Sensitivity and Low-Power Metal Oxide Gas Sensors with a Low-Cost Microelectromechanical Heater

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ABSTRACT: In this study, a simple and cost-effective metal oxide semiconductor (MOS) gas sensor, which can be fabricated utilizing only two photolithography steps, was designed and developed through the planar microelectromechanical systems (MEMS) technique. Ball-milled porous tin dioxide nanoparticle clusters were precisely drop-coated onto the integrated microheater region and subsequently characterized using a helium ion microscope (HIM). The spatial suspension of the silicon nitride platform over the silicon substrate provides superior thermal isolation and thus dramatically reduces the power consumption of the microheater. The well-designed microheater exhibits excellent thermal uniformity, which was verified both computationally and experimentally. The as-fabricated sensors were tested for ethanol gas sensing at various operating temperatures with different concentrations. At the optimal work temperature of ∼400 °C, our gas sensors demonstrated a respectable sensitivity to 1 ppm ethanol, which is the lower detection limit to most commercial products. Moreover, stable performance over repetitive testing was observed. The innovative sensor developed here is a promising candidate for portable gas sensing devices and various other commercial applications.

INTRODUCTION

A metal oxide semiconductor (MOS) gas sensor is a type of solid-state gaseous-atmosphere-dependent resistive gas sensor that has been extensively used in many practical applications, such as toxic and polluting gas alarms,1 indoor air quality control,2 environmental monitoring,3 medical diagnostics,4 domestic appliances, etc. There have been rapid developments of applications and great progress in the fundamental understandings of the MOS gas sensors since the first proposal by Seiyama et al.5 and Taguchi6 in the 1960s. There are numerous advantages of the MOS gas sensors, including simplicity, low cost, high gas sensitivity, miniature size, and thus compatibility with state-of-art electronic devices.7 MOS gas sensors typically utilize nanoscale polycrystalline n-type oxide particles, such as SnO2,8,9 ZnO,10 WO3,11 TiO2,12 and In2O3,13 or p-type oxides, such as NiO,14 Cu2O,15 Cr2O3,16 and Co3O4.17 When the oxide material is exposed to a volatile gas of a low concentration (e.g., a few ppm) in air, its electrical resistance may change accordingly, rendering it useful for gas sensitivity.18 The working principle of the MOS gas sensors can be briefly described as follows. The gas-sensitive resistor consists of a porous assembly of small grains of an n-type or p-type oxide.19 For the instance of n-type semiconductor, the oxygen (e.g., O2, O−, and O2−) in air may be adsorbed on these grains, resulting in an electron depletion layer (EDL) as well as an increased surface potential.20–24 A double-Schottky barrier for the migration of electrons is then formed across the interfaces (i.e., grain boundaries) connecting neighboring grains.20 Such barriers predominantly control the electric resistance of the sensing material.19 For example, the oxygen adsorbates are consumed if a reducing gas is in contact, which causes a decrease in the potential barrier and resistance (or increase in current) of the sensing body as a function of the partial pressure of the reducing gas.20

The MOS gas sensor typically consists of a sensing material layer that will react with the target gas, at least two electrodes to monitor the electrical resistance/current variations, and a microheater to increase the operating temperature.25 There are four most important parameters, termed sensitivity, selectivity, response transients, and stability, which define the MOS gas sensor working behaviors.19 The sensitivity is the most commonly used characteristic, and it is defined as the ratio of the sensor electrical resistance in air (R_a) to a steady level upon exposure to the mixture of target gas and air (R_g).19,21 The gas response is strongly dependent on the working...
temperature.\textsuperscript{7,21} Also, the increasing operating temperature accelerates the response process, because it influences the oxygen adsorption and the reaction rate with the target gases.\textsuperscript{23,26} The selectivity is defined as the ratio of the sensitivity of gas to that of a different gas.\textsuperscript{21} Moreover, the response transients are the time required to attain the majority of the electrical resistance (or current) change upon switching the gas concentration.\textsuperscript{21} The selectivity and response transients were demonstrated to change as a function of the working temperature.\textsuperscript{7} The stability and long service lifetime are usually required for practical applications, and they are pertinent to the operating temperature as well. Thermal stress usually accumulates during the repetitive heating/cooling cycles in the device, which may induce vertical deflection of the suspended membrane. To sum up, achieving an optimal operating temperature (typically between 200 and 400 °C\textsuperscript{18}) upon exposure to a target gas is of paramount importance for improving gas sensing performance.

