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Low power micro-calorimetric sensors for analysis of gaseous samples

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ABSTRACT

This work discusses the design, finite element method modeling (FEM), fabrication and characterization of a silicon-based, catalytic micro calorimetric sensor. The sensing area is comprised of two titanium silicide ($TiSi_2$) – polysilicon (poly-Si) resistive temperature sensors symmetrically positioned relative to a poly-Si heater on which an oxidation promoting catalyst is deposited. The resistive structures are located on a suspended, thereby, thermally isolating, low mechanical stress membrane and integrated into a glass flow channel. The micro-calorimetric sensor is applied for measuring propane and hydrogen concentrations in air.

An approach to optimization of thermal and fluidic design of the microsensor is presented based on developed models: (i) a 3D thermo-electric analysis of the suspended heater and (ii) a 2D thermo-chemical analysis of the catalytic oxidation of propane in the flow channel. Influence of the design and material of the membrane on the power consumption and temperature distribution across the sensing area are analyzed. A relationship between the thermal design of the sensor, reaction conditions and its operation as a thermal actuator and sensor of reaction heats are discussed.

Various thermo-electrical characterizations (electrical, infrared surface imaging and transient thermal response measurements) in the context of microcalorimetric sensing are performed. Microsensors with a $50\,\mu m \times 50\,\mu m$ sensing area consume ca. 12 mW at an operational temperature of 350 °C. Thermal imaging with an infrared camera indicates local heating with a temperature gradient across the active area estimated to be $4\,^{\circ}C\,\mu m^{-1}$ (at ca. 500 °C). The heating and cooling times are found to be ca. 1 and 8 ms, respectively. Temperature vs. power curves are determined for both stationary and constant flow conditions of various gases. Based on the experimental and modeling results we envision that these microsensors can be successfully used for calorimetric sensing and analysis of gaseous samples.

1. Introduction

1.1. Overview of micro hotplate sensors and applications

Thermal actuation is a process in which physical or chemical phenomena are triggered by means of heat. Microsystems capable of maintaining high temperatures only on localized/confined positions – on micrometer-sized areas – are called micro hotplates and used in a variety of thermal sensing and actuating applications. Micro hotplates have a long history of development and were traditionally addressed as multi-purpose chemical sensors [1,2]. State-of-the-art micro hotplates have power consumption of ca. 50 mW at 500 °C [3], and typically consist of an integrated heater, a temperature transducer (e.g. thermocouple, thermistor, transistor or diode), and, in some cases, a catalytic layer. With introduction of the modern microfabrication technologies enabling mass production of micro hotplates (background information on the processing techniques is described elsewhere [4,5], a large number of applications emerged [6]. These include catalytic gas sensors for the detection of combustible gases [7,8], high temperature microreactors for analysis of oxidation reactions [9,10], systems for characterization of thermo-conductive properties of thin films (e.g. lateral thermal conductivity) [11,12], gas flow- and gas direction meters [13], gas and liquid MEMS-based differential scanning microcalorimeters [14–17] and bioreactors facilitating thermal analysis [18,19]. The geometrical configuration of micro

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hotplates commonly comprises resistive heating and temperature sensing microstructures or even miniaturized hot spots [20]. These heating structures are typically positioned on a thermally isolating membrane [21], a suspended bridge [22–24] or a cantilever [25], to enhance the heat confinement and to meet strict requirements of efficient power consumption. When designed to operate at tens of milliwatts and below, these discrete micro hotplate devices can be combined in a sensor array for recognition of individual compounds in a mixture [26,27] and subjected to post-CMOS micromachining for integration with other Si-based components and electronic control [28–30].

1.2. Working principle of catalytic sensor

This study is focused on the design and fabrication aspects of micro-calorimetric gas sensors. This type of micro hotplate sensors is also commonly referred to as MEMS anemometers [31,32] micro-pellistors [33] and microscale hot surface devices. The basic working principle of such micro hotplate sensor can be described as follows. A reactive gas mixture, e.g. hydrocarbons and air, is introduced into the flow channel where the micro hotplate sensor is located. The active area of the microsensor, with a deposited catalyst, is heated up to the reaction temperature in a controlled manner. A catalyst is used to lower the operational temperature of the device and to react selectively with a gas compound of interest. When the reactive mixture is in contact with a pre-conditioned catalyst, heat is either generated (exothermic reaction) or consumed (endothermic reaction) on the surface of the catalyst. Any release or uptake of heat changes the average temperature of the active area (also referred as sensing area or heated area), and thereby causes a change in the resistance of the integrated resistive temperature sensors. The sensor output, i.e. a change in resistance of the temperature sensors or a decrease in the supply power (based on the temperature coefficient of resistance (TCR) of both heater and temperature sensor materials) needed to maintain a fixed temperature in the active area of sensor, is proportional to the heat released/consumed due to the reaction and to the concentration of a target gas. In microcalorimetric (catalytic) type of sensors, the measured sensor signal of the micro hotplate depends on the nature and ratio of reacting compounds, activity of the catalytic material, and reaction conditions (e.g. flow rate, concentration of reacting gases and reaction temperature).

