Combined smart chemFET/resistive sensor array

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Abstract

Here we describe a novel CMOS compatible gas sensor array based on a combined resistive/chemFET sensor cell. We have fabricated an array of 70 sensors with integrated drive, gain and baseline removal circuitry using an AMS 0.6 µm CMOS process. The sensing materials are carbon black/polymer composite (CB) thin films, which have been previously reported to have good vapour-sensing properties. Different CB films have been deposited onto the sensor array and have been shown to respond differently to volatile organic compounds. This combined sensing element both reduces silicon area and, more importantly, measures different physical properties of the same gas sensitive material improving discrimination and giving more insight into the sensing mechanism.

Keywords

Combined chemFET/resistive Gas Sensor, Sensor Array, Offset Cancellation.

INTRODUCTION

There have been a number of attempts over the last decade to integrate chemical sensors within a standard CMOS process. Early work used a resistive chemical sensor with separate ASIC (Application Specific Integrated Circuit) that contained signal processing circuitry [1]. Later designs directly integrated the sensor with signal processing circuitry, resulting in a number of sophisticated devices. For example Hagleitner *et al.* [2] have created a chip with four different integrated chemical sensors employing different sensing mechanisms. Work with FETs as chemical sensors has been investigated since its discovery in 1975 [3]. Such devices would seem ideal for CMOS integration due to the FET being the cornerstone of modern electronics. Unfortunately, the high operating temperature (>200 °C) and non-standard gate material, in this case palladium, makes these devices difficult to integrate. Research into polymer gate FET devices has removed some of these problems, such as the high operating temperature and using polysilicon as the contact to the sensing material [4]. The main issue with this technology is the post-processing required to expose the polysilicon gate and gate oxide. An alternative approach is to use a capacitive coupled design, where a capacitive plate is connected to the floating gate of a FET [5]. Alternative work has investigated large resistive chemical sensor arrays with an excess of 1000 sensing elements [6]. This array used a carbon black composite polymer as the sensing material and was fabricated using a standard CMOS process.

Here we report on a combined chemFET/resistive sensor array fabricated using an AMS 0.6 µm CUP CMOS process, employing carbon black (CB) composite materials, with integrated signal processing circuitry. It differs from previous work in a number of ways. Firstly, it is the first resistive CMOS compatible sensor array integrating different CB's polymers into the same array. In addition, it is the largest FET sensor array using CB materials. Lastly, it is the first time both a chemoresistor and chemFET have been combined with integrated signal processing electronics.

COMBINED SENSOR CONCEPT

The resistive sensor component is simply formed by depositing CB polymer between two sensor electrodes. The FET section is based on the floating gate concept, were the sensing material is capacitive coupled to the floating gate of a FET. As the gate is floating, any potential created through the interaction of the sensing material with the target gas or vapour appears, due to capacitive coupling, on the gate of the FET. It has previously been reported [5] that this is due to a change of workfunction within the sensing material. The floating gate will have an absolute potential subject to variations in the fabrication process. A biasing plate is added underneath the sensing plate and biased to ensure the transistor is turned-on. The capacitive plate is placed in-between the two resistive electrode elements, hence the same sensing material is used for both resistive and FET measurements. This is shown in figure 1 below.

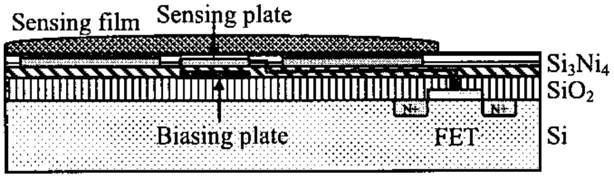

Figure 1: Combined resistive/chemFET concept and schematic of operation.

Here polymer composite materials are used as the sensing film. These combine an insulating polymer/rubber, usually used in gas chromatography with carbon black nanoparticles. The carbon endows electrical properties to the composite material. In the resistive configuration, exposure to a gas or vapour causes a swelling of the composite material, resulting in an increase in resistance [6]. Work with FET devices with these materials suggest that exposure to vapours alters the work function of the composite materials [4]. Hence our combined sensor concept can measure the change in resistance *and* workfunction of the same material, giving additional discriminatory information. Lastly, the combined sensor concept reduces the silicon area of the array, giving more space for the remaining integrated circuitry.

