A high-temperature thermopile fabrication process for thermal flow sensors

Rainer Buchner *, Christoph Sosna, Marcus Maiwald, Wolfgang Benecke, Walter Lang

Institut für Mikrosensoren, -aktuatoren und -systeme (IMSAS), University of Bremen, Otto-Hahn-Allee, NW1, 28359 Bremen, Germany

Received 31 May 2005; received in revised form 20 January 2006; accepted 1 February 2006

Available online 23 March 2006

Abstract
A new high-temperature fabrication process for thermopile-based flow sensors is presented. The high-temperature passivation of LPCVD silicon nitride leads to an improvement for liquid applications because of the low tendency towards pinholes and the good step coverage. The thermopiles are made of p-doped polysilicon and titanium–tungsten (WTi) showing a thermopower of $287 \mu V K^{-1}$ for a single thermocouple. Besides, the nitride membrane was released by a DRIE process to achieve a reduced chip size and higher yield compared to established thermal flow sensors.

Devices for different flow rates were fabricated and characterised. A very short reaction time of 2.6 ms could be achieved and the sensors show good sensitivity of $9.5 \text{ mV} \text{ mm}^{-1} \text{ s}$ in agreement to the theoretical expectations based on an analytical model.

© 2006 Elsevier B.V. All rights reserved.

Keywords: Thermal flow sensor; Calorimeter; Thermopiles

1. Introduction

Thin-film thermopiles are used as monolithically integrated high-precision thermometers in many fields of sensor applications as infrared detectors [1,2], thermoelectric gas sensors [3] and thermal flow sensors as presented in this paper [4,5]. Thermopiles measure differences in temperature between two different points based on the Seebeck effect [6] resulting in a thermopower proportional to the difference in temperature between the two junctions of the materials used. The Seebeck coefficient varies with temperature and changes in the materials used.

In the case of IR detectors and flow sensors the hot junction is placed on a membrane or free-standing bridge for thermal isolation, close to an absorbing area or heater. The cold junction is placed on the bulk material acting as a heat sink.

Polysilicon is an attractive material for thermocouples because of its high Seebeck coefficient in case of thermal flow sensors usually joined with metals like aluminium [5,7] or gold [4] to form a thermocouple. These thermocouples are connected in series to form a single thermopile in order to achieve a higher thermopower. The choice of the final passivation—recommended for measuring gaseous, but essential for measuring liquid flow—is limited by the thermal stability of this metalisation layer. Until now only PECVD passivations of silicon nitride [4] or multilayers of silicon nitride and oxide [8] have been reported, causing problems because of the tendency of PECVD films towards pinholes. For commercially available liquid flow sensors precautions for liquid insulation have been taken e.g. by measuring the flow through a tube [9] affecting the sensors dynamic behaviour because of the increased thermal capacity.

We introduce a new high-temperature thermopile process for liquid flow sensor applications using tungsten–titanium (WTi) as metallisation layer allowing a LPCVD passivation of silicon nitride with superior film quality as described in the following section. The sensors have been characterised and verified by an analytical model based on the work of Nguyen and Dötzel [10].

2. LPCVD versus PECVD

The motivation for the development of the introduced thermopile fabrication process with a high-temperature passivation lies in the central statement of Stoffel et al. [11]: “Use LPCVD whenever possible and PECVD whenever necessary.”

The important characteristics of an electrical and chemical passivation layer are resistivity, breakdown strength and pinhole density. LPCVD nitride is superior for all these characteristics as...
Table 1

<table>
<thead>
<tr>
<th></th>
<th>PECVD</th>
<th>LPCVD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deposition temp. (°C)</td>
<td>&gt;300 for good quality</td>
<td>700–800</td>
</tr>
<tr>
<td>Stress of film (MPa)</td>
<td>+600–1200</td>
<td>+2000–2000</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>85–210</td>
<td>260–330</td>
</tr>
<tr>
<td>Breakdown strength (10^6 V/cm)</td>
<td>1–5</td>
<td>10</td>
</tr>
<tr>
<td>Resistivity (Ωcm)</td>
<td>10^6–10^15</td>
<td>10^16</td>
</tr>
</tbody>
</table>

discussed by Stoffel et al. It shows almost no pinholes, because of the high surface mobility of the deposited molecules due to the high process temperature of 800 °C in our case. In addition, CVD processes show a good step coverage.

