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Monolithic integration of micro-channel on disposable flow sensors for medical applications

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Abstract

We report on the integration of a micro-flow channel with a thermal flow sensor chip. Two different technologies were investigated: first the fluid channel is performed at the back side of the standard HSG-IMIT flow sensor chip using a double KOH etching process (DKOH). The second technology consists of a bond process between the flow sensor die and a polydimethylsiloxane (PDMS) sheet containing the channel. Both sensor types can be used for detecting flow rates in the range of 0.1–5.0 μl/min (H2O) and/or pressure differences of 10–600 Pa. Compared to our previous sensor design this leads to reduced packaging costs, increased reproducibility (better than 1%) and in particular a higher sensitivity at low flow rates. Finally a concept of an adhesive-free packaging of the sensor is presented.

Keywords: Flow sensor; Integration of micro-channel; Adhesive-free packaging; Low measurement range

1. Introduction

At present, flow sensors are mainly used for air flow determination in automotive applications. However an increasing demand is observed for fluid measurements in smallest flow ranges in chemical analytics, medical diagnostics and biotechnological analysis. These new applications require highly precise measurement and reproducibility, short response time, low power consumption, smallest dimensions, adhesive-free packaging and low-cost disposable products [1,2]. Furthermore miniature flow rate sensors are used in a wide range of applications with length scales varying from flow measurements in oil wells to micro-reactors [3]. The flow range to be measured varies over several orders of magnitude. A number of the existing flow sensors are based on measurement of the pressure drop across a narrow flow channel [4–7]. The sensors are based on two separate pressure membranes placed on each side of the flow channel. These sensors are usually used as difference pressure sensor and are not really adequate for flow rate determination. The flow sensor presented here is based on one single membrane sensor similar to [8]. This solution eliminates noise from thermal, electrical and mechanical sources that would affect the two pressure membranes differently. A second possibility to integrate the channel over the sensor is presented by [9], where the silicon die is glued directly on a capillary. This last method presents relative high costs for the adhesive and calibration steps which lead to an expensive sensor.

By the integration of chip-sized micro-channels with the flow-sensor an increased reproducibility (<1%) of the sensor and a measurement range of 0.1–5 μl/min can be attained. However a minimal change of the channel geometry implies a large signal deviation by such small geometrical dimensions. Therefore a very precise alignment of the channel over the active sensor is required as well as very small tolerances for its fabrication. Two different technologies were evaluated to combine a highly precise sensor with a low cost disposable system. Afterwards the chips could be integrated in an adhesive-free packaging.

2. Realization

At first an enhanced traditional silicon technology was used as illustrated in Fig. 1. The channel fabrication is performed by a double KOH etching process (DKOH) at the back side of our standard flow sensor chip [10]. Fig. 2 presents the process flow of the device. The starting material of the sensor fabrication is a (1 0 0) n-type silicon double-side polished wafer with

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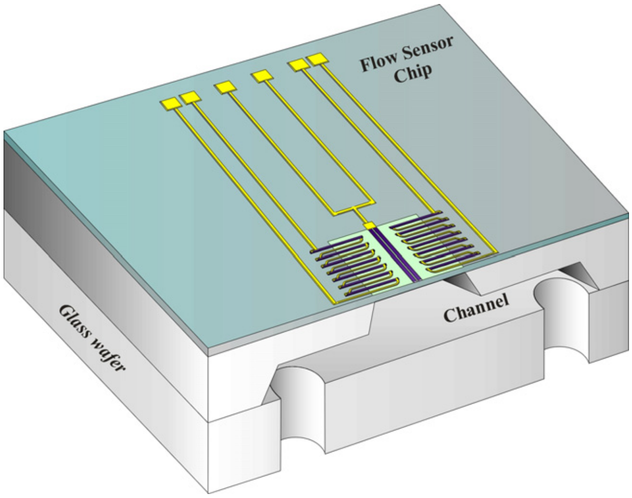

Fig. 1. Schematic cross-section of the flow sensor chip with integrated micro-machined DKOH fluid channel performed at the back side of the flow sensor chip. A glass wafer provides the fluidic connection.

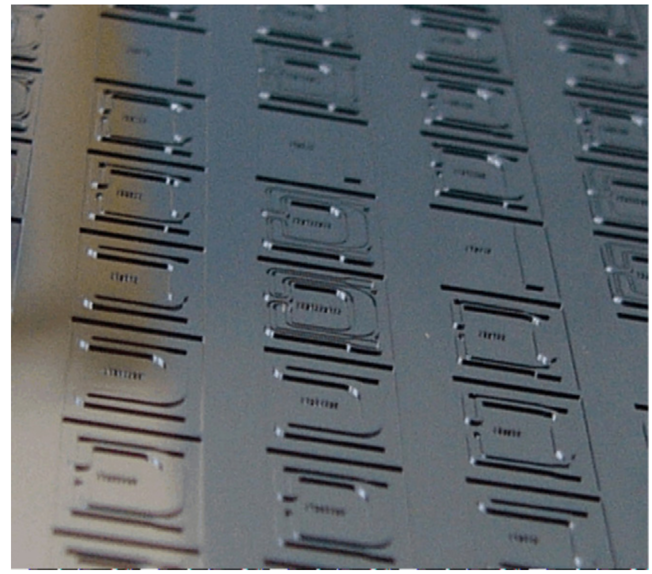

Fig. 3. Detail of the dry etched silicon negative template for the PDMS stamp.

