermal evaporation of metal in horizontal quartz tube furnaces (inner diameter:  $22\text{ mm}$ , length:  $800\text{ mm}$ ). The Sn metal-loaded  $\text{Al}_2\text{O}_3$  boat was located in the middle of the quartz tube and the alumina substrates ( $20\text{ mm} \times 20\text{ mm}$ ) with catalyst-coated Au electrodes were placed downstream of the quartz tube. After evacuating to  $10^{-2}$  Torr by using a rotary pump, the furnace was heated up to  $700^\circ\text{C}$  and then  $0.5\text{ sccm}$  of  $\text{O}_2$  was introduced as a reactive gas for  $20\text{ min}$ . After growth of  $\text{SnO}_2$  NWs for  $20\text{ min}$  (Figs. 1c and 2b), the furnace was cooled down. The morphology and phase of as-grown  $\text{SnO}_2$  NWs were characterized with field emission scanning electron microscopy (FESEM, Hitachi S-4300) and X-ray diffraction (XRD, Rigaku D/MAX-2500V/PC).

As-grown  $\text{Al}_2\text{O}_3$  substrates were diced into sensor elements, each with a size of  $5\text{ mm} \times 5\text{ mm}$ . Each sensor element was bonded to a stainless steel holder (Figs. 1d and 2c,d) and packaged with a stainless steel mesh cap (Fig. 2e). The sensor temperature of the gas sensor was controlled by modulating the power of the heater underneath the  $\text{Al}_2\text{O}_3$  substrate (Fig. 3a). The sensor temperatures at different heating voltages (heater powers) were measured by contacting a thermocouple to the upper side of the devices (inset in Fig. 3b).

Measurements of the gas sensing characteristics were performed by using a flow-through technique with a constant flow rate of  $500\text{ sccm}$ . The gas sensor was located in a specially designed chamber with a minimized dead volume ( $1.5\text{ cm}^3$ ). Details of the experimental setup for measuring the gas response are shown elsewhere [21]. The gas sensor was heated at  $450^\circ\text{C}$  to stabilize and remove residual contamination. The gas responses to  $0.12\text{--}2.5\text{ ppm NO}_2$ ,  $2.5\text{--}100\text{ ppm C}_2\text{H}_5\text{OH}$ ,  $\text{H}_2$ , and  $\text{CO}$  were measured at  $141\text{--}296^\circ\text{C}$ . The concentration of target gas was adjusted by changing the mixing ratio of the parent gas ( $5\text{ ppm NO}_2$ ,  $100\text{ ppm C}_2\text{H}_5\text{OH}$ ,  $100\text{ ppm H}_2$ , and  $100\text{ ppm CO}$ , all in air balance) and dry synthetic air. The dc 2 probe resistance of the sensor was measured using an electrometer interfaced with a computer.

## 3. Results and discussion

The 16 sensor elements (in a  $4 \times 4$  grid) were fabricated by vapor phase growth of  $\text{SnO}_2$  NW networks on the laser-scribed  $\text{Al}_2\text{O}_3$  substrate (Fig. 4a). The  $\text{SnO}_2$  NW networks were uniformly grown on the rectangular region with an Au catalyst (Fig. 4b). The high magnification SEM image shows that  $\text{SnO}_2$  NWs are several tens of micrometers long and  $30\text{--}100\text{ nm}$  thick (Fig. 4c). The uniform growth of NWs onto 16 different sensor elements demonstrates



**Fig. 2.** The sensor element and package: (a) uncoated  $\text{Al}_2\text{O}_3$  substrate with Au electrodes and heater pattern; (b)  $\text{SnO}_2$  nanowire networks on  $\text{Al}_2\text{O}_3$  substrate; (c–e) the packaging of the sensor element in a stainless steel holder and mesh cap.

