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Development of a Micro Flow Sensor for Microfluidic Systems

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Abstract A micro flow sensor based on the thermotransfer principle is developed here. The sensor consists of a micro heater with a micro thermocouple downstream of the heater. The flow sensor is microfabricated in a three step process: (a) micro heater and thermocouple fabrication, (b) microchannel fabrication and (c) system integration. The micro flow sensor was characterized using a mixture of 50% methanol and 50% water. The flow rate in the microchannel was controlled using a syringe pump. The sensor was calibrated using a pulse heat input. The results show that the sensor was sensitive for flow in the range 0.5µl/min to 0.7ml/min.

Keywords: Micro Flow Sensor, Micro Heater, Micro Thermocouple

1. Introduction

Microfluidic devices are often required to manipulate small volumes of fluid which require active sensing and control. Such microfluidic devices have found a wide range of applications such as in the pharmaceutical and biomedical fields. In such devices, accurate measurement of very small flow rates on the order of nanoliters per minute are required. Thus, there is a growing need for micro flow sensors that can be easily integrated within the device package. Several different types of micro flow sensors have been developed. Among these, thermal micro flow sensors are attractive because they can be incorporated into microfluidic devices relatively easily. Thermal flow sensors can be categorized into (i) hot film type, (ii) thermotransfer or calorimetric type and (iii) time of flight type. In hot film type sensors, the flow rate is correlated to the power required to maintain the sensor at a constant temperature above the fluid temperature. The thermotransfer or calorimetric principle is based on the temperature distribution around a heater due to thermal dissipation caused by a flowing fluid. For example, the distribution would be symmetric at no flow and skewed away from the flow direction when there is a flow. In thermal time of flight sensors, a heat pulse is generated and is detected at a known downstream distance. The time lag between the heat pulse and its detection is used to determine the flow velocity.

Micro thermal flow sensors have been developed for gases and liquids. Kim and Kim (2006) and Weiping et al. (2005) developed calorimetric based micro thermal flow sensors. Kim and Kim (2006) fabricated and characterized a micro thermal flow sensor that was fabricated on a quartz substrate using micro thermocouples that consisted of alumel and chromel legs. The sensor was characterized for flow rates in the range of 5 to100 SCCM by applying 100 W for 10s and examining the effect of the flow rates on the asymmetry of the temperature profiles. Weiping et al. (2005) used lower power levels to limit the surface temperature to less than 60 ^{o}C for use in biochemical sampling, and measured flow rates in the range 10 to 700 µl/min.

Rodrigues and Furlan (2003) designed a pulsed micro flow sensor that was operated with both sinusoidal and pulsed signals and showed an improvement to similar sensors that were operated in a DC mode. Ashauer et al. (1999) fabricated and tested a thermal flow sensor that could operate by either time of flight or thermotransfer mode. By combining both sensing principles within the same device, a larger dynamic operating range was obtained. Their sensor was bi-directional and was able to detect flow velocities from 0.1 to 150 mm/s with a resolution of 0.1mm/s. They investigated the influence of different fluids on sensor calibrations. As the thermal interactions occur within the thermal boundary layer, the response was dependent on thermal conductivity and the viscosity of the medium when operating in the thermotransfer mode. However, in the thermal time of flight mode, the fluid medium was found to have no significant effect since the time lag is directly correlated to the flow rate. Buchner et al. (2005) developed a thermal flow sensor for high temperature applications where the change in temperature was detected using a thermopile consisting of 15 thermocouples. It was fabricated on a silicon substrate and was thermally isolated using a deep reactive ion etch technique. The flow rate was determined using the thermotransfer principle, and flow rates up to 2μl/s, with a resolution of 0.2 μl/s were measured. Okulan et al. (2000) developed a pulsed mode thermotransfer micro flow sensor. The pulsed mode allowed the power input to be limited, and a novel double pulse operation was used to reduce temperature drift in the fluid. A flow range 0.01 to 10 ml/min was measured with a repeatability of measurements of ± 1.49 %.