COMBINED SENSOR DETAILS

The resistive element is two metal opening (60 μm × 60 μm) separated by a 65 μm gap, between which a CB polymer film is deposited. The extended 110 fF capacitive plate (72 μm × 15 μm) lies between the two resistor openings, and is connected to the floating gate of a MOSFET. The equivalent circuit model for the FET sensor is shown in figure 2. V_S is the potential developed by the sensing material, I_{SENS} is the current through the FET, C_{SENSOR} is the sensor capacitance, C_{SUB} is the substrate capacitance, C_{GATE} is the gate capacitance, $C_{PARASITIC}$ is the parasitic capacitance and V_{GATE} is the gate voltage.

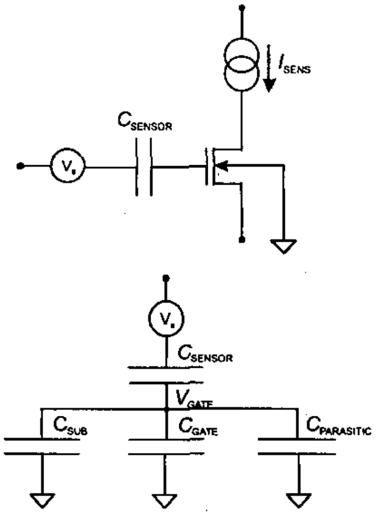

Figure 2: Circuit schematic and capacitive diagram for the chemFET sensor.

Based on the dimensions of the capacitive plates and the materials between each layers, we are able to calculate the gate voltage as a ratio of the capacitances using process parameters from the equation:

$$V_{GATE} = \frac{C_{SENSOR}}{C_{SUB} + C_{GATE} + C_{PARASITIC} + C_{SENSOR}} \times V_S$$

Using this expression we have calculated the total loss of signal by capacitive coupling from the voltage source as typically 25%.

INTEGRATED CIRCUITRY

Included with each sensor is circuitry to drive and process any response. Each resistive/chemFET sensor is driven by a constant current source and can have three different values (1 μA, 10 μA and 100 μA). This is used to compensate for variations in resistance with different coatings. To remove the DC baseline value of the sensor, an offset cancellation circuit has been added to the design. The circuit allows high gain values to be used without saturating the output. In our design, the DC offset cancellation circuitry uses a ramping DAC with a comparator. The 10-bit DAC ramps to estimate the offset voltage and the value maintained in an internal counter register. The ±1 bit error is only 5 mV and takes only 512 μs (based on 2 MHz clock) to perform a cycle of scanning the offset voltage as all cells are performed in parallel. This baseline removal can be done either before exposure to a vapour or in-situ. Hence, any long term drift of the sensor or changes in environmental conditions that alter the DC baseline can be compensated for. Once the baseline has been removed the signal is amplified with gains of 1, 5, 10 or 100. These values are programmed externally and can be altered whilst a test is running. Lastly, the signal is conditioned with a low pass filter set at a corner frequency of 1 kHz. The output voltage is at present measured externally using a data acquisition card. The control bits for each cell are contained within 4 D-type flip-flops and are loaded from an external source. A simplified schematic of the offset removal circuitry is given in figure 3.

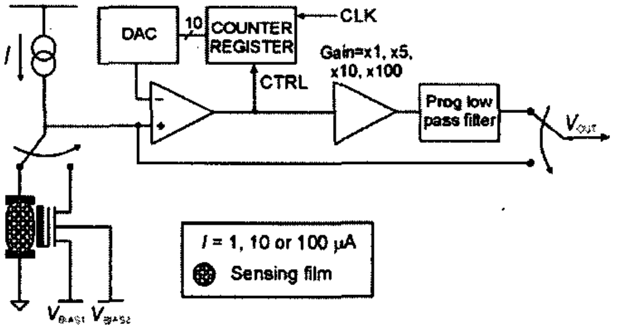

Figure 3: Simplified version of the offset cancellation circuitry.

FABRICATED CHIP AND POLYMER DEPOSITION

The sensor array was designed and implemented in the AMS 0.6 μm CUP process and has been tiled across the whole chip. Figure 4 shows a photograph of one cell of the fabricated chip, whilst a close-up of the combined resistor/FET component in shown in figure 5.

