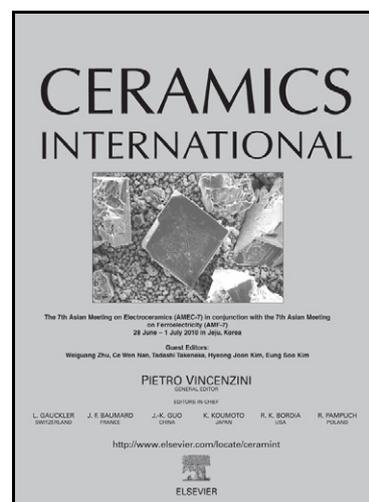


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PII: S0272-8842(13)00146-6  
DOI: <http://dx.doi.org/10.1016/j.ceramint.2013.01.112>  
Reference: CERI6406

To appear in: *Ceramics International*

Received date: 12 November 2012  
Revised date: 29 January 2013  
Accepted date: 30 January 2013

Cite this article as: E.M. El-Maghraby, Ahsanulhaq Qurashi and Toshinari Yamazaki, Synthesis of SnO<sub>2</sub> Nanowires their Structural and H<sub>2</sub> Gas Sensing Properties, *Ceramics International*, <http://dx.doi.org/10.1016/j.ceramint.2013.01.112>

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# Synthesis of SnO<sub>2</sub> Nanowires their Structural and H<sub>2</sub> Gas Sensing Properties

E.M. El-Maghraby<sup>a,\*</sup>, Ahsanulhaq Qurashi<sup>b\*</sup>, Toshinari. Yamazaki<sup>c</sup>

<sup>a</sup> *Physics Department, Faculty of Science at Damanhour, Alexandria University, Damanhour 136, Egypt*

<sup>b</sup> *Center of Excellence in Nanotechnology and Chemistry Department King Fahd University of Petroleum and Minerals, Dhahran ,31261, Saudi Arabia*

<sup>c</sup> *Graduate School of Science and Technology, Faculty of Engineering, University of Toyama, 3190 Gofuku, Toyama 930-8555, Japan*

## Abstract

SnO<sub>2</sub> nanowires were prepared on bare oxidized silicon, Au and SnO<sub>2</sub> coated substrates by thermal evaporation of tin grains in argon atmosphere at 900°C. X-ray diffraction (XRD) and field-emission scanning electron microscopy (FE-SEM) were used to characterize the SnO<sub>2</sub> nanowires. FE-SEM images indicated that the size of SnO<sub>2</sub> nanowires depend on the type of substrate. Gas sensor was fabricated by dispersing SnO<sub>2</sub> nanowires on an interdigitated Pt-electrode. H<sub>2</sub> gas sensing properties of these sensors made of nanowires prepared on three different substrates were measured at various operating temperatures and concentrations respectively. SnO<sub>2</sub> nanowires deposited on Au-coated substrates showed the highest sensitivity of 11.5 at 100°C upon exposure to H<sub>2</sub> gas of 1000 ppm.

**Keywords:** SnO<sub>2</sub>; Nanowires; Gas sensor; Hydrogen

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\*Corresponding authors. maghrabym@yahoo.com (E.M. El-Maghraby) Tel/Fax.: 0020-45-3293011

E-mail address: [ahsanulhaq06@gmail.com](mailto:ahsanulhaq06@gmail.com) (Ahsanulhaq Qurashi) Tel/Fax.: 966-3860-7264

## 1. Introduction

Metal oxide semiconductors have attracted tremendous interest due to their wide variety of optoelectronic applications. Currently, nanostructured metal oxides have emerged an exceptional material and attracted a great deal of interest from researchers. Novel physical properties arising from size reduction are very important for present and future nano-device applications in electronic, optical and sensor fields. Low cost, high sensitivity and reliability of metal oxides are essential in both the fabrication and application of gas sensors. Morphology and crystallographic structure of metal oxides have a great effect on the response of conduct-metric type gas sensors. Nanoscale structures, such as nanobelts, nanowires, and nanorings, were found to show high sensitivity as gas sensors due to the reduction of crystal size [1-5].

