ZnO nanowires grown on SOI CMOS substrate for ethanol sensing

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ARTICLE INFO

ABSTRACT

This paper reports on the integration of zinc oxide nanowires (ZnO NWs) with a silicon on insulator (SOI) CMOS (complementary metal oxide semiconductor) micro-hotplate for use as an alcohol sensor. The micro-hotplates consist of a silicon resistive micro-heater embedded within a membrane (composed of silicon oxide and silicon nitride, supported on a silicon substrate) and gold bump bonded aluminum electrodes that are used to make an ohmic contact with the sensing material. ZnO NWs were grown by a simple, low-cost hydrothermal method and characterised using SEM, XRD and photoluminiscence methods. The chemical sensitivity of the on-chip NWs to ethanol vapour (at different humidity levels) was characterised at two different temperatures namely, 300 °C and 400 °C (power consumption was 24 mW and 33 mW, respectively), and the sensitivity was found to be 0.1%/ppm (response 4.7 at 4363 ppm). These results show that ZnO NWs are a promising material for use as a CMOS ethanol gas sensor that offers low cost, low power consumption and integrated circuitry.

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1. Introduction

Gas sensors have a wide range of applications, including environmental monitoring (toxic and inflammable gases), automobiles, process control (e.g. petrochemical and food industry), personal safety, medical and even military scenarios. Wide band-gap semiconducting metal oxide gas sensors are widely used to detect toxic, inflammable gases and vapours and have been well documented in the literature [1–4]. They are low cost and can detect a large range of gases with high sensitivity at reasonable response times (~s). The working principle of these semiconductor gas sensors is based on the change in conductivity of the material upon exposure to a target gas.

In recent years, there has been increasing interest in integrating gas sensors with silicon CMOS (complementary metal oxide semiconductor) technology. CMOS comes with the 'power' of miniaturisation and allows the integration of signal conditioning hardware. The small size means more portability and potentially lower cost, but conventional metal oxides (in the form of bulk material) are less effective in such micro-gas sensors, because the lower surface area results in lower sensitivity and makes them more prone to poisoning. As a consequence, over the past cou-

ple of years, there has been a shift in sensor research towards gas sensing layers with greater surface areas. Nanomaterials are strong candidates for analytical gas detection, because of their reduced dimensions that create large surface to volume ratio and hence exceptionally high surface area, thus offering increased sensitivity even for a miniaturised sensor. Hence, it is beneficial to combine the two technologies (CMOS and nano-sensing material) and create a 'smart' sensor that could lead to lower unit costs than found in a conventional gas sensor manufacture. The combination is also additionally useful as the sensor can be combined with on-chip circuitry for signal conditioning and to compensate for some of the shortcomings of the sensing material, i.e. drift, non-linearity, aging.

Among different types of metal oxides nanowires (e.g. tin oxide, titanium oxide, tungsten oxide), ZnO (zinc oxide) is one of the most promising, with its direct wide band gap (3.37 eV at room temperature) and large exciton binding energy of 60 MeV. ZnO NWs are n-type semiconductors with high thermal stability. ZnO thin or thick films gas sensors have been widely investigated [5–8]. Various nanocrystalline morphologies of ZnO (e.g. nanowire, nanorod) grown for gas sensing using high temperature chemical vapour deposition techniques can also be found in the literature [9–17]; however the high temperatures (more than 500 °C) necessary for the growth of ZnO NWs are not suitable for a CMOS substrate. There are a few reports of growing ZnO nanostructures for the purpose of gas sensing using a low temperature hydrothermal method [18–23], though these have been deposited on a non-CMOS substrate. It has also been shown that ZnO thin/thick film

