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SOI-CMOS based single crystal silicon micro-heaters for gas sensors

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Abstract— **Here we report on novel high temperature gas sensors that have been fabricated using an SOI (Silicon-on-insulator) -CMOS process and deep RIE back-etching. These sensors offer ultra-low power consumption, low unit cost, and excellent thermal stability. The highly-doped single crystal silicon (SCS) layer of a standard SOI-CMOS process, which is traditionally used to form the source and drain regions of a MOSFET, is used, for the first time, to form a resistive heater of a micro-hotplate in a high-temperature gas sensor. Our sensors have a power consumption of only 12-30 mW at a temperature of 500 °C. We have observed that the drift in resistance of a SCS heater held at 500 °C for 500 hours, without burn-in, was less than 1 %. SCS micro-hotplates are not only suitable for chemoresistive sensors, as described here, but also calorimetric gas sensors that require these high operating temperatures. Tungsten oxide nanorods have been deposited onto our micro-hotplates by atmospheric chemical vapour deposition and have shown reasoanble sensitivity to ethanol vapour in air.**

I. INTRODUCTION

Although there is an increasing demand from the environmental, automotive and medical industries for portable, handheld gas monitors, presently the gas sensor market is still relatively small. The small uptake for these low-cost battery powered applications, can be accounted for by two major problems. Firstly, the relatively high power consumption and secondly the high price of commercial gas sensors. For example, commercially available pellistors have a typical power consumption of 350 to 850 mW for an operating temperature of 500 °C [1]. Taguchi type resistive gas sensors, (the most commonly used gas sensor) require an operating temperature of between 300 and 400 °C with a power budget of 800 mW [2]. Furthermore, the semi-manual method by which they are fabricated makes them expensive even though production volumes are significant.

Over the last 15 years, considerable effort has been directed towards reducing both the power consumption and the manufacturing costs by replacing conventional sensors with micro-hotplate type sensors based on silicon processes [3, 4, 6, 7]. However, all of these designs have some disadvantages and have not yet enjoyed great commercial success. The most widely investigated resistive heater material for micro-hotplates is platinum [3]. It is not difficult to develop a micro-hotplate using this material, as it is an inactive noble metal and thus has excellent thermal stability. However, platinum is not a CMOS compatible material and thus such designs cannot be fully integrated with drive/detection circuitry or take advantage of the low production costs associated with CMOS processes. CMOS compatible polysilicon heaters have also been widely investigated [4]. However, their long term stability has been reported to be poor due to the highly reactive grain boundary [5]. Furthermore, several studies have been made on highly boron doped single crystal silicon based micro-hotplates formed by anisotropic selective wet silicon etching [6]. These are CMOS compatible and the heater material itself is believed to be thermally stable without the grain boundary issue, which causes the resistance drift in polysilicon. However, the wet etching process makes it impossible to passivate the bottom of the heaters within CMOS process. Therefore, the heaters are easily contaminated and the commercial exploitation is difficult. Micro-hotplates using FETs have recently been proposed [7]. The FET heater can be controlled to have a higher resistivity than equivalent resistive metals (for the same area), which makes it possible to reduce the heater size further. Thus lower power consumption can be achieved. However its operating temperature is limited to less than 400 °C, because of the bipolar turn-on of the MOSFET and the use of aluminium interconnects. Thus, there is a need for a CMOS compatible,

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high temperature and reliable heater structure for gas sensors.

Recently, we have proposed, simulated and designed SOI (Silicon-on-insulator) -CMOS based highly doped single crystal silicon (SCS) micro-hotplates to achieve these goals [8, 9]. Using SOI technology, it is possible to fully passivate the SCS heaters unlike those reported in previous work [6]. We are also aiming at lower power consumption with extremely small heater radii. This paper reports on the first experimental results of our SOI CMOS SCS micro-hotplates.

II. SOI-CMOS GAS SENSOR STRUCTURES

Figure 1 shows the design of an SOI resistive gas sensor employing an SCS micro-hotplate. The micro-hotplate is comprised of a silicon nitride/silicon dioxide membrane in which a highly doped SCS resistive heater is sandwiched. The shapes of both membrane and heater were designed to be circular to reduce the possibility of membrane failure due to mechanical stress (which is more pronounced at high operating temperatures). Here, two micro-heater designs have been produced with different geometries as shown in Figure 2. The radii of the membrane and the heater are 282 μm and 75 μm for the large micro-heaters and 150 μm and 12 μm for the small micro-heaters, respectively.

