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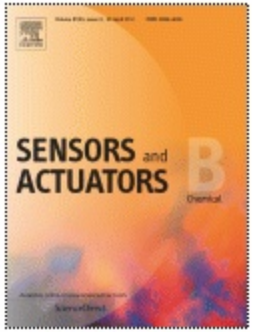

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Gas sensing properties of SnO_2 nanowires on micro-heater

In-Sung Hwang[a], Eui-Bok Lee[b], Sun-Jung Kim[a], Joong-Ki Choi[a], Jung-Ho Cha[c], Ho-Jun Lee[c], Byeong-Kwon Ju[b], Jong-Heun Lee[a,*]

[a] Department of Materials Science and Engineering, Korea University, Anam-Dong, Sungbuk-Gu, Seoul 136-713, Republic of Korea
[b] Display & Nanosystem Lab., College of Engineering, Korea University, Seoul 136-713, Republic of Korea
[c] Seju Engineering Co. Ltd., Daejeon 305-801, Republic of Korea

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ABSTRACT

The SnO_2 nanowires (NWs) network gas sensors were fabricated on a micro-electrode and heater suspended in a cavity. The sensors showed selective detection to C_2H_5OH at a heater power during sensor operation as low as 30–40 mW. The gas response and response speed of the SnO_2 NWs sensor to 100 ppm C_2H_5OH were 4.6- and 4.7-fold greater, respectively, than those of the SnO_2 nanoparticles (NPs) sensor with the same electrode geometry. The reasons for these enhanced gas sensing characteristics are discussed in relation to the sensing materials and sensor structures.

1. Introduction

SnO_2, an n-type semiconductor with a large band gap, is a representative gas sensing material. For gas sensor applications, high gas response, fast response kinetics, small size and lower power consumption are required. The gas response can be enhanced significantly when the particle size becomes comparable to the nanometer-scale thickness of the electron depletion layer [1]. However, it is very difficult to avoid agglomeration between nanoparticles (NPs) due to the strong van der Waals attraction [2,3]. This hampers the diffusion of the target gas toward the entire sensing surface, which counteracts the gas response and retards the response kinetics [4]. By contrast, the metal oxide nanowires (NWs) network shows a less agglomerated configuration without scarifying high surface area [5,6], thereby facilitating both a high gas response and fast response kinetics. Moreover, the good crystallinity of NWs enhances the stability in gas sensing reaction.

The NWs gas sensors can be fabricated either by an individual NW or by NWs network. The former provides a fundamental understanding of the gas sensing mechanism. However, the fabrication of an individual NW sensor is relatively difficult and the sensor resistance can vary significantly according to the diameter of the individual NW. The resistance of NWs network sensor is reproducible due to the averaging effect [7,8], while the junction effect between NWs can further enhance the gas response [9,10].

The size and power consumption of gas sensors are best minimized by employing the suspended, and thus thermally isolated, micro-hot plate (μHP) structure using the fabrication technologies developed for micro-electromechanical systems (MEMS) devices. Thus far, thin films [11–13], NPs [13] and nanocrystalline microhollow spheres [14] have been coated on the suspended μHP. Recently, gas sensors using an individual SnO_2 NW [15] and an individual SnO_2 nanobelt [16] on a thermally isolated μHP with embedded heater have been reported. Ryu et al. [17] fabricated In_2O_3 NW gas sensor by coating a dilute solution containing NWs on μHP. The individual NW was directly contacted between two electrodes because the length of the In_2O_3 NW was longer than the electrode spacing (3 μm). The single NW gas sensors using μHP showed an excellent gas sensing performance with low power consumption. However, expensive and sophisticated processes such as E-beam lithography and FIB are required to align and pattern a single NW on the defined electrodes.

In this contribution, we coated SnO_2 NWs network on the suspended micro-electrodes and heater patterns. To the best of our knowledge, this is the first report of a SnO_2 NWs network sensor fabricated on suspended micro-electrodes. The MEMS-based gas sensors in the literature usually employ a membrane to insulate the electrodes from the embedded heater or to sustain the sensor structure [11–17]. For effective and rapid diffusion, the co-planar configuration of suspended micro-electrode and heater (μEH) without membrane were used in this study. The gas sensing characteristics of the SnO_2 NWs network sensor on μEH were compared to those of the SnO_2 NPs sensor on the same electrode geometry.