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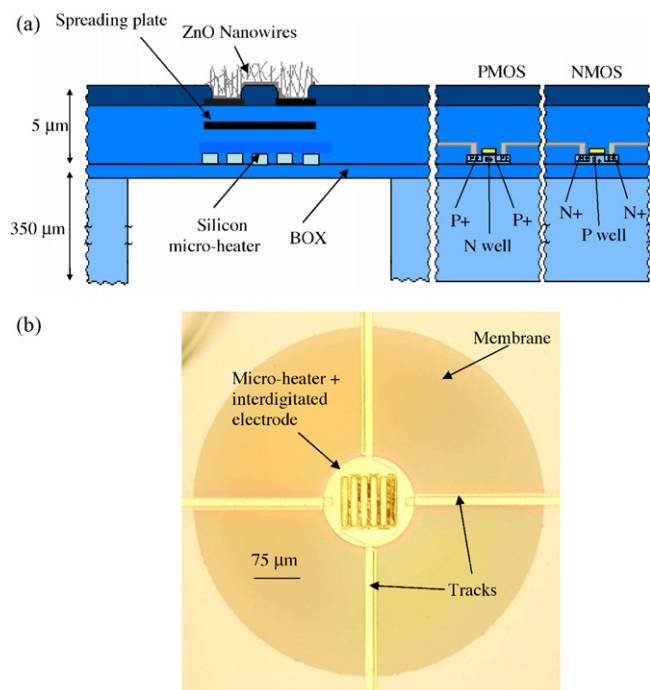
Fig. 1. (a) Cross-sectional view of the ultrathin (5 μm) SOI micro-hotplate and the CMOS electronic cells. (b) An optical microscope picture of the fabricated micro-hotplate with interdigitated electrode.

nanostructures can detect ethanol vapour [10–14,18,22,23], NO_2 [7,15,16,21], H_2 [12,16,18–20], NH_3 [5,20], humidity [9,15] and oxygen [17], however most of the measurements were carried out in dry air.

In this paper, we report on the first integration of ZnO NWs (grown using hydrothermal method) on a fully processed CMOS substrate. The NWs were grown on a thin (5 μm) SOI (silicon on insulator) membrane. The crystallinity, structure and morphology of the present ZnO NWs were characterised and their sensitivity to ethanol vapour in humid air has been evaluated.

2. Micro-hotplate fabrication

Our CMOS sensor structure comprises a resistive micro-heater (or micro-hotplate) integrated within the silicon of an SOI substrate. The top metal layer from the CMOS process is used to form interdigitated electrodes to contact to the resistive sensing layer. The micro-hotplates were designed in Cadence software (v5.0) and fabricated at a commercial foundry (XFAB, Germany) using a 1.0 μm SOI CMOS process. The cross-sectional view and the optical microscope picture of the fabricated device are shown in Fig. 1(a) and (b), respectively. The micro-hotplate comprises of a silicon nitride/silicon dioxide membrane of radius 282 μm in which a silicon resistive heater of radius 75 μm is embedded, formed from n-doped single crystal silicon. This micro-heater is also used as a temperature sensor because its resistance increases almost linearly as a function of temperature. Post-CMOS, the micro-heater is thermally isolated from the substrate using a Deep Reactive Ion Etching (DRIE) process at a commercial MEMS (Micro-Electro Mechanical System) foundry (Silex Microsystem, Sweden) to produce near vertical sides and hence minimises the chip size and reduces the power consumption. A buried oxide layer was used as an etch stop. The membrane and micro-heater shapes are circular to reduce the stress found at the corners of a square membrane.

Aluminium interdigitated electrodes tend to form an oxide when exposed to air, leading to a poor electrical contact with the sensing
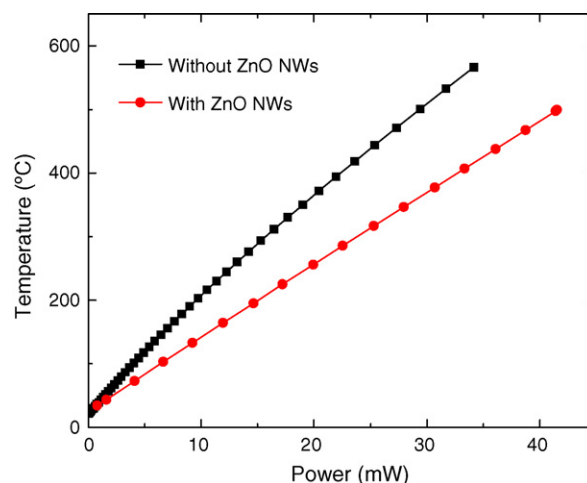
Fig. 2. Power consumption versus temperature plot of the silicon micro-heater with and without ZnO nanowires.

material. Therefore, electroless (bump bonding) plating was carried out at the wafer level to deposit nickel followed by gold (Pac Tech, Germany) on top of the aluminium electrodes.