The objective of this study was to develop a thermal pulse micro flow sensor based on the thermotransfer principle. A meandering resistance microheater was used to generate the thermal pulse with a microthermocouple placed downstream of the heater to detect the pulse. The output voltage from the thermocouple was correlated to the flow rate. This flow sensor has several advantages; it can be implemented relatively inexpensively, is amenable to standard microfabrication techniques, and has minimal disturbance to the flow.

2. Sensor Design and Microfabrication

The sensor design was based on the thermotransfer principle where the heat transfer from a micro heater to the fluid will

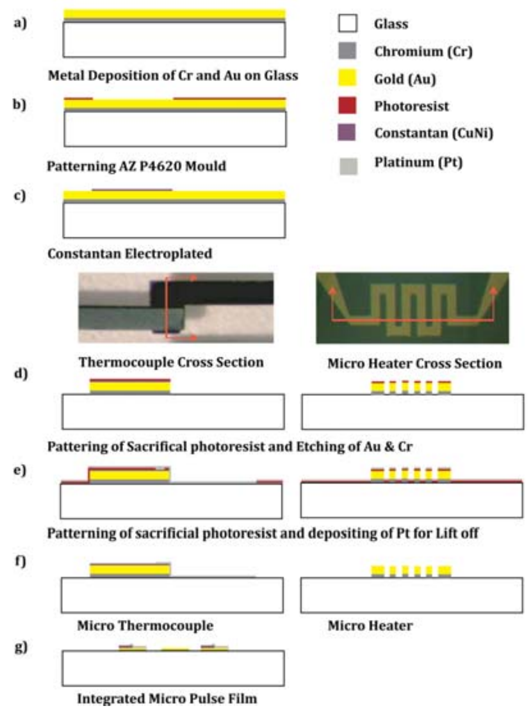

Figure 1 – Fabrication process flow for integrated micro flow sensor

increase with the flow rate. The fluid temperature downstream of the micro heater can then be correlated to the flow rate. There are two benefits of this technique: (i) scalability and (ii) no time domain information is required, after initial calibration. The calibration curves, however, would be dependent on the properties of the working fluid, and thus each different working fluid will require a new calibration. However, due to the nature of micro fabrication, several thousands of sensors can be microfabricated with a single calibration curve.

The micro flow sensor is microfabricated in a three step process: (i) micro heater and thermocouple fabrication, (ii) microchannel fabrication and (iii) system integration. The fabrication process flow for the micro heater and thermocouple is shown in Figure 1. Gold was selected for the heater base because of its high electrical and thermal conductivity, resistance to electrochemical corrosion and oxidation, and ease of integration with other sensors. Since gold does not adhere to the

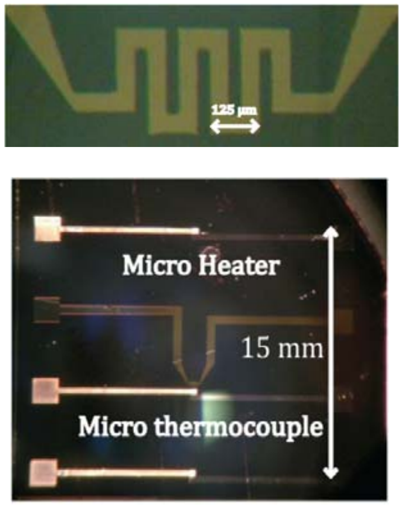

Figure 2: Photograph showing (a) micro heater and (b) micro heater with micro thermocouples on the silicon substrate

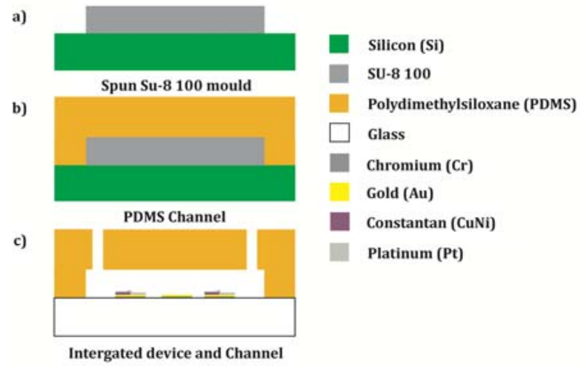

Figure 3: Schematic of SU-8 100 PDMS microchannel fabrication and integration with micro flow sensor.

