Characterisation of a micromachined Wobbe index sensor

Bogdan Breazu (s1834959), EEMCS Faculty, IDS Department, University of Twente, Enschede

Examination committee:

Dr. ir. R.J. Wiegerink

H-W. Veltkamp, MSc

Ing. R.G.P. Sanders

Dr. ir. R.A.R. van der Zee

29 January 2020

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Abstract

This report presents the calculations, measurements and simulations of the TCR (Temperature coefficient of resistance) for silicon and platinum inside multiple MEMS (micro-electromechanical system) chips used for microfluidic applications. The chips were designed as a Wobbe index meter and uses a silicon strip as a heater and a gold-platinum-tantalum alloy as a temperature sensor. The report aims to prove the working principles of the chips design as well as show the performance achieved during experiments. The theoretical calculations use models (parallel plate approximation) so all the limitations are discussed separately. The design and performance of the chips is influenced by aspects as the distribution of the heat transfer along the silicon heaters and the limitations of the SCT fabrication technology. During the experiment the chips are evaluated from the visual point of view using a microscope and also technically trough the probe measurement and joule heating process. The report records all the progress and steps taken by the chips during the experiment as well as verdicts at different checkpoints. During the experiment, one chip obtained a great performance, achieving a temperature of 325ºC at a power of 0.35W, one chip achieved an average performance, a temperature of 118ºC at a power of 0.20W and one chip broke down after only 0.20W. The paper also discussed the reliability of each design and future improvements. The simulations do not reflect the real world design and aim to prove the calculations of the theory. The heat dissipation along the silicon strip is considered to have the shape of a hyperbolic cosine function, so the most heat dissipation is in the middle and the least on the sides. Also the temperature difference between the Silicon and the platinum was calculated as a linear function that increases linearly with the power. The temperature difference varies from chip to chip depending on the length of the silicon strip but ranges from 0.5ºC to 2ºC at a dissipated power of 0.35W

Introduction

This report presents the proof of design for a MEMS (micro-electromechanical system) for calculating the Wobble index. In the last couple of years the composition of the natural gas has changed due to the introduction of LNG and biogas in the gas supply. This changes came with a set of challenges, due to the fact that the energy of the newly introduced gases is not the same as the normal natural gas. The “Integrated Wobbe Index sensor” project aims at the realization of a miniaturized Wobbe index meter for the measurement of the energy content of fuel gases, with most parts integrated on a single silicon chip. Fuel gas and air are heated and mixed on-chip, resulting in spontaneous combustion. The combustion energy is estimated from the resulting elevation in temperature and, combined with density and flow rates measured by also integrated micro Coriolis mass flow sensors, the Wobbe index can be calculated. The wobbe index defined as the heat release when a gas is burned at a constant gas supply pressure. It is defined by the following formula: [1]

$$\mathrm{I_w} = \frac{\mathrm{HHV}}{\sqrt{\mathrm{RD}}} \quad (1)$$

HHV: Higher Heating value (energy/volume)

$\mathrm{I_w}$: Wobbe Index

RD: Relative Density ($\rho_{\mathrm{gas}}/\rho_{\mathrm{air}}$)

In principle to achieve the high heat value (“amount of heat released by the unit mass or volume of fuel”) [2] the gas needs to be heated up constantly. In previous designs the heaters used in the chips were made from platinum strips. This paper presents a new microchip design that uses silicon heaters instead of platinum in order to achieve a better performance. In order to optimize the performance, multiple designs have been created. This paper presents the advantages and disadvantages for every design choice as well as measurements and theoretical calculations for the working principles for every type of chip.

First, the proposed Wobbe index sensor is made out of one or two isolated microfluidic channels with silicon heaters on the sides. The chips are made using the surface channel technology explained in depth in section SCT.

As a general proof of principle – the gas will mix with air and will flow through the microfluidic channels. Using joule heating - by applying a voltage to the sidewall silicon heaters the temperature inside the microfluidic channels will rise. The temperature can very accurately controlled and estimated using the TCR approximation. The increase of temperature in the channels create combustion in the gas air mixture, thus producing more heat and increasing the temperature even more. The new temperature can be measured and thus the energy of the high heat value of the gas can be deducted. After that, the results can be plugged in formula (1) and the Wobbe index can be calculated.

Initially, a set of 14 chips have been produced and will be used in the experiment (figure 1). Out of the 14-chips set only 4 of them were in good order - had a continuous silicon heater and reached the final stage of the measurements – the joule heating. A complete status of the chips used in the measurements can be seen in appendix.

