Experimental study of Ti/Pt thin film heater and temperature sensors on Si platform

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Abstract— **Thin film Ti/Pt heaters and temperature sensors were designed and fabricated to provide controlled heating of meandered microchannels realized on the opposite side of the Si platform. Ti/Pt heaters and temperature sensors were fabricated simultaneously by DC sputtering on SiO_2/Si substrate. Annealing temperature in the range 400-700 °C was found to influence significantly the sheet resistance and consequently the final resistance of deposited Ti/Pt layers. Measured temperature coefficient of resistance for temperature sensors and heaters was between 1440 and 1700 ppm. Improved temperature response and heating up to 250 °C with 27% decrease of power consumption was obtained by Pyrex cavity insulation.**

I. INTRODUCTION

Microfluidic devices nowadays synergize the conventional microelectronic technology and the modern techniques used in biochemical, pharmaceutical and related areas. The common problem in such applications is a microfluidic device, which can provide accurate, controlled and localized heating to enable required processes. In such lab-on-chip devices the reactions take place in a chamber closely coupled to the heating source, which preferably includes also integrated temperature sensors. Another application of thin film heaters and temperature sensors, usually made on thermally insulated suspended thin membranes, is providing and measuring required thermal operating conditions for gas, flow or humidity sensors.

One of the main advantages of thin film heaters and temperature sensors over other types is that they can operate/respond extremely fast, have relatively low power consumption and can be fabricated by standard microfabrication techniques.

In the field of temperature sensors and heating elements, linear behavior, high sensitivity and small masses are desirable especially when the sensors are to be used in control systems and in processes where the temperature may change rapidly. In this case the linearity and high sensitivity simplify the circuitry that is needed to translate the sensors output into correct temperature value [1]. Metals are the most linear elements for sensing the temperature; however disadvantage of metal temperature sensors is their low resistance what consequently means lower measuring resolution.

Platinum is commonly selected as heater material since it has a good thermal response and a resistance that exhibits a positive and highly linear temperature dependency. Besides, Pt shows excellent long term stability, it is chemically inert and has well established manufacturing processes. By appropriate design, the Pt thin film heater could be able to act simultaneously as a heater and a sensor, as reported also in the literature [2, 3].

The main objective of this work was to design and to fabricate thin film heater and temperature sensors simultaneously. The temperature sensing elements and the electrical heaters should provide accurate temperature control over the desired heating range between RT and 300 °C. In the presented design, this heater is intended to provide uniformly the evaporation heat for the various liquids flowing in the microchannels which are fabricated on the opposite side of the silicon platform.

II. DESIGN AND FABRICATION

As shown in Fig. 1, the heater design comprises a single Ti/Pt line with three meanders located along the microchannel network fabricated on the rear side of the microfluidic Si platform for maximum heat distribution uniformity.

Two Ti/Pt resistors, used as temperature sensors are positioned at the edges of the heater. Due to the high thermal conductivity of Si they should read accurately the temperature at the inlet and outlet fluid ports. This is particularly true in this case due to the fact that there are no expected fast transients.

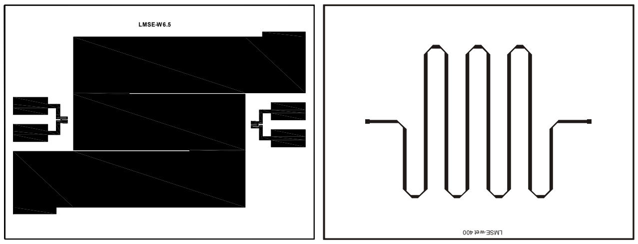

Figure 1. Mask design of the front side Ti/Pt sensors and heater (left) and mask design of meandered microchannels on the rear side (right).

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Based on the design guidelines for the resistive heater given in detail elsewhere [4], the design of photo masks was carried out, followed by the development of a suitable technological process for the Ti/Pt heater and temperature sensors on the front side and microchannels on the rear side. Since silicon is an excellent heat conductor, heat transfer from the heater to the required heating region is the most effective if the microchannels are fabricated on the opposite side of the same silicon substrate. To improve the heat distribution uniformity and to reduce thermal losses, additional Pyrex insulating cover with prefabricated cavity was implemented on the final Si platform. Fabrication process steps are presented in Fig. 2.

Silicon substrate, double side polished, 100 mm in diameter, resistance of 20 Ohmcm and (100) orientation was first thermally oxidized to grow 0.6 µm of silicon dioxide on both sides (Fig. 2a). This layer represents electrically insulating layer where the Ti/Pt heater and sensors are subsequently fabricated, and also represents the etching mask in microchannel fabrication step.

