CMOS INTEGRATED CHEMICAL MICROSENSORS AND THEIR CIRCUITRY

Special Issue Review

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1. INTRODUCTION

Chemical sensors and electronic noses are used nowadays in a variety of industrial and environmental applications, such as on-line process monitoring in the food-industry and personal safety by threshold limit value (TLV) monitoring. Commercially available desktop-size electronic noses (e.g., MOSES II [1]) are based on arrays of chemical sensors utilizing different chemically sensitive films for selectivity enhancement. Sensing structures include, e.g., quartz crystal microbalances, surface acoustic wave devices, calorimetric sensors, and metal-oxide sensors.

In recent years, industrial CMOS and BiCMOS processes in combination with silicon micromachining have been used for the microfabrication of a variety of sensors, such as infrared radiation detectors, flow sensors, and pressure sensors [2,3,4,5]. The sensors benefit from the reliability of well established IC processes and the cointegration of signal conditioning circuitry.

In this paper, we review CMOS chemical microsystems developed at the Physical Electronics Laboratory of ETH Zurich for detection of VOCs in air. Prototypes based on three different sensing principles are presented: (i) capacitive, (ii) gravimetric or resonant, and (iii) calorimetric chemical microsensors. The emphasis of the paper is on the signal conditioning circuitry cointegrated with the different sensing structures. The combination of arrays of these sensors with different chemically sensitive polymer films leads to the development of hand-held application specific electronic micronoses [6].

2. MICROSENSOR FABRICATION

The chemical microsensors presented in this paper are fabricated using an industrial 0.8 μm CMOS process from *Austria Mikro Systeme International* (Unterpremstätten, Austria) in combination with post-CMOS micromachining and polymer deposition. After completion of the CMOS process, silicon membranes and suspended n-well structures are released by anisotropic etching from the back of the wafer using a potassium hydroxide (KOH) solution in combination with an electrochemical etch-stop technique. The etching stops at the pn-junction between the CMOS n-well and the p-type substrate. The special preparation sequence for the electrochemical etching of CMOS wafers is described in [7] and is available as a foundry service from *Austria Mikro Systeme* (AMS).

The electrochemical etch-stop technique provides silicon-based microstructures that benefit from the excellent mechanical properties of silicon. Moreover, active devices can be designed within the n-well structures. By combining the described bulk-micromachining process with additional reactive-ion-etching (RIE), not only membrane structures, but also bridges and cantilever beams can be fabricated.

3. CMOS CHEMICAL MICROSENSORS

After completion of the post-CMOS micromachining steps, polymer films acting as chemically sensitive layers are deposited onto the sensing structures by spray-coating through a shadow mask. Judicial choice of the chemically sensitive polymer layers provides a trade-off between reversibility of the polymer-analyte interaction and sensitivity towards certain analytes.

The microsensor dice are mounted in standard 28-pin ceramic dual-in-line packages for testing. A flip-chip packaging technique for the chemical microsystems has also been demonstrated [8]. For characterization, the dual-in-line packages containing the CMOS gas sensors are mounted onto the measurement chamber of a computer-controlled flow setup [9].

3.1. CAPACITIVE MICROSENSORS

In the case of capacitive chemical microsensors, the change of the dielectric constant of the polymer upon absorption of analyte is sensed. To this end, interdigitated capacitor structures are formed by the two metal layers of the CMOS process [8]. In order to enable measurements at different temperatures, the sensor and reference capacitors are placed on thermally insulated island structures.

Figure 1. Photograph of the capacitive chemical microsystem. The interdigitated capacitors are located on thermally insulated n-well islands to allow measurements at different temperatures [10].

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tures (micro hotplates) released using anisotropic etching with electrochemical etch-stop [10]. On-chip heating resistors consisting of the gate polysilicon layer of the CMOS process are located on the island structures. The temperature is measured by pn-diodes integrated in every n-well island. A photograph of the complete capacitive chemical microsystem chip is shown in Fig. 1.