Among metal oxide, tin dioxide ( $\text{SnO}_2$ ), is an n-type semiconductor with a wide band gap ( $E_g \approx 3.8 \text{ eV}$ ), which is extensively used in optoelectronic devices.  $\text{SnO}_2$  nanostructures have proven as promising material due to their potential applications in nanosensors. In view of this, much attention has been paid to the synthesis and characterization of  $\text{SnO}_2$  nanostructures such as nanoparticles, nanowires and nanobelts etc. [6].

Optimization of the gas sensor parameters, for example grain size, band gap, crystal quality and thickness greatly influences the response and recovery of the sensors. It was shown that deposition parameter control, post deposition treatments, and impurity doping affect physical parameters of the metal oxides. In the case of  $\text{SnO}_2$ , an increase in pyrolysis temperature during deposition reduces the influence of water vapor on the conductivity of the sensors and, therefore, the influence on the sensor response [7,8].

SnO<sub>2</sub> is one of significant semiconductors used for sensing CO and O<sub>2</sub> gases [9]. Various groups investigated different types SnO<sub>2</sub> nanostructures. Very recently gas sensing properties of SnO<sub>2</sub> hollow spheres have been investigated [10,11]. These hollow spheres and microspheres have high surface area and show good sensor response and have potential applications in catalysis. However still sphere-like structures are limited by structural defects, crystallinity, conductivity and sluggish electron transport compared to 1D nanostructures. Beside exceptional crystal quality and high surface-to-volume ratio 1D nanostructures play an essential role for high performance of nanoscale devices. For instance a single SnO<sub>2</sub> ribbon was used to detect NO<sub>2</sub> gas at ppm-level at room temperature under UV light [12]. SnO<sub>2</sub> nanobelts showed excellent sensitivity to H<sub>2</sub> gas at room temperature [13]. SnO<sub>2</sub> nanowires film exhibited both high response and reversibility to ethanol, CO, hydrogen, acetone and other gases [14-16]. In this work, we report fabrication of SnO<sub>2</sub> nanowires by thermal evaporation on three different types of substrate, namely, bare oxidized Si substrates, gold (Au) and SnO<sub>2</sub> thin film coated respectively. The structure of the nanowires was observed by X-ray diffraction (XRD) and field emission scanning electron microscopy (FE-SEM). H<sub>2</sub> sensing properties nanowires were investigated at different operating temperatures.

## 2. Experiments

SnO<sub>2</sub> or Au films with a thickness of about 10 nm were deposited on the SiO<sub>2</sub>/Si (100) substrates by DC sputtering and vacuum evaporation, respectively. Thus, three different types of substrate (Au, SnO<sub>2</sub> and bare) were used. The apparatus used for fabrication of SnO<sub>2</sub> nanowires is shown in Fig. 1. First, an alumina boat loaded with the

substrates on which Sn grains with a diameter of 2-3 mm were placed was introduced in a quartz tube which was inserted into a horizontal tubular furnace. The one end of tube was connected with cylinder and the other end of the tube was kept open. Then, Ar gas was introduced into the quartz tube for 20 min. at a flow rate of 50 ml/min. After the flow rate of Ar gas was increased to 100 ml/min, the electric furnace was heated and kept at 900°C for 1 h. Finally, the furnace was cooled down naturally to room temperature, and the Ar gas was turned off. Finally whitish wool-like product was obtained around the Sn grains from each type of substrate.

The crystallographic structure of the products was investigated by XRD with Cu-K $\alpha$  radiation using a diffractometer (Shimadzu XRD 6100). The morphology of the products were observed by FESEM (JEOL, JSM 6700F).