* Corresponding author. Tel.: +82 2 3290 3282; fax: +82 2 928 3584.
E-mail address: jongheun@korea.ac.kr (J.-H. Lee).

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2. Experimental

2.1. Synthesis of nanowires (NWs) and nanoparticles (NPs)

The SnO_2 NWs were synthesized by thermal evaporation using Sn metal powder (99.999%). The Au (30 Å)-coated Si substrate was placed downstream of the source material in a quartz tube (inner diameter: 28 mm, length: 800 mm). The pressure of the processing tube was maintained at about 10^{-2} Torr by mechanical pumping to keep low gas pressure. NWs were grown at 750 °C for 20 min with an O_2 flow rate of 0.5 sccm. For comparison of gas sensing characteristics, the SnO_2 NPs were also prepared by the hydrothermal method suggested by Baik et al. [18]. Hydrated tin oxide precipitates were prepared by mixing aqueous solutions of ammonium bicarbonate (NH_4HCO_3, ≥95%, Junsei Chemical Co. Ltd., Japan) and tin chloride pentahydrate ($SnCl_4{\cdot}5H_2O$, GR, Kanto Chemical Co. Inc., Japan). After washing the precipitate with distilled water using a centrifuge, hydrated tin oxide gel was suspended in an aqueous ammonia solution (pH 10.5). The suspension was transferred to a Teflon-lined stainless steel autoclave and treated hydrothermally at 200 °C for 3 h to afford a clear and homogeneous SnO_2 sol solution. The SnO_2 NPs were prepared by drying SnO_2 sols at 100 °C and subsequent calcination at 600 °C for 2 h in air.

2.2. Characterization of sensing materials

The SnO_2 NWs and NPs were characterized and analyzed by X-ray diffraction (XRD, Bruker D/MAS-2500 V, Cu $K\alpha_1$ radiation, $\lambda = 0.15406$ nm), scanning electronic microscopy (SEM, Hitachi SE-4200, 30 kV), transmission electron microscopy (TEM, JEOL JEM-3011 at 300 kV), and energy-dispersive spectroscopy (EDS).

2.3. Sensor fabrication

A SiO_2 insulating layer (2 μm) was grown by thermal oxidation at 1100 °C (Fig. 1(a)). The window and electrodes were patterned by photolithography process (Fig. 1(b)). The Ti (50 nm)/Au (100 nm) seed layer was deposited by E-beam evaporator and Pt (5000 nm) electrodes were made by electrochemical deposition and lift-off process (Fig. 1(c)). The cavity was etched by KOH solutions at 80 °C for 2 h (Fig. 1(d)). As-synthesized NWs or NPs were dispersed in a solution of deionized water and isopropyl alcohol by ultrasonic treatment. The NWs- or NPs-containing solutions were dried and then the products were mixed with ethyl cellulose and alpha terpineol to control the viscosity of the slurry. The NWs or NPs were deposited on the suspended μEH by dropping the slurry. (Fig. 1(e)) To remove any organic compounds, the sensors were heat-treated at the heater voltage of 0.8 V (~500 °C) for 2 h in air. After heat treatment, the NWs and NPs were well adhered on μEH and remain rigid even under vibration.

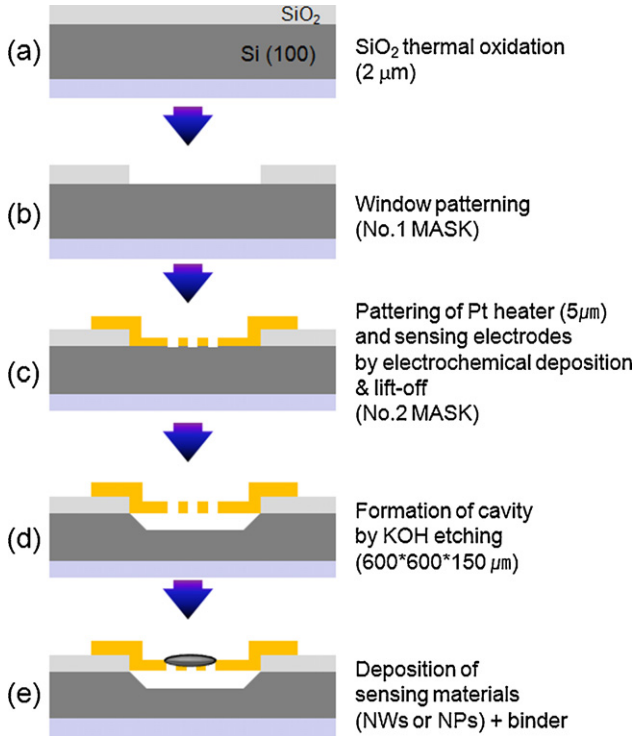

Fig. 1. Experimental procedures to fabricate micro-heater-based gas sensor.