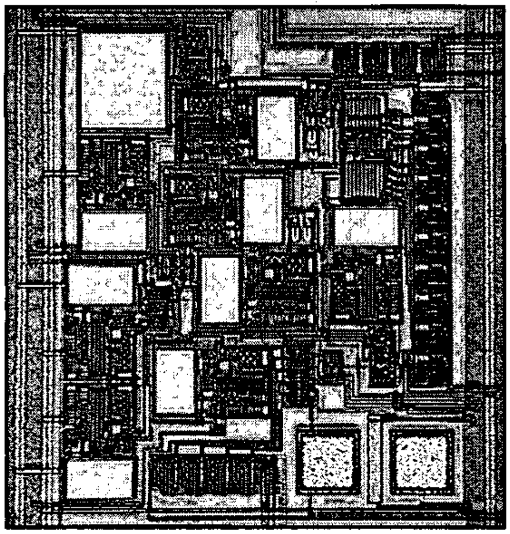

Figure 4: Photograph of the integrated electronics.

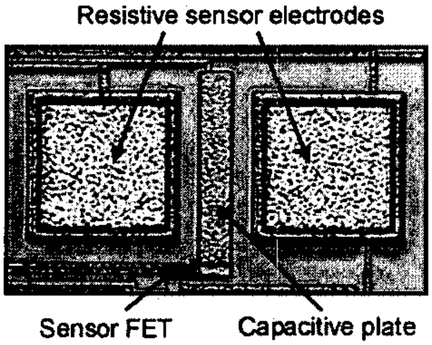

Figure 5: Close up of the combined chemoresistive/chemFET sensor.

Before CB polymer deposition, the aluminium electrodes have been gold plated. This is to ensure a stable contact between the CB polymer and the aluminium. Five different CB polymers were used as given in Table 1.

Table 1: Polymer materials used.

Type	Polymer material
1	Poly (styrene-co-butadiene), PSB
2	Poly (ethylene glycol), PEG
3	Poly (Ethyl-co-vinyl acetate), PEVA
4	Poly (4-vinyl phenol), PVPH
5	Poly (caprolactone), PCL

Polymers are mixed with 20% carbon-black loading by weight in a suitable solvent. The polymers used are obtained from Sigma Aldrich (UK) and used as received. The carbon-black is Black Pearls 2000 nanomaterial purchased from Cabot Corp. (USA). The mixture is agitated for 10 minutes and then sprayed onto the inter-electrode gap through a mechanical micromask using a micro spraying system. As each row (14 sensors) is deposited at a time to ensure the resistances are similar. Figure 6 shows the device deposited with the five different polymers.

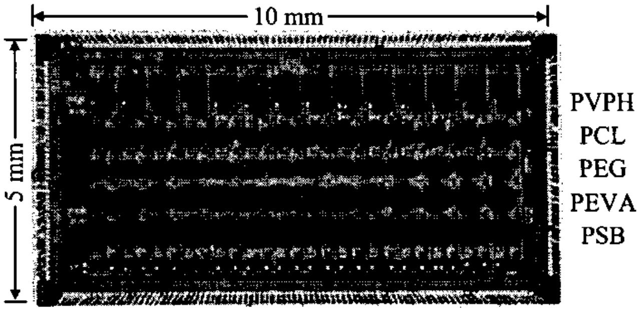

Figure 6: Photograph of fabricated device, with polymer coating

TESTING

The device is bonded into a PGA256 socket and interfaced to a data acquisition and mass flow system. A micro-lid seats on top of the device to form a sensor chamber and has a volume of 50 µl, as shown in figure 7. The host is a PC running LabVIEW with custom-designed software to automate all test cycle and data logging.

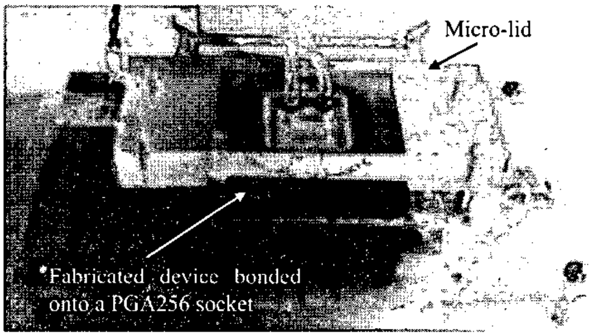

Figure 7: Assembled chemical sensing system.

Preliminary tests have shown the offset cancellation circuit can indeed remove some of the baseline offset. This circuit is still being characterised and results will be published elsewhere. Chemical tests have been performed with the circuitry in "by-pass" mode, in this case the signals are routed directly out and still contains the baseline.