On-chip readout circuitry converts the difference between the polymer coated sensing and a reference capacitance into a digital output signal. This is done by incorporating both capacitors in the first stage of a fully differential second order switched capacitor $\Sigma\Delta$ -modulator [11]. The converter provides a pulse density modulated output that can be decimated using a frequency counter.

Figure 2. First stage of second order switched capacitor $\Sigma\Delta$ -modulator. Sensor and reference capacitors are split in two parts to improve the charge transfer efficiency [10].

Fig. 2 shows the first stage of the $\Sigma\Delta$ -modulator. With $V_{ref}=V_{fb}$ [11] and given the small capacitance changes due to analyte absorption, the converter uses only a fraction of its dynamic range. By operating the first stage of the $\Sigma\Delta$ -modulator at a higher clock frequency compared to the rest of the converter, the signal can be amplified. With every clock cycle Φ_{int} , the charge $V_{ref}*(C_S-C_R)$ is accumulated on the integrating capacitor C_{int} . If the conversion takes place at a 2^n times lower clock frequency $\Phi_{\Sigma\Delta}$, the output frequency is given by [10]

$$f_{out} = \frac{f_s}{2} \left(1 + 2^n \frac{C_S - C_R}{C_{fb}} \right)$$

with C_S , C_R , and C_{fb} denoting the sensor, reference, and feedback capacitance and f_s the sampling frequency.

In order to minimize the sensitivity of the output signal towards temperature variations and aging, all feedback capacitors required in the circuit are realized as interdigitated capacitors and are located on a thermally insulated n-well island together with the reference capacitor.

After dicing, the sensing capacitors were spray-coated with a 10 μm poly(etherurethane) (PEUT) layer while the reference and feedback capacitors were covered with a 10 μm poly(dimethylsiloxane) (PDMS) layer. The limit of detection of the capacitive microsystem at 28 $^{\circ}\text{C}$ for toluene and ethanol was measured to be 8 ppm and 5 ppm, respectively [10].

Fig. 3 shows the measured frequency shift for 4250 ppm of ethanol and 2600 ppm of toluene at different sensor temperatures. The measured absolute frequency shift for a single analyte decreases exponentially with temperature because of the temperature depen-

dence of the partition coefficient. Different exponential factors of the sensor responses for ethanol and toluene reflect the different temperature dependences of the partition coefficients. Thus, the temperature dependent output signal can distinguish between different analytes. The frequency shifts for a mixture of analytes can be calculated by adding the signals from the single analytes [10].

Figure 3. Measured frequency shift of the capacitive chemical microsystem as a function of the sensor temperature for 4250 ppm ethanol, 2600 ppm toluene, and a mixture of 4250 ppm ethanol and 2600 ppm toluene [10].

3.2. RESONANT MICROSENSORS

Resonant or gravimetric chemical sensors sense the mass change of the chemically sensitive polymer layer upon absorption of analyte molecules. A CMOS resonant chemical sensor based on a 150 μm long silicon cantilever beam is shown in Fig. 4 [12].

Figure 4. Micrograph of a 150 μm long resonant beam gas sensor connected to an on-chip preamplifier [12].

The beam consists of the silicon n-well covered by the dielectric layers of the CMOS process. The cantilever structures are released by anisotropic etching with an electrochemical etch-stop technique and two additional reactive-ion-etching steps. After micromachining, the cantilevers are spray-coated with the chemically sensitive polymer film. The cantilever beams feature resistors for electrothermal excitation and piezoresistive detection of transverse vibrations. The four diffused p⁺-piezoresistors are arranged in a Wheatstone bridge configuration. An on-chip low-noise differ-

ential difference amplifier (DDA) as shown in Fig. 5 is used to amplify the weak signals from the Wheatstone bridge and eliminate the capacitive crosstalk observed with off-chip amplification.

Figure 5. Block diagram of differential difference amplifier cointegrated with the gravimetric chemical microsystem.