Microchannels were first defined on the rear side by photolithography and then fabricated using wet anisotropic etching process with TMAH etchant (Fig. 2b). To transfer the design pattern of the resistive layers on the front side, a lift-off process was utilized by using photoresist HPR 504. Photolithographic steps were performed to obtain negative pattern of the final heater (Fig. 2c). Prior to the deposition of metallic layers, a short exposure to the oxygen plasma was performed to clean SiO_2 surface.

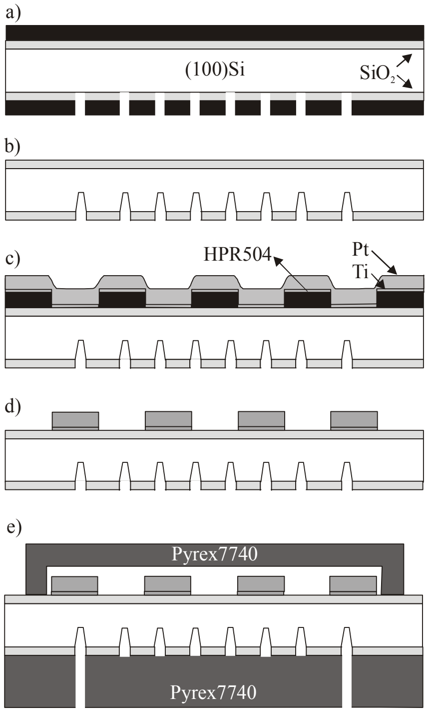

Figure 2. Fabrication process steps: a) deposition and patterning of photoresist on thermally grown SiO_2, b) fabrication of microchannels, c) deposition and patterning of photoresist, sputter deposition of Ti/Pt layers, d) lift-off process for the heater and sensors, e) double side anodic bonding of Pyrex glass.

To obtain good adhesion of Pt layer, a Ti prime layer was first sputtered on SiO_2/Si substrate. Sputter deposition of adhesive Ti layer was followed by the deposition of Pt layer (purity 99.99%) in DC sputtering system (Fig. 2c). The thickness of the layers was measured by Taylor Hobson surface profiler. Thickness of the prime Ti layer was 30 nm and thickness of the Pt layer was 200 nm. After sputter deposition of metal layers, lift-off step was performed to remove the metal with photoresist layer beneath (Fig. 2d). The Ti/Pt heater is further thermally annealed in forming gas to obtain final morphological structure of Ti/Pt layers and to stabilize thermal behavior of the heater. In order to improve thermal insulation the heater was covered by Pyrex glass, with a prefabricated 150 µm deep cavity above the heater. Pyrex was attached to the heater by anodic bonding technique, performed at 380 °C and anodic voltage of 750 V. To provide hermetical sealing of the microchannels on the rear side, additional Pyrex cover was bonded with prefabricated in/out through connections (Fig. 2e). Characterization of deposited thin film Ti/Pt was first performed on test wafers to obtain sheet resistance dependency on annealing temperature. Then, fabricated Ti/Pt heaters and sensors were characterized to obtain temperature dependency of resistance, followed by measurements on fabricated vaporizer unit assembled in the PTFE housing.

III. RESULTS AND DISCUSSION

A. *Ti/Pt deposition*

Different types of Pt deposition techniques and conditions, in addition with thermal treatment of deposited layers render also different physical properties (e.g. resistance, temperature coefficient of resistance (TCR), stress interaction with substrate) as shown also in the literature [5]. The purity of sputtered Pt layer is of utmost importance in achieving high TCR, since impurities affect the conduction properties in crystal lattice. In order to obtain stable Ti/Pt metallurgical system (interface rearrangement, stress annihilation, adhesion promotion), deposited samples were thermally annealed in forming gas in the temperature range between 400-700 °C to obtain final morphological structure of the resistive layer. The results of four point sheet resistance measurements are shown in Fig. 3. Full line in Fig. 3 represents results for separately annealed samples, while dotted line represents sample which was iteratively measured and subdued to all consecutive annealing temperatures.

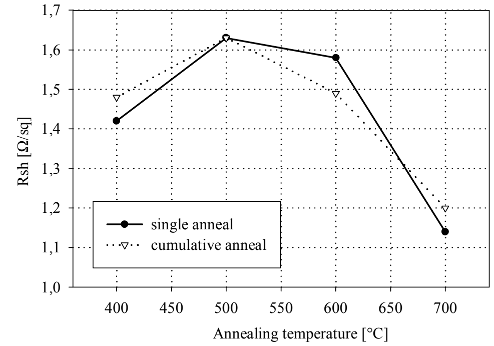

Figure 3. Sheet resistance of sputtered Ti/Pt layers vs annealing temperature.