The micro-heaters were characterised on several devices across the wafer and also from wafer to wafer. It was found that a temperature of 400 °C (shown in Fig. 2) can be obtained with 23 ± 2 mW of DC power (this measurement was done without any sensing material on the devices). The devices showed excellent reproducibility on different wafers (maximum variation found was less than 3%).

3. ZnO NWs growth and characterisation

For sensing material growth, a thin ZnO seed layer was sputter deposited (∼5 nm) on selective areas of our CMOS devices and defined using a metal shadow mask. Following seed layer deposition, these devices were dipped in an equimolar (25 mM) aqueous solution of zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, Sigma–Aldrich) and hexamethylenetetramine (HMTA, Sigma Aldrich) and were kept at 90 °C for 2 h [24,25]. The devices were removed from the solution at the end of the growth, rinsed in deionised (DI) water and dried under nitrogen flow. The advantage of this method is that ZnO NWs can be simultaneously grown on more than one micro-hotplate and hence can be extended to provide an inexpensive approach for wafer level fabrication.

The current–voltage (I/V) characteristics of ZnO NWs were measured at different temperatures (shown in Fig. 3) in laboratory air. The linear I/V characteristic confirms good ohmic contact between the sensing material (ZnO NWs) and the electrodes. This is important for the sensor measurements since the sensitivity of a gas sensor can be maximized when the metal–semiconductor junction is ohmic or has a negligible resistance. The power consumption of the devices in the presence of the sensing material was measured and a power of 33 mW was found to be necessary to reach 400 °C (as shown in Fig. 2) – which indicates an extra 10 mW compared to devices without sensing material.

The surface morphologies of the ZnO NWs with their size distribution were characterised using a field emission scanning electron microscope (FESEM) (JEOL 6340F, operated at 5 kV) as shown in Fig. 4(a). The nanowires are nearly vertically aligned as shown in Fig. 4(b). High resolution SEM images (Fig. 4(b)), revealed that the NW length and diameters are approximately, 400 nm and 70 nm, respectively. Fig. 4(c) shows an X-ray diffraction (XRD) pattern of the ZnO NWs using a Philips PW1730 diffractometer with CuKα radiation. In XRD spectrum there is a strong peak around 34.8° corresponding to the (0 0 0 2) spacing of the wurtzite structure of ZnO,
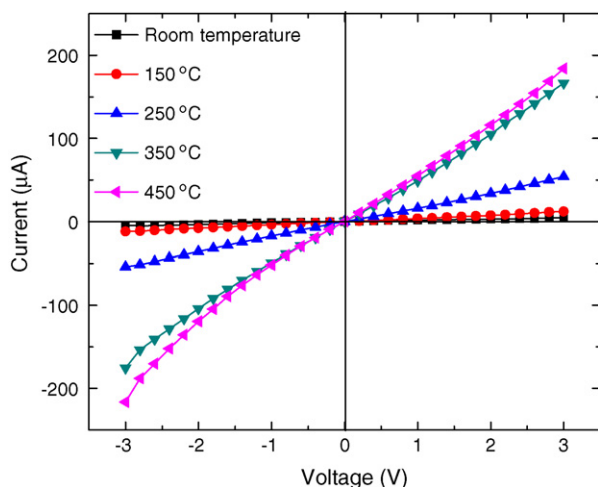
Fig. 3. *I–V* characteristics of ZnO NWs measured in air at different temperatures.

