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Micromachined calibration chip with heat source and temperature sensors for Scanning Thermal Metrology (S_{Th}M)

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Abstract

The monitoring of heat flux is becoming more and more critical for many technologies approaching nanometric dimensions. Scanning Thermal Microscopy (S_{Th}M) is one of the tools available for thermal measurement at the nanoscale. This measurement technics needs calibration samples. Therefore, micro-hotplates made of platinum heater suspended on thin silicon nitride (SiN) membranes were fabricated for the calibration of Scanning Thermal Microscopy probes. The objective is to obtain heated reference samples with localised resistive temperature sensors (RTD) on the membrane to probe the temperature on a micro-scale area (typically 10x10 μm²). This sensing area is dedicated to (1) quantify the thermal resistance between the S_{Th}M tip and hot surface contact; and to (2) evaluate the perturbation induced by the probe on the heat dissipation when the contact measurement is performed. In this communication, we report on the thermal design of low-power calibration chip and their fabrication, as well as the electro-thermal characterization of sensitive RTDs made using e-beam technology. Thermal contact measurements using a thermocouple based S_{Th}M probe validated the functionality of the calibration chip.

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Keywords: Calibration chip, Scanning Thermal Metrology, S_{Th}M probe, heat transfer, temperature sensing, RTD, micro-hotplate, thermal contact, thermocouple.

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1. Motivation, design and fabrication

Recently, SThM is being to become an effective tool for thermal metrology at the nanoscale [1]. Microelectromechanical systems (MEMS) calibration platform including heat source and temperature sensors have been designed and manufactured in order to provide active reference samples with the perspective of establishing calibration dataset to SThM community. This work and related objectives are developed in the European project named QuantiHeat¹. In order to monitor and calibrate the SThM probe/surface interactions, the instrument needs active calibration samples to be able to perform thermal metrology. Contact measurements for this specific purpose were performed in the past using standard hotplate not specifically designed for that purpose [2]. We report here on adapted devices with minimum power consumption in order to increase the sensitivity when performing measurements to determine the thermal contact resistance. We have also designed localized temperature sensor on the heating surface to calibrate the probe/surface interactions accurately.

1.1. Design

The objective of the calibration chip is to obtain heated reference samples with localized resistive temperature sensors (RTD) on a suspended membrane to probe the temperature on a micro-scale area (typically $10 \times 10 \mu\text{m}^2$). This sensing area is dedicated to (1) quantify the thermal resistance between the SThM tip and hot surface contact; and to (2) evaluate the perturbation induced by the probe on the heat dissipation when the contact measurement is performed. The RTD structures have been designed based on standard lithography and e-beam lithography. The latter thanks to its higher resolution can provide a higher RTD resistance value per area and therefore higher sensitivity.

Design and dimensions of the calibration chip are detailed bellow. Fig. 1 presents the device cross-section and planar design of RTD sensors. The main dimensions of the structure are detailed in Table 1. Different device variations have been manufactured changing the heater area and the type of RTD. RTD_a was patterned using ebeam-lithography, while the second type, RTD_b was structured using standard photolithography.

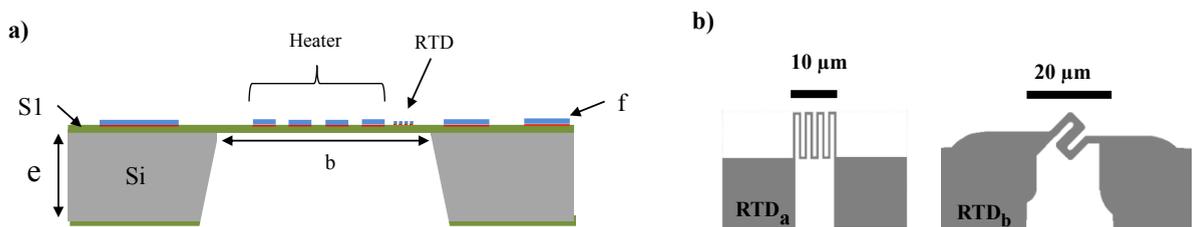


Fig. 1. (a) Schematic cross section of device ; (b) RTD design variations

Table 1. Characteristics of the calibration chip .

