NANO-FLOW THERMAL SENSOR APPLYING DYNAMIC $\omega$-$2\omega$ SENSING METHOD

M. Dijkstra, T.S.J. Lammerink, R.J. Wiegerink and M. Elwenspoek
MESA+ Research Institute, University of Twente
PO Box 217, 7500 AE Enschede, The Netherlands
m.a.dijkstra@ewi.utwente.nl

Abstract — This article presents microchannel thermal flow sensors fabricated using standard micromachining technology. The sensors comprise of a Si$_3$N$_4$ microchannel created by etching of a poly-Si sacrificial layer. The channels are released by KOH etching through inlets and outlets etched from the backside of the substrate. Liquid flow is measured by platinum resistors deposited on top of the microchannel, while the channel is thermally isolated from the substrate by a Si$_3$N$_4$ membrane. Flow rates of DI water in the order of nl min$^{-1}$ have been measured using a dynamic sensing method applying heat waves.

Keywords: thermal flow sensor, micro-fluidics, lab-on-chip

I INTRODUCTION

The general trend in miniaturisation of systems in chemistry, pharmacy and biology asks for the miniaturisation of sensors. In these fields, the accurate and reliable measurement of the amount of fluid flowing through a flow channel is one of the key issues. A number of microchannel thermal flow sensors reported [1-4], have demonstrated the possibility to measure liquid flow down to a few nl min$^{-1}$. These sensors require accurate measurement of very small flow-induced temperatures changes. Thermal fluctuations and thermal noise define a lower limit to the smallest flow rate that can be measured accurately.

With a lock-in amplifier method accurate measurements can be made with increased signal-to-noise ratios. This article demonstrates the possibility to measure nl min$^{-1}$ liquid flow using a lock-in amplifier method applying heat waves.

II SENSOR DESIGN

Figure 1 illustrates the design of the microchannel thermal flow sensor. A microchannel is suspended on a membrane providing thermal isolation to the surrounding substrate. The microchannel is provided with an inlet and outlet from the backside of the substrate.

The working principle of the thermal flow sensor is based on convective heat transfer of an oscillating heat wave [5, 6]. The heat wave is generated by a heater-resistor located at the centre of the membrane. The heat wave is transported to sensor-resistors, 100 μm upstream and downstream of the heater-resistor, by heat conduction and convection, where heat convection by liquid flow causes an asymmetric temperature profile. The flow rate is obtained by measuring the amplitude difference of the temperature waves at both sensor-resistors. Common-mode thermal ambient fluctuations are more or less rejected, depending on the matching of both sensor-resistors. $1/f$ noise and Johnson noise are filtered by measuring the heat wave amplitude using a lock-in amplifier.

III REALISATION

Processing starts by depositing a 500 nm Si$_3$N$_4$ layer on a silicon (100)-substrate by LPCVD (figure 2a). A special alignment mask is used, together with KOH etching, in order to have good alignment with the crystallographic orientation of the substrate [7]. This ensures that the designed sizes of inlets, outlets and membranes are not increased by a large under etch during KOH etching.

On the frontside of the substrate the Si$_3$N$_4$ layer is patterned to define microchannel inlets and outlets, to be opened later by KOH etching starting from the backside. A poly-Si sacrificial layer of 1 μm is deposited and patterned, defining microchannels 50 μm in width and 2 mm in length (figure 2b). The sacrificial layer is covered by another Si$_3$N$_4$ layer of 500 nm, followed by patterning of inlets and outlets on the backside of the substrate. Other areas are opened on the backside in order to create the Si$_3$N$_4$ membranes (figure 2c).

A 200 nm Pt thin-film layer is sputtered on the frontside of the substrate and patterned by lift-off to create heater/sensor structures, wires and bondpads. The Pt layer is deposited directly onto the Si$_3$N$_4$ surface without adhesion layer. To ensure good adhesion, the Si$_3$N$_4$ surface is roughened by SF$_6$ plasma. In doing so the metal thin-film is made compatible with KOH etching during the lengthy release of the microchannel.

Finally, through the wafer inlets and outlets are etched by KOH etching, after which the sacrificial layer inside the microchannel is removed [8]. At the same time the Si$_3$N$_4$ membranes are formed.
Figure 2. Microchannel thermal flow sensor process scheme.

Figure 3 shows SEM images taken from a fabricated sensor chip. Figure 3a shows a microchannel crossing a Si$_x$N$_y$ membrane, with inlet and outlet to the backside of the substrate. This particular sensor has two resistors on top of the membrane with connections for 4-point resistance measurements. Sensors used during measurement make use of an additional heater-resistor positioned at the centre of the membrane.

Figure 3b shows the backside of the substrate with inlet, outlet and freely suspended Si$_x$N$_y$ membrane. The spacing between inlet/outlet and the Si$_x$N$_y$ membrane ensures that proper fluidic interconnection to the backside of the sensor chip is possible, without leakage while keeping the channel length to a minimum.

IV EXPERIMENTAL SETUP

The experimental setup used for measuring nL-min$^{-1}$ fluid flow is depicted in figure 4. Three resistor elements on the microchannel are used for measuring DI water flowing through the microchannel. A SR830 DSP lock-in amplifier applies sinusoidal heating power at a frequency of $2\omega$ to the centre resistor by a voltage source $V_\omega$ at a frequency of $\omega$.

The temperature asymmetry between the sensor-resistors, due to convection by fluid flow, is measured by the difference in resistance change as function of temperature on the two sensing resistors.

