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High Temperature Robust SOI Ethanol Sensor

J.W. Gardner^{a,b}, T. Ahmed^b, P. T. Moseley^c, S.Z. Ali^{a,*}, M.F. Chowdhury^a,
F. Udrea^{a,d}

^aCambridge CMOS Sensors Ltd, Wellington House, East Road Cambridge CB1 1BH, United Kingdom

^bUniversity of Warwick, Coventry CV4 7AL, United Kingdom

^cAtmospheric Sensors Ltd., 5, Main Street, Little Thetford, Ely, CB6 3HA, United Kingdom,

^dUniversity of Cambridge, CAPE, 9 JJ Thomson Avenue, Cambridge CB3 0FA, United Kingdom

Abstract

In this paper we present a robust SOI-CMOS ethanol sensor based on a tungsten-doped lanthanum iron oxide sensing material. The device shows response to gas, has low power consumption, good uniformity, high temperature stability and can be manufactured at low cost and with integrated circuitry. The platform is a tungsten-based CMOS micro-hotplate that has been shown to be stable for over two thousand hours at a high temperature (600°C) in a form of accelerated life test. The tungsten-doped lanthanum iron oxide was deposited on the micro-hotplate as a slurry with terpineol using a syringe, dried and annealed. Preliminary gas testing was done and the material shows response to ethanol vapour. These results are promising and we believe that this combination of a robust CMOS micro-hotplate and a good sensing material can form the basis for a commercial CMOS gas sensor.

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1. Introduction

There has been significant research effort in recent years to develop silicon-based micro-gas sensors[1,2]. A typical design is a resistive gas sensor which consists of a sensing material (such as tin oxide) raised to a high temperature (as much as 600°C) by means of a micro-hotplate (a micro-heater thermally isolated from the substrate by means of a dielectric membrane). The resistivity of the sensing material changes depending on the presence and concentration of the target gas. Maximum responses to

* Corresponding author. Tel.: +44 – (0)1223-451302; fax: +44- (0)1223-451100.

E-mail address: zeeshan.ali@ccmoss.com.

most gases occur within a temperature range from about 100 to 500 °C. Therefore a micro-hotplate platform is needed for such sensors. Most micro-hotplates reported in the literature are not CMOS compatible, while those that are CMOS compatible are typically based on polysilicon heaters[3,4]. The lack of CMOS compatibility increases fabrication costs and circuitry cannot be integrated with the device, while CMOS based polysilicon heaters can have stability problems at high temperatures.

We have overcome these problems by using tungsten as a heater material. Tungsten is used as a high temperature metal in some SOI processes, is less susceptible to electromigration compared to aluminum, and is stable at high temperatures. It can therefore be used both as a heating element, and as an interconnect in CMOS circuitry. We have reported tungsten based micro-hotplates previously [5] and have since developed several designs for calorimetric and resistive type gas sensors. In this paper we deposit tungsten-doped lanthanum iron oxide on these micro-hotplates and show the response to ethanol.

2. Micro-hotplate Platform

The micro-hotplate platform was fabricated using a commercial SOI-CMOS process employing tungsten as an interconnect metal. This process forms the tungsten heater and top metal electrodes for gas sensing at the same time that interface circuitry can be fabricated on the chip. Interdigitated electrodes to measure the material resistance were fabricated during the CMOS processing using the top metal layer and exposing it by etching the passivation, using the same process step that is used to form the bond pads. This was followed by back etching by Deep Reactive Ion Etching (DRIE) at a commercial MEMS foundry to form a membrane. Figure 1a shows a cross-section schematic of the device while figure 1b shows a photograph of a fabricated micro-hotplate.

The devices were calibrated and thermal measurements were done using the heater as a temperature sensor. The devices showed low power consumption (0.15mW/°C) and fast thermal transient times (~20 ms). The use of a commercial CMOS process also resulted in good reproducibility (figure 2a) from device to device and wafer to wafer. Long term stability tests were also performed on an annealed micro-hotplate (figure2b), with a 1% drift in heater resistance over 2500 hours when operated at 600°C. This is considerably improved from the 10-20% drift in previous batches using different materials and designs.

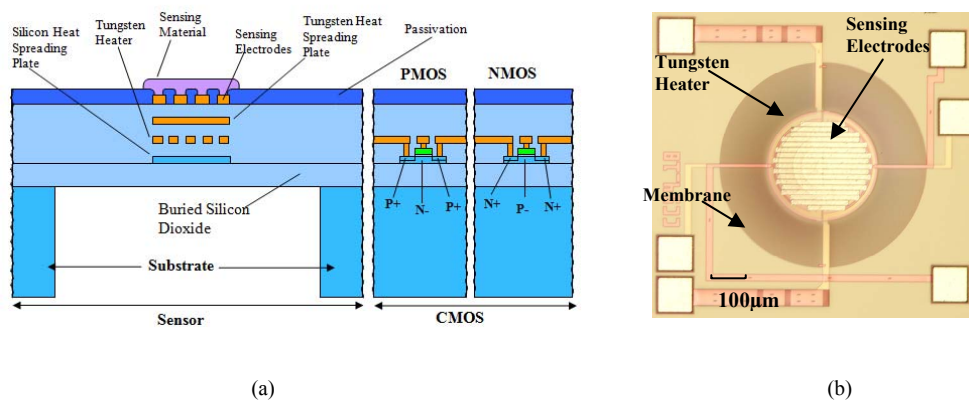
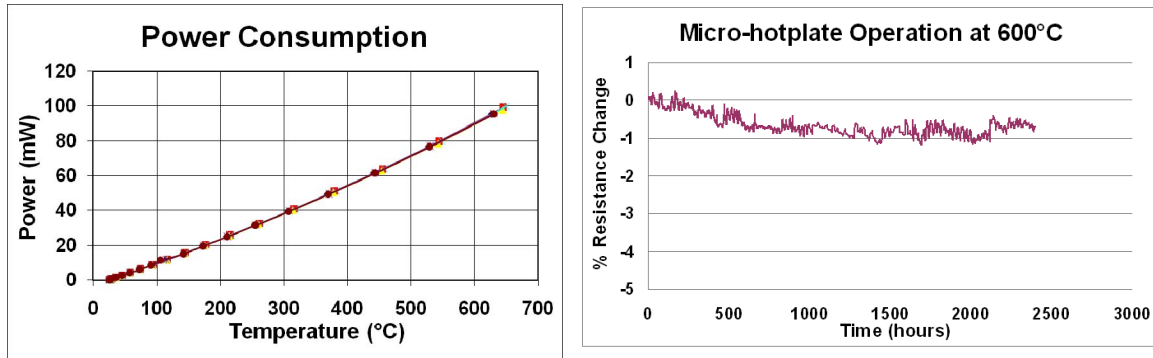


