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Silicon-Based Thermal Conductivity Detector for Gas Sensing

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Summary:

We present a novel Thermal Conductivity Detector (TCD) for gas sensing without the use of a chromatography column. Its original architecture is based on a suspended membrane on top of which are deposited a heating element and a separated sensing element made of amorphous silicon. These sensors are micro-fabricated and tested in a climatic chamber. They reach a theoretical detection limit (3 sigma) of 13 ppm for carbon dioxide in air and exhibit a signal-to-noise ratio 4 times higher than conventional platinum TCDs.

Keywords: gas, sensor, thermal, conductivity, detector

Introduction

Thermal Conductivity Detectors (TCDs) are among the most widely used sensors in gas chromatography (GC) systems [1] and well appreciated for their universality. Their working principle relies on the heat transfer between a metallic coil heated by Joule effect and the surrounding gas, resulting in a change of the coil electrical resistance. Recently, miniaturized TCDs (µTCDs) have been produced on silicon substrates using microfabrication techniques. This level of integration greatly improves the sensitivity of this technology, allowing to reach the subparts per million (sub ppm) detection limit when coupled to a GC system [2]. Moreover, it reduces the power consumption of the detector, opening the way to portable air quality monitoring systems. In that perspective, Bourlon et al. reported the use of a single µTCD chip to measure simultaneously the CO_2 concentration and relative humidity in air with respective errors of 300 ppm and 2% [3]. In this work, we present our latest investigations into the analysis of ternary gas mixtures using our novel micro-fabricated thermal conductivity sensor using a unique architecture.

Materials choice

Several materials for the sensing element have been considered but the boron-doped hydrogenated amorphous silicon (a-Si: H(B)) was chosen for its CMOS compatibility and high sensitivity. Indeed, we have estimated the temperature coefficient of resistance (TCR) at $1{,}66\cdot10^{-2}$ K^{-1} by a four-probe measurement of the electrical resistivity as a function of temperature on a full wafer. This value is one order of magnitude higher than the TCR of platinum. Regarding the heating element, we have chosen titanium nitride (TiN) for its CMOS compatibility and relatively low resistivity of $1{,}5\cdot10^{-4}$ $\Omega\cdot$cm allowing to reach ~200°C with 3V across the element.

Sensor Chip Fabrication and Architecture

TCD sensor chips are micro-fabricated in our clean room facility by bonding two silicon wafers. Silicon nitride (Si_xN_y) membranes are etched in the bottom wafer after having deposited the TiN and a-Si: H(B) layers for ensuring heating and temperature probing respectively (see Fig. 1).

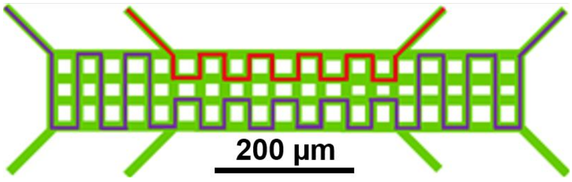

Fig. 1. Schematic of the suspended Si_xN_y membrane (green) with a-Si: H(B) sensing element (red) and TiN heating element (blue).

Fluidic channels ensuring passive gas diffusion to the suspended membrane are etched by deep reactive ion etching in the top wafer.

Four suspended membranes are used on the same silicon chip in order to have an accurate differential measurement of the resistance variations of the sensing elements using a Wheatstone bridge configuration. Two membranes are placed in an analysis cavity opened to the ambient air by fluidic channels; the other two membranes are hermetically sealed in a reference chamber (see Fig. 2).

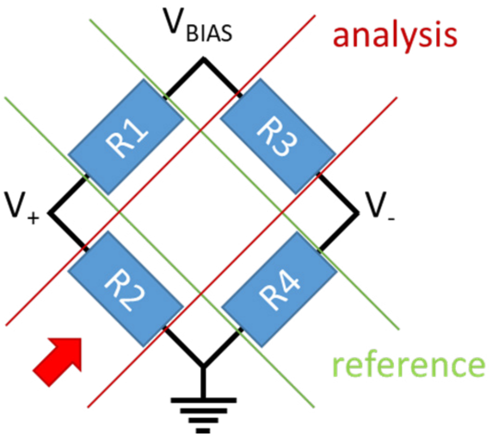

Fig. 2. Schematic of the Wheatstone bridge configuration of the four a-Si: H(B) sensing elements

Experimental Setup

The TCD silicon chip is wire-bonded onto a printed circuit board in order to connect the heating and sensing elements to a specifically designed electronic circuit. This driving electronics is able to apply sequentially two different voltages on the heating elements in order to thermalize the whole membranes at two different temperature. At the same time, it applies a voltage V_{BIAS} on the Wheatstone bridge composed of the four sensing elements and measures the corresponding signal $V_+ - V_-$.

In order to monitor the concentrations of two analytes mixed with a third component in excess (e.g. CO_2 and H_2O in air), the silicon die and its driving electronics are placed into a climatic chamber in which we control the two analytes concentrations.

After performing a calibration at a minimum of three different concentrations of the two analytes, we are able to match the TCD signals obtained for two heating temperatures with the two analytes concentrations.

Results

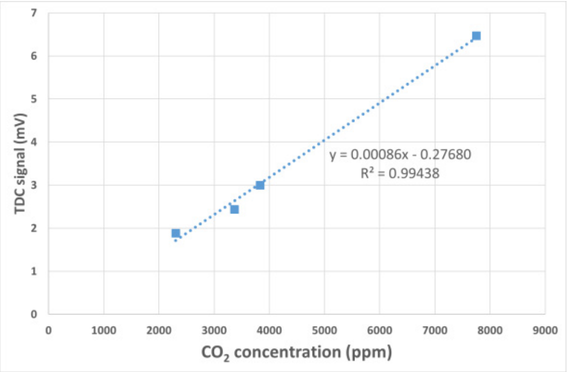

Fig.3. Measured TCD signal as a function of the CO_2 concentration

Fig.3. illustrates the measurement of the CO_2 variations in the presence of H_2O and synthetic air. The TCD sensor shows a good linearity with a coefficient of determination R^2 of 0.994. Moreover, we have compared the sensor with conventional platinum TCDs in the same experimental conditions. As can be seen in Tab.1, these sensors are almost 8 times more sensitive to CO_2 variations than conventional platinum sensor. However, they are also more sensitive to noise, resulting in a signal-to-noise ratio 4 times better than the platinum TCDs.

Tab. 1: Comparison of the conventional platinum TCD technology with the a-Si: H(B)/TiN architecture

Sensing Technology	Signal for 100 ppm variation of CO_2 (µV)	Noise average (µV)	Signal to Noise Ratio
Platinum	11.5	2.1	5.48
a-Si: H(B) and TiN	89.1	3.8	23.45

Aknowledgement

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