It is shown that due to thermal treatment the sheet resistance has a maximum value around 500 °C and then decreases by increased annealing temperature. The significant increase of the sheet resistance is attributed to the recrystallization, interdiffusion processes and possible stress relaxations within the metal structure. The specific resistivity of Pt thin film layers is commonly higher than the corresponding bulk material since the mechanism of the conduction is different due to formation of grain boundaries [6, 7].

From the measured sheet resistance of sample annealed at 700 °C, the calculated specific resistance of Ti/Pt is around 2.4 times higher with respect to the value of 1.07 μOhmcm used as standard bulk value of crystalline Pt and was taken in our design considerations. Furthermore, this difference is consistently shown also through the heater resistance values measured at different annealing temperature (see Fig. 4).
Obvious discrepancy is attributed to the deposition method of Ti/Pt layer, heat treatment effect, layer thickness tolerance and the influence of the prime Ti-Pt interdiffusion. These influencing effects will have to be taken into consideration in the future design.

B. *Characterization of the Ti/Pt heater and temperature sensors*

Fabricated heaters were attached to the alumina substrate by silicone pads and wire bonded by 350 μm Al wire to enable electrical connections. The measuring setup was used to characterize the heater resistance dependency on the annealing temperature, to determine the TCR of the Ti/Pt heater and temperature sensors and to measure the heating characteristics such as electrical power dissipation versus obtained final temperature. Characterization setup is controlled by a computer which stores measurement data, at a predefined sample rate. Heater is current driven with a dedicated DC current source. Voltage is measured by KE2700 voltmeter with KE7702 switch matrix for voltage and current measurements.

Two controlling temperature sensors (Pt100) were placed on the heating platform to assure consistent temperature readout of both sides of the silicon platform. Afterwards, current and voltage drop on the heater are measured for determination of dissipated power.

In this preliminary stage, gathered data are sufficient for adequate heater characterization, including dissipated power characteristic and sensor temperature characteristic.

To use Ti/Pt device as a temperature sensing element, the temperature coefficient of resistance α is determined according to the following relation

$$\alpha = \frac{R - R_0}{\left(T - T_0\right) \cdot R_0} \tag{1}$$

where R is the resistance at temperature T, R_0 represents reference heater electrical resistance at reference temperature T_0 (defined at 0 °C), α is the temperature coefficient of resistance (TCR), and T is the actual heater temperature.

Figure 5 shows the measured results of resistance change with temperature for both, integrated Pt sensors and the heater in the range 25-250 °C. The nonlinearity appears only at low temperature around RT and could be a consequence of temperature sensors self heating caused by measuring current. This can be reduced by fast heat transfer, i.e. the underlying SiO_2 insulation layer has to be made thinner to facilitate this. The calculated TCR for measured temperature sensors is between 1432-1437 ppm and for the heater 1732 ppm and it proved very stable and linear above 70 °C. The difference in TCR is attributed to the size effect and related stress annihilation of the heater and the temperature sensors. An encouraging result shown in Fig. 5 is that the resistance of the heater shows very linear behavior, proving that heating is homogeneous across the heater area. Heater resistance accurately reflects its temperature only if the temperature distribution in the metal film is uniform, which is usually not the case when it undergoes significant resistive heating. The influence of Pt roughness on power consumption and the TCR was shown by Kim [8]. Furthermore, TCR is dependent also on thermal mismatch between the Pt layer and underlying substrate. It is believed that by optimization of the Ti/Pt deposition process step, higher TCR can be obtained.

Temperature characterization was performed by measuring the temperature on both sides by Pt100 and integrated Ti/Pt sensors. Influence of additional thermal insulation over the heater on the electrical power consumption was characterized by comparing the two heaters with and without Pyrex glass cover.

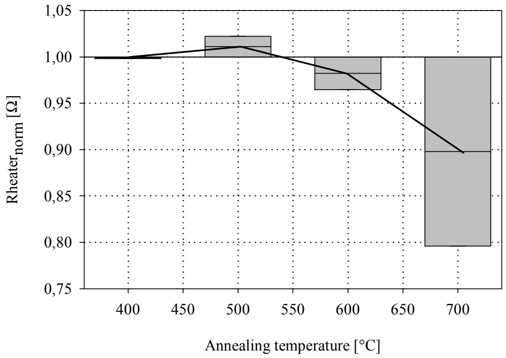

Figure 4. Heater resistance (normalized by RT value) vs annealing temperature.