2.4. Measurement of gas sensing characteristics

The gas sensing characteristics were measured in the quartz tube (inner diameter: 38 mm, length: 800 mm) at room temperature. Dry air at a constant flow rate of 500 sccm and the target gas were alternately switched on by 4-way valves. The heater voltage was supplied by dual output DC power source (Agilent E3646A). Fig. 2(a) shows the equivalent circuits for gas sensor measurement. One electrode in the heater pattern was used as the common ground (electrodes 3). The resistance (R_S) of the NWs or NPs and a reference resistance (R_L = 100 kΩ to 1 MΩ) were serially connected to the sensing electrode 2 and the voltage (V_{CC} = 5 V) was applied to between electrodes 2 and 3. A heater voltage (V_h) ranging from 0.4 to 0.8 V was applied between 1 and 3. The sensor signal (V_{out}) was attained from the potential drop across the sensor resistance under air or target gas exposure. The sensor output, V_{out}, can be

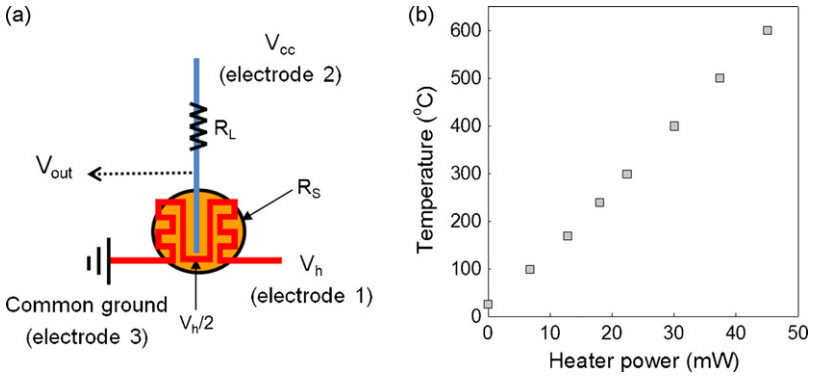

Fig. 2. (a) Equivalent circuit for gas sensor measurement and (b) sensor heater temperature as a function of the applied heater power.

expressed by the following equation [19]:

$$V_{out} = R_S \frac{V_{CC} - (V_h/2)}{R_S + R_L} + \frac{V_h}{2} \quad (1)$$

The sensor resistance can be calculated from the V_{out} value:

$$R_S = R_L \frac{V_{out} - (V_h/2)}{V_{CC} - V_{out}} \quad (2)$$

The gas responses (R_a/R_g) were measured by comparing the sensor resistance in the target gases (R_g) with that in high-purity air (R_a). The sensor temperature as a function of heater power is given in Fig. 2(b).

3. Results and discussion

3.1. Sensing materials and sensor structures

The as-grown NWs were ~100 nm in diameter and ~10 μm long and showed a rutile structure with $a = b = 4.734$ Å and $c = 3.185$ Å (JCPDS #41-1445). The selective-area electron diffraction (SAED) pattern confirmed the single crystalline structure of the SnO_2 NWs. The morphology and characteristics of the as-grown NWs have been reported elsewhere [10]. Fig. 3(a) shows an optical image of the micro-heater (electrodes 1 and 3) and sensing electrode (electrode 1) floating in the cavity with a 600 μm × 600 μm × 150 μm volume. The NWs and NPs were uniformly coated on the micro-heater and electrode (Fig. 3(b) and (c)). The NPs (size: ~50 nm) were significantly aggregated (inset in Fig. 3(c)). In contrast, the high magnification image of the NWs network show less aggregated configuration (inset in Fig. 3(b)). Note the different magnifications of the insets in Fig. 3(b) and (c).