Since the passivation layer is also a part of a free-standing membrane, the mechanical properties are crucial. The Young’s modulus of PECVD nitride ranges from 85 to 210 GPa which is in fact lower than for LPCVD nitride, ranging from 260 to 330 GPa. The stress of the deposited nitride covers a wide range depending on the detailed process parameters. For membrane material a low tensile stress is ideal to avoid any buckling of the membrane. We proved the mechanical stability of our LPCVD nitride membranes by applying a pressure up to 750 kPa on a free-standing membrane of 1 mm² without breaking it. Of course the membrane showed some deflection calculated to approximately 43 μm, making a proper flow measurement impossible. Therefore, special effort is necessary to use these thermal flow sensors in highly pressurised fluidic systems [12].

Table 1 summarises the comparison of PECVD and LPCVD nitride underlining that LPCVD nitride is the material of choice for passivation of thermal flow sensors in liquid applications, if it is compatible to the metallisation used for the thermopiles.

3. Fabrication

The sensors are fabricated on silicon substrates with 250 nm of thermal oxide. The oxide is needed as an etch stop layer for the DRIE release etch of the membrane. The thickness of the oxide is optimised to provide a safe etch stop and to avoid buckling of the membrane at higher thicknesses because of compressive stress of the thermal oxide.

The heater and the thermopiles are embedded between two layers of low stress LPCVD silicon nitride with a tensile stress of 200 MPa. Three hundred nanometer in situ p-doped polysilicon used for the heater and as one thermopile material is structured by a RIE process. WTi (90% W/10% Ti) with a thickness of 200 nm as the second thermopile material and for electrical connections is sputtered and wet-chemically etched in HCl:H₂O₂:H₂O (1:5:5). Fig. 1 shows the sensor’s membrane area with functional structures. Finally, after the opening of the passivation and fabrication of the aluminium bond pads, the free-standing silicon nitride membrane of 1 mm by 1 mm is realised by a DRIE process. Optionally the residues of the silicon oxide etch stop layer can be removed. Since the thermal conductivity of silicon oxide is very low it will not affect the sensor’s performance enough to justify an additional etching process. The detailed fabrication process is shown in Fig. 2.

Several flow sensors have been designed and manufactured with a distance between the heater and the thermopiles of 20, 100 and 200 μm, respectively for different flow rates. Fig. 3 shows sensors with 20 and 200 μm distance placed on a coin to clarify the chip dimensions of 2 mm by 4 mm.

4. Characterisation

The characterisation was performed with a sensor with a distance between heater and thermopile of 20 μm. First the See-
beck coefficient of the thermopiles is measured. The thermal time constant is qualified by measuring the thermopile’s time delay following a temperature step provided by the heater with a Hameg oscilloscope. The measurements have been taken in air.

The characteristic of the sensor is measured at different heater temperatures using water as liquid. The sensor was flush mounted in a flow channel with a rectangular cross section area of 1.2 mm² for characterisation. A pressure gradient was achieved by a difference in height between the reservoir and the channel outlet. The flow rate was measured gravimetrically using an Ohaus Adventurer AR0640 analytical balance with 0.1 mg resolution. The sensor was run in a constant temperature mode controlled by the electrical resistance of the heater since the polysilicon used shows a linear TCR in the range of 20–100 °C.

