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Micro-calorimetric sensor for vapor phase explosive detection with optimized heat profile

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ABSTRACT

A heater design, used in a micro-calorimetric sensor, has been optimized for temperature uniformity in order to increase the sensitivity and reliability of detection of trace amounts of explosives. In this abstract the design, fabrication and characterization is described. The performance of the novel heater design is characterized by measuring the temperature coefficient of resistivity (TCR) values and by mapping the temperature distribution using Raman spectroscopy. The new heater design is seen to have increased the temperature uniformity by a factor of 2.3.

1. Introduction

Security at airports and other public places need fast screening methods to ensure public safety [1]. For this reason trace explosive detection has become a predominant research activity in recent years. The leading technology in terms of sensitivity and selectivity remains to be mass spectroscopy [2] but other types of sensors are emerging.

Selectivity of gas sensors is normally achieved by immobilizing selective receptors on the sensor surface [3]. These receptors will bind only to the desired target molecule, and the binding can be detected via for example mass changes, surface stress changes or by fluorescent labeling. However it is difficult to achieve reliable and reproducible data, and regeneration of the sensor surface in order to allow for new measurements is challenging. Using calorimetric sensing, a selective surface coating can be avoided and the surface is regenerated after each measurement. In calorimetric measurements the change in desorption and/or deflagration energy is monitored until all material on the sensor is removed.

We have previously shown that the desorption and deflagration signals can reveal unique finger prints of different explosives [4] using micro-calorimetric sensors. Here, we present the design, fabrication and characterization of these calorimetric sensors and introduce an improved design of the heater elements.

2. Design

Some integral parts are important for fabricating a micro-calorimetric sensor. The thermal mass should be low to allow for the detection of minute temperature changes due to desorption of trace amounts of explosive. The sensor should be thermally isolated in order not to loose heat to the surroundings and should incorporate both heater elements and an element for measuring temperature changes.

To realize these requirements a design is made incorporating two resistors for joule heating and one resistor for temperature measurements as seen in Fig. 1. The sensor is designed as a small bridge that ensures good thermal isolation. The bridge structure is made of silicon nitride with dimensions ($l \times w \times h$) 400 μm × 100 μm × 400 nm. The resistors are fabricated in doped silicon that can endure high temperatures and at the same time the large TCR value for silicon facilitates a high sensitivity of the measurement resistor. The calculated thermal mass of the bridge is approximately 30 nJ/K. Using this thermal mass desorption of 10 pg of water corresponds to a one degree temperature change of the bridge giving an estimate of the possible sensitivity.

The goal of the optimized design is to achieve a heater shape that results in a more uniform temperature profile along the bridge. A uniform temperature profile would ensure that phase changes of material on different locations of the bridge would happen simultaneously. This should allow for a better definition of the measured signal and maximize sensitivity and selectivity. The ununiform temperature profile of the bridge is caused by conduction of heat to the silicon support. In order to compensate for this a

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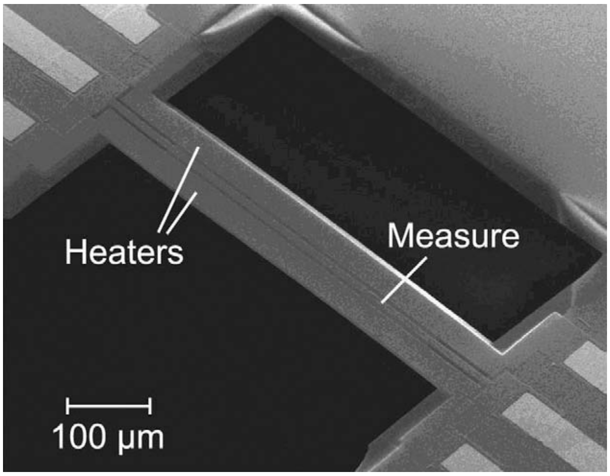

Fig. 1. SEM image of the unoptimized resistor design. Two resistors supply heat to the bridge and a measurement resistor in the middle measures the change in the temperature of the bridge. The resistors span the whole bridge and are 400 μm long. The measurement resistor is 10 μm wide and the heating resistors are 35 μm wide. The spacing between the resistors is 5 μm. All resisters are 200 nm thick and are on the top coated with 162 nm silicon nitride.