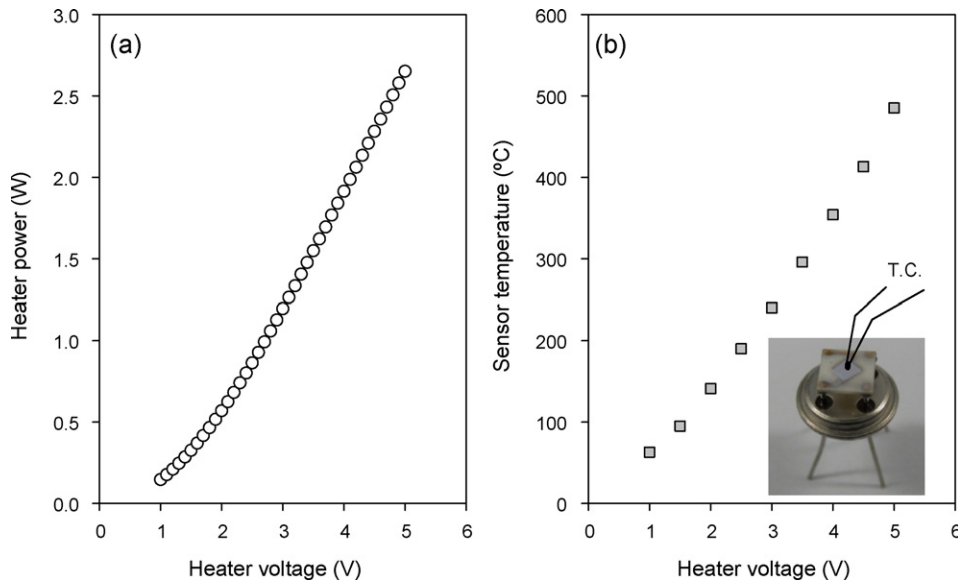


Fig. 3. The heater power and sensor temperature as a function of heater voltage: (a) heater power; (b) sensor temperature.

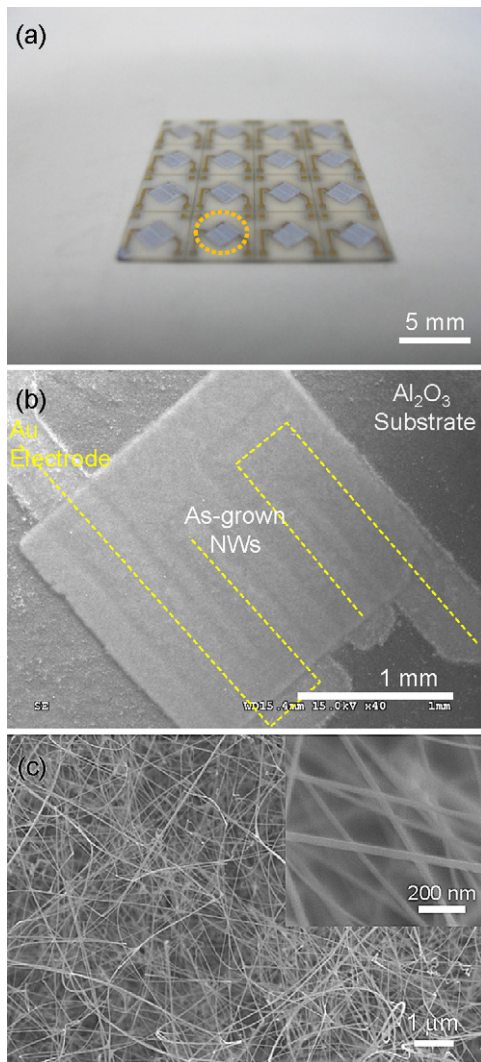


Fig. 4. The sensor elements: (a) 16 sensor elements prepared by one-pot growth of SnO<sub>2</sub> nanowire networks on laser-scribed Al<sub>2</sub>O<sub>3</sub> substrates; (b) the SEM image of one sensor element; (c) the SEM image of SnO<sub>2</sub> nanowire networks.

that this route can be used to fabricate SnO<sub>2</sub> NW network gas sensors on a large scale.

The SnO<sub>2</sub> NW networks showed the resistance increased upon exposure to oxidizing gas (NO<sub>2</sub>) and the resistance decreased upon exposure to reducing gases (CO, H<sub>2</sub>, and C<sub>2</sub>H<sub>5</sub>OH). Accordingly, the  $R_g/R_a$  and  $R_a/R_g$  ( $R_g$ : resistance in gas,  $R_a$ : resistance in air) values were used as the gas responses for oxidizing and reducing gases, respectively. The  $R_g/R_a$  value to 5 ppm NO<sub>2</sub> was as high as 1909 at 141 °C and decreased to 9.1 as the sensor temperature increased to 240 °C (Fig. 5). This tendency is consistent with the variation of the  $R_g/R_a$  values of SnO<sub>2</sub> NW networks to 0.1–5 ppm NO<sub>2</sub> at 100, 200, and 300 °C in the literature [22]. The decrease of NO<sub>2</sub> response above 141 °C can be attributed to the diminishment of NO<sub>2</sub> adsorption on the surface of SnO<sub>2</sub> NWs in the form of NO<sub>2</sub><sup>-</sup>(ads). The response to H<sub>2</sub> ( $R_a/R_g$ ) at 190 °C was low (2.9), but it monotonically increased to 54.6 as the sensor temperature was increased to 296 °C. A similar increase in gas response with sensor temperature was also observed during CO detection. The response to C<sub>2</sub>H<sub>5</sub>OH showed the highest value at 240 °C, which then decreased slightly at 296 °C. Considering the decreasing tendency of responses to CO,