To this aid a small measurement current $I_{\text{meas}}$ is applied to both resistors by a HP 3245A universal source. The obtained voltages $V_{2\omega,a}$ and $V_{2\omega,b}$ are subtracted and amplified by the DSP lock-in amplifier before digitalisation, in principle making use of the full range of the ADC. In practice a small voltage bias remains, because both sensor-resistors measure $70\pm2\Omega$. This could be compensated for by placing both resistors in a half Wheatstone bridge, increasing measurement resolution.

The final flow dependent output of the DSP lock-in-amplifier is the amplitude $\Delta V_{2\omega}$ of the frequency component at $2\omega$ in the difference between $V_{2\omega,a}$ and $V_{2\omega,b}$.

Figure 4. Schematic of expiermental setup.
The resistance change as a function of temperature needs to be measured in order to relate output signal to temperature. To this aid, the sheet resistance $R_s$ and temperature coefficient of resistance $\alpha$ of the sputtered 200 nm Pt thin-film have been measured using a van der Pauw structure [9]. The thin-film was calibrated against a PT100 temperature sensor. Figure 5 shows that the Pt thin-film has a linear response in the temperature range from 20 °C to 120 °C.

\[ R_s(T) = R_0(1 + \alpha T) \]

$\alpha = 0.0024 \text{ °C}^{-1}$

$R_0 = 0.330 \Omega$

By applying a small quantity of glue the sensor chip was glued to the Perspex block with good alignment to the drill holes.

Further connections to the Perspex block were made by LEE .062” MINSTAC fluidic tubing connectors. Having the advantage that the tube can be filled with DI water first, before connecting it to the Perspex block.

During measurements DI water was forced through the microchannel by applying pressure from a N$_2$ gas bottle, regulated by a membrane valve, with a pressure range from 0 to 1 bar (figure 4).

The flow rate in relation to the applied pressure was obtained by measuring the advancing meniscus in the connection tube for several hours using a Vernier caliper with 1 bar of pressure applied. The average speed was found to be 1.0 mm-hr$^{-1}$. The tube contains 493 nl-mm$^{-1}$, which was determined by weighing of the DI water contained inside. In reference, the tube is specified to have an inner diameter of 0.813 mm, so it should contain 519 nl-mm$^{-1}$. The flow rate determined by measurement, at 1 bar pressure, thus equals 8.2 nl-min$^{-1}$.

The restriction on the flow is mainly due to the microchannel, this allows for an approximate calculation of the flow rate. For a 2 mm long channel filled with DI water at 1 bar pressure drop the flow rate equals 7.02 nl-min$^{-1}$.

The difference to the measured value might be caused by the expansion of the microchannel under pressure. In what follows it is assumed that 1 bar of pressure corresponds to 8.2 nl-min$^{-1}$ of liquid flowing through the microchannel.

![Figure 6. Electrical and fluidic interconnection. A Perspex chip holder connects tubing to the sensor chip.](image)

**Figure 6. Electrical and fluidic interconnection.**

**V SENSOR MODELLING**

![Figure 7. Cylindrical symmetric solution of increase in temperature surrounding the membrane, with 200 nl-min$^{-1}$ flow at 4.5 mW heating power.](image)  

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![Figure 8. Simulated flow induced temperature differences, with $P = 4.5$ mW and $f = 5$ Hz](image)

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FlexPDE has been used for time-dependent modelling of the thermal flow sensor. The sensor is modelled by a 2D cylindrical grid, with the z-axis in the direction of the microchannel (figure 7). Model results were observed to be in agreement with time-independent 3D FEM simulations in [1].

The thermal conductivity was made dependent on $r$, in order to take into account the large heat transport by the Si$_x$N$_y$ membrane and Pt leads near the microchannel. Figure 8 shows simulated flow induced time-dependent temperature differences between two sensor-resistors with oscillating heating power applied to the central heater-resistor.

**VI MEASUREMENT RESULTS**

Figure 9 shows measured voltage output $\Delta V_{2\omega}$ of the thermal flow sensor (figure 6) in response to the flow rate of DI water through the microchannel, with an accuracy of 0.5 nl min$^{-1}$.

![Figure 9. Measured flow induced voltage change $\Delta V_{2\omega}$, with $P = 4.5$ mW, $I_{\text{meas}} = 1$ mA, $f = 5$ Hz and $t_{\text{sample}} = 1$ s.](image)

The sensor shows a linear response, with sensitivity $S_T$ of 1.99 µV / nl min$^{-1}$. Using $\alpha$ and $R_0$ this can be expressed in a temperature vs. flow sensitivity $S_T$ being 0.012 °C / nl min$^{-1}$.

Figure 10 shows the response of the microchannel thermal flow sensor for various flow rates against frequency. At higher frequencies the temperature difference, due to convection by fluid flow, decreases resulting in lower sensitivity.

**VII CONCLUSIONS**

Microchannel thermal flow sensors have been fabricated using standard micromachining technology using Pt resistors. The sensor is demonstrated to measure down to 0.8 nl min$^{-1}$ DI water flow, with a measurement accuracy of 0.5 nl min$^{-1}$. The measurement resolution, using the DSP lock-in amplifier, can be improved by compensating for the offset in resistors values. Increasing measurement frequency can reduce the sample time, but will reduce output sensitivity $S_T$.

The measured sensitivity $S_T$ of the sensor was found to be in agreement with simulations. The sensor shows linear behaviour starting from 0 nl min$^{-1}$, because of the fact that the temperature is not measured on the heater itself.

Simulations have shown that the Si$_x$N$_y$ membrane and Pt leads significantly contribute to the heat transfer to the substrate, requiring increased heating power. From simulations it can also be concluded that higher thermal conduction to the substrate reduces the temperature vs. flow sensitivity $S_T$.

**REFERENCES**