To supply the power to the micro-heaters, metal tracks were used to reduce both the Joule heat generated and the conduction heat loss. The second CMOS metal layer is used to form heat spreading plates to improve the temperature uniformity of the sensing material, and placed above the heaters. The electrodes to measure the resistances of the sensing materials are made of the third CMOS metal layer. These electrodes have interdigitated structures with aspect ratios of typically 16 and 1.7 for large and small micro-hotplates, respectively.

The fabrication process of the above structures strictly follows the standard SOI-CMOS process. An SOI wafer was used as an initial substrate. The SCS heaters were formed by trench etching and then doped simultaneously with source or drain by ion implantation of boron (*p*+) or arsenic (*n*+), thus no additional processing was needed. The concentration of *p*+ and *n*+ are both *ca.* 7×10^{19} cm^{-3}. After the SOI-CMOS process, the wafer was back etched by deep reactive ion etching (DRIE). Tungsten was used for metallization rather than aluminium as to avoid electro-migration at high temperatures. The thickness of the membrane is *ca.* 5 μm. Polysilicon heaters, which have the same shape as SCS heaters, were fabricated on the same wafer for comparison.

Atmospheric pressure chemical vapour deposition (APCVD) based WO_{3-x} films with nanorod structures, developed by University College London (UCL) [10], were directly deposited onto the micro-heaters. The film was deposited using WCl_6 with co-reactant of ethanol at a temperature of 625 °C. The nanorod structure is confirmed by SEM as shown in Figure 3. Similar types of metal oxide nanorod films have been previously studied due to their large surface to volume ratio [11] hence high sensitivity. However, they were synthesized by high temperature (e.g. 900 °C) annealing of metal or metal oxide powder and thus the direct deposition onto CMOS based micro-hotplates was difficult. The relatively low temperature of our CVD method and tungsten metallization of our micro-hotplates made this possible. To the best of our knowledge, this is the first time that the metal oxide nanorods have been deposited onto CMOS based micro-hotplates by CVD. The film was oxidised at 450 °C after deposition.

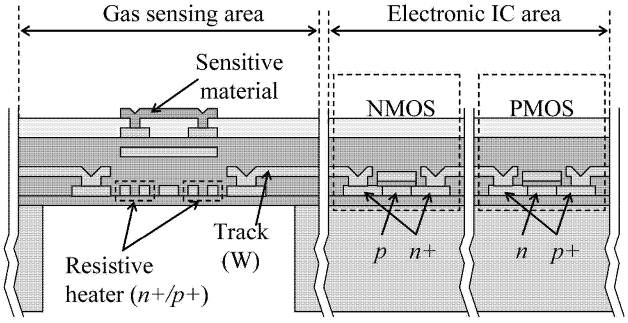
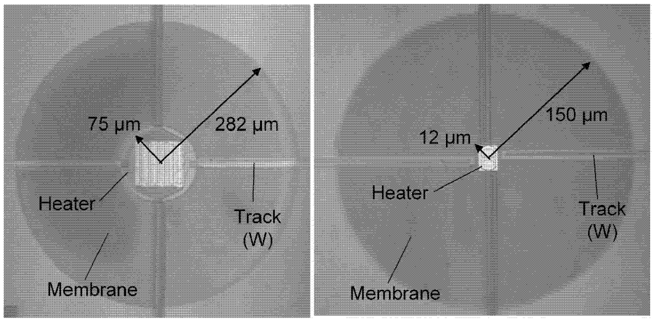


Figure 1. Schematic cross-section of a resistive gas sensor employing SCS micro-hotplate with metal tracks with integrated interface circuitry.



(a) (b)

Figure 2. Photographs of (a) large and (b) small micro-hotplate.

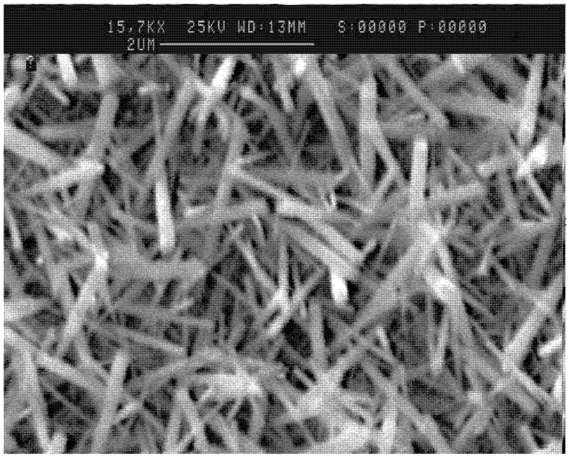


Figure 3. SEM photograph of as deposited WO_{3-x} film.