Gas sensors were fabricated by dispersing a few drops of SnO<sub>2</sub> nanowires-suspended in an ethanol on oxidized Si substrates with a pair of interdigitated Pt electrodes with a gap length of 0.12 mm. The detailed illustration of SnO<sub>2</sub> gas sensor made of nanowires is shown in Fig. 2. Two sensors for each sample were fabricated. In gas sensing test, the sensors were placed in a quartz tube inserted into an electric furnace. The operating temperature of the sensors was from room temperature (RT) to 300 °C. Dry synthetic air mixed with a desired concentration of hydrogen (100-1000 ppm) flowed through the quartz tube at 200 ml/min. The gas flow was controlled with mass flow controller. The resistance of the gas sensors was determined by measuring the electric current when a voltage of 10 V was applied between a pair of Pt electrodes. A computerized Agilent 34970A multimeter was used for the electrical measurement upon turning the test gases on and off. To determine the selectivity of the prepared sensors to H<sub>2</sub>, we chose H<sub>2</sub>S and

CO gases for practical utilization. At 250 °C when SnO<sub>2</sub> nanowires exposed to H<sub>2</sub>S and CO the sensitivity was 0.3 and 0.5 respectively. When 1000 ppm of H<sub>2</sub> gas was introduced the response reached to 2. The much lower response for both H<sub>2</sub>S and CO than that of H<sub>2</sub> implies that the current sensors have very good selectivity towards H<sub>2</sub>.

### 3. Results and discussion

Typical XRD patterns of the as-deposited SnO<sub>2</sub> nanostructures grown at 900°C are shown in Fig. 3. All the diffraction peaks can be indexed to SnO<sub>2</sub> with a tetragonal rutile structure (JCPDS card No. 41-1445). As shown in Fig. 3, no other peaks are observed in the XRD patterns. This reveals that neither other phases nor Sn metal can be found in the final product.

Figs. 4(a)–(c) show FE-SEM images of the as-deposited SnO<sub>2</sub> nanowires. The inset shows a corresponding high magnification FE-SEM image. The products deposited on the three types of substrate are nanowires/nanobelts. SnO<sub>2</sub> nanowires are about several tens to several hundreds of nanometers in diameter and a few tens of micrometers in length. In addition, the shape of SnO<sub>2</sub> nanowires depends on the type of substrate. The cross section of the nanowires is mostly rectangular for the nanowires deposited on a bare and Au-coated substrate (Figs. 4(a) and (c)). Such rectangular 1D nanostructures are reported in the literature previously by J. Wang et al [17]. While the nanowires deposited on SnO<sub>2</sub>-coated substrate were generally very thin and resembling with the shape of nanoribbons (Fig. 4(b)). It has been reported by researchers that SnO<sub>2</sub> nanowires and nanobelts were usually formed on bare and Au-coated SiO<sub>2</sub>/Si substrates [18,19]. Here, the formation of SnO<sub>2</sub> nanoribbons may be due to the lattice match of substrate which

facilitate in seeded nucleation and the grain structure of SnO<sub>2</sub> films prepared by reactive DC magnetron sputtering [20, 21]. In this case, Sn atoms evaporated from Sn metal react with oxygen atoms and then the product was probably deposited on the SnO<sub>2</sub> grains. However the growth on Au coated substrate follow the VLS (vapor liquid solid) mechanism which helps the growth of 1D nanostructures into high aspect ratio. Beside the reaction temperature, flow of gas, surface of the substrate is also important for the growth of 1D nanostructures. Here the surface of the substrate was altered by SnO<sub>2</sub> and Au films which effected on the size and shape of the nanostructures and dimensions remain similar.

The responses of three types of SnO<sub>2</sub> nanowire sensor upon exposure to H<sub>2</sub> at 1000 ppm measured at various operating temperatures are shown in Figs. 5(a)-(c). For all sensors, the resistance measured in air decreased with increasing operating temperature from 50 to 300°C. When H<sub>2</sub> gas is introduced, the resistance decreases rapidly and saturates within 10 min. The response and recovery time reduces with an increase in operating temperature. The resistance recovers to its initial value after removing H<sub>2</sub> gas, which indicates a good reversibility of these gas sensors. The response of SnO<sub>2</sub> nanowires deposited on Au-coated substrates upon exposure to H<sub>2</sub> gas is greater than that of SnO<sub>2</sub> nanowires deposited on bare or SnO<sub>2</sub>-coated substrates.