3.2. Heater characteristics

The micro-heater without sensing layer was placed within the furnace and the resistance was measured as a function of ambient temperature. The heater resistance was also estimated with varying heater voltages. The sensor temperature was evaluated by cross-checking the above two signals. The sensor temperatures were ~300 and ~500 °C when 0.6 and 0.8 V were applied to heater, respectively. The heater power required to attain the sensor temperature (250–500 °C) was as small as 20–40 mW (Fig. 2(b)).

3.3. Gas sensing characteristics

The gas responses to 100 ppm C_2H_5OH, 100 ppm CO, 100 ppm C_3H_8 and 5 ppm H_2S were measured at $V_h = 0.6$ V (heater power: 22.4 mW, sensing temperature: ~300 °C) and $V_h = 0.8$ V (heater power: 40 mW, sensing temperature: ~500 °C) (Fig. 4). At $V_h = 0.6$ V, the gas response to 100 ppm C_2H_5OH was 19.6 while those to other gases were very low (1.6–2.4) (Fig. 4(a)). As V_h was increased to 0.8 V, the response to 100 ppm C_2H_5OH was enhanced to 26.2, whereas those to other gases remain similar (1.3–2.0) (Fig. 4(a)). In order to evaluate the gas response speed, the times to reach 90% variation in sensor voltage (V_{out}) upon exposure to gas and air were defined as the 90% response time ($\tau_{res\text{-}90}$) and 90% recovery time ($\tau_{recov\text{-}90}$), respectively, and were obtained from the gas sensing response transients. The very short $\tau_{res\text{-}90}$ values (2–4 s) at $V_h = 0.6$ V (T = ~300 °C) were further shortened to 1–2 s by increasing V_h to 0.8 V (T = ~500 °C) (Fig. 4(b)). The $\tau_{recov\text{-}90}$ values at $V_h = 0.8$ V (2.5–3.5 s) were also significantly shorter than those at $V_h = 0.6$ V (3–6 s) (Fig. 4(c)). The faster response and recovery kinetics at the higher V_h were attributed to the enhancement of gas diffusion and surface reaction at the higher sensor temperature. In terms of the selective detection of C_2H_5OH and gas

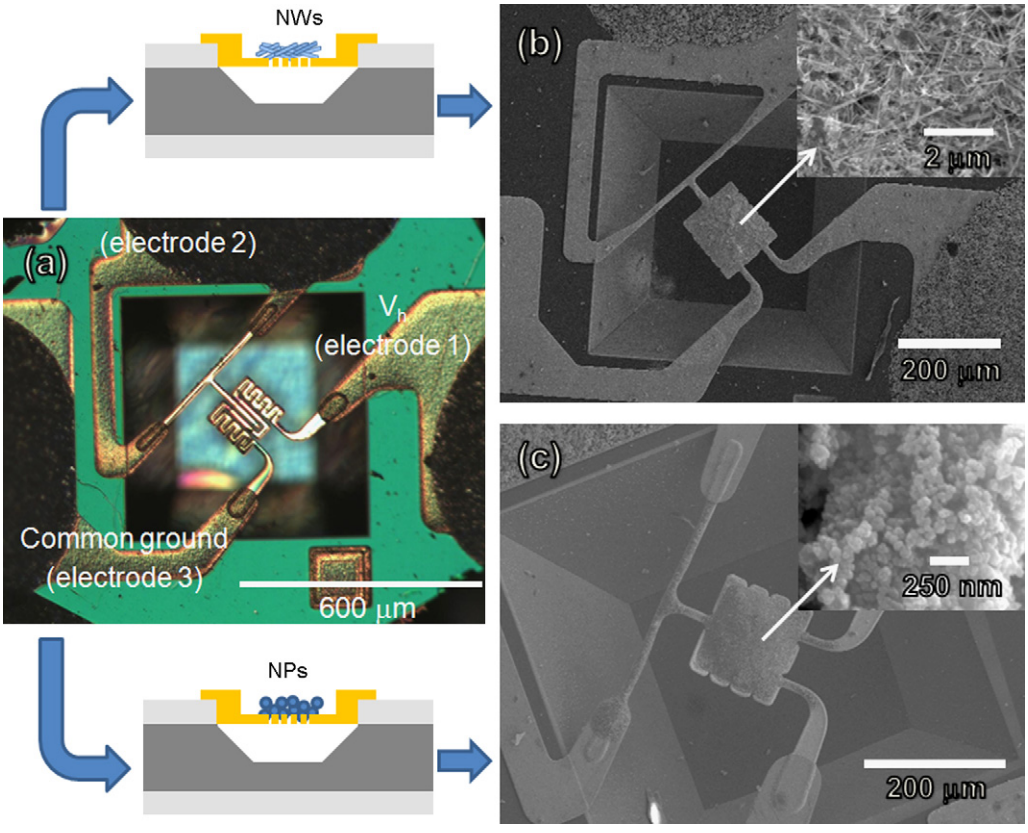

Fig. 3. Images of fabricated gas sensors: (a) optical images of micro-heater and sensing electrode, (b) SEM image of NWs gas sensor, and (c) SEM image of NPs gas sensor.