indicating preferential alignment in the *c*-axis direction. Although preferentially aligned, the NWs were found to be interconnected, providing paths for electrical transport between the electrode pads. We further investigated the structure of the ZnO NWs by photo-
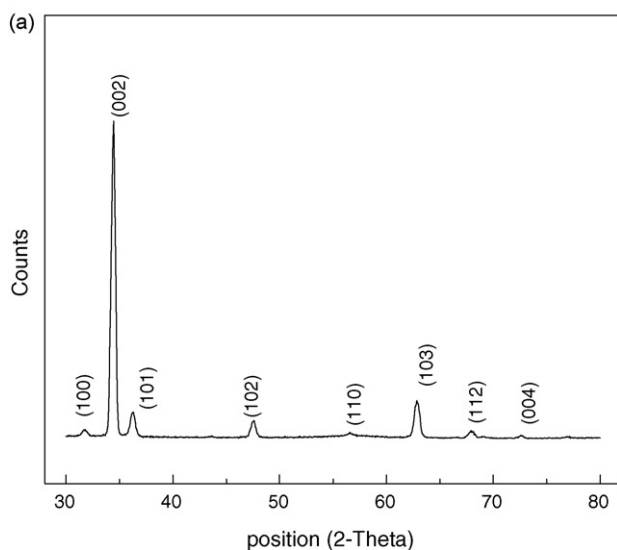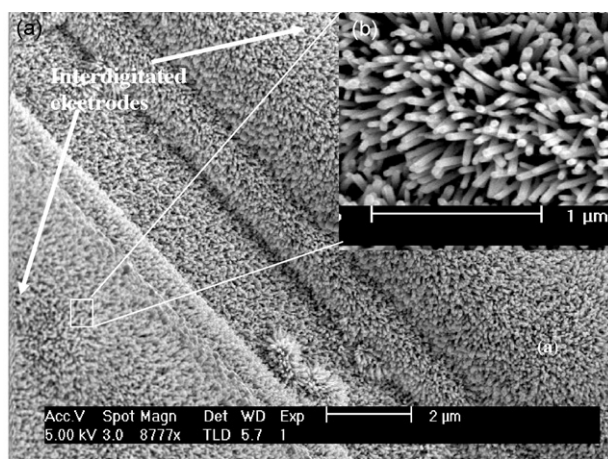

Fig. 4. (a) Top view of the SEM images of the ZnO NWs on interdigitated electrode and (b) magnified view of NWs. (c) XRD pattern of ZnO nanowires on CMOS substrate. Substrate effects have been removed.
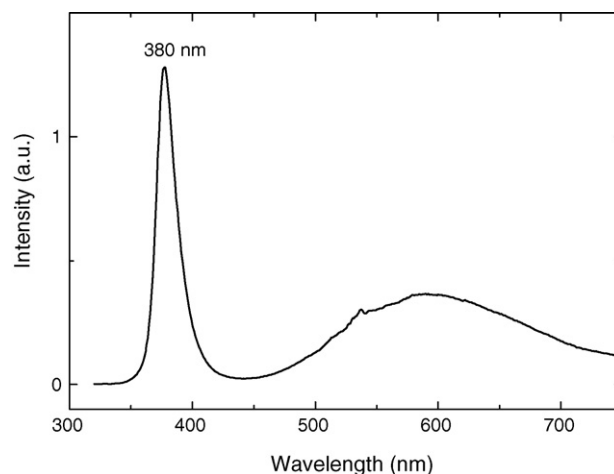

Fig. 5. Photoluminiscence spectra of hydrothermally grown ZnO nanowire.

luminescence (PL) measurements that were performed at room temperature with the 266 nm line of an Nd:YAG laser. A high intensity ultraviolet (UV) emission peak at 380 nm and a broad peak at ~600 nm can be seen clearly in Fig. 5. The UV emission peak usually originates from the near band-edge emission of the wide band gap from the recombination of the free excitons. The broad peak includes the impurities and structural defects, like oxygen vacancies, which are not negligible in our case. For the gas sensor application, the high number of oxygen vacancies is beneficial, as this can increase the interaction between the gas molecules and the ZnO NWs.

4. Ethanol test results and discussions

Chemical testing was performed at the Sensor Research Laboratory (SRL), Warwick University (UK). Here there are custom test facilities that can be used to expose sensors to specific vapours in clean air at specified water vapour levels.

The sensor was bonded onto a ceramic package (16 pin DIL, Spectrum Semiconductor, USA) and mounted in a stainless steel chamber kept at $30 \pm 1\,^\circ\text{C}$ within a Dri-bloc$^\text{TM}$ heater. The chamber was connected to a custom-made test and measurement system interfaced to a National Instruments DAQ card within a PC so that the data acquisition was recorded automatically using commercial Labview software. The sensors were operated with a constant current with the voltage drop across the sensors monitored.