III. EXPERIMENTS

The power consumption of the large and small micro-hotplates has been measured and the results shown in Figure 4. For this purpose, the SCS heater was used as both the heater and the temperature sensor. The temperature is related to the SCS resistance by:

$$R(T)=R_{27}\left\{1+\alpha(T-27)+\beta(T-27)^2\right\}, \qquad (1)$$

where $\alpha = 1.46\times10^{-3}$ [1/K] and $\beta = 9.1\times10^{-7}$ [1/K^2] (p+) are temperature coefficients of resistance obtained by the measurement of resistance in a temperature controlled furnace (Carbolite/AAF1100). Hence we found that the large and small heaters require only 30 mW and 12 mW, respectively, to operate at 500 °C.

Reliability tests were also performed. The micro-hotplates with SCS (n+, p+) and polysilicon (for comparison) heaters were continuously operated with a constant voltage. The operating temperatures were 350 °C (for chemoresistive type) and 500 °C (for calorimetric type) for 500 hours. The results are shown in Figures 5 and 6. It was found that the p+ doped SCS heater was the most stable at both 350 and 500 °C. The drift of the p+ doped SCS heater was less than 1 % after being operated at 500 °C for 500 hours.

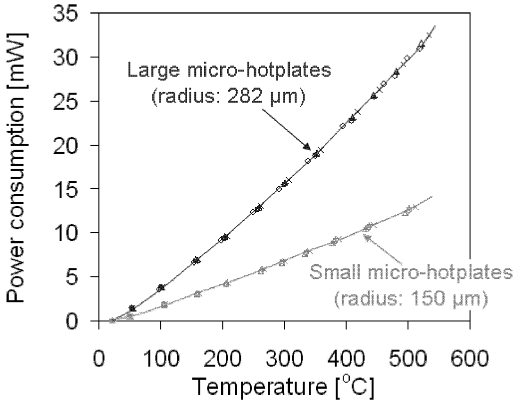


Figure 4. Observed power consumption of SOI-CMOS micro-hotplates.

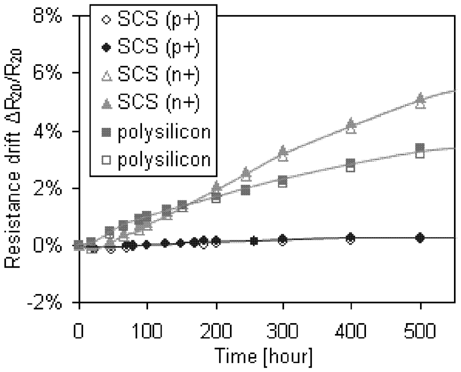


Figure 5. Resistance drift when operated at 350 °C.

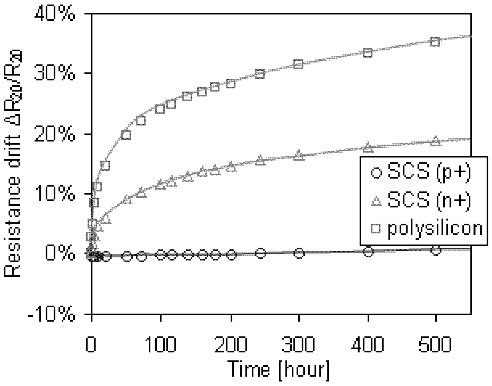


Figure 6. Resistance drift when operated at 500 °C.

Figure 7 shows the response of a resistive gas sensor coated with WO$_{3-x}$ nanorods to ethanol vapour in air. The gas sensor chip was tested in a chamber at 30 °C with a background humidity of 3000 ppm (r.h. 7.1 % at 30 °C), and the micro-hotplate was operated at a temperature of *ca.* 350 °C. The gas response follows the power law where the exponents are 0.63, which is typical for metal oxide gas sensors [12], indicating that the sensor is working properly.