Due to the poor reproducibility of the TCR of polysilicon, we changed to WTi as heater material on later sensor systems. Our sputtered WTi shows a linear TCR of $3.57 \times 10^{-4} °C^{-1}$. For keeping the temperature of the heater constant, the heater is placed in a Wheatstone-bridge circuit. The bridge voltage is applied on the heater via an operational amplifier as a feedback to provide a balanced bridge at varying flow rates. Because of this constant temperature mode the sensor uses the thermotransfer principle for flow measurement. This is clarified by measurements with different fluids as water, ethanol and isopropanol differing in thermal capacity and thermal conductivity. The different thermal flow sensor principles have been summarised by Ashauer et al. [4].

The output voltages of the thermopiles were converted by OMB-DAQ-55 data acquisition module from Omega with 22-bit resolution at a measurement range of $-62$ to $+62$ mV. The whole measurement was performed using LabView software.

### 5. Results and discussion

Each thermopile consists of 15 thermocouples. We measured a thermopower of $4.3 \text{ mV K}^{-1}$ for the thermopiles resulting in $287 \mu\text{V K}^{-1}$ for a single thermocouple. Fig. 4 shows the relationship between the difference in temperature and measured thermopower. Since the Seebeck coefficient of polysilicon is highly dependent on the dopants and doping level [13], it is difficult to put the measurements in relation to theoretical values. Therefore, we compared our values with other material combinations of realised micromachined thermocouples in Table 2.

The Seebeck coefficient of the polysilicon/WTi thermopiles shows a comparable or even superior performance than the semiconductor/metal thermocouples listed. They are only surpassed by thermocouples made of n-type and p-type polysilicon, but the fabrication of these thermocouples needs extra effort, because of the missing selectivity during structuring processes and the need of a diffusion barrier e.g. made of aluminium [14,15].

The time delay of the thermopiles in relation to a temperature step of the heater using a sensor with a distance between the heater and the thermopiles of 20 μm is shown in Fig. 5. The delay measured is 2.6 ms. In this case the heat transportation mechanisms are free convection in air and conduction in the membrane material. Radiation is almost negligible because of the low heater temperature. In case of flow the time delay will decrease because the forced convection speeds up the heat transportation towards the thermopile upstream of the heater as shown in literature [19].

Fig. 6 shows the characteristic of the flow sensor with a distance between the heater and the thermopiles of 20 μm. The measurements have been taken at heater temperatures of 16.6 and 37.1 °C above fluid temperature. The solid lines in the graph represent the theoretical expectations for the measurements achieved by an analytical model based on the work of Nguyen and Dötzel [10] and the thermal conductivity measurements of Lang [20]. The measurements show good correspondence with

### Table 2

Comparison of the Seebeck coefficient of different thermocouple materials

<table>
<thead>
<tr>
<th>Materials</th>
<th>Seebeck coefficient ($\mu$V K$^{-1}$)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bi$_2$Sb$_2$/Sb</td>
<td>135</td>
<td>[16]</td>
</tr>
<tr>
<td>$\alpha$-PolySi-PolySi</td>
<td>520–750</td>
<td>[14,15]</td>
</tr>
<tr>
<td>PolSi$<em>{70}$/Ge$</em>{30}$/AI</td>
<td>75–300</td>
<td>[17]</td>
</tr>
<tr>
<td>Ge$<em>{82}$/Ni$</em>{18}$/Cu$_{15}$</td>
<td>300</td>
<td>[18]</td>
</tr>
</tbody>
</table>
the model. For a temperature of 37.1 °C above fluid temperature we reach a high sensitivity of about 9.5 mV mm⁻¹ s⁻¹ for a measurement range up to 2 mm s⁻¹ reaching a saturation at higher flow rates. For a system with a heater to thermopile distance of 200 μm we determined a measurement range up to 1.67 mm s⁻¹ [21]. The minimum resolution could not be achieved yet because of the not fully developed measurement set up. We measured a resolution of 0.2 μs⁻¹ but as reported in literature [4,5,8], a higher resolution should be realistic.