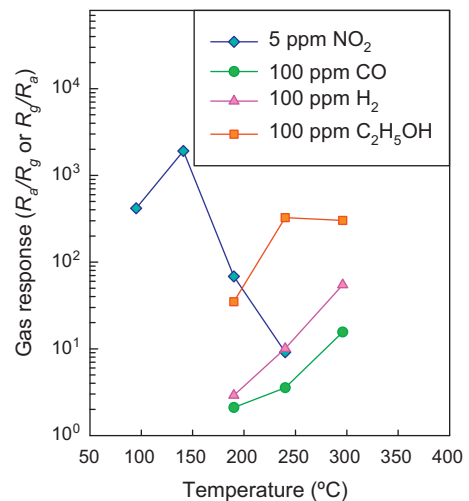
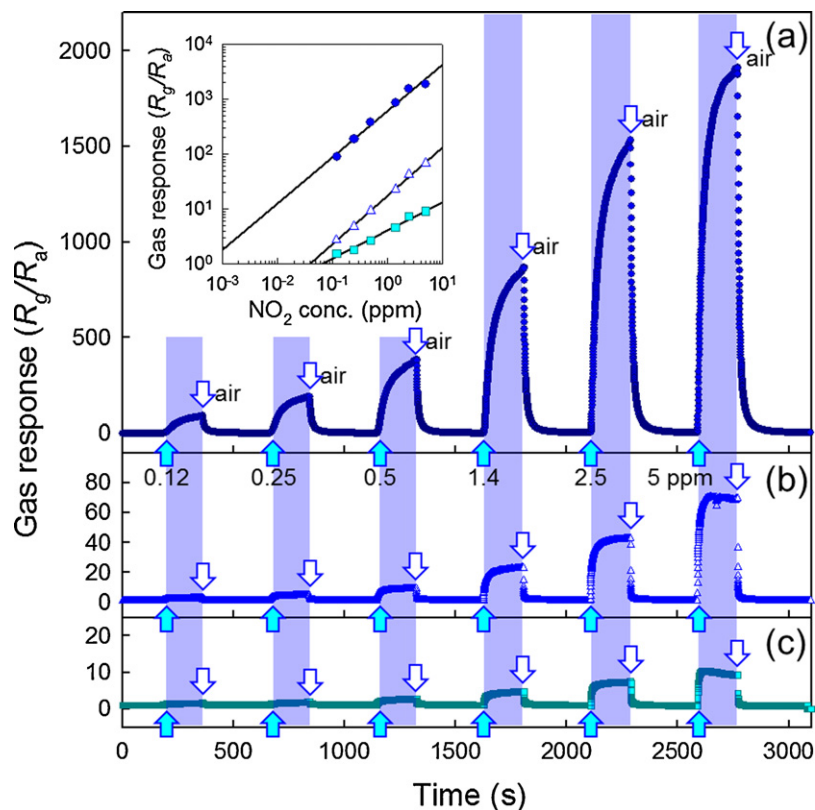


Fig. 5. Gas responses to 5 ppm NO<sub>2</sub>, 100 ppm CO, 100 ppm H<sub>2</sub> and 100 ppm C<sub>2</sub>H<sub>5</sub>OH at various sensing temperatures.



**Fig. 6.** Dynamic sensing transients to 0.12–5 ppm NO<sub>2</sub> at (a) 141 °C, (b) 190 °C, and (c) 240 °C and the corresponding gas responses as a function of NO<sub>2</sub> concentration (inset).

H<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>OH as sensor temperature decreases, the above results suggest that the selective detection of NO<sub>2</sub> and C<sub>2</sub>H<sub>5</sub>OH can be accomplished at low (141 °C) and high (240–296 °C) sensor temperatures, respectively.