a thickness of 300 nm and a diameter of 100 mm. After the deposition of the membrane materials (150 nm LPCVD silicon nitride layer on a thin oxide) a first KOH etching of 150 μm depth is performed at the back side of the silicon wafer to perform the future cavity under the membrane. The next step is the standard process for the thermopiles fabrication with a 100 nm polysilicon layer and one with 300 nm. This step needs a photolithography adjustment from back to front side. At this point a light deviation in the adjustment can occur. Afterwards – in order to protect the thermal sensor against harsh environments – a PECVD Si_3N_4 layer is applied as passivation. Then the second KOH-etching can be performed for the channel fabrication. This step represents the main delicate process. A too long etching will rapidly cause a membrane enlargement that consequently would change the heat distribution over the membrane. For example an extension of the membrane from 300 to 380 μm width induces a sensitivity increase of about 40% in air for a 1 mm^2 channel cross-section. In order to avoid this problem the last 5 μm silicon are etched using dry etching. In this way the membrane dimensions are well defined and present high fab-

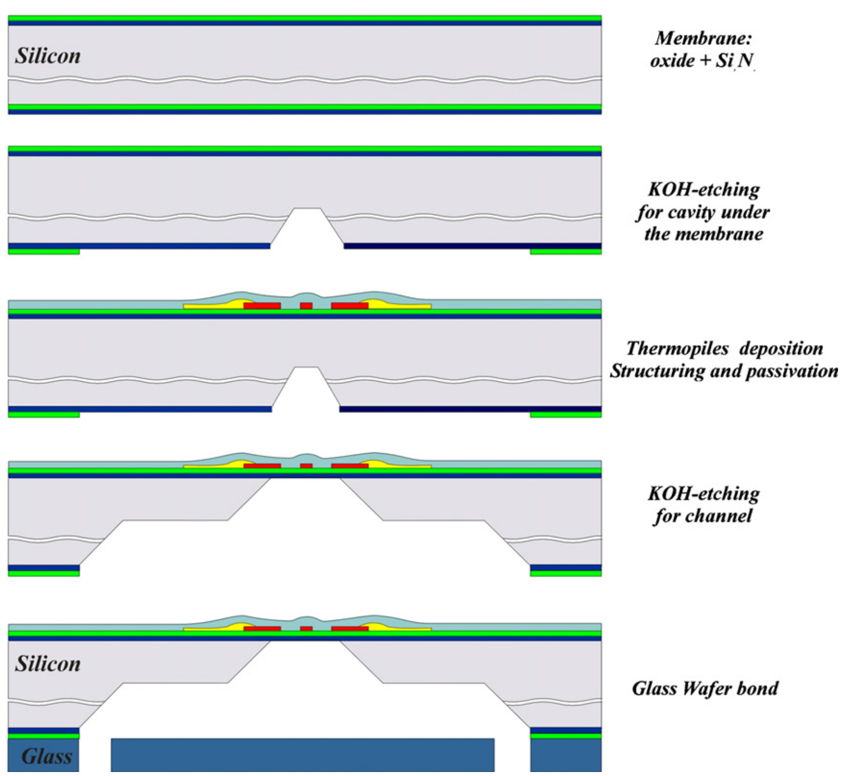

Fig. 2. Schematic drawing of the process sequence for the fabrication of the double KOH etching flow sensor chip.

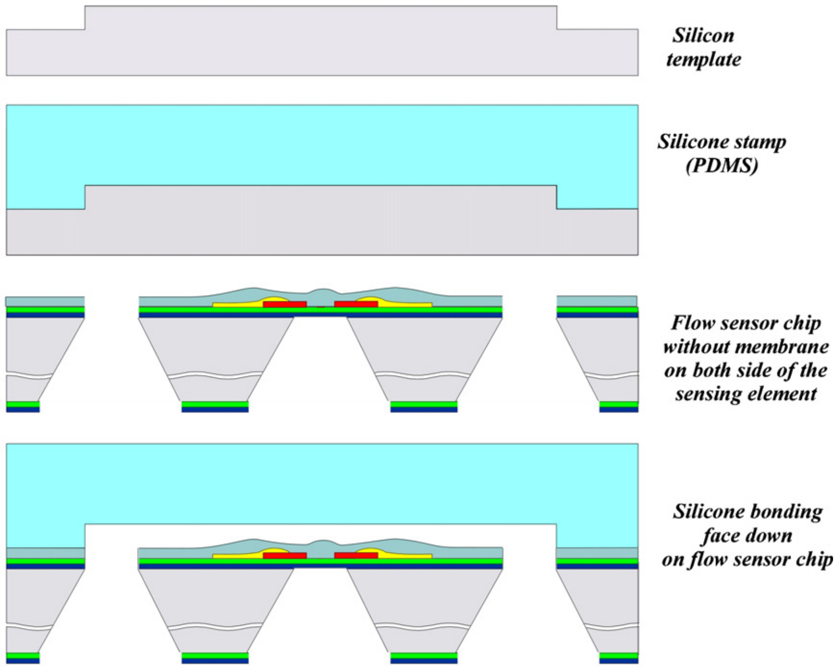

Fig. 4. Schematic drawing of the process sequence for the fabrication of the flow sensor chip with PDMS stamp with fluid channel.

rication reproducibility. Finally a full wafer bond with a glass wafer including borings as inlet and outlet provides the fluid delivery. With this technology the channel height is limited by the KOH-etching tolerances combined with the 300 μm silicon wafer thickness.

