

Proc. Eurosensors XXVI, September 9-12, 2012, Kraków, Poland

## Membrane-Type Surface Stress Sensor with Piezoresistive Readout

Frédéric Loizeau<sup>a</sup>, Terunobu Akiyama<sup>a</sup>, Sebastian Gautsch<sup>a</sup>, Peter Vettiger<sup>a</sup>,  
Genki Yoshikawa<sup>b</sup>, Nico de Rooij<sup>a</sup>

<sup>a</sup>*Sensors, Actuators and Microsystems Laboratory, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland*

<sup>b</sup>*International Center for Materials Nanoarchitectonics (MANA), National Institute for Materials Science (NIMS), Japan*

---

### Abstract

We present the fabrication and characterization of a membrane-type surface stress sensor (MSS), arranged in arrays for molecular detection in gaseous phase. Made out of SOI substrate, a round membrane with a diameter of 500  $\mu\text{m}$  and a thickness of 2.5  $\mu\text{m}$  is suspended by four sensing beams with integrated p-type piezoresistors, composing a full Wheatstone bridge. The membrane is coated with a thin polymer layer, which reacts with volatile molecules and produces a deflection of the membrane. Accumulated mechanical stress due to the deflection is applied on the sensing beams. MSS were functionalized with cellulose acetate butyrate (CAB) by inkjet spotting. They were characterized in a humidity chamber and showed a fast response time of 1.3 s for a relative humidity transition from 0% to 62%. Moreover, the membrane response is linear with a sensitivity of 87 mV/%RH.

© 2012 The Authors. Published by Elsevier Ltd. Selection and/or peer-review under responsibility of the Symposium Cracoviense Sp. z o.o.

**Keywords:** Surface stress, Gas sensor, Piezoresistive readout, Membrane

---

### 1. Introduction

Cantilever-based sensors are a frequently-used type of surface stress sensors, which have been widely studied for bio/chemical applications [1]. The major sensing principle is based on analyte-induced surface stress, which makes a cantilever bend. Depending on the functionalization layer coated on the surface of a cantilever, various analytes, such as explosives, antibodies or DNA, can be detected [2–4]. The deflection is detected either optically with a laser or electrically with an integrated sensing element, such as a piezoresistor. While the latter allows the setup to be portable and easy to handle, its sensitivity is often far

---

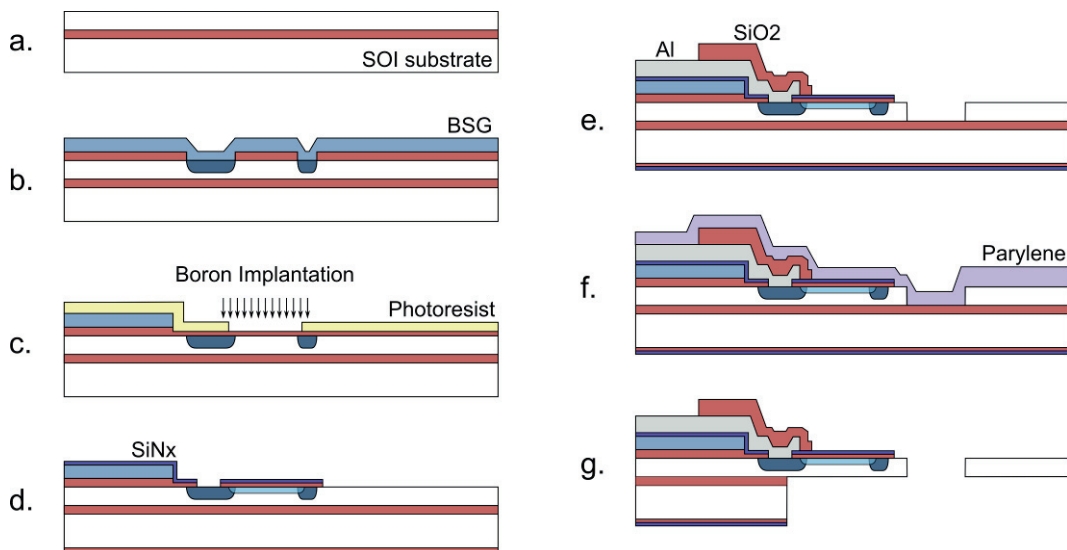
· Corresponding author. Tel.: +41 32 720 54 69, Email address: [frederic.loizeau@epfl.ch](mailto:frederic.loizeau@epfl.ch)

below than that of an optical readout system. We recently developed a membrane-type surface stress sensor (MSS) with a sensitivity increased by a factor of 20 compared to a piezoresistive cantilever-type sensor [5]. We present in this paper the microfabrication process and characterization of the latest generation.