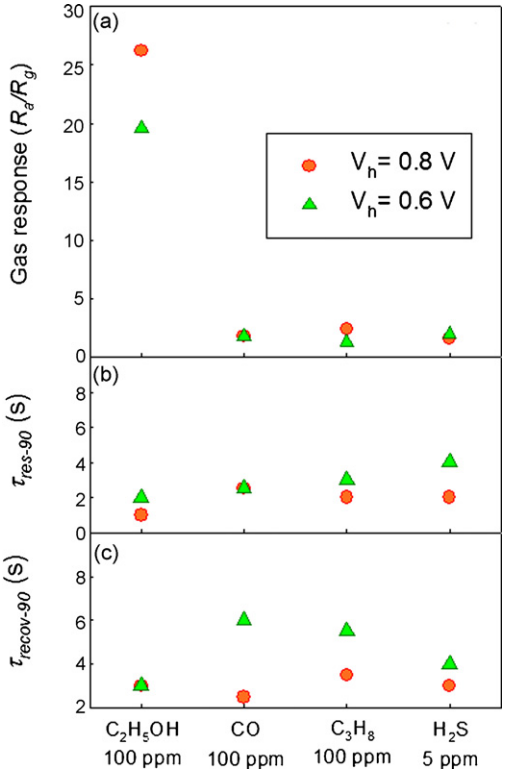

Fig. 4. (a) Gas responses (R_a/R_g), (b) 90% response times (τ_{res-90}), and (c) 90% recovery times ($\tau_{recov-90}$) of the SnO_2 NWs sensor to 100 ppm C_2H_5OH, 100 ppm CO, 100 ppm C_3H_8 and 5 ppm H_2S at the heater voltage (V_h) of 0.6 and 0.8 V.

response speed, a higher heater voltage (V_h = 0.8 V) was more advantageous.

To investigate the role of SnO_2 NWs in the gas sensing reaction, the NPs sensor with the same electrode geometry was also fabricated. Fig. 5(a) shows the dynamic gas sensing transients of NWs and NPs sensors upon exposure to 10–100 ppm C_2H_5OH. The sensor signals were very stable and reproducible. From the sensing transients, the gas response, the τ_{res-90} values and $\tau_{recov-90}$ values were estimated and the results were shown in Fig. 6. The τ_{res-90} values of the NPs sensor were very short (4.7–7.8 s) (Fig. 6(b)) and significantly shorter than the reported values of thick film sensors using agglomerated particles (30–200 s) [20–22]. This result can be explained in part by the convenient gas diffusion due to relatively thin and small-area sensing layer in the present study and indicates that the MEMS-based integrated sensors are advantageous to enhance gas response speed.

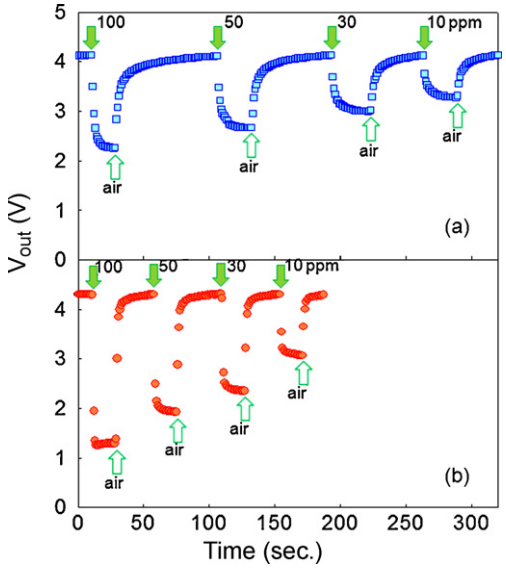

Fig. 5. C_2H_5OH sensing transients of SnO_2 NWs and NPs sensors at V_h = 0.8 V.

