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A facile fabrication of semiconductor nanowires gas sensor using PDMS patterning and solution deposition☆

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Abstract

This paper suggests a simple and cost-effective method for fabricating nanowire (NW) gas sensors. Oxide NWs were deposited uniformly over a defined electrode area by coating with a solution containing NWs after patterning with polydimethylsiloxane (PDMS). The hydrophobic PDMS guide wall prevented outstretching of the deposited solution, which allowed the density of the NWs in the sensor to be manipulated conveniently. The high-density NWs sensor enhanced the gas sensitivity toward CO, C_3H_8, C_2H_5OH, and NO_2 but reduced the gas response and recovery speed. The correlation between the NW density and gas sensing characteristics are discussed in relation to the gas sensing mechanism.

Keywords: SnO_2 nanowires; Solution deposition; Polydimethylsiloxane (PDMS) patterning; Nanowires network; Gas sensors

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ABSTRACT

This paper suggests a simple and cost-effective method for fabricating nanowire (NW) gas sensors. Oxide NWs were deposited uniformly over a defined electrode area by coating with a solution containing NWs after patterning with polydimethylsiloxane (PDMS). The hydrophobic PDMS guide wall prevented outstretching of the deposited solution, which allowed the density of the NWs in the sensor to be manipulated conveniently. The high-density NWs sensor enhanced the gas sensitivity toward CO, C_3H_8, C_2H_5OH, and NO_2 but reduced the gas response and recovery speed. The correlation between the NW density and gas sensing characteristics are discussed in relation to the gas sensing mechanism.

1. Introduction

One-dimensional oxide nanostructures are promising materials for applications in electronic and optoelectronic devices [1]. In particular, the high surface area/volume ratio and less-agglomerated structures of oxide nanowires (NWs) are beneficial for enhancing the gas sensing characteristics [2,3].

Semiconductor-type gas sensors can be fabricated using either single NWs or NW networks. The single-NW configuration allows a fundamental understanding of the gas sensing mechanism of NW-based gas sensors [4,5], which is different from that in their nano-particle-based counterpart [6–8]. The single crystalline NW of n-type oxide semiconductor establishes the electron depletion layer near the surface as a result of oxygen adsorption with a negative charge. Due to the lack of grain boundaries, electronic conduction through a single NW is determined by parallel competition between the resistive shell and semiconducting core [4,5,9]. In comparison, a number of nano-particles with core(semiconducting)-shell(resistive) configuration contact with each other, which change the resistance upon exposure to gas. According to the contact configuration, the sensing mechanism can be described either by neck model or by grain-boundary model [6–8].

However, the formation of electrodes precisely onto a single NW is a challenge requiring rather expensive e-beam lithography processes [9–11]. In contrast, the fabrication of NW networks sensor is relatively simple and cost-effective compared with that of a single NW sensor. In addition, the networked structure of NWs establishes a number of resistive contacts at the junctions between NWs. The large variation in resistance at these junctions provides an additional sensing mechanism based on the serial connection between the resistive grain boundary and semiconducting core, which leads to an increase in gas sensitivity [3]. The configuration of NWs network is less-agglomerated than that of the nano-particles counterpart, which can promote the diffusion of target gas on to the sensing surface. Accordingly, the NW-network gas sensor is of great importance not only for cost-effective fabrication but also for enhancing the gas sensitivity.

To date, several approaches to deposit NWs network or nano-particles on to the patterned area have been suggested. These include the coating of carbon nanotubes (CNTs) bundles using the combination of electrophoresis and dip coating [12], the alignment of single-walled CNTs using a funnel-shaped microchannel in a polydimethylsiloxane (PDMS) mold [13], and the patterning of slurry containing tin oxide nano-particles using micromolding in capillary [14].