In Fig. 7 the characteristic curves using water and isopropanol are put into comparison. The sensor signal using isopropanol is about four times higher than that of using water instead. These results correspond to the higher thermal capacitance and the lower thermal conductivity of isopropanol. The difference in thermal conductivity can be easily characterised by the output signal of a single thermocouple in the non-moving fluid, reaching twice the value in isopropanol than in water. These results underline the sensitivity of the sensor towards the fluid properties making a calibration for every type of fluid essential.

To prove the quality of the passivation layer we exposed a sensor to a liquid media for six months. After this period of time the sensor did not show any failures.

6. Conclusion

A new high-temperature thermopile fabrication process with improved passivation for thermal flow sensors in liquid applications has been introduced. In addition to flow sensor applications, the fabrication process presented here is also suitable for other thermopile applications e.g. IR sensors for high-temperature measurements because of the high-temperature long-term stability of the materials used.

The introduction of a DRIE etching process for the release etch of silicon nitride membranes makes a chip size reduction possible—compared to established wet-chemically etched flow sensors. A chip size of 2.5 mm by 2 mm at a membrane area of 1 mm² seems to be feasible. Since in this case the membrane release etch is the final step of process [8], the yield can be raised and no special care has to be taken for the protection of the front side structures.

For the future we plan to optimise the membrane dimensions to improve the dynamic behaviour and to decrease the chip size, which has not yet been optimised to make the non-industrial packaging and assembly possible. Furthermore we are working on other sensor applications like flow sensors for demanding environments that are based on this fabrication process and on the monolithic integration of micro-channel structures on the flow sensor.

References

Biographies

Rainer Buchner studied mechanical engineering at the Technical University of Braunschweig, Germany, and received his diploma in 2003. He joined the Institute for Microsystems, Actuators and Systems (IMSAS) in 2003 as a PhD student working on thermal flow sensors, microfluidic systems and micromotors.

Christoph Sonna studied electrical engineering, mainly emphasis micro-electro mechanical systems, and received the Dipl.Ing. in 2005 from the University of Bremen, Germany. In 2005 he joined the IMSAS as a PhD student. His main field of work are thermal sensors, especially the development of silicon based thermal flow sensors.

Marcus Maiwald studied electrical engineering at the University of Bremen since 2000. He joined the IMSAS in 2002 and is right now working on his diploma thesis about droplet characterization using miniaturized thermopile structures.

Wolfgang Benecke received his Dipl. Phys. degree from the Technical University of Clausthal (Germany) and his PhD in material science in 1982 from the Technical University of Berlin. From 1984 to 1992 he was employed by the Fraunhofer Institute (Berlin), where he was head of the Department for Micromechanics and MEMS. Since 1992 he is a full professor at the University of Bremen. He founded the Institute for Microsystems, Actuators and Systems (IMSAS), University of Bremen, Dept. 1: Physics/Electrical Engineering. W. Benecke is co-founder and shareholder of microFAB Bremen GmbH, an enterprise, offering Technology-Services (Wafer-Foundry) for MEMS-fabrication and of Campus MicroTechnologies Bremen GmbH, focusing on system integration.

Walter Lang studied physics at Munich University and received his Diploma in 1982 on Raman spectroscopy of crystals with low symmetry. His PhD in engineering at Munich Technical University was on flame-induced vibrations. In 1987 he joined the Fraunhofer Institute for Solid State Technology in Munich, where he worked on microsystems technology. In 1995 he became the head of the sensors department at the Institute of Micromachining and Information Technology of the Hahn-Schickard Gesellschaft (HSG-IMIT) in Villingen-Schwenningen, Germany, working on microsensors for flow, angular rate and inclination, sensor test and modelling. In February 2003, Walter Lang joined the University of Bremen. Together with Wolfgang Benecke he is heading the IMSAS in Bremen.

References

