

# Use of Liquid Rubber in Micromachining Focused on Flexible Large-Area Biocompatible Membranes

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In this paper we report on a novel micromachining technique for fabricating rubber membranes with an area of 20–30 cm<sup>2</sup> and a thickness of 3–5 μm. In the basic technique, a silicon wafer is spin-coated with liquid rubber, bonded to a second (sacrificial) wafer and locally thinned out by anisotropic wet etching. The potential of the technique is highlighted in a demonstration where a 20 cm<sup>2</sup> large biocompatible membrane is used to mechanically support and thermally isolate a large-active-area thermopile. The envisioned application is for high-throughput drug screening, and the large areas are needed to culture 10<sup>5</sup>–10<sup>6</sup> biological cells to confluence.

## 1. Introduction

When membranes larger than 3–5 cm<sup>2</sup> are required to be fabricated, conventional micromachining techniques have limitations. Due to residual stress in the silicon oxide and/or nitride layer(s) which form the membrane, the structures easily bend, crack or even break.<sup>(1)</sup>

The interest in polymers in the field of micromachining is increasing rapidly.<sup>(2)</sup> Their use as ion-selective membranes has been reported extensively,<sup>(3,4)</sup> but the use of silicone rubber to produce flexible large-area membranes is a novel technique.

The technique is demonstrated by fabricating a 20 cm<sup>2</sup> large flexible biocompatible membrane. The potential of this technique in sensor applications is demonstrated by transferring a (p-type) polysilicon-aluminium 1332-junction thermopile connecting two flow channels to such a membrane. A thermopile is a set of  $n$  thermocouples connected in parallel thermally and in series electrically. Each thermocouple converts the thermal difference over its two junctions into a voltage difference using the combined Seebeck coefficient  $S$  of its two structural thermoelectric materials. The total generated voltage is the sum of the  $n$  thermocouple voltages:

$$U_{tp} = nxS\Delta T.$$

The temperature difference  $\Delta T$  is the product of the generated power difference between the two junction sites and the thermal resistance:

$$\Delta T = \Delta P_{gen} R_{th}.$$

Transferring a thermopile to a rubber membrane has several advantages:

- It renders excellent thermal isolation properties to the thermopile since the thermal conductance of the rubber is very low ( $0.2 \text{ Wm}^{-1}\text{K}^{-1}$ ) and the membrane can be made very thin ( $\mu\text{m}$  range). Therefore,  $R_{th}$  in the above formula can be increased.
- It offers the possibility of preparing a large thermopile which is needed if good thermal isolation and high sensitivity are desired.<sup>(6)</sup> Large areas are also needed if the metabolism of biological cells is being tested, since cells are preferentially tested in monolayers<sup>(7)</sup> and a large number of them are needed to obtain a significant signal (power production of a single cell is on the order of picowatts). Therefore,  $\Delta P_{gen}$  in the above formula can be increased. A cross-sectional drawing and a photograph of the sensor are shown in Fig. 1 and Fig. 2, respectively.

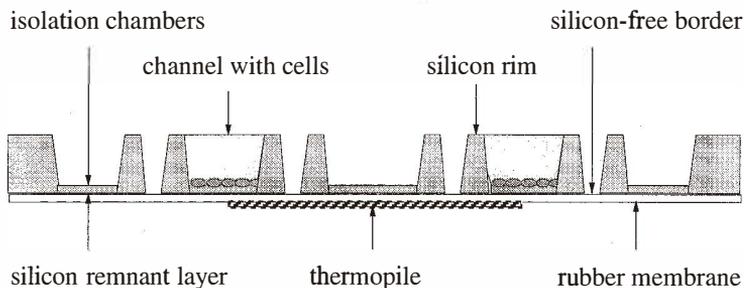


Fig. 1. Cross-sectional drawing of the thermopile sensor.

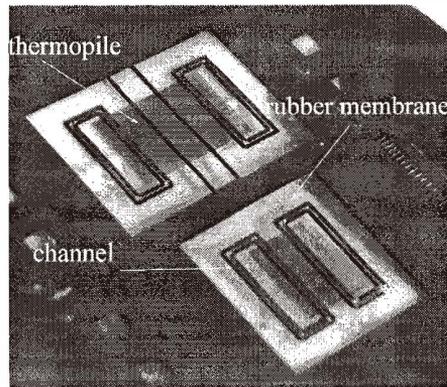


Fig. 2. Thermopiles transferred to a rubber membrane, for use in flow-through device for medical use.

## 2. Materials and Method

### 2.1. Materials

The material used is the two-component liquid silicone rubber ELASTOSIL LR3003/10A, B (Wacker Chemie). This rubber has a high mechanical strength (2.5 MPa tensile strength; elongation at breakage: 620%), perfect biocompatibility,<sup>(5)</sup> low thermal conductivity ( $0.2 \text{ Wm}^{-1}\text{K}^{-1}$ ), low linear thermal expansion coefficient ( $300 \times 10^{-6} \text{ mm}^{-1}\text{K}^{-1}$ ), high electrical resistivity ( $5 \times 10^{15} \text{ }\Omega\text{cm}$ ), low water uptake, high gas permeability and relatively low viscosity. The last property makes a spin-coating technique feasible.

### 2.2. Method

The technique used for fabricating rubber membranes consists of three steps. Figure 3 shows the basic sequence.