In this study, another facile route for fabricating a well-defined NWs gas sensor in a networked configuration was suggested using a

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PDMS patterning and solution deposition method. The NW density was manipulated by controlling the coating parameter. The effect of the NW density on the gas sensing characteristics, such as the gas response and response time, were investigated.

2. Experimental

SnO_2 NWs were grown by a vapor phase transport using Sn metal (99.999%). The source loaded in the Al_2O_3 boat was located in the center of a quartz tube (inner diameter: 28 mm; length: 800 mm) in a horizontal furnace. A Si wafer coated with Au (thickness: 30 Å) was placed 5 cm downstream from the source. After evacuating the quartz tube to $\sim 10^{-2}$ Torr using a rotary pump, the furnace temperature was increased from room temperature to 750 °C, and the NWs were formed by a reaction between the source and O_2 gas (0.5 sccm) for 20 min.

For sensor fabrication, the Ti (50 nm) and Pt (300 nm) layers were deposited in sequence on a 4-in. SiO_2 (300 nm)/Si wafer by DC sputtering, and comb-like electrodes with a $500 \times 500\ \mu m^2$ area were formed using a lift-off process (Fig. 1(a)). The substrate was coated with a solution containing PDMS and hardener (9:1 by volume) and subsequently hardened at 60 °C for 5 h. The PDMS patterns with a square type hole were formed by cutting the PDMS layer above the electrode area and its subsequent removal by tweezers (Fig. 1(b)). The as-grown 0.01 g SnO_2 NWs were dispersed in a mixture of deionized water and isopropyl alcohol (IPA) (5 ml:5 ml) by ultra-sonication. A slurry droplet containing SnO_2 NWs (10 μl) was dropped onto the PDMS patterned substrate using a micro-pipette (Fig. 1(c)) and dried gradually (Fig. 1(d) and (e)). The coating of NWs on a defined area was confirmed by the observation using optical microscope (OLYMPUS, BX51M). The density of NWs was controlled from low to high by manipulating the number of droplets deposited. Two sensors were fabricated by coating one and five droplets of the slurry, which were referred as low-density nanowires (LD-NWs) and high-density nanowires (HD-NWs) sensors, respectively. In order to decrease the density of NWs further, 0.005 g SnO_2 NWs were dispersed in a mixture of deionized water and isopropyl alcohol (IPA) (5 ml:5 ml) and 1 droplet of slurry was dropped and dried. This sensor was referred as very-low-density nanowires (VLD-NWs). The gas sensing characteristics of three sensors were measured and compared.

The sensor was contained within a quartz tube and heat-treated at 400 °C for 12 h to decompose any residual PDMS that might deteriorate gas sensing characteristics. And the temperature of furnace was set to the gas sensing temperature. The gas concentration was controlled by changing the mixing ratio of the parent gases and dry synthetic air. A flow-through technique with a constant flow rate of 500 cm^3/min was used. The gas responses ($S = R_a/R_g$ or R_g/R_a) to nitrogen dioxide (NO_2), carbon monoxide (CO), propane (C_3H_8), and ethanol (C_2H_5OH) were measured at 300–400 °C by comparing the sensor resistance in the target gases (R_g) with that in high-purity air (R_a). The dc 2 probe resistance of the sensor was measured using an electrometer interfaced with a computer.

3. Results and discussion

The as-grown SnO_2 NWs were typically 50–100 nm in thickness and several tens micrometers in length (Fig. 2(a)) and showed a rutile structure (JCPDS #41-1445) (Fig. 2(b)). High resolution transmission electron microscopy (HR-TEM) confirmed the NWs to be single crystalline SnO_2 (Fig. 2(c)). A solution containing the SnO_2 NWs, water and alcohol was prepared and coated on the electrodes. In the absence of a covering PDMS pattern, the droplet with hydrophilic characteristics spread over a wide area of substrate, which hindered the deposition of SnO_2 NWs onto a defined area (dotted rectangular area in Fig. 1 and Fig. 1(f)). The introduction of a PDMS guard pattern with a hydrophobic nature effectively prevented the spread of the droplet and confined the solution to within the designated area (Fig. 1 (d) and Fig. 1(g)). Accordingly, without outstretching, high-density NWs could be coated on the electrodes by repeating the solution deposition and drying processes.