The sensors were heated locally by the silicon micro-heater, which is just underneath the sensing material area. Sensors were kept at two different temperatures ($300 \pm 5\,^\circ\text{C}$ and $400 \pm 10\,^\circ\text{C}$). The measurements were carried out at six different concentrations of ethanol vapour in 3000 ppm (parts per million) humid air. The sensors were first exposed to filtered air to create a baseline signal for 25 min, then exposed to ethanol vapour (at the same humidity) for 25 min. This was repeated for the six different ethanol concentrations.

The ZnO NWs response at different ethanol concentrations (809–4563 ppm) were measured, as shown in Fig. 6(a) at two different temperatures, 300 °C and 400 °C. Here, the sensor resistance increases with exposure to ethanol vapour. A magnified view of 809 ppm response at 400 °C operation is shown in Fig. 6(b). The response time (which is defined as the 90% of the full response) is slow and varies from 200 s to 125 s as we increased the concentrations from 809 ppm to 4563 ppm and recovery time is approximately 10 min. But the recovery time can be improved by increasing the temperature of the NWs, as also shown in Fig. 6(a).
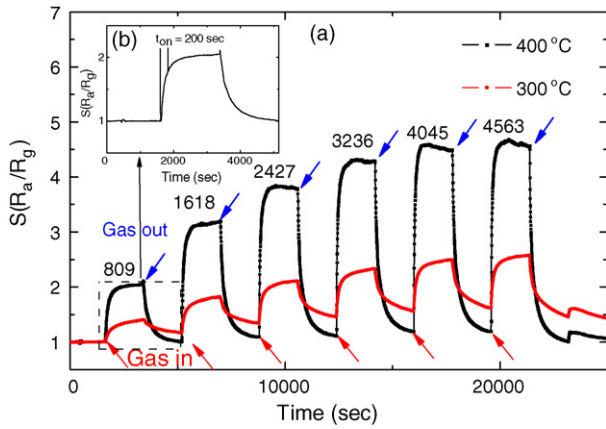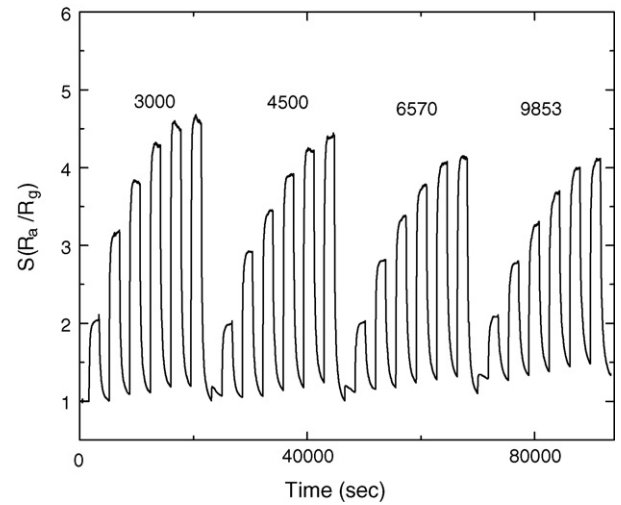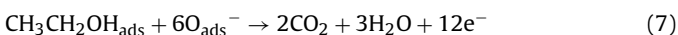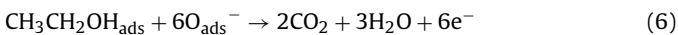
Fig. 6. (a) Change in resistance of the fabricated ZnO NWs ethanol gas sensor at different temperatures and different concentrations at a constant humidity of 3000 ppm and (b) magnified view of the response of 809 ppm at 400 °C operation.

The response of the sensor is defined in this work as R_a/R_g where R_a is the baseline resistance of ZnO NWs in the presence of humid air and R_g is the resistance in presence of ethanol and humid air. The response increases with the increase in ethanol concentration. The sensitivity values (response per unit concentration) are 0.2%/ppm (2) and 0.1%/ppm (4.7) at 809 ppm and 4563 ppm, respectively (response values are shown in brackets).