1.3. Optimization of micro hotplate design

Optimization of the micro hotplates typically undergoes several development stages aiming at the reduction of power consumption, an enhanced surface temperature uniformity and stability at elevated temperatures. This improves the overall reliability of the system and increases the sensitivity of the system to a measured signal. A reduction in power consumption is critical for portable gas sensing applications. A homogeneous temperature distribution in the heated area is an important requirement in microreactors applied for kinetic studies and catalysts screening. Once deployed gas sensors are typically used over a longer period of time (months and years), therefore high temperature stability of all constituting parts is important. Commonly the thermal characterization of the micro hotplates involves measurement of the TCR of heating and sensing elements, and evaluation of the temperature distribution across the hot surface area using infrared (IR) or Raman spectroscopic methods [20,21]. The publications dedicated to thermo-electrical characterization of various design aspects of micro hotplates can be found elsewhere [34–37]. These studies focus primarily on the analysis of power vs. surface temperature uniformity, elimination of hot spots on the surface [3,38] as well as optimization of heater geometry [39]. Additionally, mechanical and structural analyses of both the heating elements and the suspended membrane can be found in literature [9,40,41].

The development of a good theoretical model for thermochemical actuation and possible heat loss mechanisms is important for the design of catalytic microsystems. Such a model allows reducing the amount of lab-bench experiments, whilst predicting device performances with respect to a specific application. The optimization approaches are broadly represented in the literature [42–47].

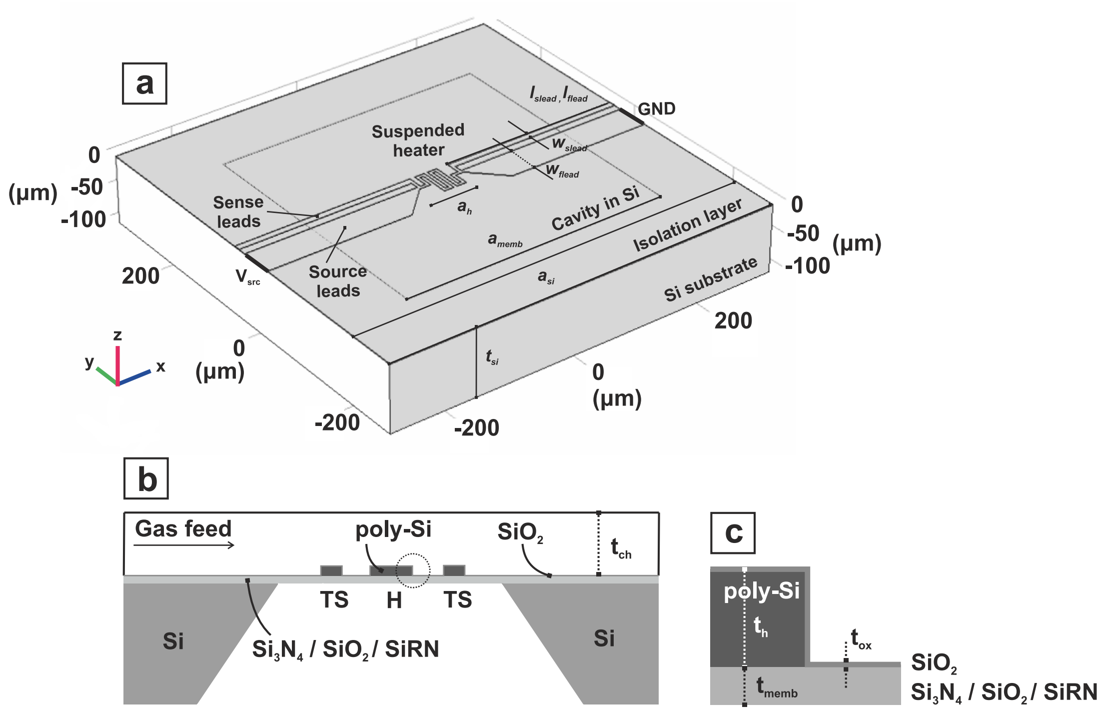

Fig. 1. Geometry of the micro hotplate in 3D (a), and a 2D cross-section (b), and close up 2D view (c).