Fig. 3(a) shows a low magnification SEM image of a typical sensor (LD-NWs sensor). The SnO_2 NWs were coated uniformly over the comb-shaped electrodes. Only limited region was cov-

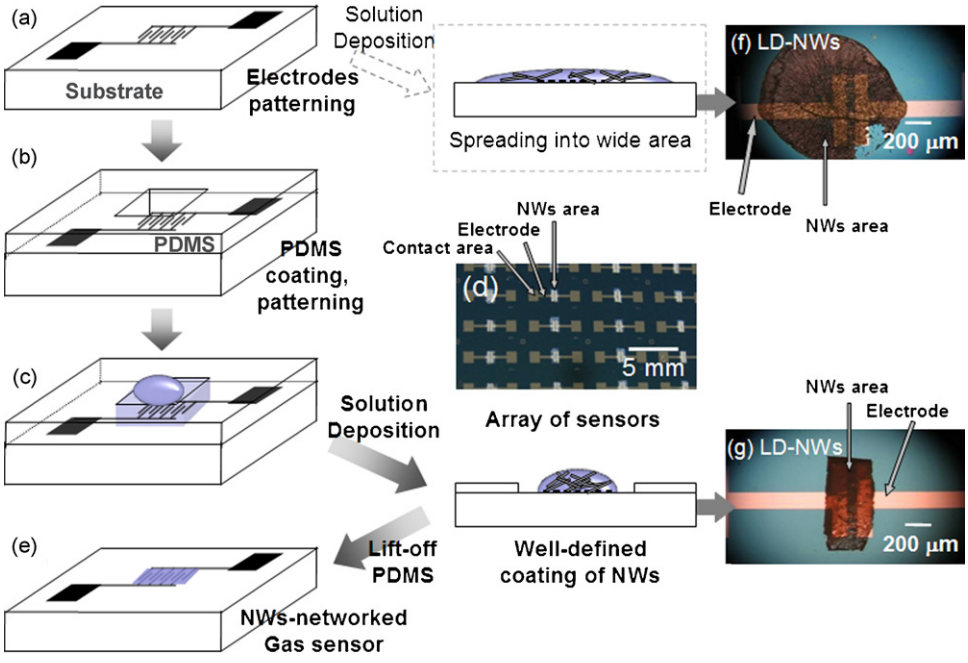

Fig. 1. Experimental procedures to prepare the networked NWs gas sensor.