Results from resistive component are shown in figure 8. Here, a device coated with PSB has been exposed to five different concentrations of ethanol (180, 260, 370, 520 and 1150 PPM) and toluene (100, 160, 220, 310 and 700 PPM) vapour in air at a constant temperature of 30 °C and humidity (20 % r.h.). The percent response refers to the percentage change ($\Delta R/R$) in resistance.

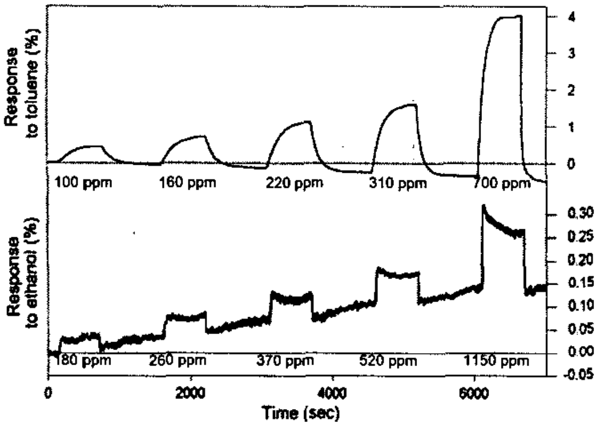

Figure 8: Results of PCB resistive sensor to six different concentrations of ethanol and toluene vapour in air.

From these results we can plot the static effect of toluene and ethanol vapour in air as shown in figure 9. Here the results from a column (14 sensors) are combined.

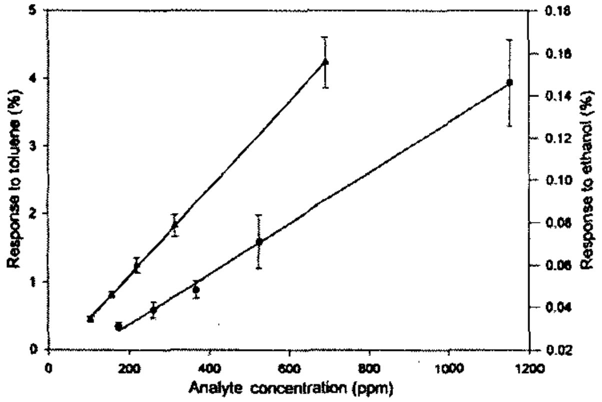

Figure 9: Static sensor output, fitted with a linear model.

The sensitivity to ethanol vapour is about 0.00012 %/ppm and 0.00644 %/ppm to toluene vapour. We have also tested other sensors with different coatings (figure 6) to ethanol vapour in air and the results are shown in figure 10. Here the sensor array is exposed to a 5 sec ethanol vapour pulse at a flow rate of 25 ml/min. The results show that different types of polymer films provide different response profiles (magnitude and response time), thus providing added discriminatory information.

CONCLUSIONS

In this paper we have described a novel sensor array with a combined chemFET/resistive sensing element. We have described a design where the FET component is placed *in-between* the resistive electrodes and so uses the *same sensing material* for both resistive and FET elements. The FET section is based on a capacitive coupled plate connected to the floating gate of the MOSFET. By employing this configuration both the size of the sensor cell is reduced, and two different sensing properties of one polymer film can be used to increase the discriminatory power of the array. In addition, full signal processing electronics have been integrated with the sensors.

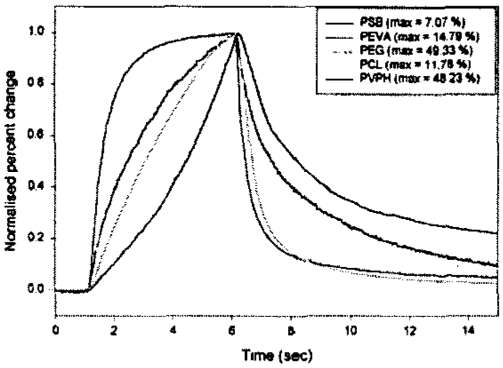

Figure 10: Results of five resistive sensors of different sensing materials to a 5 sec ethanol vapour pulse in air.

Preliminary results have shown that the resistive component of the combined sensor array operates as expected. In addition, the offset cancellation circuit has been shown to perform its required function, though it has still to be tested with the sensor. Lastly, work is presently being undertaken to characterise the chemFET sensor components.

ACKNOWLEDGEMENTS

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