In order to obtain more freedom in channel design (e.g. height or realization of meander structures to alter the fluidic resistance), new bonding technologies based on biocompatible materials such as polydimethylsiloxane (PDMS) were developed. Low cost PDMS channel structures were used to define rapidly the influences of different channel geometries on the sensor sensitivity and the measurement range. The used PDMS is a two-component heat-curing system consisting of a base and a curing agent part. After mixing the two components the silicone is dispensed on a dry etched silicon template (Fig. 3) presenting the negative form of the future channels. After spreading and degassing a heat curing at 140 °C is performed for about 15 min. Consequently, the PDMS structures shrink of almost exactly 3%. Afterwards the PDMS plate containing the channel structures is pealed off the silicon template and stamped in small dies. The following bond process is performed after plasma activation of both partners. The process steps are summarized in Fig. 4. The bond step is performed chip per chip using a fine placer. Under these conditions the reproducibility of the fabrication and especially the adjustment of the channel over the membrane are not as good as with the alignment obtained by a mask aligner. A failure of about 40 μm in adjustment is observed over the channel length, causing an eventual change in the flow status between laminar and turbulent. In the PDMS channel fabrication itself a deviation of the channel height is observed that has several causes. First there is a material shrink during the curing process. Secondly the precision of the dry etching for the silicon template is of about ±2 μm which leads to a high failure in percent for the low channel heights which are in our case the focus of the current development. At least a small tolerance is induced by the bond process itself. Fig. 5 presents a flow sensor with a fluid channel integrated in PDMS [11]. On both sides of the flow sensor membrane, holes are performed in the silicon chip to realize the fluid connection.

3. Results and discussion

Flow measurements, sensitivity and measurement range depends on several parameters which highly interact with each other. For example the sensor sensitivity is strongly correlated to the flow resistance and the channel geometry.

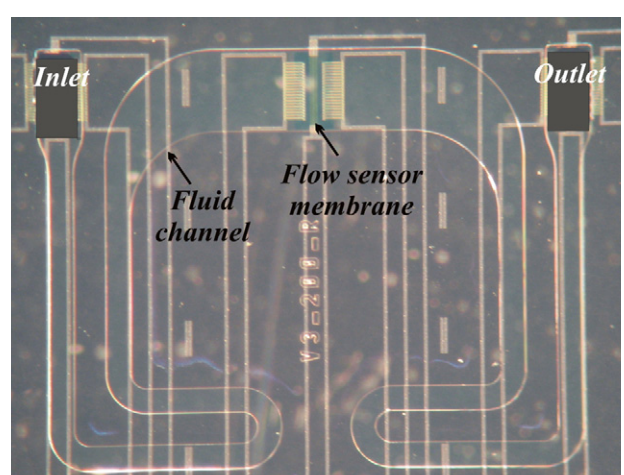

Fig. 5. Photography of flow sensor chip with integrated fluid channel by bonding a PDMS part containing a channel on top of the flow sensor die.

The increase of the flow resistance induces a decrease of the flow rate in the sensor and thereby a lower output signal. With a smaller channel height the fluid velocity increases at the same time and the output signal rises. Both phenomena take place simultaneously which means that the sensor sensitivity will follow the most important parameter. Under these conditions an analytical description of the thermal flow sensor would premise a high complexity.

The aim of this study is a technological research correlated to the characterization of the sensor to proof the technologies themselves. However the influence of the significant parameters is shortly discussed in relation to the experimental results using FEM simulations [11].

3.1. DKOH sensor

To determine the influence of the tolerances of the "traditional" fabrication technologies, air flow measurements with DKOH sensors were carried out.

The flow resistance depends only on the fluid parameters and the channel geometry. This dimension is independent from the flow velocity and can be used for a direct comparison of the different sensor types. One goal of the study is the realization of a high sensitive difference pressure sensor. For this purpose a high fluid resistance is suitable. The reproducibility of the fluidic resistance of flow channels over a wafer batch for the double KOH-etching is presented in Fig. 6. The pressure drop over the sensor system is a measure for the cross-section of the fluid channel and indicates the reproducibility of the flow sensor fabrication. An excellent reproducibility is achieved, since the observed deviations are in the range of the accuracy of the used reference pressure sensor.

Using the Hagen-Poisseuille law for a square cross-section the flow resistance can be calculated as follows:

$$R_{\mathrm{Rf}} = 8\varphi \frac{l(b+h)^2}{(bh)^3} \upsilon\rho.$$

Therein b and h represents width and high of a rectangular channel, ρ the fluid density, ν the cinematic viscosity and φ the coefficient for the cross-section form in the flow channel. The KOH etched channel height can be extrapolated from the curve slope assuming a channel width of 820 μm. In this way the flow sensor presents at its back side a channel height of about 164 μm. The calculated value fitted very well with the experimental results. Unfortunately the channel height is correlated to the silicon wafer thickness so that no variation of the channels can be achieved with this technology. It leads to a limitation of the sensitivity and therefore of the flow sensor resolution in a measurement range below 1 ml/min in air and 0.5 μl/min in water.

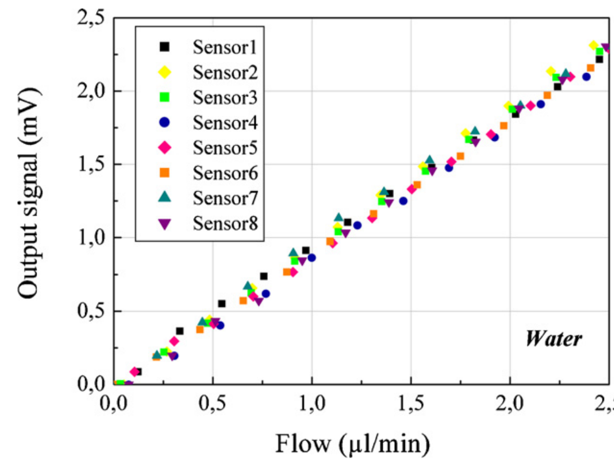

Fig. 7. Output signal for different sensors vs. flow. Heat power = 10 mW.