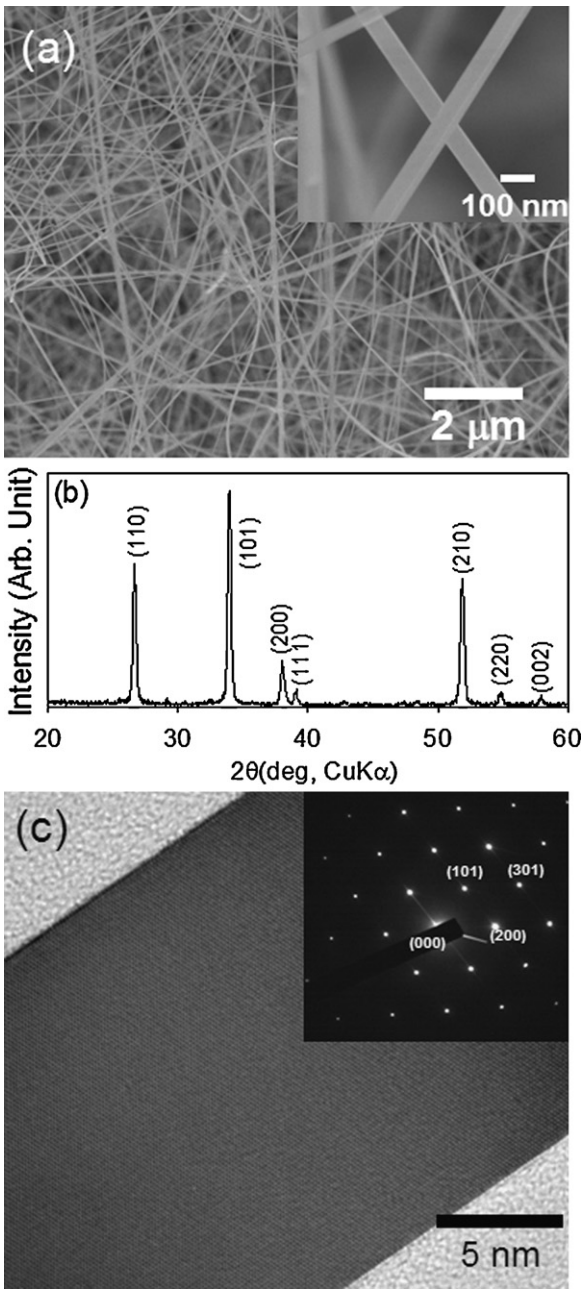

Fig. 2. (a) SEM image, (b) X-ray diffraction pattern, and (c) high-resolution TEM image of the as-grown SnO_2 nanowires.

ered by NWs in the VLD-NWs sensor (Fig. 3(b)). At the LD-NWs sensor (Fig. 3(c)), approximately one monolayer of SnO_2 NWs was loosely packed with each other. Five repetitions of the solution deposition and drying procedure increased the NW density significantly (HD-NWs sensor at Fig. 3(d)). The NWs were connected in a rather compact manner and showed a multilayer configuration. It should be noted that the NWs coated on the electrodes were approximately 5 μm long, which are shorter than the as-prepared ones (Fig. 2(a)). This means that the NWs are broken into the shorter ones during the ultrasonic treatment for dispersion.

The dynamic gas sensing characteristics toward 100 ppm CO, C_3H_8, and C_2H_5OH were measured at 400 °C, and the results are shown in Figs. 4 and 5. The gas response (R_a/R_g) values of the VLD-NWs sensor upon exposure to 100 ppm CO, C_3H_8, and C_2H_5OH were 1.81, 1.6, and 3.3, respectively. In the LD-NWs sensor, these values became 1.66, 1.8, and 23.8, respectively. Finally, in the HD-NWs sensor, the R_a/R_g values upon exposure to 100 ppm CO, C_3H_8, and C_2H_5OH increased to 3.95, 2.98, and 42.6, respectively. The R_a/R_g values tended to increase as increasing the density of NWs although a small fluctuation was found in the sensing of CO. This suggests that the density of NWs is a key parameter determining the R_a/R_g value. It should be noted that the response and recovery kinetics are also closely related to the NW density. The times to reach 90% variation in resistance upon exposure to gas and air were defined as the 90% response time ($t_{90\%}$(air-to-gas)) and 90% recovery time ($t_{90\%}$(gas-to-air)), respectively, and were obtained from the response transients shown in Fig. 4. The $t_{90\%}$(air-to-gas) and $t_{90\%}$(gas-to-air) values increased up to 5.5 and 27.5 times with increasing NW density (Fig. 5(b) and (c)), respectively. The response and recovery kinetics should be understood in the framework of a gas diffusion and surface reaction. In this study, the same NWs were deposited at different densities. Therefore, at a constant sensing temperature in the absence of a catalyst, gas diffusion rather than a surface reaction should be considered to be the primary factor for determining the reaction kinetics, even though further studies will be needed to understand the sensing reaction. The slow response and recovery kinetics of the HD-NWs sensor can be explained by the sluggish diffusion of the target gas onto the sensing surface through the agglomerated network of NWs as well as the slow counter diffusion of the reactant gas toward the ambient atmosphere, respectively.

