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Effective design and fabrication of low-power-consumption self-heated SnO₂ nanowire sensors for reducing gases

Trinh Minh Ngoc¹, Nguyen Van Duy^{1,*}, Nguyen Duc Hoa¹, Chu Manh Hung¹, Hugo Nguyen²,
Nguyen Van Hieu^{3,4,*}

¹International Training Institute for Materials Science (ITIMS), Hanoi University of Science and Technology, No.1 Dai Co Viet str. Hanoi 100000, Vietnam

²Uppsala University, Department of Engineering Sciences, Lägerhyddsvägen 1, 751 21 Uppsala, Sweden

³Faculty of Electrical and Electronics Engineering and Phenikaa Institute for Advanced Study, Phenikaa University, Yen Nghia, Ha-Dong district, Hanoi 10000, Viet Nam

⁴Phenikaa Research and Technology Institute (PRATI), A&A Green Phoenix Group, 167 Hoang Ngan, Hanoi 10000, Viet Nam

***Corresponding authors**

Nguyen Van Duy, Associated Professor

nguyenvanduy@itims.edu.vn (NV Duy);

Nguyen Van Hieu, Professors

hieu.nguyenvan@phenikaa-uni.edu.vn (NV Hieu)

<Highlight>

- An effective design has been realized for self-heated sensors using network nanowires.
- The density of nanowires was optimized via controlling of the gap size of heat-to-heat electrodes.
- The dense of network nanowires consumes a high power but is effective for detection of reducing gas.

Abstract: Developing metal oxide gas sensors for internet-of-things (IoT) and portable applications require low-power consumption because of the limited battery in devices. This requirement is challenging because metal oxide sensors generally need high working temperatures, especially for reducing gases. Herein, we present an effective design and fabrication method of a SnO₂ nanowire (NW) sensor for reducing gases by using the Joule heating effect at NW nanojunctions without needing an external or integrated heater. The sensor's low-power consumption at around 4 mW was controlled by the size and nanojunction density of the device. The sensor has a simple design and is easy to fabricate. A proof-of-concept of a portable gas sensor module can be realised for monitoring highly toxic reducing gases, such as H₂S, NH₃ and C₂H₅OH, by using the developed self-heated NWs.

Keywords: *SnO₂ nanowires; Nanojunction; Self-heated sensor; Low-power consumption.*

1. Introduction

In recent years, low-power-consumption gas sensors [1] have been developed for medical diagnostics [2] and internet-of-things applications [3] because of their simple operation and rapid and non-invasive operation with high reliability [4]. Resistive gas sensors are an excellent choice due to their low cost, small size and relatively high sensitivity [5]. The principal working mechanism of resistive gas sensors based on metal oxides relies on the interaction or adsorption/desorption of analytic gas molecules to the surface of sensing materials [6]. Accordingly, reducing gases react with the pre-adsorbed oxygen species and hence modify the sensor resistance [7]; thus, heat is required to activate the sensing layer for gas reaction [8]. To monitor reducing gases such as H_2 , H_2S , NH_3 and C_2H_5OH , metal oxide gas sensors generally, require high working temperatures ranging from 200 °C to 450 °C [9,10].

Conventionally, an external or integrated heater is used to provide heat for gas reaction in the sensors and thus causes significant power consumption [11]. To reduce power consumption, researchers have developed microelectromechanical system (MEMS) gas sensors [3]. For instance, a micro-gas sensor can be fabricated with a micro-heater by using a CMOS-compatible MEMS process, and the ink jetting technique reaches a low power consumption of approximately 24 mW [12]. The state-of-the-art membrane MEMS sensors have been proven feasible in reducing the energy consumption down to ~20