Parallel to the channel geometry investigation the output signals of the flow sensors are studied. Fig. 7 presents the results for a measurement range under 2.5 μl/min in water. The curves present a linear behaviour with flow in this low measurement range. A perfect overlap of all curves can be pointed out. This leads to conclude to a high reproducibility of the sensor signal with the DKOH technology.

Furthermore the flow sensor contains a thin membrane (1 μm thick) which has a burst pressure of about 2–3 bar. For high-pressure applications a solution was found to increase the mechanical resistance of the membrane: bonding a full PDMS

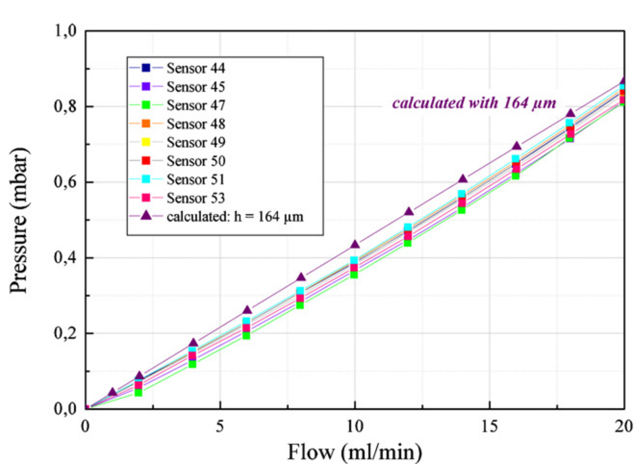

Fig. 6. Air pressure drop over the flow sensor vs. flow rate for several DKOH-silicon chips with a 4 mm channel.

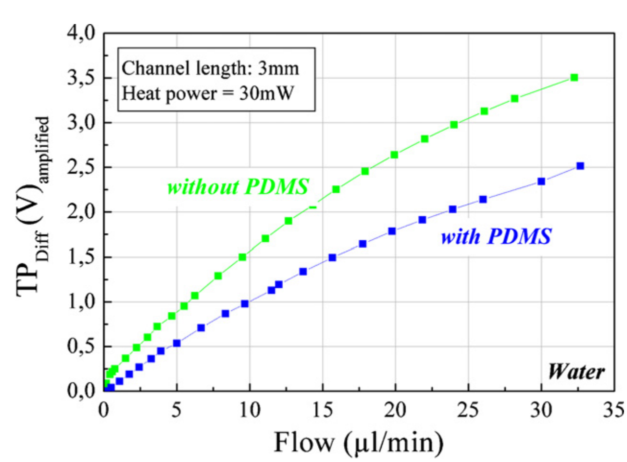

Fig. 8. Sensor signal over flow of the same sensor with and without PDMS on the membrane top.

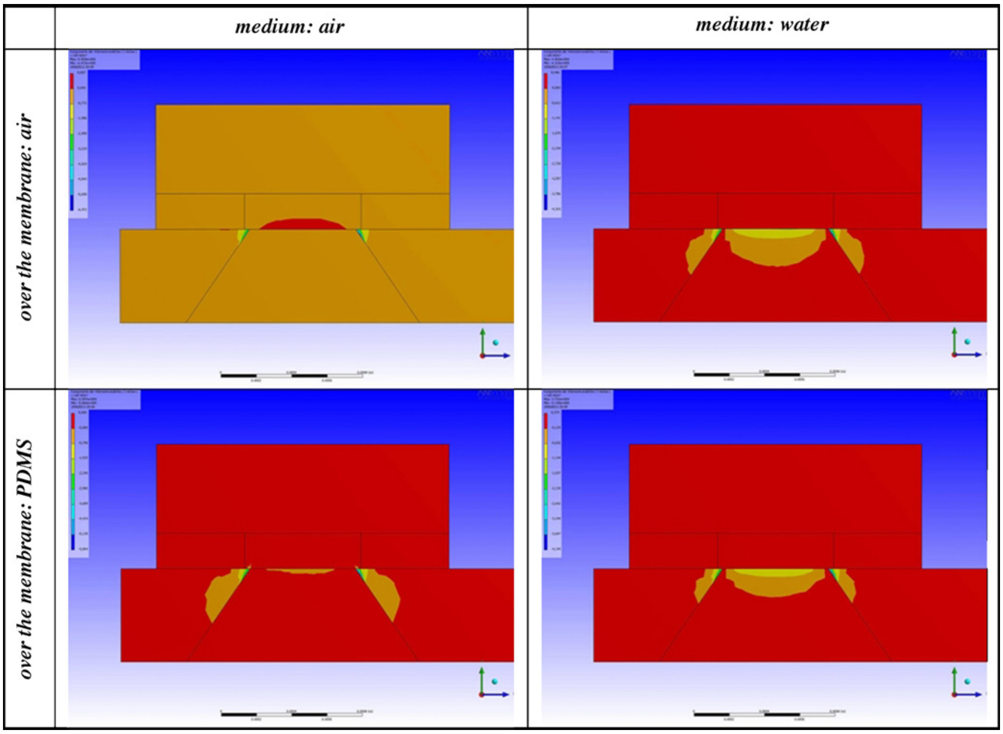

Fig. 9. Simulation of the heat density distribution in vertical direction in case of DKOH-sensors with and without PDMS for air and water.

die over the sensor membrane. Due to the thermal conductivity of silicone a loss in sensor sensitivity is expected. A DKOH flow sensor was measured with water before and after the silicone bond. Fig. 8 compares the two measurements. As expected a decrease of a factor 1.5 of the sensor sensitivity is observed for the sensor with PDMS on the top of the membrane due to the thermal transfer through PDMS. This factor between with and without PDMS is relatively small. As comparison a glass die is glued directly over the membrane. The sensor shows a decrease of a factor 2.5. The mechanical resistance against pressure was tested with air pressure up to 8 bar without any crack in the membrane.