It is well known that the sensing mechanism for most semiconducting oxide gas sensors is surface-controlled and is controlled by the species and amount of oxygen ions (O_{ads}^-, O_{2ads}^- and O_{ads}^{2-}) on the surface. An oxygen molecule adsorbs on the surface of the ZnO NWs upon air exposure. As a result, an oxygen ion is formed by capturing an electron from the conduction band and there is a formation of depletion layers on the surface regions. It is well known that at temperatures between 100 °C and 500 °C the adsorption of oxygen leads to ionised molecular (O_{2ads}^-) or atomic species (O_{ads}^- and O_{ads}^{2-}), depending on the temperature. The reaction kinetics can be described as follows:

$$O_{2gas} \leftrightarrow O_{2ads} \tag{1}$$

$$O_{2ads} + e^- \leftrightarrow O_{2ads}^- \tag{2}$$

$$O_{2ads} \leftrightarrow 2O_{ads} \tag{3}$$

$$O_{ads} + e^- \leftrightarrow O_{ads}^- \tag{4}$$

$$O_{ads}^- + e^- \leftrightarrow O_{ads}^{2-} \tag{5}$$

When these sensors are exposed to ethanol (reducing gas) at high temperatures (>200 °C, O_{ads}^- and O_{ads}^{2-} are main adsorbed species), the gas reacts with the surface oxygen species and releases the trapped electrons back to conduction band increasing the conductivity of the ZnO NWs. The reaction between the ethanol and ionic oxygen species is described by

$$CH_3CH_2OH_{ads} + 6O_{ads}^- \rightarrow 2CO_2 + 3H_2O + 6e^- \tag{6}$$

$$CH_3CH_2OH_{ads} + 6O_{ads}^- \rightarrow 2CO_2 + 3H_2O + 12e^- \tag{7}$$

Fig. 7. Response of the ZnO NWs measured at different humidities (3000 ppm, 4500 ppm, 6570 ppm and 9853 ppm).

Most of the measurements reported in the literature [9,10,12,16–21,23] are performed in dry air. So the effect of water concentration on the response of ZnO NWs sensors exposed to ethanol in air was investigated, as would be used in a real life instrument. The reaction kinetics of water and ZnO is described by:

$$H_2O + Zn_{lat} + O_{lat} \leftrightarrow (HO–Zn_{lat}) + (O_{lat}H)^+ + e^- \tag{8}$$
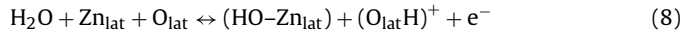
The vapour response has been measured at four different humidity values (3000 ppm, 4500 ppm, 6570 ppm and 9853 ppm ~10–40% relative humidity at 20 °C). It was found that an increase in water concentration caused a decrease in the sensor response, as shown in Fig. 7. It was also reported in the literature [9,15] that the ZnO NWs are used as a humidity sensors. So when the measurements were performed in the presence of humidity, it is proposed that ethanol vapour is in competition for sites with the water. As the ethanol concentration increases the sites preferentially bond with the ethanol over the water, with the ethanol replacing the water within the ZnO NWs.
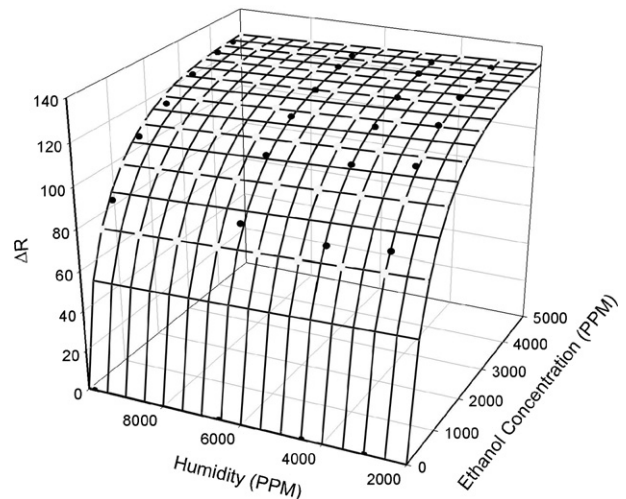
Fig. 8. The effect of water concentration on the response of ZnO NWs sensors to ethanol vapour in air at 400 °C.
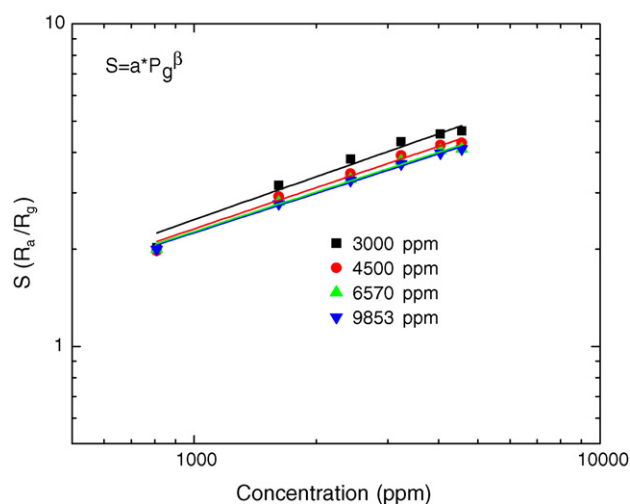
Fig. 9. Response of the ZnO NWs measured as a function of ethanol concentration at different relative humidities.