mW at 247 °C [13]. However, the MEMS-based designs of gas sensors have their drawbacks, such as the long and complicated fabrication process, poor adhesion of the on-chip heater and the gas-sensitive material layer on the substrate surface. None of the MEMS-based gas sensors can reach the power consumption level of microwatts (μ W) or lower. The working principle of external or integrated heaters relies on the Joule heating phenomenon, in which an electric current pass through a conductor (heater) and produces heat with a power (P) calculated by $P = I^2R$, where I is the applied current and R is the heater resistance. Researchers have also utilised the gas-sensing layer as a heater. This sensor is often called a self-heated sensor and has been studied for different gases. The first reported self-heated gas sensor was based on a SnO₂ thin film for carbon monoxide monitoring, where the temperature can be controlled in the range of about 30 °C to 175 °C by supplying power from about 0.1 W to approximately 2 W [14]. Thus, the power consumption of this self-heated thin film sensor is very high relative to that of the MEMS-based heater. Also, the thin film was not thermally stable because the local heating region was much warmer than the average temperature of the device. The first self-heated single SnO₂ nanowire (NW) sensor was reported to have a power consumption of only a few microwatts due to the small thermal capacitance and hampered thermal losses from the NW to its surroundings [15]. However, single NW devices require complicated processes and expensive fabrication techniques, such as a focused ion beam system [16], [17]. In [18], the self-heated hydrogen gas sensor based on Pd wire network that was fabricated by crackle lithography technique needed a power density of 0.6 W/cm² to retain a temperature of approximately 100 °C. Another self-heated single NW sensor fabricated by a post-synthesis method required a power of 35 μ W as well as a supplied voltage of 25 V to achieve this power because of the very high resistance of the device [19]. The fabricated device was used for oxidising NO₂ gas but not for reducing gases. In [20], a single NW indium–tin–oxide (ITO) gas sensor was designed and nanofabricated via laser writing and subsequent etching. This sensor can respond to

oxidising and reducing gases. The single Pd NW sensor fabricated by top-down approach in [21] needs a power of approximately 4 mW to approach the temperature of 384 °C.

On-chip growth NW sensors have a strong advantage over sensors fabricated from a top-down or post-synthesis method; that is, they are simple to fabricate. Kim et al. [22] reported a self-powered NW sensor based on the SnO₂–ZnO core-shell NWs functionalised with Pt nanoparticles for monitoring toluene. The SnO₂ NWs were grown by the chemical vapour deposition (CVD) method, and the ZnO shells were made by an atomic layer deposition technique, followed by functionalisation with Pt nanoparticles, which was performed by gamma-ray radiolysis. In [23], the single SnO₂ NW device fabricated by transfer processes had a power consumption of 1.6 µW for oxidising NO₂ gas. In [24], the self-heated hydrogen gas sensor based on Pt-coated W₁₈O₄₉ NW networks achieved a power consumption of approximately 60 mW at an applied voltage of 6 V and was found to be useful for portable devices. For the self-heated CO gas sensor based on the Au-functionalised network of SnO₂–ZnO core-shell NWs presented in [25], the power consumption at a supplied voltage of 20 V was estimated to be 8.3 µW. Functionalisation of NWs can reduce the working temperature of the sensor and hence lead to a reduced power consumption. However, functionalisation requires additional steps in the fabrication of a sensor after the on-chip growth.

Recently, we have successfully synthesised different NW sensors by using on-chip techniques, where the density of the NWs can be easily controlled by tuning the growth time or growth temperature [26]. The self-heated NW sensors consume a power of approximately 30 mW when used for NO₂ gas detection [27]. However, for detecting reducing gases, the working temperature is substantially higher than that for detecting oxidising gases, and an increase in the supplied power to reach a high working temperature can damage the device. Lowering power consumption is desirable not only for saving the already limited battery in portable devices but also for developing a battery-free sensing device. At the

consumed power level of tens of microwatts, a self-heated sensor can already be powered by an energy-harvesting source. However, the microwatt power consumption of a self-heated sensor can only be obtained in devices with a single NW or with functionalised NWs. Because the former sensor type has a large disadvantage in fabrication, optimising the latter sensor type is the preferable route to lower the power consumption of the device. However, achieving a sufficient sensing performance with an appropriate supplied voltage for portable devices remains a difficult task. The low resistivity of a material is very important because high resistivity requires a high applied voltage to achieve the desired Joule heat; as such, these high-resistivity materials become unsuitable for mobile device applications. In self-heated NW sensors, power consumption is strongly dependent on heat loss via convection, radiation and conduction. Also, the ‘heater’, which is the sensing material itself, should be adequately designed to lower the power consumption but still sufficient to maintain the temperature for gas reaction.

In the present study, we aim to design and fabricate self-heated NW gas sensors, where an NW network bridges the gap between two electrodes. The gap size is controlled to obtain a proper density of NWs and their nanojunctions to minimise the power consumption and maximise the response for reducing gases. The local heating at nanojunctions in sparse NWs enables the ultralow-power-consumption NO₂ sensor, whereas the network heating in dense NWs consumes a higher power but is effective for reducing gases. The gas-sensing mechanism of the devices, the limit of the supplied power and the low manufacturing cost because of their simple designs and fabrication processes will also be discussed.