The gas responses of the NWs sensor to 10–100 ppm C_2H_5OH were 1.7–4.6-fold higher than those of the NPs sensors (Fig. 6(a)). Moreover, the τ_{res-90} values of the former (1–2.5 s) were significantly shorter than those of the latter (4.7–7.8 s) (Fig. 6(b)), demonstrating the high sensitivity and fast response of the NWs gas sensors. In our previous study [23], we prepared SnO_2 thick film sensors using dense microspheres containing primary NPs and porous hierarchical nanostructures. At 400 °C, the hierarchical nanostructures showed 1.7–3.3-fold higher gas response and 50–90-fold faster response speed to 10–50 ppm C_2H_5OH than dense microspheres. Benkstein and Semancik [13] reported that the CH_3OH responses of mesoporous TiO_2 NPs film on μHP are more than double than those of dense TiO_2 thin film prepared by chemical vapor deposition. The response time of mesoporous TiO_2 NPs film on μHP was as short as 5 s. When the diffusion of analyte gas through the dense agglomerated structures is sluggish and restricted, the gas sensing reaction is slowed and occurs only near the surface region of secondary particles [4]. This decreases the gas response speed and gas response. The enhancement of both gas response and gas response speed achieved in the present study by using NWs can therefore be understood in the same viewpoint.

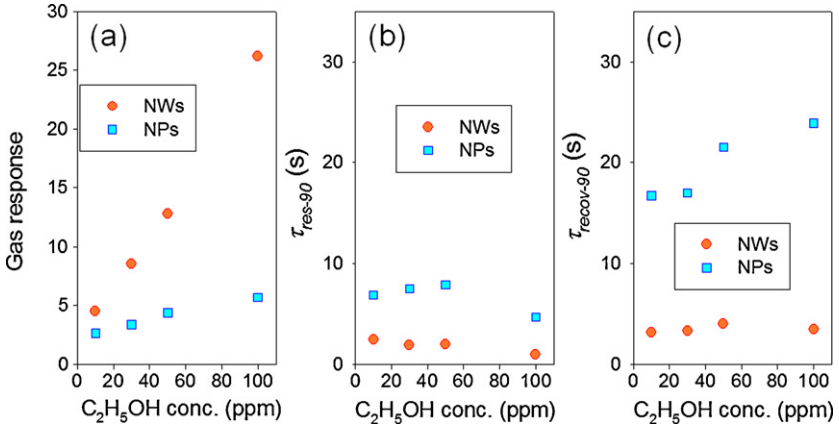

Fig. 6. (a) Gas responses (R_a/R_g), (b) 90% response times (τ_{res-90}), and (c) 90% recovery times ($\tau_{recov-90}$) of SnO_2 NWs and NPs sensors to 10–100 ppm C_2H_5OH at V_h = 0.8 V.

The $\tau_{recov\text{-}90}$ values of both the NWs and NPs sensors were 2–3-fold longer than the $\tau_{res\text{-}90}$ values (Fig. 6(c)). In the sensing of reducing gases using n-type oxide semiconductors, the recovery times are usually longer than the response time [10,13,22,24,25]. At 200–400 °C, the n-type SnO_2 semiconductor formed an electron depletion layer near the surface by adsorption of negatively charged oxygen (O^- or O^{2-}). The fast response reaction when using well-dispersed nanostructures revealed the rapidity of the oxidation reaction between the reducing gas and the negatively charged oxygen. In contrast, substantial recovery time is still required even at very fast response time (that is, rapid gas diffusion). The recovery reaction requires the following reactions: (1) the diffusion of oxygen gas toward the sensing layer, (2) the dissociation of molecular oxygen, and (3) the formation of adsorbed negatively charged oxygen. Assuming rapid gas diffusion from the short response time, the elongated recovery time can be attributed to the sluggish surface reaction to form the negatively charged oxygen adsorption. Nevertheless, the $\tau_{recov\text{-}90}$ values of the NWs sensor are some of the shortest values in the literature, indicating the promising potential of the NWs sensors on μEH to provide both rapid recovery and quick response.