Furthermore a FEM simulation with ANSYS Workbench is performed to investigate the thermal heat energy lost due to the PDMS die placed on the sensor membrane for its stabilization. Four different situations are considered. The material over the

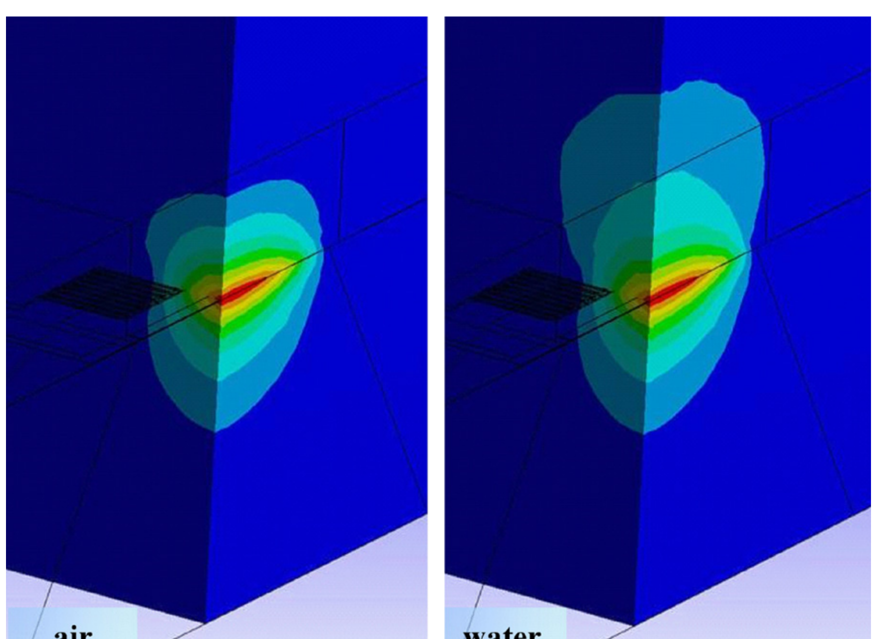

Fig. 10. Simulation of the heat distribution in PDMS sensors with a 200 μm height channel in air (left) and water (right).

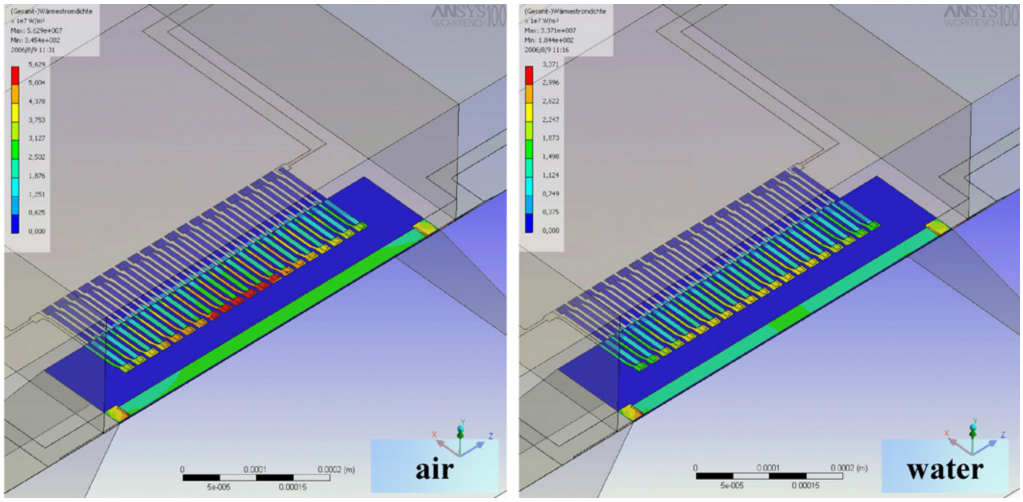

Fig. 11. Temperature distribution on the heater and thermopiles for a 200 μm channel height PDMS sensor. Left: air; right: water.

membrane, the channel walls and the channel top are assigned to air and PDMS respectively. The fluid under the membrane is thereby air or water in each case. The simulation results of the heat flow distribution in vertical direction are represented in Fig. 9. In all four cases the heat dissipation from the membrane edges into the silicon is recognizable. For both fluids the heat flow dissipation increases above the membrane in the case of PDMS bonded on it. A heat lost in the thermal flow leads to a

