Silicon microheater based low-power full-range methane sensing device
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\textbf{A B S T R A C T}

For many industrial applications of the methane sensor in a wireless node, the primary requirements are low power consumption and wide detection range. These requirements are considerably challenging as compared to the requirements related with conventional methane sensors. To meet such requirements of Internet of Things (IoT) development for methane monitoring in underground coal mines, we propose a full concentration range methane detector using a micro silicon device with low power consumption. The microheaters are fabricated with a CMOS-compatible SOI (silicon-on-insulator) MEMS (microelectromechanical system) process. Joule heating is utilized to enable micro-local high temperature for methane sensing. To obtain signals with high sensitivity and low heating power under an appropriate supplied current, a working point is determined by means of the maximum voltage variations from the voltage-current characteristics of the microheater. In general, the methane sensitivity of the micro heater increases and the power consumption decreases, as the length of the heater increases. The device has an exponential-decay response in the entire methane concentration range. A typically low power consumption around 27 mW yields an average sensitivity of approximately 20 mV/% CH\textsubscript{4} for the methane concentration ranging from 0 to 17%.

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1. Introduction

Methane is an extremely flammable gas, and it has the potential to form an explosive when mixed with air. It also contributes significantly to the global warming. Methane is found in industrial productions such as coal mines, oil wells, natural gas wells; transported and stored through pipelines, storage stations, and residential buildings. Methane in high level concentration (within or above the explosion range of 5–15%), for example, resulted from accumulation or gas outburst in coal mines has caused deadly explosions. The methane should be diluted under 1% and removed by a ventilation system to prevent the potential explosion hazard in coal mines. Hence, the concentration of methane in underground coal mines must be correctly detected according to the mandatory safety regulation. The IoT (Internet of Things) based methane monitoring technology could effectively avoid the explosion hazard by the deployment of the high-density wireless methane sensing nodes. However, the methane sensors which are currently available cannot satisfy the requirement of the wireless and battery-powered monitoring nodes, mainly because of their power-hungry nature. Another desired ability of the methane sensors is the wide detection range which is beneficial in real-life application because diffused methane in high concentration is a high-potential explosive threat. For the methane sensor to be embedded in the wireless nodes, qualities such as low cost, small size, fast response are also expected.

The current methane sensors \cite{1,2} are based on various sensing mechanisms, such as colorimetric, optical, semiconductor, thermal-wave, photoacoustic, and electrochemical. Overall, only the pellistor and optical-based sensors can meet the strict application requirements in terms of accuracy and response time. However, most of the optical sensors have high power consumptions and require special analyses and additional measurement apparatus via complicated alignment procedures for optical detections. Though microfabrication technologies could decrease the heating power of pellistor for catalytic methane combustion, the nominal measurement range of the pellistor sensors is only up to five percent by volume in the air as the output signal depends on

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both the concentration of methane and oxygen. Table 1 summarized the main performance of various methane sensors.

In recent years, many studies have focused on using nanomaterials for gas sensing due to their unique chemical and physical properties, e.g., the large surface-to-volume ratio for high sensitivity. A broad variety of sensors have been proposed based on 1D and 2D nanomaterials, such as nanoparticles, nanowires, nanotubes, and nanobelts. Recently, the self-heating effect of the nanomaterials has been utilized as a power-efficient strategy for various gas sensing applications. For example, Salehi demonstrated the detection of carbon monoxide by self-heated SnO₂ [15]. Kawano et al reported on the gas sensing properties of a self-heated single multilayered carbon nanotube [16] and Chikkadi exploited the self-heating effect of the suspended carbon nanotube for NO₂ sensing at a low power of 2.9 μW [17].

In the aforementioned examples, sensing technologies for low gas concentration with low power consumption have been achieved; there are however several shortcomings. One is the limited measurement range caused by sensing saturation at low concentration. Other issues with these sensors lie in manufacturing cost and the possibility of possible mass production. Silicon-based MEMS (microelectromechanical system) fabrication has been a developed technology, and the self-heating effect has been a useful approach for MEMS-based accelerometers, anemometers, vacuum gauges, and gas sensors. For example, microthermal conductivity gas sensors based on the self-heating effect have been proposed [18,19]. In the present study, in order to meet the IoT-based methane monitoring demand for low to high concentration range with low power consumption in the coal mine, we have explored the methane sensing capability of the Joule self-heated silicon microheaters.

