

# Detection of Moisture and Methanol Gas Using a Single Electrospun Tin Oxide Nanofiber

Y. Wang, I. Ramos, and J. J. Santiago-Avilés

**Abstract**—This letter reports the fabrication of a gas sensor based on a single tin oxide nanofiber made from dimethyldi-n-octadecanoate tin using electrospinning and metallorganics decomposition techniques. The fabricated sensor has been used to detect moisture and methanol gas. It showed high sensitivity to both gases and the response times of the complete testing system are in the range of 108–150 s for moisture, and 10–38 s for methanol gas, respectively.

**Index Terms**—Gas detectors, nanotechnology, tin compounds, wire.

## I. INTRODUCTION

Binary oxide semiconductors have important sensing and optoelectronic applications [1], [2]. Usually, such oxides have a wide band gap and are good insulators in their undoped and stoichiometric state. However, oxygen vacancies leading to nonstoichiometry can easily be formed in their lattice, donate electrons, and greatly increase their conductivity. Their conductivity is also modulated by species chemisorbed on their surface and the subsequent interaction between the chemisorbed species and the nonstoichiometric oxygen vacancies. Tin oxide ( $\text{SnO}_2$ ) sensors represent some of the early commercialized chemical sensors (the Taguchi sensor) and as such its bulk properties are well known. Usually, the  $\text{SnO}_2$  sensing element is used as thin films because of their higher surface-to-volume ratio  $1/t$  ( $t$  is the film thickness) than bulk. With an even higher ratio  $2/r$  ( $r$  is the nanofiber radius,  $r \ll t$ ),  $\text{SnO}_2$  nanofibers are expected to have better sensing capabilities (such as higher sensitivity and shorter response time) than thin films. However,  $\text{SnO}_2$  nanofibers have been fabricated only recently in a limited number of ways, such as thermal decomposition [3], laser ablation [4], template oxidization [5], vapor deposition [6], and electrospinning [7], even though numerous methods have been developed to deposit  $\text{SnO}_2$  thin film [1], [2]. We fabricated  $\text{SnO}_2$  nanofibers using electrospinning and metallorganics decomposition (MOD) techniques [7], [8]. The conductivity of our synthesized fiber is highly sensitive to its environment [9], suggesting promising sensing applications. In this letter, we fabricated a gas sensor based on a single electrospun  $\text{SnO}_2$

nanofiber and used it to detect moisture ( $\text{H}_2\text{O}$ ), and methanol ( $\text{CH}_3\text{OH}$ ) gases. Although  $\text{SnO}_2$  nanowires fabricated in other ways have been used to detect  $\text{CO}$ ,  $\text{O}_2$ ,  $\text{CH}_3\text{OH}$  and other gases [5], to our knowledge, there have been no such reports in literature using an electrospun  $\text{SnO}_2$  nanofiber.

## II. EXPERIMENT

The sensing element of the single  $\text{SnO}_2$  nanofiber [Fig. 1(a)] was synthesized using electrospinning and MOD techniques. A homogeneous precursor solution with appropriate rheological properties was made from commercial poly (ethylene oxide) (molecular weight 900 000), chloroform, and dimethyldi-n-octadecanoate tin ( $\text{C}_{22}\text{H}_{44}\text{O}_4\text{Sn}$ ) in a pertinent ratio. Single precursor fibers were electrospun from the solution using a homemade electrospinning setup [7] onto single-crystal silicon wafers with their surface thermally oxidized and patterned with gold contacts. The as-deposited precursor fibers on the  $\text{SiO}_2/\text{Si}$  substrates were subsequently isothermally decomposed at  $600^\circ\text{C}$  in air for 2 h, and finally gold wires were bonded to the contacts, as schematically shown in Fig. 1(b). Our previous characterization showed that the synthesized nanofibers were rutile-structured  $\text{SnO}_2$  [7], [8]. The sensor was tested at room temperature, as shown in Fig. 1(c). During operation, the pressure inside the chamber was maintained about 1.1–1.3 atmosphere by flowing nitrogen ( $\text{N}_2$ ) gas to prevent the reflux of the ambient atmosphere. The  $\text{N}_2$  gas was used also to purge the system, to generate a testing gas from its liquid source, and to transport it into the chamber. A Keithley 6475 A electrometer was used to apply a 0.1 V bias between two probes and to measure the current ( $I$ ) and conductance ( $G$ ). With the fiber size measured under a JOEL JSM-6360 scanning electron microscope (SEM), conductivity ( $\sigma$ ) was also calculated [9]. After the sensor was loaded, purging  $\text{N}_2$  was fed directly into the chamber, as well as onto the fiber surface, until  $I$  was stabilized. At that time,  $\text{N}_2$  was directed to go through and bubble deionized water or liquid methanol until  $I$  was stabilized at another level. Finally, the  $\text{N}_2$  gas was redirected to bypass the liquids and flow directly into the chamber.