The sensor sensitivity, defined as  $(R_a - R_g)/R_g$ , where  $R_a$  and  $R_g$  are the electrical resistances before and after the introduction of H<sub>2</sub> gas, is shown in Fig. 6 upon exposure to 1000 ppm H<sub>2</sub> as a function of operating temperature for the three different sensors. The sensitivity of SnO<sub>2</sub> nanowires on SnO<sub>2</sub>-coated substrates is relatively low, and even the highest sensitivity is 1.96 at an operating temperature of 150°C. The low sensitivity of

this sensor may be attributed to the relatively small surface-to-volume ratio and low conductivity of SnO<sub>2</sub> nanostructures. The sensitivity is higher in the sensor of the bare substrate than the sensor prepared from SnO<sub>2</sub>-coated substrate. The maximum sensitivity of 11.5 was obtained for the sensor of the Au-coated substrate at an operating temperature of 100°C. This value for a pure SnO<sub>2</sub> nanowire sensor is higher than those observed for the SnO<sub>2</sub> nanowire sensor reported elsewhere [15, 18]. It is also noticeable that this sensor shows a high sensitivity at a low temperature of 100°C, which is favorable for lower-power consumption. The synthesis of SnO<sub>2</sub> nanowires on Au coated substrate follows VLS (vapor solid liquid) growth mechanism [22, 23]. The presence of Au on the surface of nanowires may improve the conductivity of nanowires which helps in hydrogen dissociation and trigger the reaction. From these results we can suggest that using noble metals (Au, Pt, Pd, Ag etc) as catalyst might be helpful for successful growth VLS of nanowires and simultaneously enhance the sensitivity of the metal oxide nanowire based sensors.

Since the dissociation of the H<sub>2</sub> molecules on the surface of the sensor is the first step in the H<sub>2</sub> gas sensing mechanism. The large surface-to-volume ratio of nanostructures also allows more efficient dissociation of H<sub>2</sub> molecules to form atomic hydrogen. Then high accumulation of H<sub>2</sub> atoms on the surface of the sensor takes place. As seen from the FE-SEM images, the nanowires deposited on Au-coated substrates are of high aspect-ratio compared to other substrates. The longer nanowires may be a favorable factor for a change of resistance by chemical interaction of the gas with the sensing layer. Therefore, the sensitivity is greatly improved.

The changes in the resistance of three types of sensor upon exposure to H<sub>2</sub> gas at 100 °C is shown in Fig. 7. It is clear that the decrease in the resistance of SnO<sub>2</sub> nanowires deposited on SnO<sub>2</sub>-coated substrates is relatively small. The decrease in the resistance of SnO<sub>2</sub> nanowires formed on SiO<sub>2</sub>/Si substrates is obviously larger than that of the nanowires deposited on SnO<sub>2</sub>-coated substrate. As stated earlier the maximal decrease in the resistance upon exposure to H<sub>2</sub> gas was found for SnO<sub>2</sub> nanowires formed on Au-coated substrates. In this case, the resistance does not saturate and still decreases after the introduction of H<sub>2</sub> gas for 10 min. The response times of three types of SnO<sub>2</sub> nanowire gas sensors are less than 2 min. For the nanowires formed on Au-coated substrates, the resistance recovers to its initial value in 30 min after removal of H<sub>2</sub>. This long recovery time, compared with the other two sensors, is sufficient and may be attributed to the large decrease in the resistance and the slow desorption of hydrogen atoms from the surface of the nanowires. It is noteworthy to mention that the gas sensing properties were found to be similar and reproducible. Previously we have investigated In<sub>2</sub>O<sub>3</sub> and ZnO nanowires, however the stability was not better than SnO<sub>2</sub> [24, 25]. Moreover, when the evaluation of the sensor was performed one month later again for the same devices, the results were found reliable. This indicates reasonable stability and durability of the SnO<sub>2</sub> nanowire based sensors.

