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Investigating time-resolved response of micro thermal conductivity sensor under various modes of operation



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ABSTRACT

This article provides measurements of thermal conductivity carried out on a microelectromechanical system (MEMS) structure for the application of selective gas sensing. MEMS thermal conductivity based gas sensors work by measuring the interaction of a heated polysilicon bridge with an ambient gas and sensing temperature changes. The tiny MEMS element provides an ability to measure noble gases and is especially applicable to binary mixtures. In this work we conducted experiments on the KWJ Engineering nano-powered thermal conductivity detector (TCD) elements to investigate the sensitivity of the sensor transient responses to gas composition. Measurements are made at constant power, constant resistance, and constant energy. By introducing feedback to maintain constant sensor temperature, the relative change of gas thermal conductivity with respect to temperature can be estimated and provide information about the concentration as well as insight into the composition of the gas mixture.

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1. Introduction

Gas sensors serve an important purpose in environmental sensing, medical diagnosis, health and safety monitoring, industrial gas detection applications, and an ever-increasing role in consumer products. For example, gas sensors can be used in the home to check for gas leaks, radon, or carbon monoxide. Industrial applications of gas sensors include checking for pipe and valve leaks or pollution levels generated from industrial processes [1,2]. Medical applications of gas sensors include measuring environmental exposure to toxic gases and the emerging area of breath analysis for rapid medical diagnostics that include metabolic and bacterial conditions [3].

There are many types of gas sensors based upon different measurement principles that include but are not limited to: 1] electrochemical devices, which rely upon the oxidation or reduction of the target molecule after diffusion through a porous barrier, 2] Infrared spectrometers, which rely upon measuring the wavelengths of radiation absorbed or emitted by the sampled gas, 3] electronic sensors, which consists most commonly of a thermally controlled semiconductor material that changes resistance upon reactions with the target molecule, and 4] catalytic thermal platforms often called pellistors that are solid-state devices selected

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http://dx.doi.org/10.1016/j.snb.2017.07.142 0925-4005/© 2017 Elsevier B.V. All rights reserved. for a high temperature coefficient of resistance and used to detect gases that are either combustible or react with a catalyst wherein the heat of reaction causes a measureable change in temperature of the pellistor that includes a Pt-resistance thermometer to sense the temperature changes.

Using microfabrication technology, the size, power consumption and cost of gas sensors can be reduced significantly [4]. Through a reduction in the size of the device, using MEMS technology, the heat capacity and power consumption are reduced in our case, enabling a very fast, nano to micro-second response time. Coating a MEMS fabricated micro-TCD [or a hotplate] can result in an electronic sensor wherein the temperature can be controlled by using the sensing element as a fast response low power heater. For example, the tin oxide sensor for measurement of oxidizing or reducing gases, at 500 °C, with a power input of 3.6W was demonstrated in the work of Graf [5]. Ali et al. [6] has demonstrated microhotplates with a temperature of 600 °C using only 12 mW of power.

Microhotplates and pellistors that are microfabricated in general, have lower operating power than miniature analogues based upon hand assembly of the component parts. Gas sensors based on thermal conductivity methods, compared to traditional metal oxide gas sensors [7], have lower power consumption, greater stability, and a longer lifetime [8]. Mahdavifar tested micro thermal conductivity sensors in a variety of gases and measured the time constant and the resistance sensitivity as well as the limit of detection associated with both transient and steady-state calculations [8]. Micromachined thermal conductivity gas sensors are produced



Fig. 1. Typical Voltage Output During a Single Square Wave Pulse Illustrating the 4 Portions of the Response With Microscopy Image of Sensor Showing Microbridges and Electrical Runners in Corner.

through microfabrication process, and hence result in small devices that have a small thermal mass resulting in a fast response time. Simon [9] and Capone [10] have both worked on the miniaturization of gas sensors for commercial and residential applications, lowering the power consumption as well as the overall size. Kaanta et al. [11] have also worked on μ TCD sensors with detection limits of 260 parts per billion for hexane in helium that exhibit linear responses with a response time of 150 μ s. Stetter, et al. has used micro TCD sensors for methane, CO₂, H₂, He, and air detection at room and cryogenic temperatures [12,13] and similar sensor elements are used herein. Manginell, et al. have experimented with polysilicon microbridges in pulsed mode operations [14], comparing experimental and simulated temperature distributions in varying surrounding mediums.

1.1. Principle of operation

An electrothermal gas sensor is based on the heat loss from a heated filament-shaped element. The microfabricated sensor has heat generation by passing a current through an electrically conductive portion of the suspended thermally isolated element. The rate of heat loss from electrical power dissipation is greater for gas mixtures with higher thermal conductivity that are in the vicinity of the heated portion of the element. The primary mode of heat transfer from the beam is conduction, with convection and radiation composing less than 1% of the total heat transfer in a certain region of temperature operation. The operating region was selected to have a low Grashof number (less than 0.01), and this was further confirmed by experiments [15]. Using a material, such as polysilicon, for the sensing element, with a high temperature coefficient of resistance as the heating element, the changes in heat loss rate can be related to changes in gas thermal conductivity. This also allows an estimate of the average temperature of the heating element, calculated by assuming a linear relationship between resistance and temperature.

A square wave voltage is used to drive to the sensor (Fig. 1). During both the rise and fall of the square wave, the system acts like an overdamped system, approaching two different temperatures in an exponentially asymptotic manner. Heat transfer is primarily through conduction and characterized by the time constant associated with the exponential growth and decay of the responses as well as the difference between temperature asymptotes.



Fig. 2. A B, C Sensor 1, 50 $\mu m \times 3 \ \mu m$, Sensor 2, 100 $\mu m \times 6 \ \mu m$ with a 50 $\mu m \times 3 \ \mu m$ Section in the Middle, and Sensor 3, 100 $\mu m \times 3 \ \mu m$, U structure, all Sensors are 1 μm in Depth.

2. Methods

The experimental set-up consisted of two mass flow controllers (Alicat MC model) connected to two different gas species: nitrogen and a 5% mixture of a diluent gas in nitrogen, with the exception of hydrogen gas which was limited to a maximum of 2%. Adjusting the mass flow rates, any mixture containing between 0 and 5% of the species of interest can be delivered into the test chamber. The test chamber was a 150 ml beaker with inlet and outlet ports on each side. This beaker was enclosed within a Yamato ADP200C oven to ensure constant temperature within the enclosure as well to ensure that the gas composition within the control volume reached the desired composition after a reasonable time. During a typical experiment gas flow was continuous at 500 sccm, with data only being collected after several minutes had passed to allow for stabilization of the sensor baseline resistance.

The circuit used is the same as the circuit in [8], consisting of a voltage controlled current source. The circuit uses a reference resistor ($R_f = 2k\Omega$), two auxiliary resistors ($R_{1,2} = 300 \Omega$), and an op-amp (LT 1028). Resistors were chosen to have low temperature coefficient of resistance (<5 ppm/°C). The circuit was designed apply constant current pulses across the sensor and translate the corresponding resistance into a measurable voltage. The sensor resistance can be calculated via the following relation between the input and output voltages: $V_o = V_i (R_f - R_s) / (R_f + R_1)$, the current can be calculated via: $I_s = V_i (R_f + R_1)$, and the power dissipation in Download English Version:

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