The sensing transients to 0.12–5 ppm NO<sub>2</sub> at 141, 190, and 240 °C are shown in Fig. 6. The sensor showed stable sensing and recovery characteristics regardless of sensing temperature. The responses to 0.12–5 ppm NO<sub>2</sub> at the sensor temperature of 141 °C ranged from 89.6 to 1909, which are among the highest values reported in the literature for pristine SnO<sub>2</sub> NW gas sensors [19,22,23]. The NO<sub>2</sub> detection limits of the sensors at 141, 190, and 240 °C were calculated to be 0.62 ppb, 49 ppb, and 93 ppb, respectively, when  $R_g/R_a > 1.2$  was used as the criterion for gas detection (inset in Fig. 6). These results indicate that the present SnO<sub>2</sub> NW network sensors prepared by single step vapor phase growth can detect even sub-ppb levels of NO<sub>2</sub> at the sensor temperature of 141 °C.

Fig. 7 shows the sensing transients to 100 ppm H<sub>2</sub>, CO, and C<sub>2</sub>H<sub>5</sub>OH at 190, 240, and 296 °C. The response to 100 ppm C<sub>2</sub>H<sub>5</sub>OH at 240 °C ( $R_a/R_g = 326.4$ ) is significantly higher than the responses to 100 ppm CO and H<sub>2</sub> (3.6 and 10.1) (Fig. 5 and 7b). As the sensor temperature is increased to 296 °C, the response to 100 ppm C<sub>2</sub>H<sub>5</sub>OH slightly decreases to 301.8, while the responses to 100 ppm CO and H<sub>2</sub> increase to 15.6 and 56.4, respectively (Fig. 5 and 7c). From the sole viewpoint of selective C<sub>2</sub>H<sub>5</sub>OH detection, the operation of the sensor at 240 °C will be more advantageous. However, not only the selectivity but also the responding and recovering speed should be also taken into account to determine the optimum sensor temperature. For this, the times to reach 90% variation in resistance upon exposure to 100 ppm of gas in air, i.e. the 90% response ( $\tau_{res}$ ), and the recovery ( $\tau_{recov}$ ) times were determined. At 240 °C, the  $\tau_{res}$  values to 3 different gases were very short (0.27–0.82 s). This suggests that the analyte gases rapidly diffuse to the sensor surface through the highly porous NW networks and the oxidation of

analyte gas with negatively charged oxygen occurs very quickly. In contrast, the  $\tau_{recov}$  values from the exposure to 100 ppm CO, H<sub>2</sub>, and C<sub>2</sub>H<sub>5</sub>OH were 41.3, 14.3, and 465.8 s, respectively (Fig. 7b). The slow recovery can be attributed to the slow surface reaction regarding the adsorption, dissociation, and ionization of oxygen because the in-diffusion of oxygen can be regarded as relatively fast. The long recovery time (465.8 s) from the exposure to C<sub>2</sub>H<sub>5</sub>OH could be shortened to 108.8 s by increasing the sensor temperature to 296 °C (Fig. 7c), probably due to the thermal promotion of the surface reaction during recovery. Considering the gas response, selectivity and response/recovery speed together, the optimum sensor temperature to detect C<sub>2</sub>H<sub>5</sub>OH was determined to be 296 °C.

The sensing transients to 2.5–100 ppm C<sub>2</sub>H<sub>5</sub>OH at 296 °C are shown in Fig. 8. Stable sensing and recovering characteristics were observed (Fig. 8a). The  $\tau_{res}$  values tended to decrease from 1.27 to 0.42 s as the concentration of C<sub>2</sub>H<sub>5</sub>OH is increased, while the  $\tau_{recov}$  values tended to increase from 30.1 to 108.8 s (Fig. 8c). The low detection limit of C<sub>2</sub>H<sub>5</sub>OH was calculated to be 0.33 ppm when using the  $R_a/R_g > 1.2$  criterion for gas detection (Fig. 8b). This indicates that the present SnO<sub>2</sub> NW networks can be also used to detect sub-ppm levels of C<sub>2</sub>H<sub>5</sub>OH in a selective and reliable manner. The validity of the present large-scale fabrication route based on the selective growth of SnO<sub>2</sub> NW networks can be supported by the fact that the responses to 2.5–100 ppm C<sub>2</sub>H<sub>5</sub>OH in this study are some of the highest values reported in the literature for pristine SnO<sub>2</sub> NW-based gas sensors [24,25].