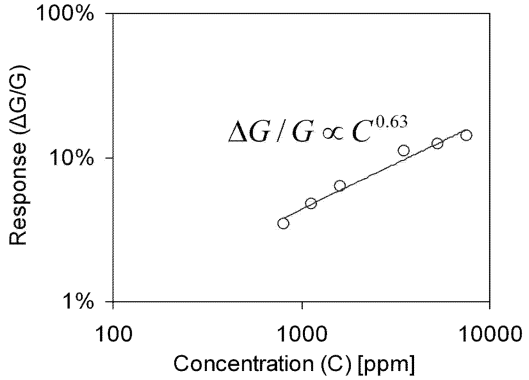


Figure 7. Typical response of APCVD based WO$_{3-x}$ nanorods at 350 °C.

IV. DISCUSSION

The 12 mW power consumption of these SCS heaters at 500 °C and 8 mW at 350 °C are, as far as we know, the smallest that have ever been reported. Previous work by Sheng, reports a power consumption of 12 mW to operate at 300 °C [4]. The extremely large ratio of membrane to heater radii makes this possible. The main power loss of the proposed micro-hotplate is by conduction through the membrane [8], which is given by the following (for a circular membrane):

$$Q_{conduction} \propto 1/\ln\left(r_m/r_h\right), \qquad (2)$$

where r_m and r_h denote the radii of the heated and membrane area [13]. The membrane to heater ratio of our small micro-hotplate is 12.5, whereas that of previous work was *ca.* 2 [4].

Although it has been observed that the thermal stability of the *p*+ SCS is much higher than the *n*+ SCS or polysilicon, the precise cause is not known. A possible reason, for this resistance drift of the SCS heater, is the stress induced by the deformation of the membrane at high temperatures. Optical interferometer (Wyko/NT2000) was used to measure this deformation and it was found that the membrane deforms significantly at high temperatures (maximum deflection is *ca.* 10 µm at 500 °C) the results are shown in Figure 8. This is due to the difference in temperature expansivity of the layers within the SOI membrane. This deformation induces a stress in the SCS heater. Interestingly, Demenet *et al.* reported that the yield stress of *n*-type heavily doped SCS is much lower than that of intrinsic silicon, whereas *p*-type doping has only a small effect on yield stress [14]. The reported values of yield stress at 500 °C are 60, 180 and 210 MPa for *n*-type (6×10^{18} cm^{-3}), *p*-type (1×10^{18} cm^{-3}), and intrinsic silicon. This lower *n*-type value is natural because the dislocation velocity of heavily doped *n*-type silicon is much faster (*ca.* 10 ×) than that of *p*-type silicon at 500 °C [15]. Therefore, it is possible that the membrane deformation is causing an increased plastic deformation of *n*+ SCS over *p*+ SCS during operation. Furthermore, it is also known that the dislocation motion during plastic deformation causes multiplication of dislocations [16], and it is these dislocations that cause the increase in the resistivity, hence resistance [17]. This would result in an increased drift in *n*-type SCS heaters over *p*-type, as observed here.

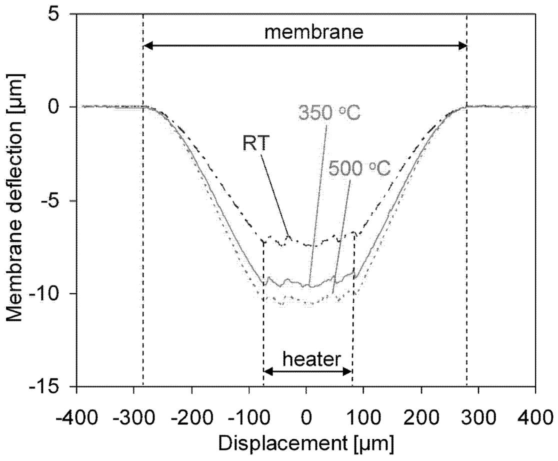


Figure 8. Membrane deformation at RT, 350 and 500 °C.

V. CONCLUSIONS

Here, novel gas sensors based on highly doped SCS micro-hotplate have been designed and characterised. These gas sensors were fabricated using a standard SOI-CMOS process that could offer low production costs, with a power consumption of only 12-30 mW to operate at 500 °C. The drift of resistance for *p*-type SCS was found to be significantly less than that of polysilicon or *n*-type, (less than 1 % when operated at 500 °C for 500 hours). APCVD based novel tungsten oxide nanorods were deposited directly onto the micro-hotplate to form a resistive gas sensor, and its operation has been demonstrated.

ACKNOWLEDGEMENT

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