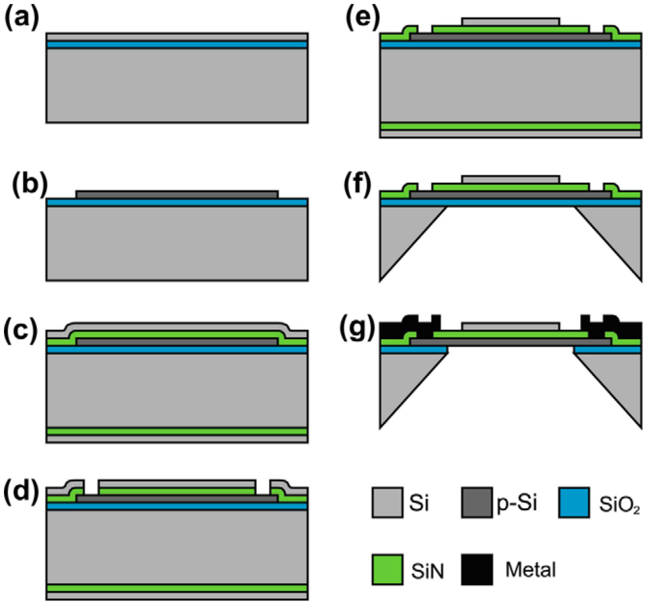

Fig. 2. Fabrication sequence of calorimetric sensor. (a) An SOI-wafer with a 200 nm device layer is used. (b) The resisters are p-type ion implanted using boron and defined by RIE. (c) Low stress silicon nitride and poly-silicon are deposited on top by a LPCVD process. (d) Contact holes to resistors and outline of bridge is etched using RIE and a phosphoric acid etch. (e) Poly-silicon layer is defined using RIE. (f) Bridge is defined from the back side using KOH etching. (g) Metal deposition and final release of bridges in hydrofluoric acid.

more heat should be dissipated in the regions close to the support. This can easily be achieved by changing the geometry of the heater and narrowing the width of the heater like a lens. The lens shaped heaters are 14 μm wide at the edge of the bridge and 32 μm wide in the middle of the bridge compared to the 35 μm width of the original design.

The readout is performed by measuring the change in resistance, it is therefore important to consider the possible contributions to the change in resistance. In this case basically two factors can give rise to a change in resistance, a piezoresistive effect and a thermal contribution. For our device the change in resistance due to temperature change is approximately 100 times larger than the contribution from the piezoresistive effect, thus the piezoresistive effect can be ignored. The resistivity in silicon ρ is given by [5]:

$$\rho = \frac{1}{q\mu_n n + q\mu_p p} \tag{1}$$

where q is the elementary charge and, n and p are the electron and hole concentration, μ_n and μ_p are the electron and hole mobility in silicon

$$\mu_p = 54.3T_n^{-0.57} + \frac{1.36 \times 10^8 T^{-2.23}}{1 + \left[p/\left(2.35 \times 10^{17} T_n^{2.4}\right)\right] 0.88 T_n^{-0.146}} \tag{2}$$

$$\mu_n = 88T_n^{-0.57} + \frac{7.4 \times 10^8 T^{-2.33}}{1 + \left[n/\left(1.26 \times 10^{17} T_n^{2.4}\right)\right] 0.88 T_n^{-0.146}} \tag{3}$$

where T is the temperature in kelvin and $T_n = (T/300\ \text{K})$. Silicon also has an intrinsic concentration of electrons and holes, which heavily depends on the temperature, thus if not careful it could become a problem for our application. Therefore the silicon is heavily p-doped, and thus the intrinsic concentration can be ignored for temperatures that we are operating at.

3. Fabrication

The fabrication of the micro-calorimetric sensor is illustrated in Fig. 2. A 4 in. SOI-wafer (silicon on insulator) with a device layer of 200 nm is boron doped by ion implantation to a resistivity of 1.54 mΩ cm. Heating and measurement resistors are defined in the device layer by reactive ion etching (RIE) using the buried silicon dioxide layer as an etch stop. Using low pressure chemical vapor deposition (LPCVD) a 162 nm silicon rich nitride and subsequently a 90 nm poly-silicon layer is deposited. The outline of the bridge and the contact holes are defined by RIE of the poly-silicon, and a wet phosphoric acid etch of the silicon nitride. Next, a poly-silicon pad is defined on the middle of the bridge also using RIE. The pad is used for enhanced adsorption of explosives [6]. The bridge is finally released in a potassium hydroxide etch (KOH) from the back side of the wafer. During KOH etching the front side of the wafer is protected by a plasma enhanced chemical vapor deposition (PECVD) silicon nitride layer. The KOH etch is stopped on the back of the buried oxide layer. The oxide layer is then removed by BHF to release the bridge completely and the protective PECVD silicon nitride is removed in phosphoric acid. The electrical connections on the front side are now defined using e-beam evaporated metal (15 nm Ti and 400 nm Au) and subsequent wet etching.

The wafer is diced using a diamond saw and the individual chips are mounted on PCBs and wire bonded. A SEM image of the finished sensor can be seen in Fig. 1.

4. Characterization

To characterize the fabricated calorimetric sensor in terms of improved temperature uniformity and sensitivity, Raman spectroscopy temperature measurements have been performed [7].