The gas sensing characteristics toward NO_2 were also examined. Because the sensor resistance increased upon exposure to oxidizing NO_2 gas, the gas response to NO_2 was defined as R_g/R_a. The R_g/R_a values to NO_2 increased with decreasing sensing temperature from 400 °C to 200 °C. However, the gas response kinetics at 200 °C was very slow. The optimum temperature for detecting NO_2 was 300 °C when both the gas response (R_g/R_a) value and response kinetics were considered (Fig. 6). The R_g/R_a values of the LD-NW sensor upon exposure to 0.2, 0.5, and 1 ppm NO_2 were 2.5, 6.2 and 10.6, respectively. These increased to 4.2, 7.3, and 21.0, respectively, in the HD-NW sensor. The detection limits of NO_2 were estimated to be approximately <100 and <60 ppb for the LD-NW and HD-NW sensors, respectively, when the criterion for gas detection was set to $R_g/R_a > 1.3$. The $t_{90\%}$(air-to-gas) and $t_{90\%}$(gas-to-air) values increased by up to 20.6 times with increasing NW density. The increase in the response and recovery times is analogous to the sensing characteristics to CO, C_3H_8, and C_2H_5OH, and can be also explained in part by gas diffusion. It has been reported that the undoped SnO_2 nanowires and nanostructures show high sensitivities to C_2H_5OH and NO_2 [3,15–18]. Thus, the higher gas responses to C_2H_5OH and NO_2 compared to those to CO and C_3H_8 in the present NWs network sensor are consistent with the literature. Note that the difference of response and recovery times between the LD-NW and HD-NW sensors in the sensing of NO_2 (Fig. 6(d) and (e)) is higher than those in the sensing of CO, C_3H_8, and C_2H_5OH (Fig. 5(b) and (c)). At this moment, the reason is not clear and should be studied further. However, the differences in the gas sensing temperature and gas sensing mechanism (reductive interaction of CO, C_3H_8, and C_2H_5OH and oxidative interaction of NO_2) can be considered as the possible reasons.

Zhang et al. [11] prepared single-NW and NW-networks sensors using In_2O_3 NWs and reported that the detection limit of NO_2 in the NW-networks gas sensor was lower that that in a single-NW sensor. The increase in sensitivity was attributed to the effect of the NW/NW junctions. The higher gas sensitivity with the higher-density NWs sensor in this study has a similar physical meaning