An example of a column heading	Material	Thickness (nm)	Area (μm^2)
RTD	Ta + Pt	$d = 6 + 50$	10×10
Membrane	SiN (layer S1)	$S1 = 500$	$b * b, b = 1000$
Heater	Ta + Pt	$f = 15 + 150$	220×220 to 440×440
Chip	Si	$e = 390 \cdot 10^3$	3000×3000

¹ European Union Seventh Framework Program FP7-NMP-2013-LARGE-7 under GA N°604668 Project “QUANTHEAT”, www.quantitheat.eu.

1.2. Fabrication

Silicon-nitride micromachined suspended heaters were manufactured on 390 μm -thick double side polish silicon wafers. First silicon wafers a 500 nm-thick silicon oxide was thermally grown on both side of the wafers. Then a 500 nm low-stress Low Pressure Chemical Vapor Deposition (LPCVD) SiN was deposited. Then 10 nm Ta/100 nm Pt thin films were e-beam-evaporated and patterned via a lift off process in order to define the resistive heating elements and the standard RTD. High resolution RTDs were structured using e-beam lithography to lift-off a thinner (THICKNESS) platinum film also deposited by e-beam evaporation. Finally, the release of the SiN membrane consisted in the bulk etching of the silicon wafer by Deep Reactive Ion Etching (DRIE) process. The SiO_2 layer used as etch stop was etched away in BHF solution. Illustrations of the released membrane and RTD structured by ebeam lithography are presented below.

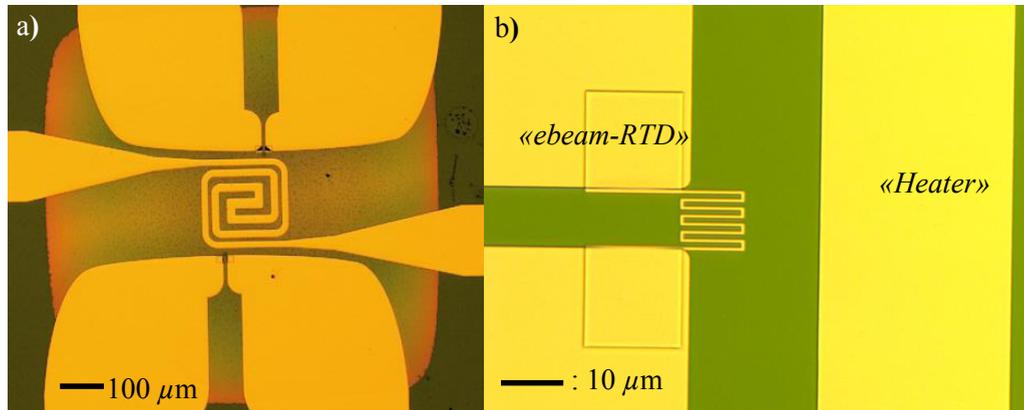


Fig. 2. (a) Example of CC suspended device implementing; (b) RTD structured using ebeam lithography

The RTD designs patterned using standard lithography provided typical resistance values between 12 and 100 Ω , while the RTD fabricated using ebeam lithography exhibited higher sensitivity thanks to their larger resistance values ($>300 \Omega$). The heat source and the side RTD sensors have been tested and used with SThM equipment.

2. Characterizations

The temperature dependence of the different heaters and RTDs implemented has been measured. A dedicated climatic chamber has been used to measure the resistance variation between 30 and 120 $^{\circ}\text{C}$. The Temperature Coefficient of Resistance (TCR) of all heating elements and RTDs have measured between 0.0025-0.0034 $\Omega\cdot\text{K}^{-1}$. The sensitivity achieved with the RTD obtained using e-beam lithography is three times higher than the 100 Ω RTD patterned by standard photolithography.