2. Experimental

In this study, a sensor design with the so-called head-to-head electrodes was used (Fig. 1). The width of the electrode heads was fixed at 10 μm , whereas the gaps between the heads were set to 2, 5, 10 and 20 μm . The sensors with these gaps are hereafter denoted as G2, G5, G10 and G20, respectively. The sensors were fabricated in only two steps: electrode deposition and SnO₂ NW growth.

In the first step, a simple fabrication process was realised by using the conventional UV-photolithography technique on a heat-resistant Pyrex glass substrate with only one mask. Each glass chip unit with a dimension of 15 mm × 10 mm and thickness of 500 µm contained all four sensors, from G2 to G20 (Fig. 1(B) and (C)). The sputter-deposited layers from the bottom to the top were Cr (10 nm)/Pt (30 nm)/Au (10 nm)/Pt (50 nm)/ITO (20 nm). The electrodes with this sandwich structure were obtained after a lift-off step in acetone.

In the second step of sensor fabrication, SnO₂ NWs were grown from the edges of the electrodes by using CVD method. The length and density of the SnO₂ NWs were controlled by their growth time, growth temperature and the size of the electrode gap (Fig. 1(B)). A scheme of the CVD system is shown in Fig. S1(A) (Supplementary information). In a typical synthesis, a quartz boat containing approximately 0.1 g of tin (purity of 99.9%) with a sensor chip on top was placed at the centre of a quartz tube horizontal furnace. The system was firstly purged with Ar (99.99%) with a flow rate of 300 sccm for 5 min. The temperature was increased at a rate of 36 °C/min from room temperature to 730 °C. The CVD process was subsequently carried out at a temperature of 730 °C, with an O₂ gas flow of 0.5 sccm at a pressure of 1.8×10^{-1} Torr. The growth time was maintained for 20 min to ensure that the NWs bridge the two opposite electrodes. These parameters were important because a shorter growth time and/or lower temperature were impossible for the SnO₂ NWs to bridge the electrodes (Fig. S1, Supplementary information). After the CVD growth step, the furnace was naturally cooled down to room temperature. The sensors were then removed from the setup and examined using SEM. The gas-sensing characteristics were measured under static condition, in which the sensing chamber with a volume of 500 mL was injected with analytic gas or dry air, whilst the sensor resistance was continuously measured. A typical sensing measurement system (here for NO₂ gas) is shown in Fig. S2 (Supplementary information). The supplied electric power was maintained by a millisecond feedback

function (Keithley 2602A) during measurement to hold the sensing material at different constant working temperatures [27].

<preferred position of Figure 1>

3. Results and discussion

Alumina or silicon dioxide is often used as a substrate material for gas sensors because of its inertness and thermal stability. However, alumina substrates have high thermal conduction, which leads to high heat loss. Herein, the low-cost Corning Pyrex glass substrate was adopted to reduce the heat loss and lower the cost. An image of six sensor arrays fabricated on Corning Pyrex glass substrate is displayed in the inset of Fig. 1(B).

The power consumption of self-heated sensors is strongly affected by the heated area of the sensing material. Therefore, using small head-to-head electrodes is the first measure to reduce power consumption. The next measure is focused on the number of heated nanojunctions. Figure 1(A) illustrates that the SnO_2 NWs are not stoichiometric with their outer poor conduction depletion region. The nanojunction between two n-type SnO_2 NWs constitutes a barrier with a height $\Delta\Phi$; thus, the resistance at the nanojunction (R_j) is higher than that of the NW (R_{NW}). Therefore, when powering the device with an electric current (I), the generated heat at the nanojunction (Q_{NJ}) is higher than that at the NW (Q_{NW}). Hence, in the case of sparse NWs, the nanojunctions play the main role in gas sensing. Meanwhile, with dense NWs, many nanojunctions are close to one another, which leads to collective heating (called network heating in this report). Thus, NW heating is more important than that in the sparse NW case.