To achieve gas sensing at an elevated temperature, a typical semiconductor gas sensor requires not only a microheater (e.g., a serpentine track of a metal resistor) for maintaining a good temperature distribution but also interdigital electrodes (IDEs) for measuring the \textit{in situ} electrical resistance or current of the sensing material. According to the type of heating components, the MOS gas sensor products may be divided into three categories, termed ceramic tube heater, planar ceramic substrate heater, and microelectromechanical systems (MEMS) microheater. Both the ceramic tube and planar substrate exhibit high power consumption, and therefore, they may not be the optimal choices for lithium ion battery-powered sensor devices.\textsuperscript{29} MEMS sensors recently attract increasingly more academic and industrial attentions due to their miniaturization (e.g., through microfabrications), excellent sensing performance, and dramatically lowered power consumption since the suspended heater design significantly reduces the heat loss of the substrate.\textsuperscript{29} Currently, there are only a few planar substrate-type MEMS gas sensors available in the market.\textsuperscript{30} The primary limitation for high-volume industrial and commercial applications of the MEMS technique is its high costs due to the complex fabrication process.\textsuperscript{31} Specifically, the fabrication usually involves four sequential lithography steps: (a) Mask one creates a layer of a patterned serpentine metal resistor for ohmic heating, (b) mask two produces a patterned \textit{Si}$_x$\textit{N}$_y$ insulating layer where the bonding pads are exposed, (c) mask three defines the sensing electrode, and (d) mask four forms selective openings on the insulating membrane for the subsequent anisotropic wet etching. More recently, some researchers use a single photolithography step to define both the microheater and IDE structure on the suspension platform, where IDEs are enclosed by the serpentine microheater to minimize unwanted thermal diffusion.\textsuperscript{32,33} Therefore, the fabrication process can be reduced to three lithography steps. The remaining two steps aim to pattern an isolation layer between the microheater and the sensing film, and to make a mask to expose the silicon for wet etching, respectively.\textsuperscript{32} In general, more process steps usually lead to longer development time, lower yield and ultimately higher cost. Hence, to facilitate the high-volume adoption of MEMS-type gas sensors, a strategy to further simplify the fabrication process is highly desired.

In this work, we introduce the design, simplification, and fabrication of a low-cost MEMS microheater for MOS gas sensor applications. By optimizing the design of the layout and etching processes for the two metal electrodes and the silicon nitride insulation layer, we successfully consolidate the fabrication process into only two photolithography steps. To demonstrate the excellent performance of our re-designed microheater for gas sensing, \textit{SnO}$_2$ is used as the metal oxide sensing material for ethanol detection. This particular material is chosen because of its high mobility of electrons, high chemical and thermal stability under the sensor operating conditions.\textsuperscript{24} The properties of the fabricated novel gas sensors are characterized in detail. The results show that the optimal operating temperature of our device is \textasciitilde 400 °C at a power consumption of 39 mW, which is significantly lower than that of commercially available non-MEMS gas sensors. The microheater also exhibits excellent thermal uniformity with a small temperature gradient of \textasciitilde 0.03 °C/μm. In addition, the gas sensor yields a respectable sensitivity to gaseous ethanol. More specifically, the sensitivity was recorded to be 10.2 upon exposure to 1 ppm of ethanol, which is at least three times higher than most of the commercially available MEMS-type gas sensor. Overall, the fabricated sensor in this work demonstrated a high response to the target gas with relatively small power consumption, which can be a crucial step toward the high-volume manufacturing of low-cost, high-yield MEMS-based gas sensors.