2. Fabrication, sensing principle and experiment setup

We chose monocrystalline silicon as the structure and heating material of the microheaters because of its high mechanical strength and high melting temperature. The microheater was designed to be a U-shaped cantilever and then fabricated with a CMOS-compatible SOI (silicon-on-insulator) process. Fig. 1 illustrates the SEM photo of the silicon microheater. The manufacturing process mainly consists of two steps of deep silicon etching process on front and backside sides, respectively. The U-type silicon structure was finally obtained by the etching of the exposed oxide layer of SOI with a wet etching process. Fig. S1 depicts the fabrication process of the silicon microheater.

The Voltage-Current (V-I) characteristic experiment was conducted in ambient atmosphere by using a Keithley 4200 semiconductor parameter analyzer in the current sweep mode. The current was supplied to the microheater with an incremental rate of 0.02 mA, and the corresponding voltages were recorded. The resistance and voltage difference (Vdifference) were derived from the V-I characteristic. We calculated the Vdifference from the V-I data as follows:

\[ V_{\text{difference}} = V_n - V_{(n-1)} \]

where \( V_n \) and \( V_{(n-1)} \) are the voltage of the \( n^{\text{th}} \) and \( (n-1)^{\text{th}} \) points, respectively, of the sweeping measurement with the current incremental step of 0.02 mA (I\text{step}).

Fig. 2(a) shows the operation principle of the cantilever microheater. The silicon devices were supplied with a constant current to obtain a high temperature by Joule-heating effect. Relying on the relatively high thermal conductivity, the presence of the methane will lead to a voltage decrease due to the decrease of the temperature and resistance of the microheater. To test the response, the desired mixtures of methane and air were delivered to the sensor at the flow rate of 200 cm³/min (SCCM) by a computer-controlled gas mixing system. The experimental setup for methane response is shown in Fig. 2(b).

3. Results and discussion

Fig. 3 depicts the resistance-current and voltage-current curves of a silicon cantilever microheater. The maximum resistance value corresponds to the intersection of the positive and negative temperature coefficient of resistance (TCR) during the heating process. The temperature at the tip corresponding to the maximum resistance of the cantilever resulted from Joule self-heating, and was previously characterized to be approximately 740–780 °C [20].

In order to have a high sensitivity for methane detection, a specific working point of the sensing device should be preferred. The sensitivity of the device can be defined as \( S = \Delta V / \% \text{CH}_4 \), where \( \Delta V \) is the voltage variation caused by one percent alteration of methane concentration in air. For a specific device under a specific current, the sensitivity depends on the voltage ramp rate \( \Delta V / (S \times \Delta V) \). Considering the increment current step (I\text{step} = 0.02 mA) is quite small, we can use the \( V_{\text{difference}} \) instead of \( \Delta V \) for evaluating the methane sensitivity under different applied current and thus \( V_{\text{difference}} \) and supplied current can be used together to define the working point.
Fig. 2. The methane sensing principle of the silicon microheater under Joule-heating supplied with current (a) and the experimental setup for methane response (b).

of the device. Hence, for the device with a direct current supply, we take the current corresponding to the maximum of $V_{\text{difference}}$ as the current of the working point. The working point is also labeled according to its definition with the curve of the derived voltage difference ($V_{\text{difference}}$) in Fig. 3.

Fig. 4 plots the experimental results of microheaters with different lengths. The working points are first determined by the maximum voltage differences. It is found that the value of the maximum $V_{\text{difference}}$ increases as the length of the microheater increases. Furthermore, there is an interesting observation, which is different from the previous report [21], that the power consumption decreases as the length of the silicon cantilever increases. As such, the devices with longer length will have better sensitivity and lower power requirement at the working points.