humidity has a significant effect on ethanol detection through the tested range. We believe this ZnO NW growth on fully processed CMOS substrate could potentially be used as a low-cost ethanol microsensor.

Hence, we can combine the expression for the response to ethanol concentration with the effect of water concentration on the change in resistance, using a competitive binding model:

$$\Delta R \approx \left(\frac{k_W a_W \Gamma C_W + k_E b_E \Gamma C_E^\beta}{1 + k_W C_W + k_E C_E^\alpha} \right) = \left(\frac{a C_W + b C_E^\beta}{1 + k_W C_W + k_E C_E^\alpha} \right) \qquad (9)$$

where a_W and b_E are sensitivity coefficients to water and ethanol, k_W and k_E are binding coefficients, Γ is the number of binding sites, C_W and C_E are the concentrations of water and ethanol vapour, respectively, $a = k_W a_W \Gamma$, $b = k_E a_E \Gamma$. In Fig. 8, the sensor response, to ethanol concentration and the effect of water concentration has been approximated to a competitive binding model.

The measured responses were calculated with different concentrations at different humidities as shown in Fig. 9. The response of the semiconducting gas sensor can usually be represented as: $S = A P_g^\beta$, where P_g is the target gas partial pressure, which is in direct proportion to its concentration, A is the prefactor and β is the exponent on P_g [26,27]. Generally, the ideal value of β is 0.5, which is derived from the surface interaction between oxygen ions and the reducing gas, such as ethanol. In our case, the value of β varies from 0.44 ± 0.04 to 0.40 ± 0.04 as we changed the humidity from 3000 ppm to 9853 ppm, determined by the power law fit shown by the solid line in Fig. 9.

5. Conclusions

This paper describes a novel method for the growth of ZnO NWs on SOI CMOS membranes. This method, as shown, is simple, economical, CMOS compatible and hence ideal for large scale wafer level production. The basic gas sensor device is a micro-hotplate structure containing a silicon micro-heater embedded in a thin dielectric membrane, which allows us to achieve high temperatures with low power consumption. SEM, XRD and photoluminescence methods were utilized for the structural characterisation of the ZnO NWs. Electrical characterisation shows good ohmic contact between the NWs and the electrodes. The sensors were exposed to different concentrations of ethanol vapour in air (relative humidity 3000 ppm) and the sensitivities of 0.2%/ppm and 0.1%/ppm at 809 ppm and 4563 ppm, respectively, were observed. The response was much better at 400 °C compared to 300 °C. The measurements were repeated at four different humidities and it was found that

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Biographies

Sumita Santra obtained her MSc degree in physics form Calcutta University, India in 1999. She received her second Masters in physics and her PhD in atomic physics & spectroscopy from Saha Institute of Nuclear Physics, India, in 2001 and 2007, respectively. She is currently a research associate at the University of Cambridge (UK). Her research interests include temperature sensors and gas sensors. She is also involved in fabrication and characterisation of different nanomaterials on CMOS platform for smart sensors.

Prasanta Guha received his PhD in smart CMOS gas sensor from the Department of Engineering, University of Cambridge, UK in 2008. He has a BSc (Hons) degree in physics and a BTech degree in 'Radiophysics and Electronics' from the University of Calcutta, India in 1997 and 2000, respectively. He obtained his MPhil degree from the Microelectronic Research Center, University of Cambridge, UK in the year 2003. He is presently a research fellow at the University of Warwick. His research interests include smart CMOS sensors and interface electronics.