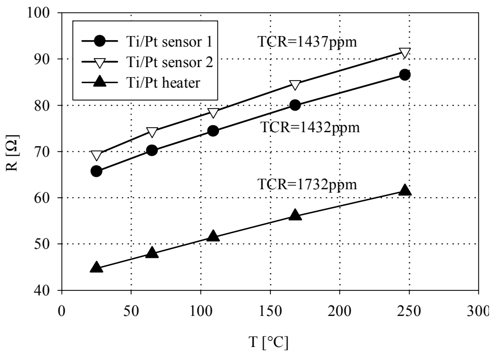

Figure 5. Temperature dependency of integrated Pt sensors and heater resistance with calculated TCR values.

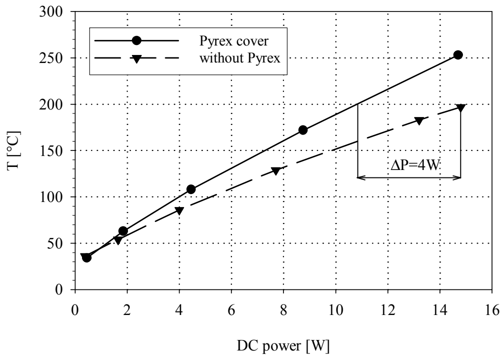

Figure 6. Influence of Pyrex cover insulation on dissipated power of the heater.

Thermal insulation was improved by Pyrex cover that was pre-etched to obtain the insulating cavity above the heater. The anodically bonded Pyrex cover proved stable behavior under all heating experiments, without any adhesion failure. The results in Fig. 6 show that a significant reduction of heat loss is obtained; e.g. to maintain the temperature of 200 °C, almost 27% less supply power is required for the heater with Pyrex cover insulation compared to the heater without Pyrex cover. As shown in Fig. 6, the thin film heater having a uniform temperature distribution under the Pyrex cavity could act simultaneously as a heater and as a temperature sensor. This would simplify the temperature control significantly.

Beside the measured power consumption, the heating dynamics was also evaluated, from temperature vs. time characteristics. By comparing both type of heaters (with and without Pyrex insulation), it was found that temperature rise time (defined as the time needed to reach 90% of final temperature) was decreased by 15% using the Pyrex cover. This is an important issue, since fast response of heating element is often a basic requirement in many applications.

C. Characterization of the Ti/Pt heater and temperature sensors during vaporization in microchannels

A PTFE housing was designed and realized to provide fluid connections (1/16 inch SS tubing) on the microchannel side as well as electrical connections to the heater and temperature sensors on the heater side of the silicon platform.

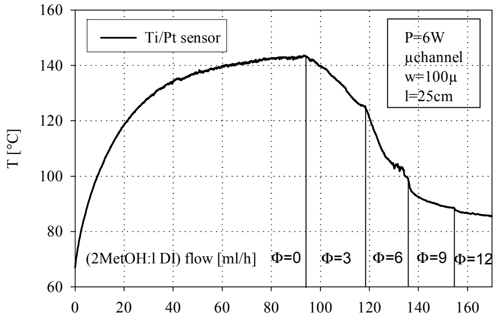

Figure 7. Temperature vs. time characteristic of integrated vaporizer supplied by constant heating power of 6 W. Evaporization process substantially decreases the temperature in the microchannels.

By applying the developed housing, the microchannels and heater together were utilized and characterized as vaporizing unit. Fig. 7 shows the heating temperature dependency of vaporizer, measured by the integrated Ti/Pt sensor. After reaching a steady temperature (145 °C) with constant heating supply power of 6 W, a volumetric mixture of two parts of methanol and one part of deionized water was introduced into the microchannels by a syringe pump. Due to evaporization heat needed to evaporize required inlet quantity of liquid, e.g. 3 ml/h, the temperature decreases by 15 °C for specific setup and given insulation.

Vaporization process in microchannels was simultaneously monitored also optically, providing the information on the location of phase transformation dynamics. By each increase of the inlet liquid it was observed that evaporization point position in the microchannel moves significantly toward the outlet port of the microchannels. By in-situ measurements of temperature with fabricated Ti/Pt sensors, the appropriate temperature control loop can be provided for evaporization of adjustable input liquid flow.

IV. CONCLUSION

Design, fabrication and characterization of Ti/Pt heater and integrated temperature sensors, to provide heat and simultaneously monitor the temperature in Si microchannels, are presented. It was found that thermal annealing of deposited Ti/Pt layers has significant influence on the sheet resistance and consequently on the final resistance of heater and temperature sensors. Thermal insulation of the heater was successfully implemented by anodically bonded Pyrex cover with prefabricated cavity. It is shown that this approach reduces the power consumption of the heater by nearly 27%. Linear dependency of the heater resistance vs. temperature and obtained TCR value of 1732 ppm proves that the heater can be used as the temperature sensor for temperature control in vaporization process.

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