Silicon substrate, a 60 Å layer of chromium was deposited as a seed layer followed by a 200 Å layer of gold (Figure 1a). Subsequently, the wafer was plasma oxidized and a thick positive photoresist (AZ P4620) was spun cast. CuNi and platinum were chosen for the two legs of the thermocouple. The first mask containing the CuNi leg of the thermocouple was exposed and developed (Figure 1b). CuNi was electroplated onto the exposed gold surface (Figure 1c), the details of which are provided in Loane at al. (2011). This was followed by spin casting a thin layer of S-1808 photoresist. The second mask containing both the micro heater and the second leg of the micro thermocouple was exposed and developed (Figure 1d). The process is then divided into two stages: (i) section with the micro thermocouple and (ii) section with the micro heater. The gold and chromium layers are etched away to reveal the micro heater. A thin layer of S-1808 photoresist was spun cast and the final lift off mask was exposed and developed, to fabricate the second leg of the thermocouple. A 60 Å layer of chromium followed by a 1200 Å layer of Platinum was deposited on the substrate to form the second leg of the micro thermocouple (Figure 1e). Chromium acts as an adhesion layer between the platinum and the substrate. The platinum was released, upside down, in a bath of acetone. Fabricating both the micro heater and the micro thermocouples on the same substrate provides the framework for the micro pulse film flow sensor, as shown in Figure 1g. Photographs of the microheater and the fabricated micro pulse film flow sensor are shown in Figure 2. In this figure, two additional micro thermocouples, further upstream and downstream of the micro heater are present.

The microfluidic channel was fabricated by casting polydimethylsiloxane (PDMS) on top of an SU-8 (MicroChem Corp.) mould which was patterned photolithographically to define the micro channel structure, as shown schematically in Figure 3. A silicon wafer was plasma oxidized for 1 min at 50 W to improve the adhesion properties. SU-8 100 was spun cast at 3000 rpm on a silicon wafer for 30 sec to spread a 100µm thick layer. The resist was soft baked for 10 minutes at 65^{o}C, and for 30 minutes at 95 ^{o}C. Subsequently the sample was exposed for 90 seconds at 7.2 mJ/sec using a negative microchannel mask. Following exposure, the sample was post baked for 1 minute at 65^{o}C, and for 10 minutes at 95 ^{o}C. A 1:10 curing agent to base PDMS pre-polymer mixture was cast on the SU-8 moulds to produce a replica. The cast PDMS pre-polymer was cured at 65^{o}C for 1 hour. The cured PDMS elastomer was peeled off from the Su-8 mould, and holes were punched in

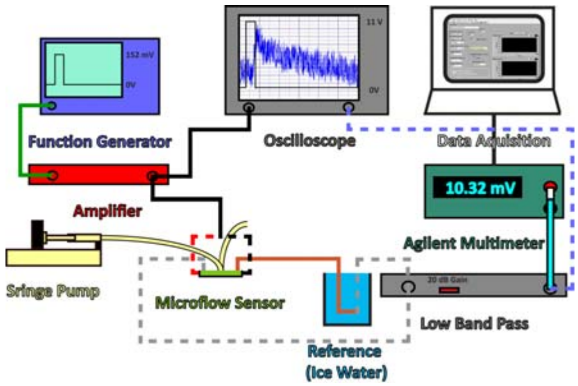

Figure 4: Schematic of experimental set up to characterize micro flow sensor

them in order to attach glass tubing for the inlet and outlet of the channel.