## III. RESULTS AND DISCUSSION

Fig. 2 shows the sensor's typical time response to the turn-on and turn-off of  $\text{H}_2\text{O}$  and  $\text{CH}_3\text{OH}$  gases with main response parameters listed in Table I. Upon exposure to  $\text{H}_2\text{O}$  carried by  $\text{N}_2$  at a higher flow rate, the sensor showed a higher relative  $G$  change and shorter response times, defined as the times it takes  $G$  to change by 90% of the measured change. Such response is reasonable in that a higher  $\text{N}_2$  flow rate generates a higher moisture concentration in the chamber, accumulates and ventilates it in a shorter time. In fact, the measured response times are those of the whole testing system, and are affected not only by the sensor's intrinsic response time but also by gas generation, transport and venting times. A higher  $\text{N}_2$  flow rate decreases the

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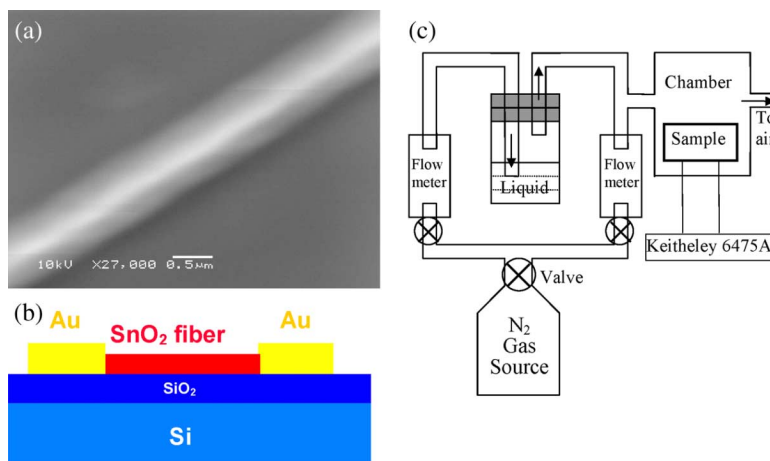


Fig. 1. (a) SEM micrograph of a single  $\text{SnO}_2$  nanofiber sensing element, (b) schematics of the tested sensor, and (c) schematics of the homemade sensor-testing system (the liquid methanol and deionized water were the analytes for these tests).

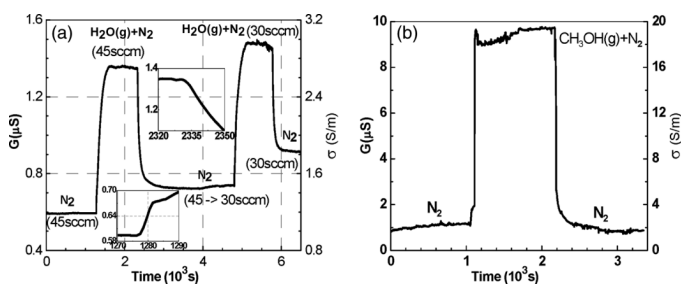


Fig. 2. Response of the sensor to (a) moisture and (b) methanol gas.

TABLE I  
MAIN RESPONSE CHARACTERISTICS OF THE SENSOR

Analyte Gas	$\text{N}_2$ flow rate (sccm)	$G(\text{high})/G(\text{low}) - 1$		90% Response time (s)	
		Rise	Fall	Rise	Fall
$\text{CH}_3\text{OH}$	45	845%	845%	38	10
$\text{H}_2\text{O}$	45	229%	88%	108	150
$\text{H}_2\text{O}$	30	105%	62%	218	187

latter three times. Additionally,  $G$  increased after the first exposure to the moisture, suggesting a not totally reversible change (such as  $\text{H}_2\text{O}$  adsorption) during that exposure. This may have contributed to the higher  $G$  level during the second exposure than during the first.

With the same  $\text{N}_2$  flow rate of 45 sccm,  $\text{H}_2\text{O}$  and  $\text{CH}_3\text{OH}$  increased  $G$  by 229% and 845%, respectively. The rise and fall times are 108 and 150 s for  $\text{H}_2\text{O}$ , and 38 and 10 s for  $\text{CH}_3\text{OH}$ , respectively. The higher relative  $G$  change and the shorter response times with  $\text{CH}_3\text{OH}$  do not necessarily mean higher sensitivity to  $\text{CH}_3\text{OH}$  than to  $\text{H}_2\text{O}$  since their concentration in the chamber could not be measured. It could, at least partially, be attributed to the higher volatility of  $\text{CH}_3\text{OH}$ , which may have accumulated a higher  $\text{CH}_3\text{OH}$  concentration in the chamber within a shorter time. The effect is consistent with that of the  $\text{N}_2$  flow rate on the sensor's responses to moisture.

Although the sensor's response times are not exactly the same as the measured system response times, they cannot be much less than the latter and are expected to be several to tens of seconds. Such response times, although close to the lower limit of the response time range of sensors based on  $\text{SnO}_2$  thin films [1], are still dictated by surface adsorption and desorption processes, which, after triggered by the introduction and exhaustion

of reducing or oxidizing analytes, involve atomic diffusion and surface species migration.

Currently, we are enhancing the testing system in three ways: first, we are adding a vacuum pump to evacuate the chamber and suppress effects of the initial surface state of the fiber and the initial residual gas in the chamber, and to reduce the gas transport time within the system; second, we are using gaseous, as well as more typical, analyte sources (such as  $\text{H}_2$  and  $\text{CO}$ ) to eliminate the gas generation time; and finally, we are using a mass flow controller to monitor the flow rate of analytes and to calculate their concentration in the chamber.

#### IV. CONCLUSION

A gas sensor has been fabricated based on a  $\text{SnO}_2$  nanofiber made through electrospinning and thermal decomposition of  $\text{C}_{22}\text{H}_{44}\text{O}_4\text{Sn}$ , and been used to detect moisture and methanol gases. It shows high sensitivity to both gases and the response times of the complete testing system are 108–150 s for moisture and 10–38 s for methanol gas, respectively.

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