The SEM images in Fig. 2 show that the SnO_2 NWs grew from the electrode edges and bridged the gaps between them, and almost no NW grew on the glass surface nor on top of the electrodes. The effect

of the ITO layer hindered the SnO₂ NWs from growing on top of the electrodes; moreover, the very thin Au layer buried between the two Pt layers of the electrodes, when exposed to the growth environment, functioned as a catalyst in the vapour–liquid–solid growth of the NWs [27]. Thus, the electric conducting channel of the sensor was composed of the NWs and their nanojunctions. The SEM images also show that the SnO₂ formed a mat of porous entangled NWs with similar morphologies on all sensors (Fig. 2(A–C)). However, for sensor G20, at the centre of the gap, the density of the NWs was clearly lower than those of the other sensors. This result confirms that the gap size determines the density of the NWs, that is, for the same batch of NW growth, a large gap will result in a low density. This effect is due to the fact that over a large gap between electrodes, only the long NWs can meet, but they are not as many as the short and long NWs collectively, which can bridge a small gap.

<preferred position of Figure 2>

The SEM and TEM images of the synthesised SnO₂ NWs are shown in Fig. 3. The surface of the NWs was very smooth, and the average diameter of the NWs was about 80 nm. The NWs were very long and reached tens of micrometres in length. The TEM image in Fig. 2(C) reveals clearly the lattice fringes, confirming the single crystalline nature of the synthesised SnO₂ NWs. The spacing between adjacent lattice fringes was approximately 0.24 nm, which corresponds to the interspace between the (200) planes of the tetragonal rutile SnO₂. The fast Fourier transform image is shown in Fig. 3(D) displays two bright dots, confirming again the single crystallinity of the NWs [28]. Single crystalline SnO₂ NWs with high quality are the proper materials for self-heated sensors because they can avoid grain growth and thus distortion of sensor performance.

<preferred position of Figure 3>

Generally, in the development of self-heated sensors, selecting the appropriate power level is a highly challenging task. An exceedingly high supplied power can easily damage or degrade the device, whereas

an exceedingly low power level cannot be effective for heating the sensor. In the case of Pyrex glass substrate, high supplied power can even crack the substrate by high shock temperature [14]. Herein, we studied for the first time the damaging supplied power for self-heated sensors with different NW densities. For each sensor, we measured continuously the resistance while increasing the supplied power until the device was damaged. The SEM images of the damaged sensors are shown in Fig. 4. As the supplied power increased, the temperature increased, leading to a decrease in sensor resistance due to the semiconductive nature of the NW material. For sensor G2 (Fig. 4(A)), when the supplied power increased from 8 mW to 28 mW, the sensor resistance decreased from about 2 k Ω to approximately 0.5 k Ω , indicating the increase in sensor temperature. At the power supply of 32 mW, the resistance exceeded the measured range of the system, indicating the total damage of the sensor. Therefore, for sensor G2, sufficient power supply should be in the range of 8 mW to 20 mW, which means a safety factor of roughly 1.5, to avoid the damage. An SEM image of sensor G2 after damage is shown in Fig. 4(B). The damage across the centre of the NW mat can be explained by the excessive local heating effect. The damage occurred at the nanojunctions between NWs because of their high resistance and thus high temperature. The self-heated energy under the supplied power was not enough to heat the SnO₂ NWs to the melting point and damage the device because the melting temperature of SnO₂ is very high approximately 1630 °C. Thus, the damage, in this case, should be caused by the electromigration in the conducting path, where the mass transport was induced by the high electric current [29]. At a relatively high temperature caused by the self-heating effect, the collisions between electrons and material ions around lattice points and the interface between two single NWs (meaning junction) increase, and thus, mass transport rises [30]. As a result, the damage occurred at the nanojunctions by electromigration. With the increase in the electrode gap, the damaging supplied power is decreased. The critical power supply values were about 32, 26, 20 and 16 mW for the sensors G2, G5, G10 and G20, respectively. The SEM images of these sensors after damage are shown in Fig. 4(B, D, F, H), respectively. On the basis of

the change in resistance of the sensors with increased supplied power, we can easily conclude that device damage occurred gradually before the sensors suddenly went off. At the beginning, the smaller and thus, hotter, nanojunctions were damaged initially, followed by the larger and less hot ones.

<preferred position of Figure 4>

The gas-sensing characteristics of the fabricated sensors were studied for $\text{C}_2\text{H}_5\text{OH}$ (250–2000 ppm) detection, and the data are presented in Fig. 5. For sensor G2, the $\text{C}_2\text{H}_5\text{OH}$ sensing characteristics were tested at the supplied powers of 10, 12, 14 and 18 mW (Fig. 5(A)). The sensor exhibited a reasonable response to $\text{C}_2\text{H}_5\text{OH}$ at all supplied powers with reversible characteristics. We observed that the response and recovery speeds of the sensor increased with the increase in the supplied power and working temperature. The other sensors, namely, G5, G10 and G20, showed similar trends in gas-sensing characteristics, where the responses improved with the increase of the supplied power (Fig. 5(B–D)). The lowest supplied power levels for sensors G5, G10 and G20 were 8, 6 and 4 mW, respectively.