In contrast to an operational amplifier, the DDA amplifies the difference between its differential input voltages. The gain is defined by the feedback resistors R_a and R_b :

$$A_v = 1 + \frac{R_a}{R_b}$$

Compared to the standard instrumentation amplifier configuration with two operational amplifiers, the DDA offers a reduction in circuit complexity and an independent choice of the output common mode voltage.

The gravimetric chemical sensor is operated at its fundamental resonance frequency using an external feedback loop [12]. To this end, the output signal of the DDA is limited by a comparator and then applied to the heating resistors. The performance of the DDA and feedback system are summarized in Table 1.

Differential Difference Amplifier (DDA)	
Gain	30 dB
3-dB bandwidth f_{3dB}	500 kHz
Equivalent input noise	15 nV/ $\sqrt{\text{Hz}}$
Feedback Loop	
Fundamental frequency	354 kHz
Allan variance (short time stability)	0.03 Hz

Table 1. Performance of differential difference amplifier cointegrated with the resonant gas sensor and stability of the feedback loop.

The linear response of the resonant gas sensor coated with a 2.4 μm poly(etherurethane) (PEUT) layer to the analyte concentration is shown in Fig. 6 for different analytes. Assuming a minimal detectable frequency shift of 1 Hz, the limit of detection (LOD) is less than 10 ppm for toluene and 70 ppm for ethanol [12].

3.3. CALORIMETRIC MICROSENSORS

The calorimetric chemical sensor measures the enthalpy change upon absorption and desorption of analyte molecules into the chemically sensitive polymer. The CMOS calorimetric gas sensor shown in Fig. 7 consists of a 595 μm by 2162 μm suspended n-

Figure 6. Frequency shift of a resonant beam gas sensor as a function of different concentrations of various analytes. The gas sensor was covered with 2.4 μm of PEUT [12].

well membrane with 300 thermocouples covered by the chemically sensitive polymer [13]. Enthalpy changes during absorption and desorption of gaseous analytes into the polymer cause (transient) temperature variations of the thermally insulated micromachined membrane. Polysilicon/aluminum thermopiles are used to measure these temperature variations. A polymer coated sensing membrane and an uncoated reference membrane are connected in parallel to the input stage of a low-noise amplifier to measure the temperature differences between the two membranes.

Figure 7. Micrograph of the calorimetric chemical sensor with a rectangular n-well island structure, thermopile, metal heater, and on-membrane diode [13].

Fig. 8 shows the block diagram of the low-noise chopper-stabilized instrumentation amplifier that has been designed to measure the weak sensor signals. In order to avoid the large $1/f$ -noise in CMOS input stages, the input signals are first mixed to higher frequencies by the input chopper. Then, the signal is amplified by a three stage amplifier and finally demodulated by the output chopper. The bandpass filter after the first amplification stage avoids saturation of the system by the DC-offset of the first amplifier and reduces the residual offset caused by demodulated charge injection from the input chopper [14]. The amplifier features a gain of 75 dB, a bandwidth of 500 Hz, and an equivalent input noise of 12 nV/ $\sqrt{\text{Hz}}$. The equivalent residual input offset was measured to be smaller than 1 μV .

Figure 8. Block diagram of low-noise chopper amplifier [14].

In contrast to the capacitive and gravimetric chemical sensors, the calorimetric sensor detects transient (non-equilibrium) signals only, i.e., changes in the analyte concentration. Figs. 9 (a) and (b) show the output voltage of the microsystem while switching 1500 ppm of toluene in air on and off, respectively, at 33 sccm flow rate and a temperature of 28 °C [13]. The sensing membrane was coated with a 10 µm thick poly(etherurethane) layer. The enthalpy changes are calculated by integrating the sensor output signal and calibration using a known heating power dissipated in a metal heating resistor located on the membranes. The resulting enthalpy changes of about 30 µJ are identical for switching on and off the analyte.

Figure 9. Output voltage of the calorimetric chemical sensor system while switching (a) off and (b) on 1500 ppm of toluene in air at 28.0 °C. The resulting enthalpy change was calculated by integrating the sensor output signal and calibration using the integrated heating resistor [13].

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