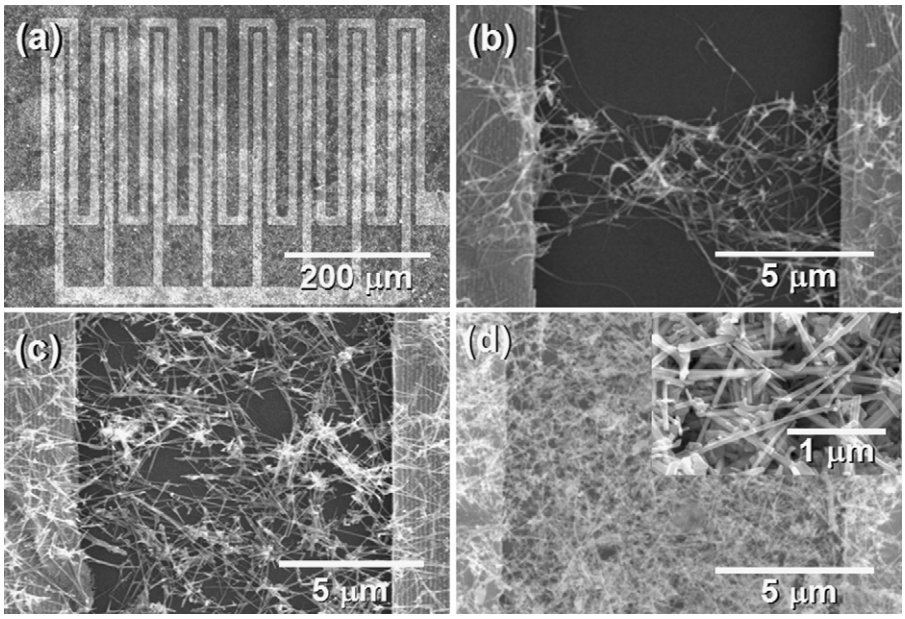

Fig. 3. SEM images of the sensors: (a) low magnification, LD-NWs sensor; (b) high magnification, VLD-NWs sensor; (c) high magnification, LD-NWs sensor; (d) high magnification, HD-NWs sensor.

and can be explained as follows. At 200–400 °C, the single crystalline SnO_2 NWs form an electron depletion layer near the surface through the adsorption of oxygen with a negative charge (O^- or O^{2-}). In a single NW configuration, the overall sensor resistance was determined by parallel competition between conduction along the semiconducting core and that along the resistive shell. Therefore, a NW diameter comparable to the thickness of the electron depletion layer (several nm) is essential for accomplishing high gas sensitiv-

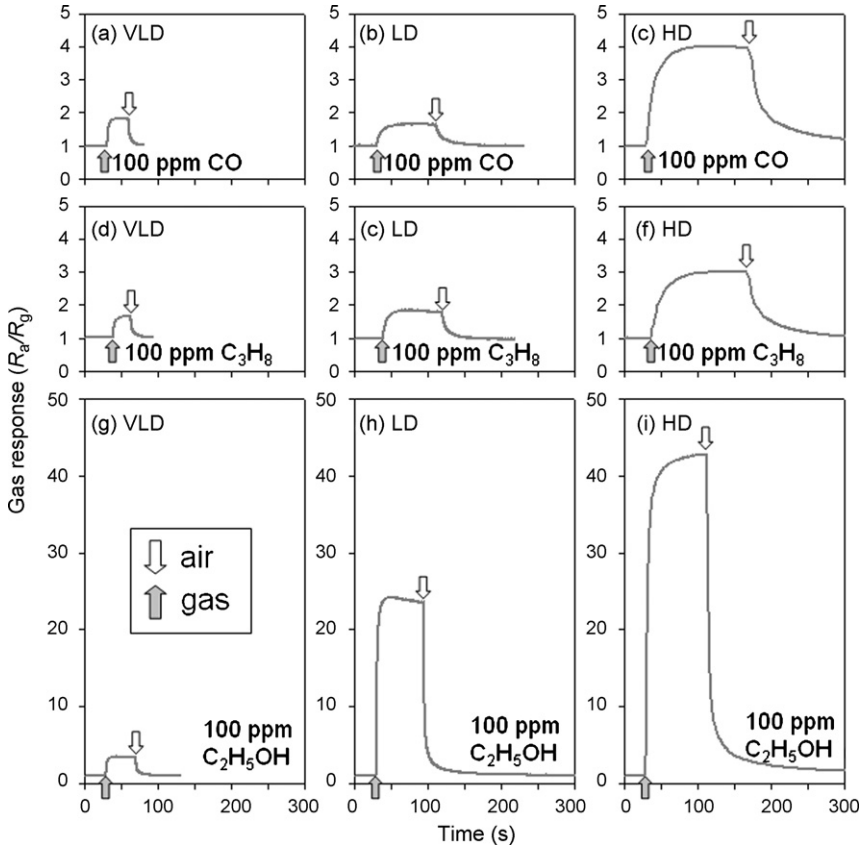

Fig. 4. Dynamic gas sensing characteristics at 400 °C: (a) VLD-NWs, 100 ppm CO; (b) LD-NWs, 100 ppm CO; (c) HD-NWs, 100 ppm CO; (d) VLD-NWs, 100 ppm C_3H_8; (e) LD-NWs, 100 ppm C_3H_8; (f) HD-NWs, 100 ppm C_3H_8; (g) VLD-NWs, 100 ppm C_2H_5OH; (h) LD-NWs, 100 ppm C_2H_5OH; (i) HD-NWs, 100 ppm C_2H_5OH.