<preferred position of Figure 5>

Notably, for metal oxide-based gas sensors, the diagram of their response as a function of working temperature has a bell shape [31]. This shape means that at a given concentration of an analytic gas, the sensor response increases with the increase in temperature and then decreases with further increase in temperature beyond the optimal value. Herein, the response diagrams possess only the first half of the bell shape, suggesting that the temperature of the sensor is lower than the optimal value. However, we could not increase the supplied power to reach the optimal working temperature due to the risk of damaging the devices. In self-heated NW sensors, the temperature at the nanojunctions is the highest. Hence, the variation of sensor resistance was essentially caused by the resistance change at the NW-NW junctions. As a result, the response time and recovery times of sensor are dominated by the

nanojunctions. On the other hand, the lower-temperature NWs still contribute to the total sensor resistance, and therefore the nanojunctions do not dominate but together with the NWs determine the sensor response. The plots of the responses as functions of $\text{C}_2\text{H}_5\text{OH}$ concentrations of all self-heated sensors G2–G20 at various applied powers are shown in Fig. 6. For all sensors, the response increases with the increase of the supplied power and $\text{C}_2\text{H}_5\text{OH}$ concentration. By comparing the responses of different sensors, we noted that sensor G20 achieved the highest response value, although the supplied power was lower than those of the other sensors. Sensor G20 can measure $\text{C}_2\text{H}_5\text{OH}$ (250–2000 ppm) effectively at the supplied power of 4 mW. This power consumption is five times lower than that of MEMS devices [12]. This result is very interesting because despite having the lowest power consumption, sensor G20 (with the largest electrode gap) still provided the highest response. This effect was likely due to the gap size because the width of all the sensors was fixed at 10 μm . As mentioned above, the NWs on all the sensors G2–G20 were grown in the same batch, and G2 had the densest nanojunctions, whereas G20 had the sparsest. Therefore, sensor G20 required less power to approach the optimal temperature than did the other sensors. In addition, the large gap was easily fabricated using the conventional photolithography technique, making this sensor very attractive. Hence, the following sections focus more on this sensor than on the other sensors.

<preferred position of Figure 6>

The gas-sensing characteristics of sensor G20 were studied further against different gases, such as H_2 , NH_3 and H_2S , to compare with the $\text{C}_2\text{H}_5\text{OH}$ -sensing characteristics. The data are shown in Fig. 7 are for the measurements with a supplied power of 10 mW, because at this supplied power, the sensor showed a remarkable response to $\text{C}_2\text{H}_5\text{OH}$ (250–2000 ppm), H_2 (100–1000 ppm), NH_3 (100–1000 ppm) and H_2S (1–10 ppm). Recent works focused on self-heated gas sensors for sensing oxidising gases, such as NO_2 , because they have a low working temperature [19] [32]. All the self-heated sensors in this work could

operate effectively for detecting NO_2 at a power consumption of 10 μW (Figs. S3–S6, Supplementary information). Sensor G20 showed a good reversible response to all the tested reducing gases, such as $\text{C}_2\text{H}_5\text{OH}$, H_2 , NH_3 and H_2S (Fig. 7). The sensor responded rapidly within 15 s upon exposure to $\text{C}_2\text{H}_5\text{OH}$, H_2 , NH_3 and H_2S . The response time of the SnO_2 NW sensors strongly depended on their working temperatures; that is, the response time decreased with increasing working temperature [31]. The very fast response times obtained in this study at the supplied power of 10 mW were comparable to the values for the SnO_2 NWs decorated with Pd nanoparticles operated at temperatures ranging from 300 $^\circ\text{C}$ to 400 $^\circ\text{C}$ under an integrated heater [33].