Syed Zeeshan Ali graduated from GIK Institute (Pakistan) in 2003 with a BS in electronic engineering. He finished his PhD in 2007 at the University of Cambridge (UK) focusing on CMOS micro-hotplates for smart gas sensors and electro-thermo-mechanical modelling of membrane devices. At present he is a research associate at the University of Cambridge researching on micro-hotplates and materials for smart gas sensors.

Pritesh Hiralal has a background in physics with a MPhys degree from the University of Manchester. He is currently a PhD student at the EPEC group at the engineering department at Cambridge. Current research interests are in the growth of 1-D nanomaterials and their application in energy generation and storage, including photovoltaics, photoelectrochemical cells, batteries and supercapacitors.

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James Covington BEng, MRes, PhD, MIET is presently an associate professor in the School of Engineering at the University of Warwick. He received his BEng in 1996 in Electronic Engineering and remained there receiving his PhD in 2000. His PhD was on the development of CMOS and SOI CMOS gas sensors for room temperature and high temperature operation. He worked as a research fellow for both Warwick University and Cambridge University on the development of gas and chemical sensors and was appointment as a lecturer in 2002, being promoted to associate professor in 2006. Current research interests focus on the development of silicon devices with novel materials using CMOS and SOI ASIC technology (nose-on-a-chip), and biologically inspired neuromorphic devices with applications based on environmental and biomedical engineering.

Gehan Amaratunga FREng, FIET, CEng obtained his BSc (1979) from Cardiff University and PhD (1983) from Cambridge, both in electrical/electronic engineering. He currently holds the 1966 Professorship in Engineering at the University of Cambridge. He has research interests on the synthesis and electronic applications of carbon nanotubes and other nanoscale materials. His group has many 'firsts' emanating from his research in carbon, including field emission from n-doped thin film amorphous carbon and diamond, laboratory synthesis of carbon nanonions, tetrahedral amorphous carbon ('amorphous diamond')-Si heterojunctions, deterministic growth of single isolated carbon nanotubes in devices, high current nanotube field emitters and the polymer-nanotube composite solar cells. He also has research interest in nanomagnetic materials for spin transport devices. Professor Amaratunga was elected a fellow of the Royal Academy of Engineering in 2004. In 2007 he was awarded the Royal Academy of Engineering Silver Medal 'for outstanding personal contributions to British engineering'.

W.I. Milne FREng, FIET, FIMMM has been the head of electrical engineering at Cambridge University since 1999, director of the Centre for Advanced Photonics and Electronics (CAPE) since 2004 and head of the Electronic Devices And Materials group since 1996 when he was appointed to the "1944 Chair in Electrical Engineering". He obtained his BSc from St Andrews University in Scotland in 1970 and then went on to read for a PhD in Electronic Materials at Imperial College London. He was awarded his PhD and DIC in 1973 and in 2003, a DEng (Honoris Causa) from University of Waterloo, Canada. He was elected as a fellow of the Royal Academy of Engineering in 2006 and was awarded the JJ Thomson medal from the IET in 2008. He is a guest professor at HuangZhou University in Wuhan, China and a distinguished visiting professor at SEU in Nanjing, China and at NUS, Singapore. He is also a distinguished visiting scholar at KyungHee University, Seoul. From 1973 until 1976 he worked at the Plessey Res Co., Caswell after which he joined Cambridge University Engineering Department as an assistant lecturer. On arriving in Cambridge Prof. Milne set up the Electronic Devices and Materials group, which now has 7 Staff members, approximately 30 Post doctoral research staff and fellows and over 50 PhD students. The yearly income is of order Eu 15 million. His research interests include large area Si and carbon based electronics, thin film materials and, most recently, MEMS and carbon nanotubes and other 1-D structures for electronic applications. He currently collaborates with various companies including Thales, Hitachi, Nokia and FEI and is also currently involved in 5 EU projects and several UK Government funded EPSRC projects. He has published/presented ~600 papers, of which ~120 were invited.
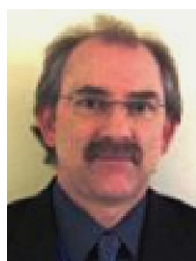
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