Fig. 5 shows the responses of the silicon microheaters of different lengths to methane in the concentration from 0 to 17% covering the entire range of explosive nature. The responses of all devices are obtained under their own specific working points. The response comparison for the devices operating at a same current and at the current corresponding to their own working points is listed in the Table S1. The voltage of the microheaters decreases with the increase of methane concentration. The voltage of microheater corresponding to the zero concentration of methane is taken as the reference, and the response is expressed as the absolute value of the voltage variations under different levels of methane. The sensitivity increases with the length of the microheater. It agrees well with the relationship between the value of the maximum difference and the length of the microheater as shown in Fig. 4. For the
Fig. 3. The electrical characteristics of a microheater methane sensor (voltage, derived resistance and voltage difference versus the supplied current).

Fig. 4. Power consumption and maximum voltage difference versus the cantilever length of the devices.

Fig. 5. Responses (in voltage) to methane in the concentration from 0 to 12% of the microheaters with different length. The voltages corresponding to the zero concentration of methane are taken as the reference (zero) and the response (in voltages) is the absolute value of the voltage variations caused by different level of methane.

600 μm-long microheaters, the average sensitivity is 20 mV/%CH₄ with a typically low power consumption of 27 mW.

The response to the full concentration range (0–100%) of methane of the typical sensor with 600 μm-long microheater is displayed in Fig. 6. The fitted curve of the response is of an exponential function. The monotonic response of the proposed device is an outstanding advantage against with the pellistor sensor (inset in the Fig. 6). For the pellistor, there is a peak output signal around the stoichiometric mixture (10%). Thus, one output signal would mean two different concentration levels in the full range. Such behavior of non-uniqueness between output and input is not desirable for sensors. The proposed device also has other merits such as anti-thermal shock and anti-poisioning, as no catalytic materials was used. In comparison, the pellistor sensors can easily poison and even be inhibited by many chemicals such as lead and sulfur compounds. Furthermore, the combustion or explosion of methane and air mixtures has to be avoided even at the maximum temperature of the microheater, which could be higher than 600 °C as the local high-temperature surface area is too small.

The total absolute value of the voltage response to the pure methane is higher than 1.2 V as shown in Fig. 6. However, the sensitivity decreases with the increase in methane concentration increase in the entire concentration range. The sensitivity reduction could be ascribed to the drop in the operating temperature of the microheater devices as the methane concentration increases.
The thermal response time (thermal time-constant) is taken as the response time since no chemical reaction is involved during methane sensing and is measured with an oscilloscope. The test result is shown in Fig. S2. The thermal response time in different concentration levels of methane is listed in Table S2. The long-term stability has been evaluated with the sensitivity variations after continuing the test for three months. The sensitivity shows a good repeatability as no discernible change in sensitivity was observed. Compared with a pellet, the property of the proposed device won’t be affected by the deterioration of the supporting material such as catalyst poisoning, sintering, and coking. Fig. S3 presents the standard deviation (σ) that is calculated based on the recorded data. The maximum standard deviation is about 1 mV, which is much smaller than the sensitivity of about 20 mV. Lack of selectivity is a disadvantage of the proposed device. Nevertheless, the proposed sensing device can still be used like the pellet in broad actual application environments, such as coal mine, where no interference gas exists.

4. Conclusion

In this work, we have demonstrated a full-range methane sensor with a monotonic exponential response based on the silicon microheater at operating by local high temperature Joule self-heating. The proposed silicon device is fabricated from an SOI substrate with a CMOS-compatible MEMS process. We defined the working point for the devices to evaluate their performance. The power requirement and sensitivity of the devices with different heater lengths are characterized. Test results suggest that the longer heater has a better performance. Sensitivity about 20 mV/% CH₄ by the power consumption of 27 mW was obtained in the range from 0 to 17% for the 600 µm-long microheaters. Benefitting from the high operating temperature and optimized structure, the device offers attractive advantages such as high sensitivity, small size, small current and power requirement, cost-effective, wide sensing range. These features can make it a competitive candidate for methane leakage monitoring in various hostile environments.

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Appendix A. Supplementary data

Supplementary material related to this article can be found. in the online version, at doi:https://doi.org/10.1016/j.sna.2019.05.039.

References


Biographies

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