Power consumption and maximum operating power of the hotplate have been characterized. In order to characterize the maximum power consumption that the fabricated hotplates can reach, a voltage ramp with steps of 50mV/50ms is applied to the structure until its breakdown. The suspended heater (shown in Fig. 2, area = 220x220 μm^2) can be powered up to 120 mW before destruction of the membrane. According to SThM measurements results, the same structure consumes 24 mW to reach 300 $^{\circ}\text{C}$.

More SThM characterizations were performed in order to evaluate the calibration chip functionality by measuring temperature with a SThM probe put in contact with the localized RTD. The SThM probe is a micro-thermocouple probe made of thin wires, already reported in previous work [2, 3]. Fig. 3 presents an example of results obtained with a 5 μm diameter thermocouple. The difference of the measured temperature before and after contacting the probe gives directly the probe thermal response, called τ , which corresponds to the ratio between the elevation of the probe temperature ($T_p - T_a$) on the elevation of the RTD temperature ($T_s - T_m$). An averaged value of 0.7 was extracted from these measurements.

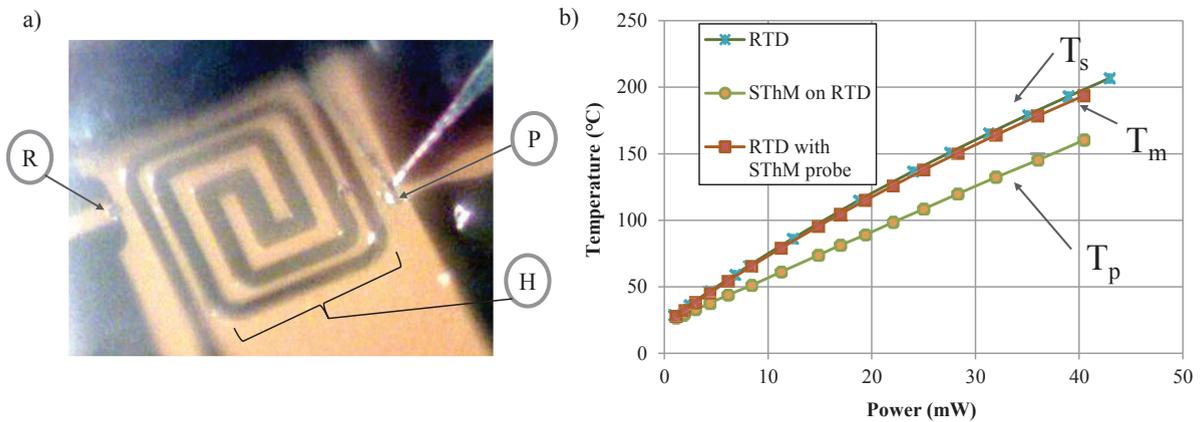


Fig. 3. (a) Optical view of the device and the 5 μm SThM thermocouple probe (P) in contact with a RTD (“R” indicates the RTD locations and “H” the heater area); (b) Evolution of the temperature with respect to the power consumption: (x) on area R using RTD, (o) measured with SThM probe (P), (■) measured with RTD when the SThM probe (P) is in contact with the RTD;

A difference of 5°C (without correction) is observed at 30mW between the RTD measurement when the probe is in contact with the resistor and when there is no contact. Therefore the thermal influence on the SThM probe in contact with the heated surface could be observed and the successful operation of the calibration chip highlighted.

3. Conclusion and perspectives

In this work the manufacturing and the characterization of calibration chips made of platinum heater suspended on a thin silicon nitride (SiN) membrane was achieved. Localized resistive temperature sensors (RTD) have been used as local thermometer directly implemented on the silicon nitride membrane. Fabricated devices consume 24 mW at the temperature of 300°C and maximum applied power can go up to 120mW. SThM measurements could be performed on the heated membranes and it was demonstrated that influence of the probe/surface contact can be evaluated using this dedicated calibrated chip.

In the future, the functionality of the active calibration platform will be demonstrated for several types of SThM probes and a variety of heated materials. The thermal contact between the SThM probe and the heated membrane will be analyzed and calibrated. Additionally the limit of sensitivity (and therefore the spatial resolution of the measurement) of the manufactured RTD will be explored by reducing the probe/surface contact area.

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