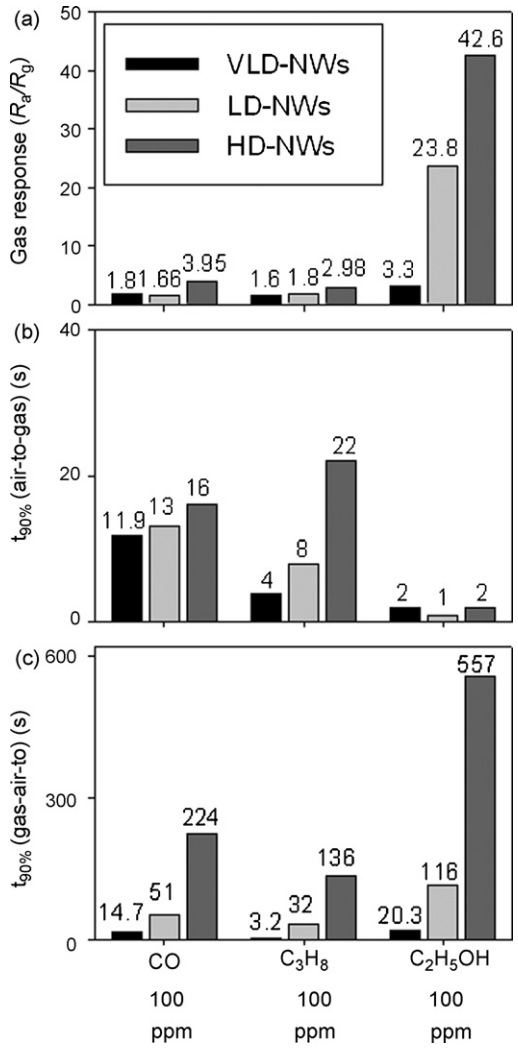

Fig. 5. (a) Gas responses (R_a/R_g), (b) 90% response time ($t_{90\%}$(air-to-gas)), and (c) 90% recovery time ($t_{90\%}$(gas-to-air)) to 100 ppm CO, C_3H_8, and C_2H_5OH at 400 °C.

ity: the sensitivity decreases significantly with further increases in the NW diameter [19].

In contrast, in the NW networks sensor, conduction should occur across the potential barriers established at the junction between the NWs. This is analogous to the 'grain-boundary model' in the gas sensing mechanisms based on nanocrystalline particles [20]. Thus high gas sensitivity can be attained using NW network. However, the nanoparticles tend to aggregate easily through strong van der Waals forces [21]. When the secondary aggregates are large and dense, the gas diffusion toward the inner region of secondary particles becomes sluggish or difficult, which slows the gas response kinetics to a great extent or even deteriorates the gas sensitivity by decreasing the active surface area. Although the high-density NW networks showed slightly slower response kinetics, the 90% response times to 100 ppm CO, C_3H_8, and C_2H_5OH at 400 °C were still quite short (1–22 s). Accordingly, the major advantages of NW-based sensors over the nano-particles-based ones can be found from the rapid response kinetics as well as the high gas sensitivity due to the less-agglomerated configuration. As shown in Fig. 2, HD-NW sensors have more junctions between NWs. The overall sensor resistance will be dominated more by the resistive junctions at the higher NW density. Accordingly, the dramatic change in resistance near the junction upon exposure to a gas will enhance the gas sensitivity of the HD-NW sensor significantly.