The SnO<sub>2</sub> NW networks are known to show high gas responses to various reducing gases on account of their high surface area to volume ratio and chemo-resistive contacts between NWs [3–6,13,26,27]. The gas accessibility due to the less-agglomerated network configuration is advantageous not only in achieving rapid in-diffusion of the analyte gas but also in the uniform loading of catalytic additives onto the entire sensor surface, both of which facilitate fast response speeds and the effective control of gas



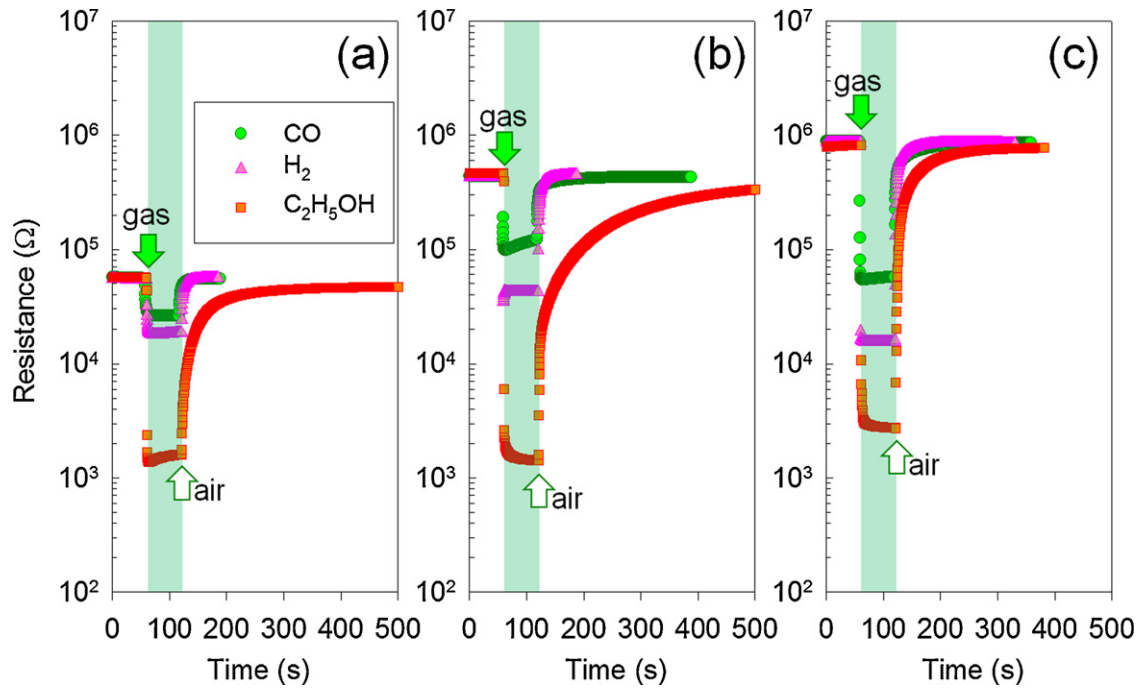


Fig. 7. Dynamic sensing transients to 100 ppm CO, H<sub>2</sub> and C<sub>2</sub>H<sub>5</sub>OH at (a) 190°C, (b) 240°C, and (c) 296°C.