From room temperature to 500 °C the change in resistance of the highly doped silicon resistor is expected to be approximately linear. This is verified by measuring the temperature on the top of a fabricated bridge, see Fig. 3. These measurements are done both on an optimized bridge and on a bridge with a standard rectangular heater design. Measurements are performed at the center of the bridge at different power levels. It can be seen that the slope for the optimized heater design is slightly steeper yielding higher

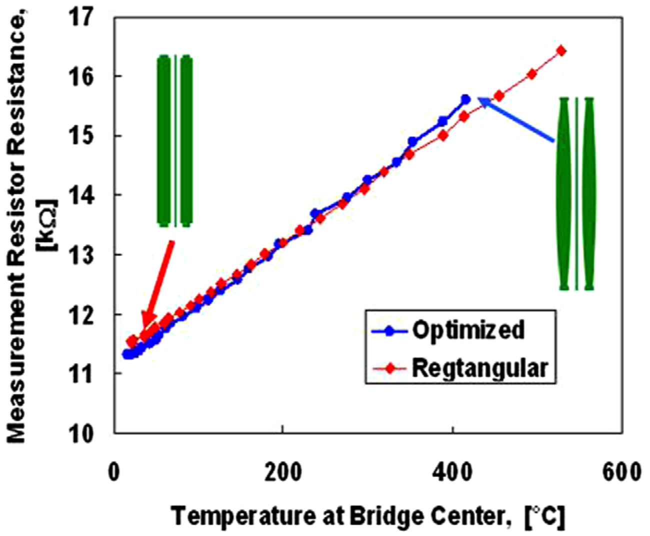

Fig. 3. Raman spectroscopy temperature measurements measured at the center of an optimized bridge and on a bridge with rectangular heaters for different heater power levels. It can be seen that the resistance change is linear as a function of temperature for both designs within the measured range.

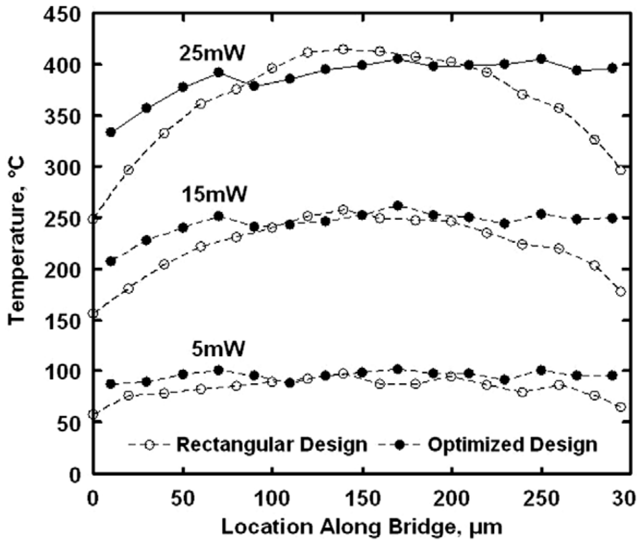

Fig. 4. Raman spectroscopy temperature measurements along middle 300 μm of an optimized, and a standard bridge for three different power consumptions. Results are normalized for same input power.

sensitivity. By linear regression a TCR value of 0.9×10^{-3}/K can be found for the optimized design, and of 0.85×10^{-3}/K for the rectangular heater design. This increased sensitivity is most likely due to a larger part of the bridge contributing to the resistance change. In the old design a large part of the bridge would be at a lower temperature not contributing to the change in resistance with the same magnitude.

For these measurements it should be noted that part of the resistance comes from parasitic resistances in the contact regions and at the resistor regions that are not heated. But significant sensitivity is in the present design lost due to parasitic resistances.

A laser has been scanned along the length of the bridge, following the center axis of the measuring resistor. This has been done for both an optimized heater and a rectangular heater design at different power levels. The measurements are shown in Fig. 4. At low power levels the temperature uniformity is good for both the rectangular and the optimized heater design. As power increases the temperature distribution gets parabolic for the rectangular design. The optimized design is better at keeping a uniform temperature distribution at high powers, especially when looking at the center region.

To quantify the improvement, a measure for the non uniformity can be calculated using:

$$\text{Nonuniformity} = \sigma\left(\frac{T(x) - T_{avg}}{T_{avg}}\right) \times 100\% \tag{4}$$

Here T_{avg} is average temperature and $T(x)$ is temperature measured at a given point. Measured on three different bridges with optimized and rectangular heater designs this gives an average of 12.4% nonuniformity for the standard rectangular bridges and 5.4% nonuniformity for the optimized version. This is an improvement by a factor of 2.3.

5. Conclusion

A micro-calorimetric sensor with an improved heater design has been fabricated and characterized. Characterization has been done using Raman spectroscopy temperature measurements both to investigate sensitivity and temperature distribution. It was found that a lens shaped heater has a slightly higher sensitivity of 0.9×10^{-3}/K compared to 0.85×10^{-3}/K for the rectangular heater design. Temperature nonuniformity was improved from 12.4% to 5.4% when scanning across the length of the bridge. Future work will include comparative measurements of trace amounts of explosives using the rectangular and the optimized heaters.Initial result show improved reliability and increased sensitivity.

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