<preferred position of Figure 7>

Figure 8(A) shows a plot of the responses of sensor G20 measured at 10 mW to various concentrations of different gases. The sensor successfully monitored H_2S gas at concentrations down to the sub-ppm level (1 to 10 ppm), whereas the detection concentrations of $\text{C}_2\text{H}_5\text{OH}$, H_2 and NH_3 were roughly ten times higher (100–1000 ppm). Also, the response to $\text{C}_2\text{H}_5\text{OH}$ was much higher than that towards H_2 and NH_3 , suggesting that sensor G20 is effective for monitoring H_2S and $\text{C}_2\text{H}_5\text{OH}$. Given that the concentration of a mixture of these gases can be controlled (e.g. by dilution), this single sensor can potentially be used for discriminating gases instead of using an array of sensors. The reliability and stability of the self-heated sensor were tested over six cycles of filling the gas chamber with dry air and subsequently with 2000 ppm $\text{C}_2\text{H}_5\text{OH}$ at a supplied power of 10 mW. As shown in Fig. 8(B), no substantial change in the sensor response was noted, which demonstrated that the sensor has good stability.

<preferred position of Figure 8>

By using an IR-camera (FLIR System, Thermovision A40) with a resolution of $8\ \mu\text{m} \times 8\ \mu\text{m}$ per pixel, we documented clear images of sensor G2 with powering (Fig. S7, supporting information). However, we failed to obtain IR-images of sensor G20. This result was because although the electrode gap on the G2 was only $2\ \mu\text{m}$, the amount of heat was large enough for the very small IR-light source to ‘blossom’ largely. Consequently, its image was registered by the actual IR-camera. G20 did not provide a good IR-image because the density of the nanojunctions was much lower than that of G2. Hence, an exceedingly low amount of heat was produced, although the heat at the nanojunctions was high and the size of the IR-light source (electrode gap of $20\ \mu\text{m}$) was more than twice larger than the camera resolution per pixel. These were the effect of the network heating on the G2 and the local nanojunction heating on the G20. Obviously, the very small and heating source, along with a small amount heat, in sensor G20 is an advantage for detecting H_2 and other explosive gases because these attributes can help avoid the ignition of these gases. Notably, the working temperatures of the sensors in this work were calculated from the supplied power and not measured using the IR-camera.

4. Conclusions

We have presented an effective design and fabrication method of self-heated SnO_2 NW sensors by using only one mask for UV-lithography. Fabricating the sensors required only two steps: (I) sputter deposition of all electrode materials and lift-off after UV-lithography and (II) CVD synthesis of SnO_2 NWs. The size of the electrode gap on the sensors was used to control the NW network density, which also means the nanojunction density between the electrodes. For the same batch of NW synthesis, the sensor with a narrow gap obtained a dense NW network, and the large gap obtained a sparse network. The sensor with an electrode gap of $20\ \mu\text{m}$ demonstrated better sensing performance for $\text{C}_2\text{H}_5\text{OH}$ at the lowest supplied power of $4\ \text{mW}$ compared with the other sensors with narrower electrode gaps. The proposed sensor was also effective in sensing H_2S at the applied power of $10\ \text{mW}$. In terms of power

consumption and sensing performance, we conclude that local nanojunction heating is more effective than network heating for self-heated NW sensors. Such self-heated sensors are good candidates for integration into portable devices.

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<Figure captions>

Figure 1. (A) Design and band structure of the self-heated n - n nanojunction. (B) Sensor electrode array and (C) SEM image of sensors with different electrode gaps: 2, 5, 10 and 20 μm . The inset of (B) is an image of six sensor arrays.

Figure 2. SEM images of SnO₂ NW gas sensors (A) G2, (B) G5, (C) G10 and (D) G20.

Figure 3. (A) SEM image, (B and C) HRTEM images and (D) the corresponding FFT of the SnO₂ NWs.

Figure 4. (A, C, E, G) Variation in resistance of sensors G2, G5, G10 and G20 at different supplied powers. (B, D, F, H) SEM images of the respective sensors after damage.

Figure 5. Ethanol (250–2000 ppm)-sensing characteristics at different supplied powers of SnO₂ NW sensors (A) G2, (B) G5, (C) G10 and (D) G20. The sensor NWs were grown at 730 °C under 20 min.

Figure 6. Response (R_0/R) as functions of ethanol concentrations at different applied powers of sensors (A) G2, (B) G5, (C) G10 and (D) G20.

Figure 7. Transience versus time of sensor G20 upon exposure to various concentrations of different gases at a supplied power of 10 mW: (A) C_2H_5OH , (B) H_2 , (C) NH_3 and (D) H_2S .

Figure 8. (A) Selectivity and (B) stability of sensor G20 at a supplied power of 10 mW.