### Experimental Procedures

#### Design and Fabrication of the Device

The schematics of the fabrication process are shown in Figure 1. As mentioned,

![Figure 1. Schematic illustration of the fabrication process of an MHP: (a) an n-type (100) silicon substrate; (b) a thin layer of silicon nitride layer deposited by LPCVD, which provides thermal isolation between the microheater and the silicon substrate; (c) the serpentine platinum resistor, the IDEs, and the bonding pads were fabricated by double resist lift-off processes; (d) the silicon nitride insulating layer coated by PECVD; (e) wet etching windows, IDEs, and the bonding pads were uncovered through photolithography and dry etching; (f) the microheater and IDE structure was suspended by anisotropic wet etching.](https://dx.doi.org/10.1021/acsomega.0c04340)
films on both sides of a 4 inch silicon (100) wafer through low-pressure chemical vapor deposition (LPCVD). Electron beam evaporation and a double layer lift-off processes were carried out to pattern the platinum serpentine resistor, the IDEs, and bonding pads with a thickness of ~200 nm on the front side (i.e., mask 1). The wafer was then coated with an ~500 nm-thick silicon nitride film (i.e., an isolation layer) by plasma-enhanced chemical vapor deposition (PECVD). Afterward, the silicon nitride membrane was selectively dry-etched by inductively coupled plasma (ICP) etching with gaseous CHF$_3$ and O$_2$ to uncover the releasing windows, bonding pads, and the contact areas of IDEs (i.e., mask 2). Lastly, the platinum heater and IDEs were suspended over a silicon cavity by four beams through anisotropic wet etching with a 40 wt % KOH at 60 °C for 12.3 h.

**Measurement of Thermal Properties.** The temperature distribution of the MHP was measured using a thermal emission microscope (Optotherm Sentris, U.S.A.) with a spatial resolution of ~5 μm and a detection waveband of 7~14 μm. The device was heated to 45 °C by placing it on a hot stage for ease of focusing. The 2D thermograph was constructed using Matlab from the outputting raw data. It is worth noting that some of the thermal image contrasts may result from different emissivity of various materials, since the surface emissivity was assumed to be one for all materials in the thermograph. In addition, a finite element tool of COMSOL was utilized to computationally investigate the thermal distribution. The boundary conditions here are as follows: (1) The silicon substrate was fixed with no displacement, and (2) the substrate was set as an ambient temperature. The parameters used in this simulation were extracted from the COMSOL material library.

In addition, the temperature coefficient of resistance (TCR) of the device was determined for accurate temperature calibration. The temperature of the MHP was raised from an ambient temperature to 295 °C through immersing the device in silicone oil, while the temperature was maintained by a water bath heater. The temperature and the corresponding resistance were measured using a thermocouple and a resistance meter, respectively. Once the TCR is calibrated, the actual temperature of the device can be calculated by measuring the resistance at such temperature according to the following equation:

$$\text{TCR} = \frac{1}{T_2 - T_1} \frac{R_2 - R_1}{R_1}$$

where $R_1$ is the resistance of the MHP at temperature $T_1$, and $R_2$ is the resistance at temperature $T_2$, respectively. The temperature resistance method is an appropriate and convenient approach to estimate the working temperature of the device at a given applied voltage and power consumption.

**Synthesis and Characterization of the Sensing Material.** The synthesis approach utilized in this work basically follows ref. 34. In a typical experiment, 500 g of as-received SnO$_2$ micropowders with particle sizes ranging from 2 to 5 μm (Jinxin Advanced Materials, China) was added into 1425 g of deionized water with magnetic stirring, followed by mixing with 75 g of triethanolamine (Usolf Chemical, China) as dispersant. The stirring and dispersing were continued for 10 min. Afterward, the mixture was ball-milled (WG-1 L, Vgreen Nanometer Technology, China) with a ball diameter of 0.3 mm for 2 h to create a uniform dispersion. At last, 1 g of tetraamminepalladium nitrate (Aladdin, China) was added into 12 g of the synthesized dispersion as a palladium dopant. Consequently, the stable nanodispersion with a solid content of 15% was acquired. The gas sensitive material was deposited on the MHP through drop coating.