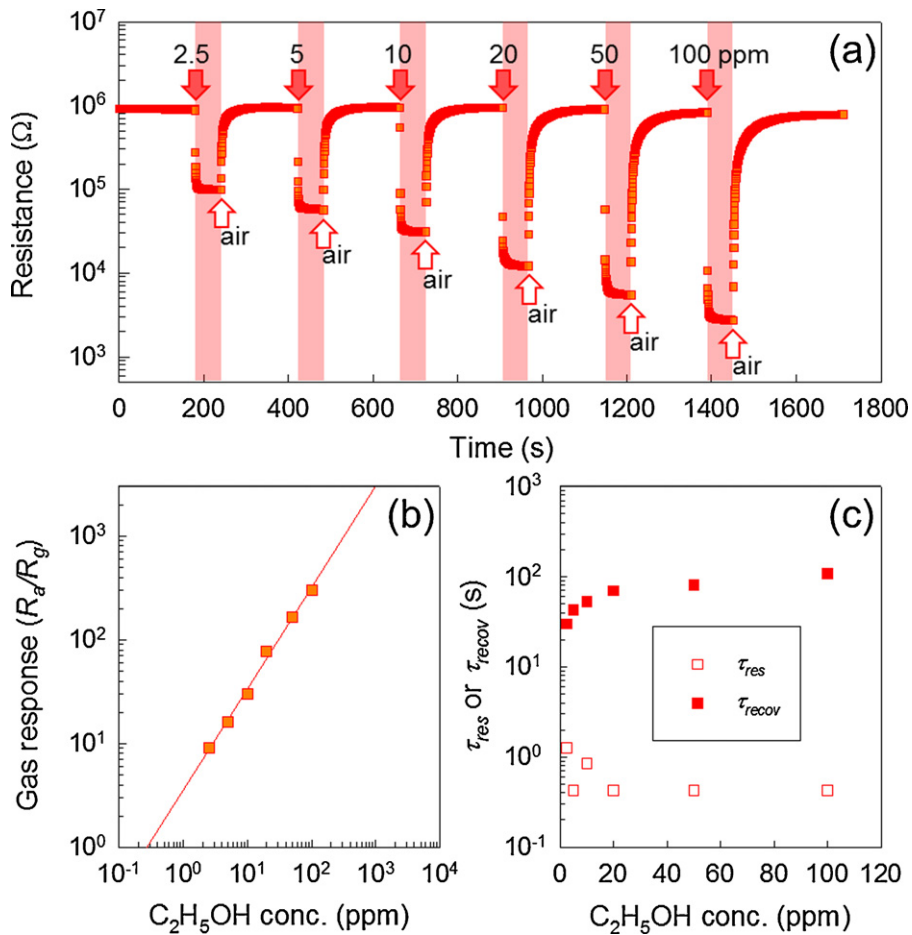


Fig. 8. (a) Dynamic sensing transients to 2.5–100 ppm C<sub>2</sub>H<sub>5</sub>OH at 296°C, (b) gas responses as a function of C<sub>2</sub>H<sub>5</sub>OH concentration, and (c) the 90% response times ( $\tau_{res}$ ) and 90% recovery times ( $\tau_{recov}$ ).

sensing characteristics [8,18,28–31]. It is reported that the gas response of the NW network sensor is higher than that of a single NW sensor [26] and increases with increasing nanowire density [6,13]. This can be attributed to the high chemoresistive variation in the NW contacts. In spite of these advantages, the large-scale production of oxide NW network-based sensors by a vapor phase reaction has been scarcely studied. The advantage of the present sensors is the cost effective and facile processing route by which we can prepare highly crystalline SnO<sub>2</sub> NW network gas sensors on a large scale.

The laser-scribed Al<sub>2</sub>O<sub>3</sub> substrate used in the present study is 2 cm × 2 cm in area and consists of 16 sensor elements (each measuring 5 mm × 5 mm). Considering the screen printing technology used to form Au electrodes and heater patterns and the uniform temperature zone of the conventional box furnace, the area of the laser-scribed Al<sub>2</sub>O<sub>3</sub> substrate for one-pot growth of SnO<sub>2</sub> NWs can be simply widened to an area of 20 cm × 20 cm and the down-sizing of single sensor elements to 1 mm × 1 mm is not a difficult task. This means that 40,000 NW network sensors can be fabricated by a one-pot NW growth reaction, which demonstrates the potential for the mass production of reliable NW network gas sensors. Moreover, the selective growth of SnO<sub>2</sub> NW networks using patterned catalysts can also be applied to the suspended micro-electrode and heater patterns fabricated by micro-electromechanical systems (MEMS) in order to reduce the heater power consumption and to increase the integrity of sensors.

#### 4. Conclusions

A facile route to fabricate single crystalline SnO<sub>2</sub> NW network gas sensors on a large scale has been suggested. The SnO<sub>2</sub> NW networks were grown on 16 different sensor elements via a single step vapor phase reaction using laser-scribed Al<sub>2</sub>O<sub>3</sub> substrates with patterned Au catalyst layers, Au electrodes, and Pt heaters. The sensor showed highly sensitive and selective detection of NO<sub>2</sub> and C<sub>2</sub>H<sub>5</sub>OH at 141 and 240–296 °C, respectively. The present method provides a high-throughput fabrication method of SnO<sub>2</sub> NW network gas sensors using a vapor phase reaction.

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