It was previously reported [15] that the sensitivity and selectivity to C_2H_5OH of the SnO_2 NW gas sensor can be enhanced significantly by dropping a La-salt solution with subsequent heat treatment. This suggests another method to tune the gas sensitivity and selectivity of NW-based gas sensors in the application of artificial olfaction based on a sensor array. Indeed, an electronic nose has been fabricated using oxide NWs by varying the NW density and using a temperature gradient [22]. The present method provides a simple and cost-effective way of realizing the NW-based gas sensors and NW-based sensors array with high sensitivity and selectivity.

4. Conclusion

This paper suggests a facile route for fabricating oxide semiconductor nanowire (NW) gas sensors employing polydimethylsiloxane (PDMS) patterning and solution deposition. The

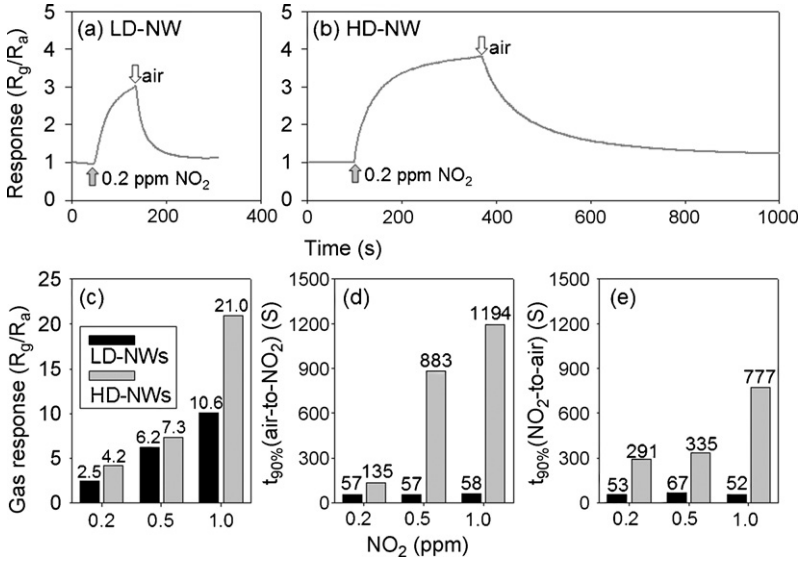

Fig. 6. Dynamic gas sensing characteristics of (a) LD-NWs and (b) HD-NWs sensors to 0.2 ppm NO_2 and (c) gas responses (R_g/R_a), (d) 90% response time ($t_{90\%}$(air-to-NO_2)), and (e) 90% recovery time ($t_{90\%}$(NO_2-to-air)) to 0.2, 0.5, and 1 ppm NO_2 (measured at 300 °C).

hydrophobic PDMS guide wall prevented outstretching of the deposited solution, which facilitated a well-defined, high-density coating of NWs on the electrodes. The gas sensitivity was increased up to ~13 times by increasing the NW density, while the response and recovery speeds decreased. The enhanced gas sensitivity at the high NWs density was attributed to the increase in the number of NW/NW junctions that vary the resistance significantly upon exposure to gas. The retardation of the response and recovery reactions was explained by the slow gas diffusion caused by the soft agglomeration of NWs.

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Byeong-Kwon Ju received his MS degree from University of Seoul in 1988, respectively. He received his PhD degree from the department of electronic engineering, Korea University in 1995. He joined the Korea Institute of Science and Technology from 1995 to 2005. Since 2005, he is a professor of the department of electrical engineering, Korea University. His current research interests include the application of carbon nanotubes to sensor, MEMS, and nanotechnology.

Jong-Heun Lee has been a Professor at Korea University since 2008. He received his BS, MS, and PhD degree from Seoul National University in 1987, 1989, and 1993, respectively. He then spent 7 years developing automotive air–fuel-ratio sensors at Samsung Advanced Institute of Technology. His current research interests include chemical sensors, functional nanostructures, and solid oxide electrolytes