Fig. 1. (a) A cross-section schematic of a micro-hotplate device; (b) A fabricated micro-hotplate chip



(a)

(b)

Fig. 2. (a) Power consumption of the micro-hotplate; (b) Results of stability measurements on the micro-hotplate

3. Material Deposition

Many semi-conducting oxides exhibit resistance-sensitivity to the presence of small concentrations of oxidizing or reducing gases in air [6]. N-type materials such as tin dioxide, for example, respond with resistance decreases to the presence of reducing gases in air and p-type materials respond in the opposite sense (resistance increases when in contact with reducing gases). In an earlier publication [7] it was shown that thick films of tungsten-doped lanthanum iron oxide respond sensitively, with the p-type characteristic, to the presence of reducing gases in air at temperatures up to 500°C. However, such responses are more secure at the low end of the temperature range where the responses are not degraded by the high oxygen ion mobility of the material at high temperature.

In the present work the sensing material was prepared as reported previously [7] and then deposited in the form of a slurry in terpineol onto the micro-hotplate sensing electrodes using a micro-syringe. The slurry was heated using the micro-hotplate for a few hours to drive off the terpineol and anneal the remaining oxide. Figure 3 shows the chip with the sensing material in place.

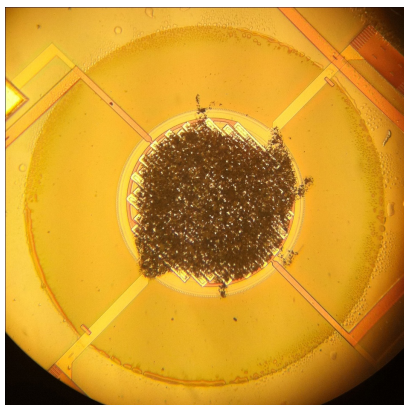


Fig. 3. Lanthanum Iron Oxide deposited on the micro-hotplate

4. Gas Sensing

The device was then placed in a gas test chamber and the sensing material was heated to 450°C using the micro-hotplate. Figure 4a shows the test setup. 90% Ethanol vapour in air was introduced into the chamber, and the sensing results are shown in figure 4b, and as can be seen, there is a large increase in resistance when the ethanol vapour is introduced.

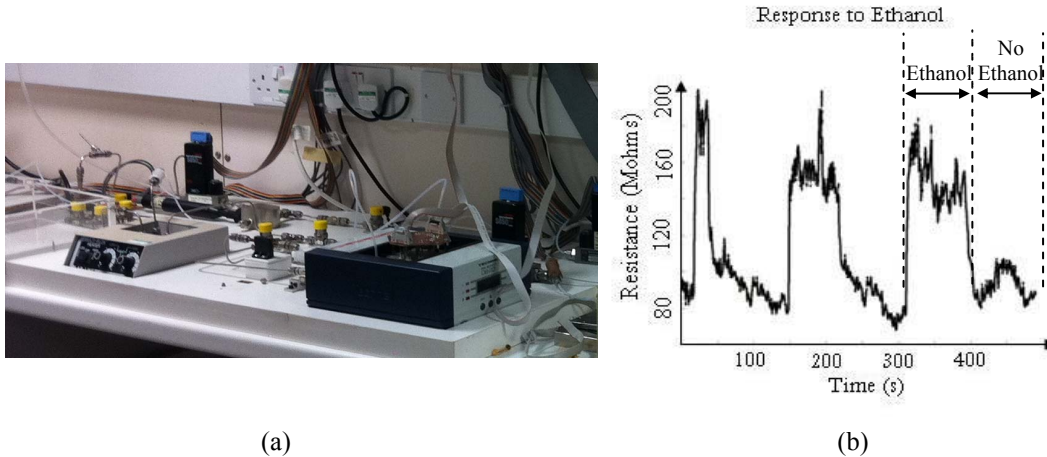


Fig. 4. Response of tungsten-doped lanthanum iron oxide (at 450°C) to ethanol

5. Conclusions

In this paper we have shown a CMOS based ethanol sensor. We have developed and demonstrated a CMOS micro-hotplate capable of operating reliably at high temperatures at 600°C. We have deposited tungsten doped lanthanum iron oxide as a sensing material and preliminary tests show response to ethanol. We believe that this device can form the basis of an integrated ethanol sensor.

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