The morphology characterization of the as-fabricated gas sensor and the sensing material was conducted via secondary electron detection using a helium ion microscope (HIM, Zeiss Orion NanoFab, Germany) at an accelerating voltage of 30 kV, a beam current of 0.6 pA, and a working distance of ~8.7 mm.

**Gas Sensing Testing.** The gas sensing performances for ethanol were studied using a homemade testing system. An 8 L chamber was initially filled with ambient air. A certain amount of liquid ethanol was then injected onto the evaporator (i.e., to facilitate diffusion) through the gas inlet. The fans mounted on the chamber sidewalls also assist the gaseous flow within the chamber. The sensing material which acts as a chemiresistor may interact with the injected gas, and the change of its current measured by IDEs with respect to a reference current was monitored as a function of time. The temperature of the microheater was carefully controlled through supplying a certain voltage by a source meter (Keithley 2602A, U.S.A.).

**RESULTS AND DISCUSSION**

The optical and HIM images of the as-fabricated gas sensor are shown in Figure 2. The separation distance between the coplanar platinum electrodes was designed to be 9.8 μm allowing electrical bridging with the sensing material to form a chemiresistor. The three IDEs were surrounded by the serpentine microheater to provide necessary heating for gas sensing. The serpentine track of resistor yields analogous thermal uniformity compared to stack-type microheaters. In order to reduce the thermal strain during operation and to improve the mechanical strength of the serpentine micro-
heaters, corner compensations were introduced via replacing sharp corners by rounded corners. In addition, only the platinum IDEs were exposed, while other regions were covered with a silicon nitride layer in the central active region (Figure 2a,b). Therefore, the circuitous heating resistor was electrically insulated in-plane from the platinum sensing electrodes. Furthermore, a silicon pit (i.e., for thermal isolation purposes) created via anisotropic etching under the membrane is visible in the tilted view image (Figure 2d).

The measured resistance of the resistor as a function of temperature is shown in Figure 3a. The TCR value of the platinum resistor was experimentally determined to be $2.39 \times 10^{-3} \, \text{°C}^{-1}$. This is in good agreement with the literature values of the TCR of platinum, $2.70 \times 10^{-3} \, \text{°C}^{-1}$ and $2.19 \times 10^{-3} \, \text{°C}^{-1}$. The relationship between the applied heating voltage and the corresponding power consumption was also measured, and the obtained results along with the estimated temperature via the TCR method were shown in Figure 3b. Consequently, the temperature of the microheater at various applied voltages can be estimated. In addition, the suspended MHP from the silicon substrate confines the heat within the platform. Thus, the device exhibits low power consumption. For example, the power consumption is approximately 39 mW for achieving a working temperature of $\sim 400 \, \text{°C}$, which is significantly lower compared to other commercially available non-MEMS microheaters for gas sensing applications (e.g., 400 mW for the SP series of Nissha FIS; 210 mW for Figara TGS2620).