The microfluidic channel was integrated with the microheater and microthermocouple by adhering it to the substrate using a combination of plasma oxidization and liquid PDMS as glue. Both the PDMS channel and the silicon substrate were plasma oxidized for 1 minute at 50 W. A 1:3 curing agent to base mixture of PDMS was spun cast on a glass slide at 3000 rpm for 1 minute. The microchannel was placed gently onto the spun PDMS, to attach the thin film of PDMS only in the elevated regions, preventing the channel being clogged. The PDMS elastomer with microchannels was subsequently aligned using alignment markings on the substrate with the thermocouples and micro heater. Then a 1:3 curing agent to base mixture of PDMS pre-polymer was poured on the microchannel border and baked at 150 ^{o}C. The PDMS pre-polymer on the microchannel boundary bonded the microchannel with the underlying substrate.

3. Experimental Setup and Instrumentation

The micro flow sensor was calibrated by correlating the output voltage from the thermocouple to the flow rate due to a pulse heat input by the microheater. One advantage of this technique is that following the initial calibration of the sensor, there is no requirement for time shift data. Even though the calibration correlations are dependent on the working fluid properties and operating conditions, a single calibration reproducible to thousands of other devices can be bulk micro machined.

The experimental setup for the sensor calibration is shown schematically in Figure 4. The power input to the micro heater was generated using a function generator with a maximum frequency of 250 MHz with 5V_{p-p}. The output voltage from the function generator was amplified to achieve higher voltages. Different duty cycles, amplitudes, and pulse durations were examined. To reduce the noise in the signals, all wires and instruments were shielded using coaxial cables.

A syringe pump with a range 0.1μl/min to 0.7 ml/min was used to control the flow in the microchannel. A mixture of 50% methanol and 50% of water was used as the working fluid. Methanol is miscible in water and also enables wetting of the mixture in PDMS microchannels. The flow in the channel was allowed to establish for 45 minutes prior to taking any measurements. After each test the fluid was pumped for a period of at least one minute and was again allowed to attain steady state for 5 minutes.

4. Results and Discussion

The micro flow sensors were fabricated on silicon dioxide substrates, and thus the heat conduction from the heater through the substrate was significant compared to convection through the fluid for the flow rates considered here. Thus, the micro thermocouple will detect the temperature change of the substrate and not of the fluid temperature in this instance. One method of reducing the conduction through the substrate is to thermally isolate the thermocouple from the heater. However, due to time constraints in this study, and to remove any ambiguity and show proof of concept, a meso T-type thermocouple of wire diameter (Φ 0.002') was placed in the PDMS micro channel 0.5 mm downstream of the microheater. This eliminated the effect of conduction through the substrate on the thermocouple output. The results presented here are from this thermocouple.

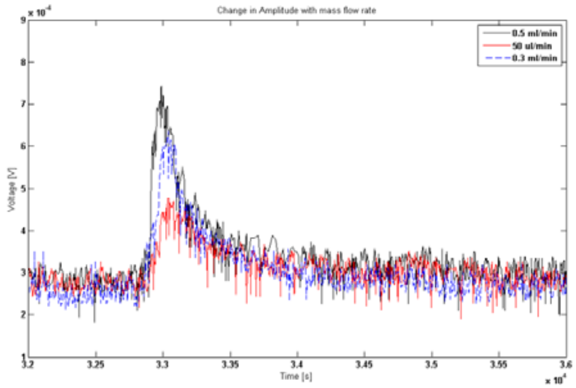

Figure 5: The response of the thermocouple at different flow rates for an input pulse voltage of 10 V, duty cycle of 2.5%, and period of 4 seconds.

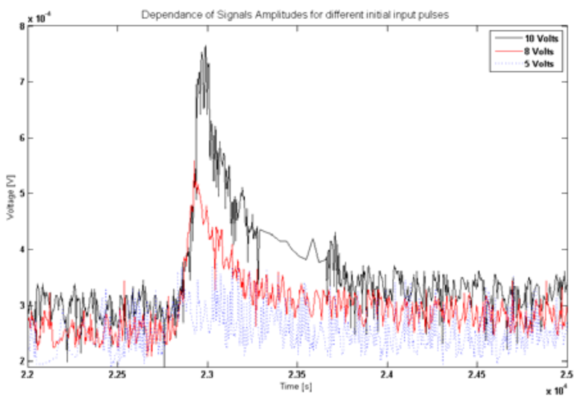

Figure 6: The response of the thermocouple for different input pulse voltages with duty cycle of 2.5%, period of 4 seconds and a flow rate of 0.1 ml/min.

