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Differential Thermal Conductivity Hydrogen Sensor

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Summary:
In this work, a thermal conductivity sensor for the detection of hydrogen in fuel cell applications is presented. The sensor uses a differential measuring concept with two cavities combined on one semiconductor-die where one of those cavities is hermetically sealed and filled with nitrogen as a reference gas. The sensors were characterized with hydrogen concentrations between 0 and 3.5 % at different heater temperatures. A 3σ-noise level of 0.06 % hydrogen was achieved with the prototype sensor.

Keywords: thermal conductivity, MEMS, hydrogen, gas sensor, automotive

Introduction

Greenhouse gases such as carbon dioxide are drivers of climate change. Many governments are increasing their commitment to climate protection in order to slow down the warming of the planet. For example, Germany is aiming for greenhouse gas neutrality by 2050 [1]. Within the transportation sector, activities toward electrification and zero-emission drives continue to increase. Using hydrogen as a fuel is one pillar to allow decarbonization where electrification is not possible or reasonable [2].

To avoid endangering passengers or pedestrians, the use of hydrogen as a vehicle fuel also increases the demand of hydrogen gas sensors. Since the thermal conductivity of hydrogen at room temperature is seven times higher than that of air, measuring thermal conductivity is a feasible method to detect hydrogen. Even though other sensor principles like electrochemical and catalytic sensors may show higher sensitivities and selectivity toward hydrogen, they show some major drawbacks regarding an application in the automotive sector such as a short lifetime and vulnerability to poisoning, respectively [3]. A thermal conductivity sensor implemented as a micro-electrical-mechanical system (MEMS) offers additional advantages such as miniaturization, low production costs and low power consumption.

The measurement principle itself is based on resistivity changes of an electrically heated free-standing resistive element. This hot element is a resistor which is heated by short pulses. The resistor should be thermally decoupled from the substrate so that most thermal energy is transferred via the surrounding gas. The hot element cools down when hydrogen is present, and a change of resistivity can be measured.

A MEMS-based sensor using thermal conductivity to measure hydrogen concentrations below the lower explosive limit of 4 % is presented. A differential measurement concept with four geometrically identical elements is used, two located in a hermetically sealed cavity containing a reference gas.

Method

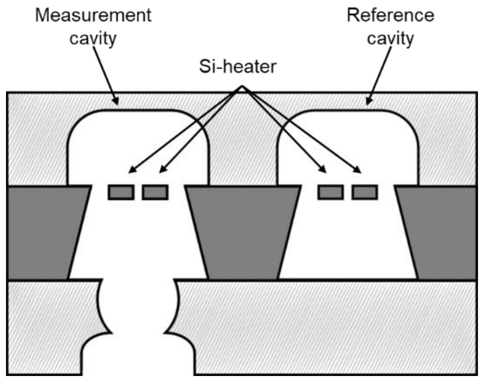

Fig. 1. Schematic cross-section of the MEMS structure. A sensor detects the thermal conductivity of the ambient gas while a reference sensor in a hermetically sealed cavity is surrounded by a defined reference gas.

The MEMS consists of a silicon wafer into which two cavities are etched. One is later used as a measurement cavity with an open port to the surrounding gas. The other cavity is hermetically sealed and filled with a reference gas which should most closely resemble the measurement environment. For the application as a leakage sensor in fuel cell vehicles, the cavity is filled with nitrogen.

Certain areas of the wafer are made conductive by doping. These areas are exempted during the process and later form the electrical leads and sensing elements.

The structured silicon wafer is encapsulated by two glass wafers under nitrogen atmosphere, resulting in a hermetically sealed nitrogen-filled cavity (fig. 1).

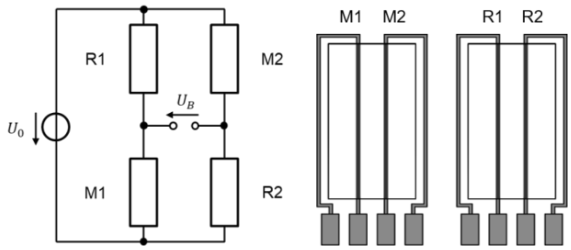

Fig. 2. Schematic top view of sensor structure with four resistors separated in a measurement (M1, M2) and a reference cavity (R1, R2) connected as a Wheatstone bridge.

Each cavity contains two resistors made of doped silicon which act as heater and sensor elements. Two heaters are exposed to the measured medium while the other two heaters are in the reference gas. The heaters are connected in a Wheatstone bridge. Only two of the four resistors are exposed to a change of thermal conductivity in the presence of hydrogen. This differential measurement concept leads to very precise and low-noise measurements. The bridge circuit reduces temperature and aging effects.

Results

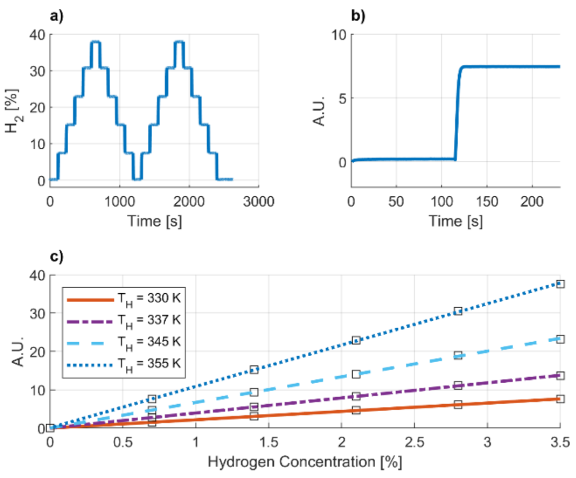

Fig. 3. (a) Raw data of hydrogen concentration measurement from 0 to 3.5 % in 0.7 % steps (T_H = 355 K), (b) the first two concentration steps on a smaller scale and (c) mean values of concentration measurements at four different temperatures T_H of the heaters.

The gas sensing attributes of the sensor were characterized during exposure to varying hydrogen concentrations ranging from 0 to 3.5 % in dry air at a constant gas flow of 500 sccm, at room temperature and ambient pressure. Each concentration step was held for two minutes. The carrier gas was dry air generated by a zero-air generator. The hydrogen concentration was gradually increased from 0 to 3.5 % in 0.7 % steps, then reduced back to 0 %. Then, this sequence was repeated.

The measurement was repeated at four different supply voltages of the bridge. Changing the supply voltage leads to different temperatures of the Si-heater. The performance of the sensor was determined at four different heater temperatures T_H. Fig. 3 a) shows the raw data of a measurement at T_H = 355 K as a visualization of the measurement sequence and the resulting sensor output. Fig. 3 b) shows the first change of concentration on a smaller scale. The response time of the sensor is a few seconds.

Tab. 1: 3σ-noise level of the sensor at different heater temperatures T_H.

T_H [K]	330	337	345	355
3σ-noise [%H_2]	0.17	0.12	0.09	0.06

With this sensor and measurement configuration a 3σ-noise level of 0.06 % hydrogen in air (at T_H = 355 K) is achieved. Table 1 shows the noise level at all measured temperatures.

The measurements show that a higher temperature of the heater results in a higher sensitivity and a better resolution of the sensor. Lower temperatures though reduce power consumption which is very valuable for many applications. The sensor still shows good results at lower heater temperatures and could be used as a leakage detection sensor for hydrogen in the future.

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