(a) On the top of a 6-inch p-type <100> wafer, silicon oxide and silicon nitride windows are etched to define the membrane pattern. On the bottom, the silicon oxide and silicon nitride layers serve as an etch stop. The ratio of the thicknesses of the oxide and the nitride layer is  $\sim 3$  (470 nm and 150 nm respectively) to balance out the tensile and compressive forces. The bottom side is coated with liquid rubber. The relatively low viscosity of the rubber allows for a spin-coating technique. By varying the speed and the time of spinning, the thickness of the layer can be adjusted in the range from 5 to 50  $\mu\text{m}$ . For greater thicknesses, successive layers are spun on top of each other. A spin rate of 4,000 rpm and a spin time of 15 s yields a layer thickness of 20  $\mu\text{m}$ . The wafer surface is chemically modified to make it water repellent by treating the wafer surface with hexamethyldisiloxane (HMDS). The viscosity of the rubber, and thus the layer thickness, can be reduced by adding small amounts of silicone oil.

(b) A second 6-inch wafer is bonded onto the first using unvulcanized rubber. The bonding is performed in a low vacuum to avoid air bubble formation at the wafer-rubber interface. To cure the rubber, the 3-layer stack structure is baked for 3 min at 170°C on a hot plate.

(c) To form the membrane, a chemical back etch is performed in 35 w% KOH at 60°C. The last 10  $\mu\text{m}$  of silicon is etched at room temperature to minimize the risk of breaking the oxide/nitride layer underneath. The rubber used in this work is not attacked by KOH at room temperature so the sacrificial wafer requires no etch stop. If the bottom oxide/nitride layer of the first wafer is not a structural element of the design, it can be omitted from the beginning, making the rubber layer an etch stop for both etching sites. The oxide/nitride layer is necessary in cases where, *e.g.*, gas impermeability is needed (the rubber is permeable to gases) or chemicals which attack the rubber need to be transported.

### 3. Results

As an example, a rubber membrane for use in a flow-through thermal biosensor is fabricated. In this application, a thermopile, positioned on the rubber membrane, connects two twin channels and measures the differential temperature between them. The rubber membrane fulfills the stringent biocompatibility requirements, allows for relatively large pressures to be build up during the pumping of liquid through the device, renders the sensor excellent thermal isolation properties, enables the active area to be very large and makes it possible to have optical access thanks to its transparency. The fabricated structures are 16

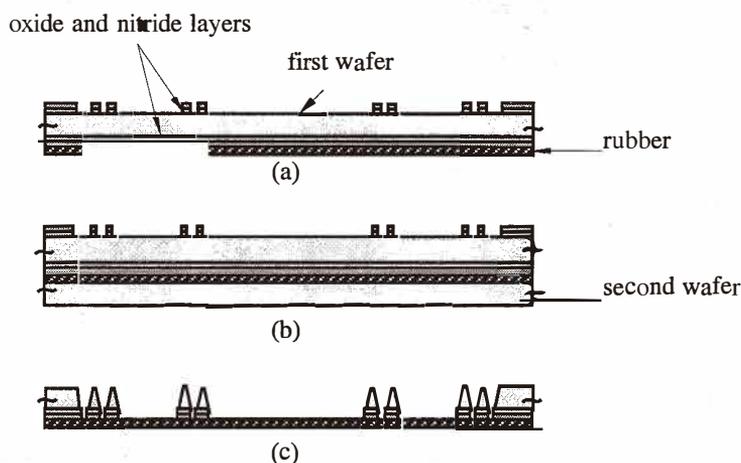


Fig. 3. Overview of the process sequence. (a) Liquid rubber spin-coating, (b) bonding of second (sacrificial) wafer, (c) etch-back in KOH.

cm<sup>2</sup> and 20 cm<sup>2</sup> large membranes of 10  $\mu\text{m}$  thickness. We fabricated a thermopile composed of 666 thermocouples covering an area of  $20 \times 15 \text{ mm}^2$  on a 6-inch wafer, and transferred it to a rubber membrane (Fig. 2).

The transfer of the thermopile to the rubber membrane is carried out following the basic technique described above, except that instead of a second wafer, a glass plate and wax are used to protect the bottom (processed side coated with rubber) of the wafer while the top is being etched in KOH at 40°C. The wax is removed using 1,1,1-trichloroethane. A thin layer of silicon is left on the entire membrane surface, except in the close vicinity of the channels (Fig. 1). This silicon-free bordering of the channels provides excellent thermal isolation between the two channels and to the environment. The differential etch depth is accomplished without an additional step. It is found that the etch rate in between the silicon rims is higher than on the rest of the membrane areas, probably due to localized stress concentration. When the silicon in between the rims is fully etched, a semi-transparent layer of silicon is left on the other membrane areas. The thin layer of silicon on the channel floor provides a uniform temperature distribution along the channel surface.

The temperature sensitivity of the device is 130 mV/K. The power sensitivity of the device is tested by monitoring the generated thermopile voltage, using 1.4 mW dissipation heat in calibration resistors in the channels, as plotted in Fig. 4. It was measured to be 23 V/W. The time constant of the sensor under atmospheric conditions is 12 s.

#### 4. Conclusion

A novel micromachining technique using an appropriate silicone rubber was described. The biocompatibility, high mechanical strength, high degree of transparency and low thermal conductivity of this material, enable its use in many applications where conventional micromachining techniques fail. We demonstrated its use as a mechanical support layer for a large-active-area thermopile in a medical flow-through device.

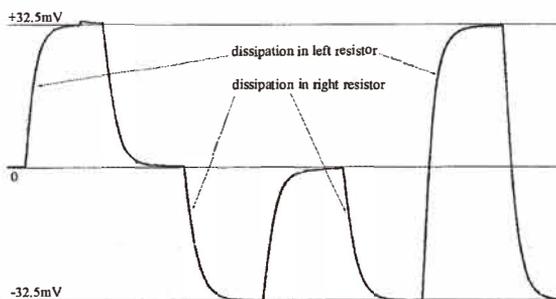


Fig. 4. Generated voltage of the transferred thermopile in response to 1.4 mW dissipated in the channels.

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