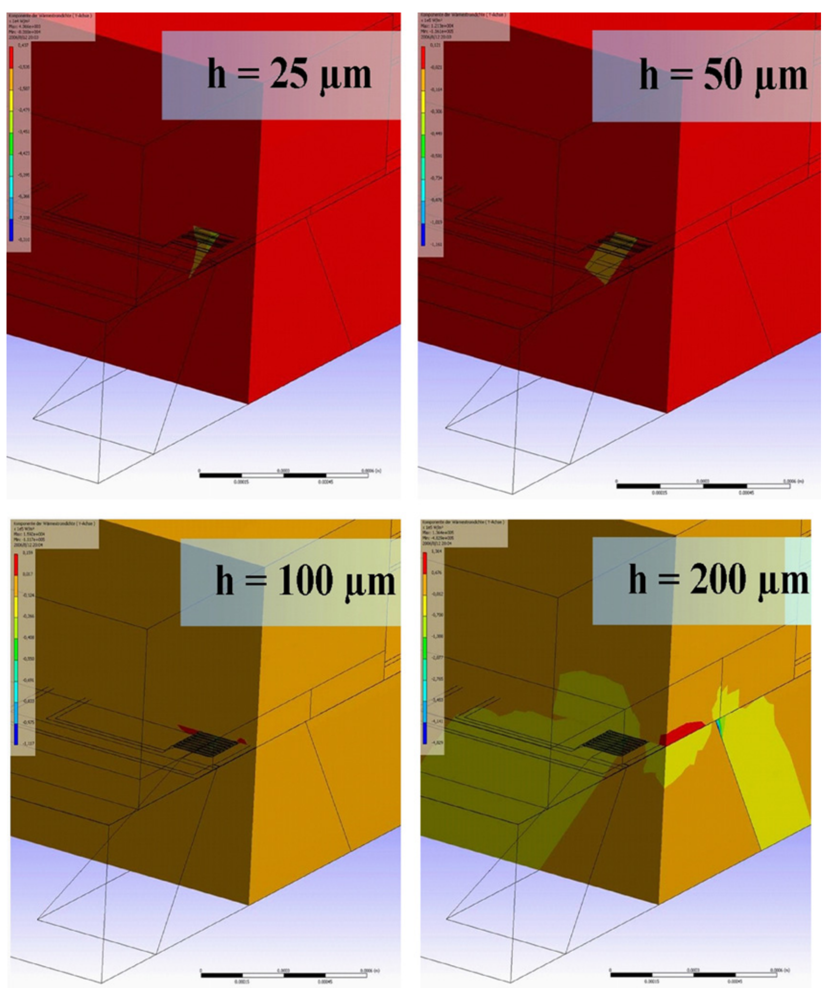

Fig. 12. Heat density distribution for PDMS sensors with different channel heights (25, 50, 100, 200 μm).

decrease of the sensor signal. With air as medium the difference of the heat distribution is clearly higher. The simulation results are in good agreement with the experimental results.

3.2. PDMS sensor

To achieve a better resolution and accuracy of the flow determination in a measurement range below 1 μl/min, the sensors with a PDMS layer on top, containing flow channels were characterised.

An investigation of the influence of the fluid (water and air) on the heat distribution in the channel in PDMS is performed to assist the experimental data analysis. A first simulation shows a sensor with 200 μm channel height without flow (Fig. 10). The effect of the different thermal conductivities of the medium can be clearly seen. Due to the higher thermal conductivity of water the thermal heat transfer in the medium is strongly increased leading to large heat dissipation in the PDMS material itself. Consequently the maximum temperature in the channel and the output signal of the flow sensor decreases. Additionally the thermal dissipation in the membrane is also investigated. The heat transfer of the heater over the membrane and of the thermopiles to the silicon chip is simulated. Using workbench it is possible to see in a defined restricted area the temperature distribution. Fig. 11 summarizes the simulation results. Both pictures show the tempering membrane which is covered left and right with water and air respectively. To obtain a sensor signal the two ends of the thermopiles need to have different temperatures. The end of the represented thermopiles on the membrane is visibly heating up. Already after few micrometers from the transition between thermopiles and silicon frame, the chip takes the ambient temperature because of the high thermal heat conductivity of the silicon material. Thereby the chip temperature, thus the ambient temperature, serves as reference dimension.

The last simulation deals with the influence of the height of the channel in PDMS. Fig. 12 shows the heat distribution with PDMS sensors in water for different channel heights: 25, 50, 100 and 200 μm. The red colored areas stand for a high heat flow density. Here the heat produced by the heater is efficiently

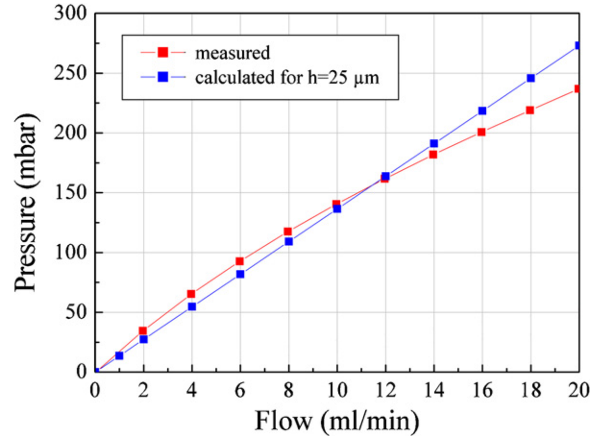

Fig. 14. Measured and calculated pressure over flow in air for a 25 μm channel height.

dissipated. The heat flow density in the channel walls and in the silicon chip is very large with small channels, as seen for 25 and 50 μm channel height. The heat dissipation over the channel wall increases with the flow channel dimension drop. The largest heat flow density in the fluid between the heater and Thermopiles arises with the 200 μm channel height. The heat flow is transferred not over the channel wall to the environment, but distributed in the fluid itself. Under these conditions the measurement range will be shifted into a higher flow rate.

Fig. 13 shows the influence of the channel height on the sensor measurement range and sensitivity, respectively. As expected, there is a significant increase of the sensor signal with decreasing channel heights. For a 2 μl/min reference flow the sensor output signal is about ten times higher for a 25 μm channel height than for the one with a height of 200 μm. For a 25 μm high channel a detection limit of 0.05 μl/min is obtained. However, the width of the measurement range decreases considerably with smaller channel heights. The same measurements are performed in air. A linear increase of the sensitivity is observed with channel height from 200 to 50 μm. By 25 μm channel height a lower sensitivity as expected is obtained. One reason for this behaviour is the heat dissipation in the upper opposite channel wall.