In an MOS gas sensor, homogeneous temperature distribution in the active membrane enclosing the IDEs is vital for the optimal sensing performance. Heat transfer usually occurs in three different pathways, termed heat conduction, heat convection, and thermal radiation. In an MHP with four supporting beams, the heat conduction occurs in the lateral plane, while heat convection and possible thermal radiation take place above and below the suspended membrane through neighboring air molecules and infrared photons. The temperature distribution of the fabricated microheater was simulated and experimentally investigated. Figure 4a shows the simulated temperature distribution using a COMSOL multiphysics simulator. The active area of the MHP is at $\sim 394 \, \text{°C}$ under 4.3 V, while the other area is approximately at an ambient temperature. In comparison, the temperature was estimated to be $400 \, \text{°C}$ under 4.3 V according to the TCR correlation. The good accordance between the simulated microheater temperature and the extrapolated temperature using the TCR approach corroborates the reliability of the simulation method. The thermal isolation and the consequent minimized power consumption may be attributed to the air gap between the microheater and the silicon substrate, as well as the four suspended slender beams where the temperature gradient is high, since the relatively small dimensions of the beam suppress significant heat flow from the heated membrane. The temperature profile along the diagonal line in Figure 4a is shown in Figure 4b. The temperature distribution within the membrane region yields good thermal homogeneity with a subtle temperature gradient of $\sim 0.03 \, \text{°C}/\mu\text{m}$ enclosing the active sensing area.

The emitted radiation distribution (Figure 5) measured using the infrared microscope shows, however, some deviations from the simulated temperature distribution, especially at the platinum IDEs. These measurement errors are possibly because of the material-dependent emissivity. That is, materials with lower emissivity emit less radiation and may appear to be cooler in an uncorrected infrared thermograph. Typically, the infrared thermal microscope detects the infrared radiation emitted from the material’s surface, and the temperature or thermography is estimated through the well-known Stefan–Boltzmann law: $R = \varepsilon \sigma T^4$, where $R$ is the emitted radiation, $\varepsilon$ is the material’s emissivity, $\sigma$ is the Stefan–Boltzmann constant, and $T$ is the temperature. Hence, the measured infrared temperature is directly correlated to the emissivity of the material. In practice, most of the infrared thermal systems
emissivity-adjusted results infer the temperature distribution in Stefan for various materials can be extrapolated according to the emissivity is 1 for all materials. Therefore, the true emissivity (damaged) while measuring the thermography assuming that the true temperature difference of 17.4 ± 0.7%. The emissivity-corrected results infer the temperature distribution in the active region is isothermal (e.g., \( \frac{T_{\text{Pt}} - T_{\text{SiN}}}{T_{\text{Pt}}} = 0.3 \pm 0.2\% \)), which is in good agreement with the COMSOL simulation result. Therefore, the microheater fabricated in the experiment exhibits a uniform thermal distribution, which facilitates the subsequent gas sensing performance.

An HIM is one of the ideal techniques to characterize small-scale structures, because the He\(^+\)-sample interactions induce backscattered electrons with no high energy, therefore, reveals better secondary electron image and surface morphology fidelity.\(^{43}\) The SnO\(_2\) nanostructure imaged using an HIM is shown in Figure 6. The as-synthesized SnO\(_2\) nanoparticles seem homogeneous without additional phases. They are almost spherical with a tiny particle size of \( \sim 30 \) to 40 nm. The nanoparticles were aggregated tightly with their neighbors and form large aggregations or clusters of particles. The voids and channels between the aggregations not only facilitate the diffusion of the sensing gas but also increase the surface-to-volume ratio of the sensing material. The observed aggregation effect might depend on the nanoparticle size, for example, the aggregation occurrence may intensify while the particle size is moving toward nanoscale.\(^{44}\)