A pulsed voltage with a low duty cycle of 2.5 % and pulse widths between 50 to 150 ms was selected for the heater input. This low duty cycle pulse voltage was found to minimize the temperature drift within the working fluid, since the cooling of the fluid back to the ambient was found to take longer than the initial heating of the fluid due to the pulse input. Increasing the duty cycle resulted in an increase in the response time of the sensor, albeit at an increase of the temperature drift in the fluid.

The thermocouple output response to the pulsed voltage was calibrated to the flow rate.

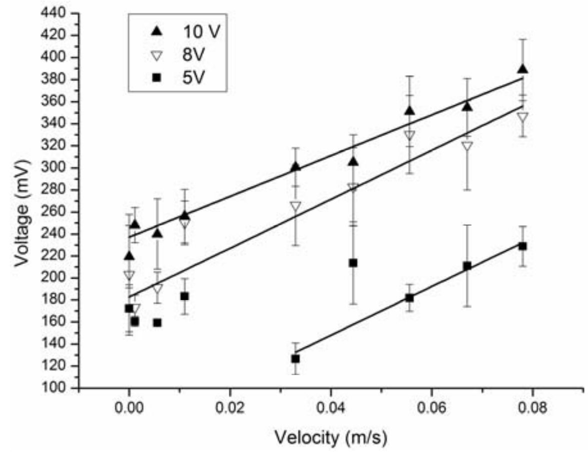

Figure 7: Variation of the thermocouple voltage output for the single pulse mode of the signal amplitude with flow velocity for different input voltages.

The calibration principle is that the heat transfer from the micro heater to the fluid will increase with flow rate, thus resulting in a higher voltage output from the downstream thermocouple. The response of the thermocouple that was placed 0.5 mm downstream from the micro heater for a pulse input voltage of 10 V at duty cycle of 2.5% (pulse width of 100 ms) and period of 4 secs is shown in Figure 5 for different flow rates. Clearly, the thermocouple output signal increases with an increase in the flow rate. The response of the thermocouple to different input voltages for a duty cycle of 2.5% and period of 4 secs at a fixed flow rate of 0.1 ml/min is shown in Figure 6. As expected, there is an increase in the thermocouple output peak voltage with an increase in the input voltage.

The flow sensor calibrations were performed by averaging three sets of data at each flow rate. The thermocouple output voltage is plotted against the flow rate for input pulsed voltages of 5, 8 and 10 V with a pulse width of 100 ms, duty cycle of 2.5 % and period of 4 secs in Figure 7. The output voltage is nearly linear with the flow rate, and the data were fitted with a linear curve for each input voltage. At a low heater input voltage of 5 V the sensor detection range was 0.1 ml/min to 0.7 ml/min. Below 0.1 ml/min

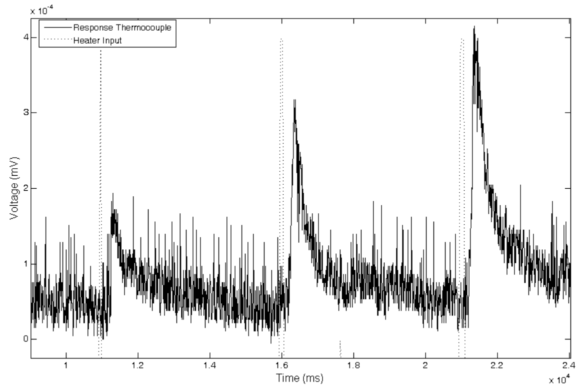

Figure 8: The response of the thermocouple to change in pulse width of 50, 100 and 150 ms for a 10 V input at flow rate of 0.5 ml/min