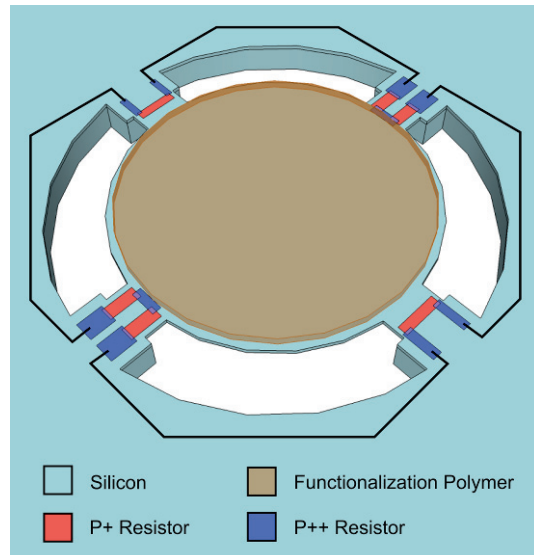
## 2. Methods and Results

Figure 1 shows a schematic of an MSS. A typical MSS has a round membrane with a diameter of 500  $\mu\text{m}$  and a thickness of 2.5  $\mu\text{m}$ . The membrane is supported with four sensing beams, on which transverse and longitudinal piezoresistors are implemented. Depending on the target analyte, a specific polymer is coated on the membrane. Its swelling, due to the absorption of the analyte, will produce the deflection of the membrane.

Figure 2 shows the main steps in the fabrication process of the membrane. The cross section shows one of the four constricted beams with a partial view of the suspended membrane. We start with an SOI wafer with a device layer of 3.5  $\mu\text{m}$  that is thinned down to 2.5  $\mu\text{m}$  by thermal oxidation and subsequent removal of the oxide (Fig. 2a). The p-type piezoresistors are then created with two distinct doping processes: deep and high conductivity diffusions with boron silicate glass (BSG) and shallow diffusions by ion implantation (Fig. 2b and 2c). After the deposition of passivation layer (Fig. 2d), aluminum wires are structured and encapsulated with a thick oxide layer. The membrane is then structured into the device layer with deep reactive ion etching (DRIE) (Fig. 2e). A 5  $\mu\text{m}$  thick layer of Parylene is deposited on the front side to protect the membrane prior to its final backside release by DRIE (Fig. 2f and 2g). Figure 3 shows SEM pictures of the released membranes.

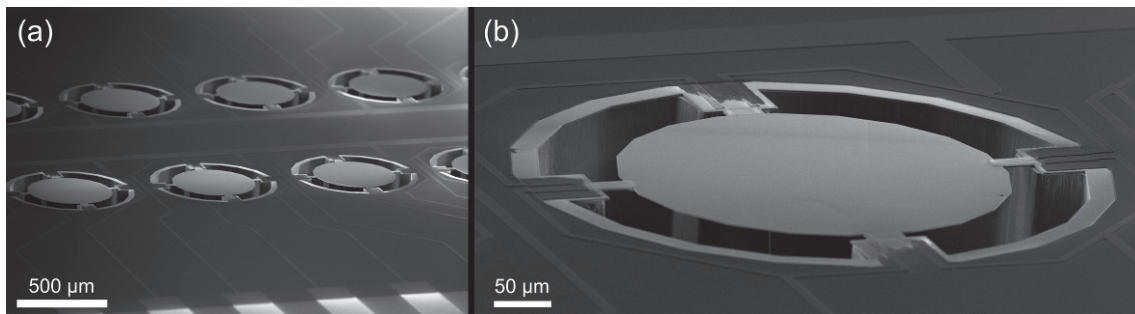


**Fig. 2:** Process flow of the membrane-type sensor fabrication. The cross section represents one of the four constricted beams with an integrated piezoresistor.