Fig. 8 shows the dynamic resistance response of SnO<sub>2</sub> nanowires synthesized on bare substrate to H<sub>2</sub> gas at various concentrations (100-1000 ppm) at 150°C. It can be found that the resistance decreases upon exposure to H<sub>2</sub> gas, and the resistance further decreases with increasing H<sub>2</sub> concentration. The resistance recovers to its initial value after removing H<sub>2</sub> gas, indicating a good reversibility. It should be noted that all sensors show

good reversibility at every temperature, irrespective of the type of template. This good reversibility is attributed to the large surface-to-volume ratio of SnO<sub>2</sub> nanowires, making the adsorption and desorption of H<sub>2</sub> gas on the nanowires more simple owing to high chemical reactions.

#### 4. Conclusions

SnO<sub>2</sub> nanowires with a tetragonal rutile structure were prepared on bare oxidized silicon Au and SnO<sub>2</sub> coated thin films at 900°C in argon atmosphere. The size and shape of 1D SnO<sub>2</sub> nanostructures were dependent on the nature of substrate. Rectangular cross section is found for the nanowires prepared on the bare and Au-coated substrates while nanoribbons-like structure is observed on the SnO<sub>2</sub>-coated substrate. Gas sensors based on SnO<sub>2</sub> nanowires prepared on different substrates were fabricated. These SnO<sub>2</sub> nanowire gas sensors showed a reversible response to H<sub>2</sub> gas at an operating temperature of RT-300°C. The highest sensitivity upon exposure to 1000 ppm H<sub>2</sub> gas obtained in this study was 11.54 at 100 °C, which was measured for the SnO<sub>2</sub> nanowires prepared on Au-coated substrates. This sensitivity is high for pure SnO<sub>2</sub> gas sensors.

#### Acknowledgements

E. M. El-Maghraby and Ahsanulhaq Qurashi, are supported by the VBL (venture business laboratory), Toyama University, Japan

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## Figure Captions

Fig. 1. Schematic diagram of vapor transport tubular furnace used for SnO<sub>2</sub> nanowires synthesis.

Fig. 2. (a) Photographic image of an electrode and (b) schematic diagram of SnO<sub>2</sub> nanowire based gas sensor device.

Fig. 3. XRD pattern of SnO<sub>2</sub> nanowires synthesized on (a) bare, (b) SnO<sub>2</sub>-coated, and (c) Au-coated SiO<sub>2</sub>/Si substrates.

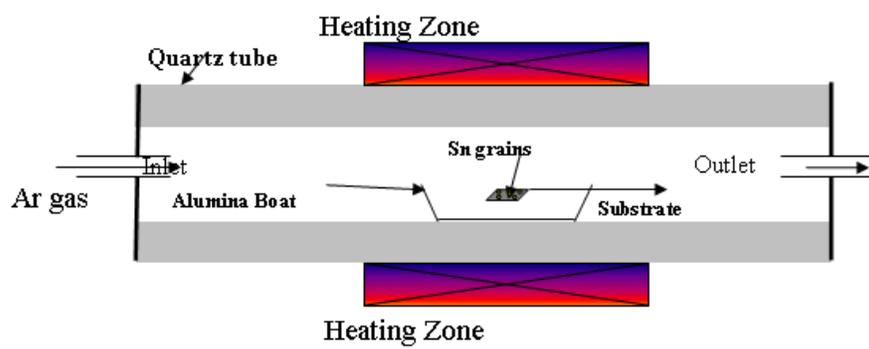
Fig. 4. FE-SEM images of SnO<sub>2</sub> nanowires synthesized on (a) bare, (b) SnO<sub>2</sub>-coated, and (c) Au-coated SiO<sub>2</sub>/Si substrates.