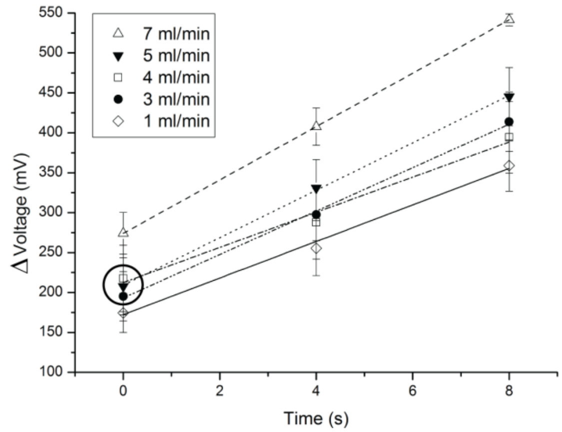

Figure 9: The difference in voltage amplitude for change pulse widths with time between pulses

the amplitude was within the noise region of the signal. This made the detection of an individual pulse harder and reduced repeatability. At 8V input there was a higher dynamic measurement range from 50 μl/min to 0.7 ml/min. The standard deviation however was 8.36 mV which was higher than that of the 5V input (8.09 mV). This was due to the fact that the calibration was made over a larger range. When the input was increased to 10 V, the dynamic measurement range increased from 10 μl/min to 0.7 ml/min. Interestingly, the standard deviation decreased to 4.9 mV, meaning that with an increase in voltage input there was an increase in the dynamic range and repeatability. The increase in repeatability with increased voltage input was expected. An increase in the micro heater power would produce a stronger heat pulse, resulting in more heat transfer to the fluid.

The effect of changing the width of the input voltage pulse on the response of the flow sensor was investigated. The response of the thermocouple to a voltage input that was cycled between pulse widths of 50 ms, 100 ms, and 150 ms is shown in Figure 8. There is an increase in the thermocouple voltage output with an increase in pulse width. This provides an additional method of calibrating the flow sensor. The difference in voltage response can be calculated for each pulse, and the change in amplitude plotted against the mass flow rate. The slope and offset of this curve can then be correlated to the mass flow rate. The advantage of such a scheme is that the sensitivity of the sensor can be maintained, while reducing the overall thermal effects on the fluid since the overall power consumption and heat input can be reduced. A sequence of weak, medium and strong pulses is now used and detected by the sensor. The difference in voltage response for pulse widths of 50 ms, 100 ms and 150 ms with a period of 4 secs that was cycled through are shown in Figure 9 for different flow rates. The voltage response of the thermocouple increases with an increase in pulse width in a linear manner. Consequently, the linear slope associated with the change in the output voltage with the pulse width and the offset of this curve can be related to the flow rate. The first point associated with the 50 ms pulse at the lowest flow rate is erroneous, which decreases the slope for this particular flow rate. The reason for this is that at the lower pulse widths there is a wider range or error in the thermocouple output.

5. Conclusion

A micro flow sensor based on the thermotransfer principle was developed for flows up to 0.7 ml/min with a 5% precision. The sensor consists of a microheater and a microthermocouple placed downstream of the heater. One advantage of the sensor developed here is its scalability, as it has a significantly reduced foot print requiring only a heater and a temperature sensor. One important consideration is to ensure that the heat diffusion through the substrate and the fluid is small compared to that convected by the flow to improve the signal-to-noise ratio. The heat diffusion through the substrate can be reduced by using non conducting substrates or by having thermal barriers between the heater and microthermocouple in the design of the sensor. The ratio of the thermal diffusion to convection within the fluid will depend on the fluid thermal properties and the flow velocity. This is an inherent limitation of this type of flow sensor, and will impose a limit on the minimum flow rate that can be measured by the sensor.

The current sensor was operated using a pulsed voltage with a low duty cycle of 2.5 percent. The power consumption can be further reduced by introducing novel pulsing schemes, making the sensor particularly applicable to low temperature applications. No time domain information is required once the initial calibration is performed. This is a significant advantage of the thermotransfer method as it reduces the amount of hardware and software required for operation of the flow sensor. The technique, however, requires an initial calibration technique such that if it were required for each individual device thereafter would make this method too cumbersome and expensive. However, due to the batch fabrication method of MEMS devices, one device calibration per batch is sufficient making this mode of operation particularly attractive.

6. References

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