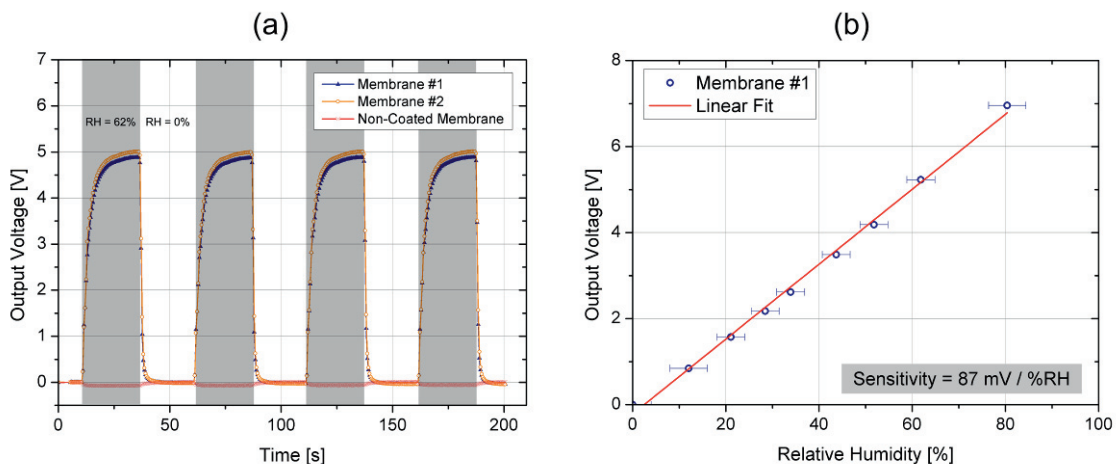


**Fig. 1:** Graphical representation of a membrane suspended by four constricted beams with integrated piezoresistors connected in a Wheatstone bridge. The membrane is coated with a polymer that reacts to surrounding molecules.

Two membranes within an array were functionalized by inkjet spotting with cellulose acetate butyrate (CAB) diluted in hexyl acetate. As a preliminary characterization, the membranes were tested as humidity sensors since polymers react strongly with water molecules. A commercial temperature and humidity sensor (Sensirion SHT21) was placed near the membranes to record the humidity level in the chamber. Figure 4a shows the dynamic response of the membranes to four relative humidity pulses of 62% with an electrical amplification gain of 500 and a bridge voltage of -1.0 V. An output of the non-functionalized membrane is also displayed as a reference. The functionalized membranes react quickly to the humidity changes with a rising time constant ( $\tau_{62\%}$ ) of 3.5 s and a purging time constant of 1.3 s. The difference between the two time constants is due to the mixing of the two gases in the chamber, which is, in reality, not immediate. Therefore, the purging time constant is closer to that of the sensor since it is not limited by the mixing inertia. Figure 4b shows the static response of the first membrane to different humidity levels. The error bars on the x-axis represent the measurement uncertainties of the commercial sensor. The response of the membrane is linear and shows a sensitivity of 87 mV/%RH.



**Fig. 3:** SEM pictures of (a) a silicon chip containing two arrays of membrane-type sensors and (b) a close view on one membrane suspended by four constricted beams.



**Fig. 4a:** Dynamic response of three membranes to four humidity pulses of 62%. One of the membranes was used as a reference without the functionalization of the CAB polymer. The others coated with CAB layers showed a response time of 1.3 s during the purging step. The gain of the amplification stage is 500. **Fig. 4b:** Static responses of membrane #1 to humidity values from 0% to 80%. The response is linear with a sensitivity of 87 mV/%RH.

### 3. Conclusion

A new type of surface stress sensor, which consists of a suspended membrane, and its fabrication process have been presented in this paper. By clamping the membrane in the four directions, the whole stress is efficiently transduced on the constricted beams, where the piezoresistors are located. Its sensitivity is therefore increased compared to a standard piezoresistive cantilever with a free-end. Characterized as a humidity sensor, the time response of the membrane is faster than that of a commercial humidity sensor. Moreover, their static response is linear in the measured range, between 0% and 80% of relative humidity. By selecting the right functionalization layer, this sensor would be able to detect various analytes in both gaseous and liquid environments while being embedded in a portable and easy-to-use setup.

### Acknowledgements

This project is supported by Nano-Tera, a Swiss federal program funding scientific projects. We gratefully thank the ComLab staff of CSEM SA and Dr. Laure Aeschmann from NanoWorld AG for their technical assistance and support during the microfabrication of the devices. We finally thank Dr. Giorgio Mattana from SamLab for his help during the CAB functionalization.

### References

- [1] M. Baller et al., A cantilever array-based artificial nose, *Ultramicroscopy* **82**, 9 (2000).
- [2] L. Pinnaduwaage et al., Detection of 2,4-dinitrotoluene using microcantilever sensors, *Sensors and Actuators B: Chemical* **99**, 223-229 (2004).
- [3] Y. Arntz et al., Label-free protein assay based on a nanomechanical cantilever array, *Nanotechnology* **86** (2003).
- [4] R. Mukhopadhyay et al., Nanomechanical sensing of DNA sequences using piezoresistive cantilevers, *Langmuir: the ACS Journal of Surfaces and Colloids* **21**, 8400-8 (2005).
- [5] G. Yoshikawa et al., Nanomechanical membrane-type surface stress sensor, *Nano Letters* **11**, 1044-8 (2011).