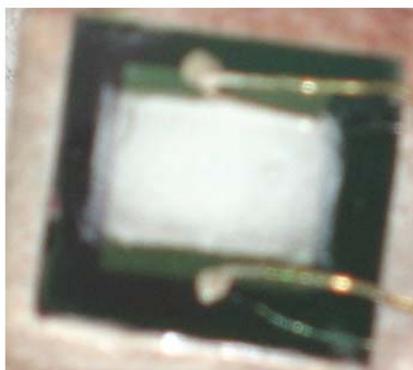
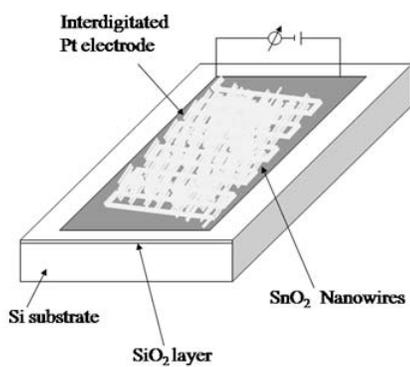
Fig. 5. Changes in the resistance of three types of SnO<sub>2</sub> nanowire gas sensors upon exposure to 1000 ppm H<sub>2</sub> gas at various operating temperatures.

Fig. 6. Sensitivity of three types of SnO<sub>2</sub> nanowires gas sensors upon exposure to 1000 ppm H<sub>2</sub> gas at various operating temperatures.

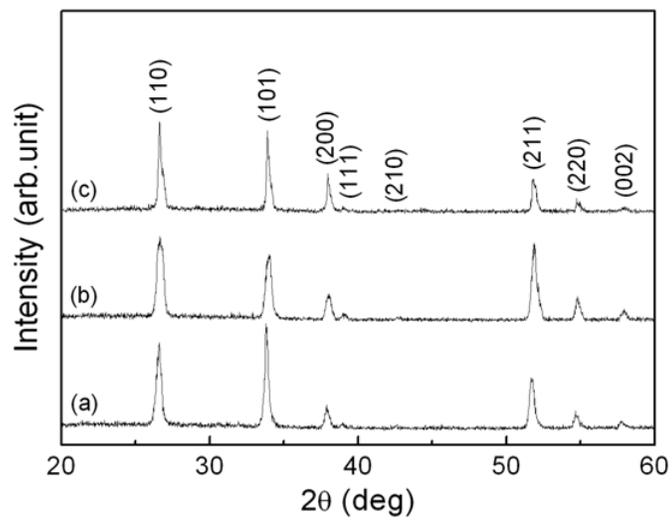
Fig. 7. Changes in the resistance of three types of SnO<sub>2</sub> nanowire gas sensors upon exposure to 1000 ppm H<sub>2</sub> gas at 100 °C.

Fig. 8. Dynamic response of the SnO<sub>2</sub> nanowires gas sensor synthesized on bare- SiO<sub>2</sub>/Si substrate upon exposure to H<sub>2</sub> gas with various concentrations at 150 °C.