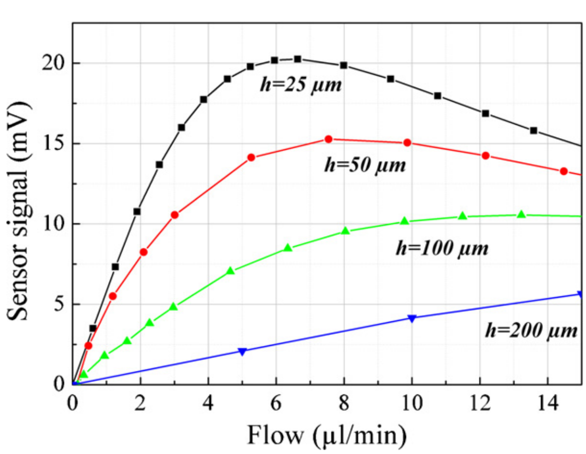

Fig. 13. Sensor signal over flow for different fluid channel heights. Measurements are performed at 10 mW heating power and water as fluid.

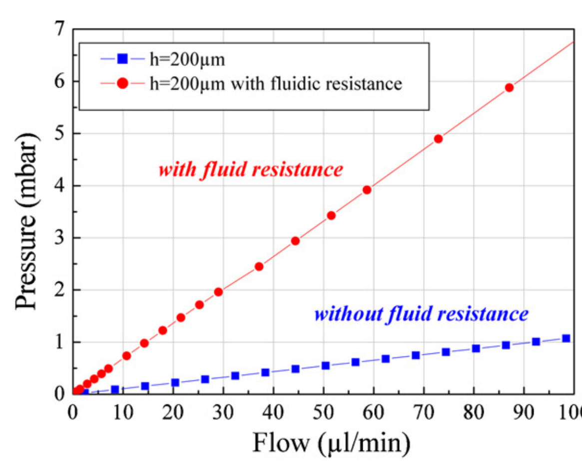

Fig. 15. Pressure signal over flow for different channel geometries (with and without fluid resistance) at 10 mW heating power and water as fluid.

Furthermore, one of the drawbacks of PDMS material is its elasticity. For applications with air, under a relative pressure of about 100 mbar and a 25 μm high channel structure a deformation of the channel with a dilation of its cross-section is observed. This phenomenon induces a change of the fluidic resistance and thus a failure in the flow measurement. Fig. 14 presents the results of the 25 μm channel height sensor compared to theory. The pressure over the sensor has lost its linear behaviour in air. For small flow the measured pressure is higher than the calculated. The channel is constricted. With increasing flow and pressure the measuring curve rises. The soft PDMS expands from the inside by the relatively high positive pressure. The duct cross-section becomes larger and flow resistance becomes smaller. In water this effect cannot be observed, since the flow like the pressures lies lower here.

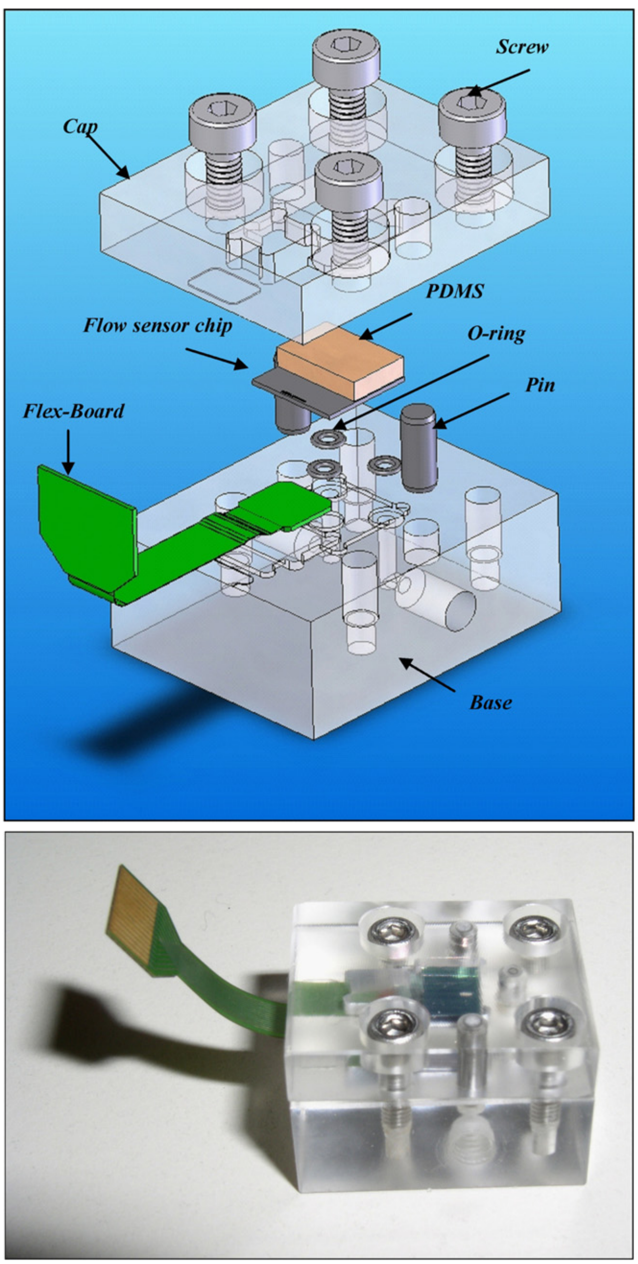

Fig. 16. CAD model of the adhesive-free flow sensor package (top). Photograph of the whole package for fluid leak tests (bottom).