The work temperature is vital and dictates the gas sensing performances. The desired temperature of the microheater can be obtained by adjusting the heater voltage. The gas sensitivity to 10 ppm ethanol of the fabricated gas sensor at stepped temperatures are shown in Figure 7a. The sensor response initially increases with temperature, then reaches a maximum sensitivity (e.g., 29.7 at \( \sim 400^\circ\text{C} \)), and decreases along the operation temperature. Such temperature dependence of the gas sensing sensitivity is in good accordance with the literature.\(^{21}\) The optimal work temperature with the best response signal to ethanol was determined to be \( \sim 400^\circ\text{C} \). Furthermore, Figure 7b shows the sensitivity as the gas sensor is sequentially exposed to ethanol at different gaseous concentrations ranging from 1 to 25 ppm at \( \sim 400^\circ\text{C} \). The current rise upon exposure to gaseous ethanol with a response time of 17.6 ± 1.2 s is clearly observed. The rapid recovery of the current with a transient of 16.7 ± 0.9 s after ethanol off also demonstrates good reversibility of the gas sensor. Figure 7c shows the gas sensor response increases with the increasing gaseous concentration, e.g., 10.2 to 57.4 from 1 to 25 ppm. Typically, the gas sensing sensitivity can be expressed as \( \ln S = \ln a + b \ln C \)\(^{45,46}\) where \( S \) is the sensitivity, \( C \) is the gas concentration of ethanol, and \( a \) and \( b \) are the gas sensor/gas sensing material-dependent constants. The logarithmic relationship, which can be utilized to predict the target gas concentration, is shown in the inset of Figure 7c. If we presume a smallest acceptable sensitivity value of 3, the lowest detection limit for ethanol was calculated to be \( \sim 120 \) ppb according to the logarithmic correlation. Moreover, the response measure-

**Figure 7.** Gas sensing performance: (a) the sensitivity (e.g., \( R_a/R_b \) or \( I_a/I_b \)) to 10 ppm ethanol under stepped temperatures, where \( I_a \) and \( I_b \) represents the sensor electrical current in the mixture of target gas and air and that of upon exposure to merely air, respectively. (b) Dynamic response of the sensor current for 1–25 ppm of ethanol at \( \sim 400^\circ\text{C} \). (c) Sensitivity as a function of various concentrations of target gas; the insert shows their logarithmic correlation. (d) Repeated sensing test to 25 ppm ethanol confirms repeatability and reproducibility of the device. The sensor voltage was maintained at 5 V.
ment was repeated for 8 cycles to ethanol at 25 ppm (Figure 7d). The reproducible results with a small variation of approximately 0.5 μA (i.e., ~1% of the measuring currents upon injecting gaseous ethanol) suggest satisfied repeatability of the device.

The gas sensor fabricated in this work yields excellent and reliable gas sensing performances. For example, the gas sensor response to 1 and 10 ppm of ethanol was measured to be 10.2 and 29.7, respectively, which are superior compared with other commercially available MEMS gas sensors (Table 1).

Table 1. Comparison of Gas Sensing Performance to Ethanol with Other Commercially Available MEMS Gas Sensors. Gas Sensing Performances Were Extracted from the Product User Manuals

| product name | sensitivity at 10 ppm | lower detection limit (ppm) | work power consumption (mW) |
|--------------|-----------------------|-----------------------------|-----------------------------|
| this work    | 29.7                  | 1                           | 39                          |
| SGX MiCS-5524| 3.3                   | 10                          | 76                          |
| ams AS-MLV-P2| 11.1                  | 10                          | 34                          |
| ams CCS801   | 4                     | 10                          | 33                          |
| FIS SM-30    | 3.3                   | 1                           | 20                          |
| Figaro TGS8100| 4                     | 1                           | 15                          |

CONCLUSIONS

An easily fabricated, low-cost, simple gas sensor was designed and developed by planar MEMS technology with only two photolithographic steps, whereas conventional MEMS-based gas sensors require at least three masks. Porous and uniformly distributed clusters of SnO₂ nanoparticles were carefully synthesized and loaded onto the suspended MHP. Gaseous ethanol was utilized to test the performance of the fabricated gas sensor. The optimal work temperature was determined to be ~400 °C with a small microheater power consumption of 39 mW and a reliable gas sensing sensitivity to 1 ppm ethanol. In addition, the gas sensor exhibited excellent sensing repeatability and reproducibility. The sensor reported in this experiment reduces the fabrication cost in terms of minimized photolithography steps (e.g., higher total yield) compared to traditional gas sensors; therefore, the insights obtained here will be attractive and promising for large-scale industrial manufacturing of MEMS microheaters and gas sensors.

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