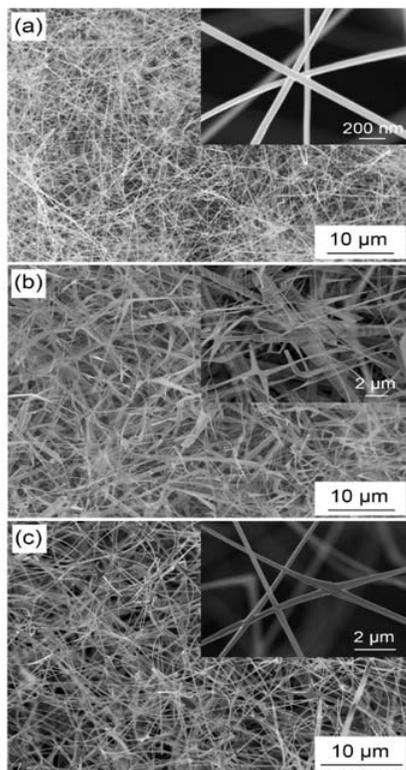


**(a)****(b)**

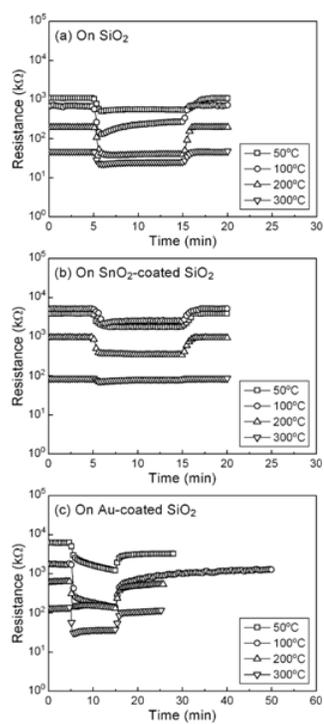
Accepted manuscript



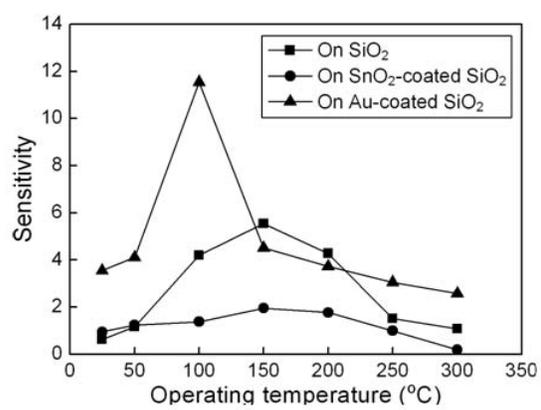
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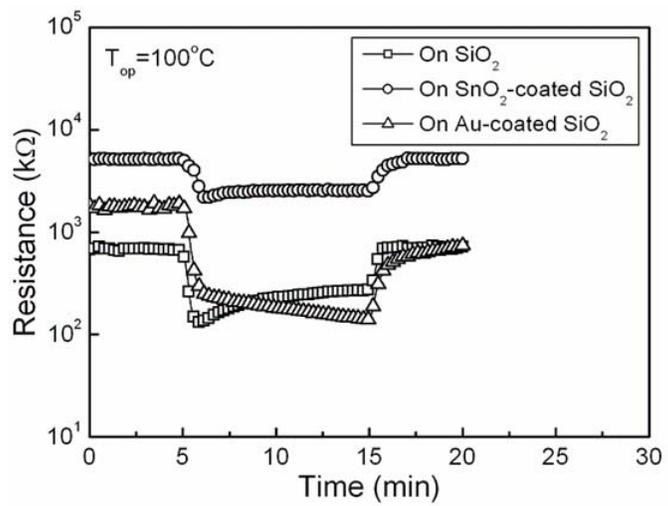
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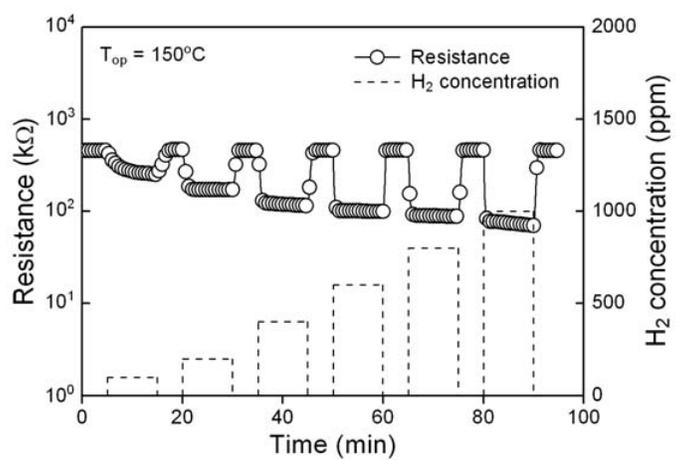
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