Another application of the thermal flow sensor is its use as differential pressure sensor. To achieve high pressure sensitivity the internal fluidic resistance is increased by adding a meander structure to the fluid channel in PDMS on chip. An enhancement of the sensitivity by a factor of 6.3 is achieved (Fig. 15).

This result opens new industrial applications for the flow sensor as precise differential pressure sensor.

3.3. Adhesive-free packaging

Finally, a concept for a biocompatible adhesive-free packaging of the sensor is presented in Fig. 16. A DKOH chip with a PDMS protection layer is clamped between two plastic parts. The PDMS layer protects the sensor membrane and thus increases its robustness and compensates tolerances of the packaging. Tightness is performed with two o-rings placed at the in- and out-let of the chip. The electrical connection is provided by a flexible circuit board on which the sensor chip is bonded. This package demonstrates the possibility to realize a hermetically sealed snap packaging in injection molding technology.

4. Conclusion

We presented a new generation of flow sensors with integrated micro-channels and demonstrated the realization of small channel structures using improved fabrication processes. The obtained sensor characteristics over different flow rates in water and air correspond with the expected improvement in sensitivity and resolution. Realizing the fluidic channels by a double KOH etching process from the backside of the sensor element provides the highest geometrically reproducibility. The new DKOH sensors present a failure in reproducibility under 1% where the standard ones show about 12%. The process of producing the channel structure in PDMS could be used as rapid prototyping to evaluate the influence of different channel structures and sizes. An accuracy of about 4% is obtained with this last technology.

In addition to standard mass flow rate measurements the realization of a high precise differential pressure sensor was possible. Therefore the internal fluidic resistance of the flow sensor was increased by integrating different micro-channel structures on chip. This leads to a very compact system having the size of the silicon chip (36 mm^2).

The new technological development combined with the adhesive-free packaging concept allows the usage in medical technology and makes a sensor size reduction possible.

For the future we plan to optimize the channel structure and its integration on the flow sensor chip. A SU8 technology will be developed to realize the free formed channel structures in an inelastic material with a full wafer bond process.

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Biographies

Billat Sophie studied physics at Grenoble University (France) and received her Diploma in 1991. Her PhD in solid state physics at institute of spectrometry physics in Grenoble was on the electroluminescence of porous silicon. In 1995 she joined the institute for ion and thin layer technology of the KFA in Jülich, where she worked on supperlattices with porous silicon. Since 1998 she works on research and development of thermal sensors, especially on inclinometers, heat conductivity, and flow sensors in the microfluidics department at the Institute of Micro- and Information Technology of the Hahn-Schickard Gesellschaft (HSG-IMIT) in Villingen-Schwenningen.

Kliche Kurt studied mechanical engineering at the University of Stuttgart with specialization in microsystem and precision engineering and finished it with his Diploma Thesis in 2006. His diploma thesis done at the Institute of Micromachining and Information Technology (HSG-IMIT) in Villingen-Schwenningen, Germany, discusses the characterization of micromachined thermal flow sensors with integrated fluid channel. Until finishing the thesis he works as engineer at HSG-IMIT in the department microfluidics in the group thermal sensors. His current main tasks are CAD-construction and CFD-simulation.

Gronmaier Roland studied electrical engineering with specialization in microelectronics at the University of Ulm, Germany, where he received his diploma in 2003. In his diploma thesis he worked on the development of CVD-diamond structures for micro-membrane pumps. Since 2003 he works in the department of microfluidics at the Institute of Micro- and Information Technology of the Hahn-Schickard-Gesellschaft (HSG-IMIT) in Villingen-Schwenningen, Germany. His work focuses on micro-dosage systems and microfluidic platforms.

Nommensen Peter received the MSc in physics from the University of Tübingen, Germany, in 1996. Thereafter he joined the HSG-IMIT in Villingen-Schwenningen, Germany, as a Process Engineer for PVD and bonding techniques. Since 2001, he is the head of the Department of Micro-technology at the HSG-IMIT. His current research interests include silicon micromaching and device integration techniques, with a focus on entire fabrication processes for MEMS applications and their suitability for serial production.

Auber Johannes studied in Furtwangen (Germany) and received his diploma in Micro Systems and Engineering in 1995. He worked as AS Process Engineer by the Robert Bosch company where he was responsible for wet etching and dry etch processes (ASE). Until 1998 he has joined the HSG-IMIT where he focussed mostly on etch processes.

Hedrich Frank studied engineering at Dresden University and received his Diploma in 1992 on preparation of thin TiNi shape memory alloy layers by sputtering processes. Since 1992 he works in the sensors and microfluidics departments at the HSG-IMIT in Villingen-Schwenningen. He works especially in the areas of electrical measurements like programmes, porous silicon technology and the development of thermal sensors.

Zengerle Roland is Head of the Laboratory for MEMS Applications at the Department of Microsystems Engineering (IMTEK), University of Freiburg, Germany. He also is a Director with the Institute for Micro- and Information Technology, Hahn-Schickard-Gesellschaft (HSG-IMIT), a nonprofit organization supporting industries in development of new products based on MEMS technologies. His and his team's research is focused on microfluidics and covers topics such as miniaturized and autonomous dosage systems, implantable drug delivery systems, nanoliter and picoliter dispensing, lab-on-a-chip systems, thermal sensors, miniaturized fuel cells, and micro- and nanofluidics simulation. He has coauthored more than 200 technical publications and 25 patents. He is the European Editor of the Springer Journal of Microfluidics and Nanofluidics. He is a member of the Technical Program Committee of several international conferences. Dr. Zengerle is a member of the International Steering Committee of the IEEE-MEMS conference.