Various kinds of MEMS-based gas sensor have been proposed. In the representative configuration, the gas sensors are fabricated on the micro-machined hot plates with the embedded heater. For this, Si_3N_4 and SiO_2 were used as the membrane and the sensing layers were deposited on the top of the membrane layer [11,13–17]. In the present study, the suspended micro-machined hot electrode and heater were fabricated without any membrane structure. The gas sensing reaction could occur rapidly over the entire surface of the thin and porous NWs network, through both the upper part of the sensing layer and the lower part exposed toward the cavity, which enhanced the gas response and response speed. Therefore, the NWs network sensor on μEH in the present study promises high gas response, fast response and recovery kinetics, small heater power, and integrated structures.

4. Conclusions

The SnO_2 NWs network sensors were fabricated on a microelectrode and heater (μEH) suspended in a cavity without membrane structure. The NWs sensors selectively detected C_2H_5OH. The gas responses of the NWs-based gas sensors to 10–100 ppm C_2H_5OH were 1.7–4.6-fold higher than those of NPs sensors with the same electrode geometry. Moreover, the NWs sensor on μEH showed ultra fast response (90% response time: 1–2 s) and recovery (90% recovery time: 2.5–3.5 s) to C_2H_5OH, as well as low heater power consumption (30–40 mW). These enhanced gas sensing characteristics were attributed to the rapid and effective diffusion of analyte gases through the porous SnO_2 NWs network coated on the thermally isolated and integrated μEH.

Acknowledgements

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Biographies

In-Sung Hwang studied materials science and engineering and received his B.S. from Kumoh National University, Korea, in 2004. In 2006, he received his M.S. degree from Korea University. He is currently studying for a Ph.D. at Korea University. His research interest is oxide nanostructure-based electronic devices.

Eui-bok Lee received his B.S. and M.S. degrees from the Department of Electronic Materials Engineering, Kwangwoon University in 2004 and 2006, respectively. He is currently a Ph.D. candidate in the Department of Electrical Engineering, Korea University. He has been involved in the development of the next generation memories such as PRAM in the Nano Device Reach Center, Korea Institute of Science and Technology(KIST), Seoul, Korea. His main interests are Nonvolatile memory technology and micro-machined devices.

Sun-Jung Kim studied materials science and engineering and received his B.S. and M.S. degrees in 2006 and 2008, respectively, at Korea University in Korea. He is currently studying for a Ph.D. degree at Korea University. His research interests are oxide nanostructures for chemical sensor applications and the combinatorial design of gas sensing materials.

Joong-Ki Choi studied materials science and engineering and received his B.S. degree from Korea University in 2009. He is currently a master course student at Korea University. His research topic is oxide nanowire gas sensors.

Jung-Ho Cha received his B.S. and M.S. degrees from the Department of Semiconductor Science, Dongguk University in 2000 and 2002, respectively. Currently, he works at Seju Engineering Co. Ltd. as manager of the research & development center. His research interests include MEMS-based gas sensors.

Ho-Jun Lee received his B.S. degree from the Department of Telecommunication Engineering, Korea Aerospace University in 1986, and his M.S. and Ph.D. degrees from the Department of Electrical Engineering, Korea Advanced Institute of Science and Technology (KAIST) in 1991 and 1996, respectively. Currently, he works at Seju Engineering Co. Ltd. as chief of the research & development center. His research interests include MEMS-based gas sensors.

Byeong-Kwon Ju received his M.E. in electronics engineering from the University of Seoul in 1988 and a Ph.D. in semiconductor engineering from Korea University in 1995. In 1988, he joined the Korea Institute of Science and Technology (KIST), Seoul, where he was engaged in the development of mainly silicon micro-machining and microsensors as a principal research scientist. In 1996, he spent 6 months as a visiting research fellow at the Microelectronics Centre, University of South Australia, Australia. Since 2005, he has been an associate professor of Korea University with his main interest in flexible electronics (OLED, OTFT), field emission devices, MEMS (Bio and RF) and carbon nanotube-based nanosystems.

Jong-Heun Lee has been a Professor at Korea University since 2008. He received his B.S., M.S., and Ph.D. degrees from Seoul National University in 1987, 1989, and 1993, respectively. Between 1993 and 1999, he developed automotive air-fuel-ratio sensors at the Samsung Advanced Institute of Technology. He was a Science and Technology Agency of Japan (STA) fellow at the National Institute for Research in Inorganic Materials (currently NIMS, Japan) from 1999 to 2000, a research professor at Seoul National University from 2000 to 2003, and an associate professor at Korea University from 2003 to 2008. His current research interests include chemical sensors, functional nanostructures, and solid oxide electrolytes.