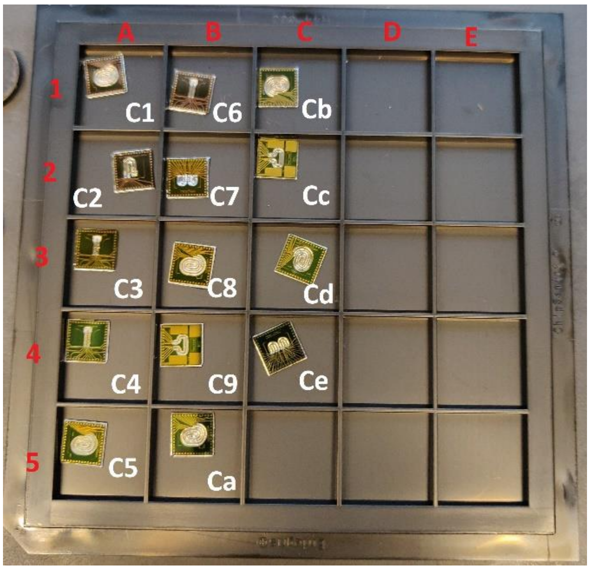


Figure (1) – The chips that will be used in the paper – and the corresponding notation and reference of each of them.

Method

SCT fabrication process

The chips were produced using the SCT (surface channel technology) and are split into 2 categories: the SMS 3-channel - 2 heater configuration and SEM, a 2-channel - one heater configuration.

The SCT method is used to be able to create multiple layers of materials and the channels between them. The steps of the SCT fabrication process for the chips discussed in this paper are presented in figure (2) [2].

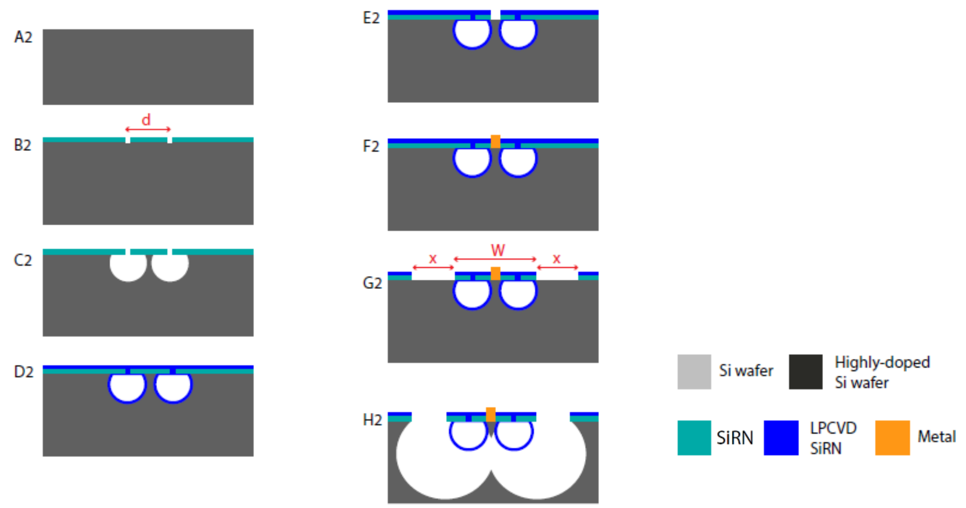


Figure (2) – From A2 to H2 there are presented the fabrication steps of a SEM chip. Figure retrieved from 2019 MFHS paper [3]

Initially, a bulk silicon wafer is covered on top with a thin layer of SiRN. After, at a distance ”d” two extrusions are made. The distance is the same with the distance between the middle of the channels. Next the first step of the etching procedure happens: the $SF6$ gas is applied through the etches of the SiRN (green in figure 2) and the shape of the channels are made. After etching, a rich nitride silicon material is applied on the edge of the channels. In this way the walls of the channels are complete. In the next step, in image E2, the platinum sensor is applied between the channels, on top of SiO_2. Afterwards, 2 more extrusions are made next to the channels, where the etching procedure takes place again. This steps ensures that channels are floating freely and they are surrounded by air. Ideally, after the second etching stage, a small triangular piece of silicon should remain in the middle of the channels. The manufacturing process of the chips is now complete.

Below the 4 chip designs that reached the final stage are presented:

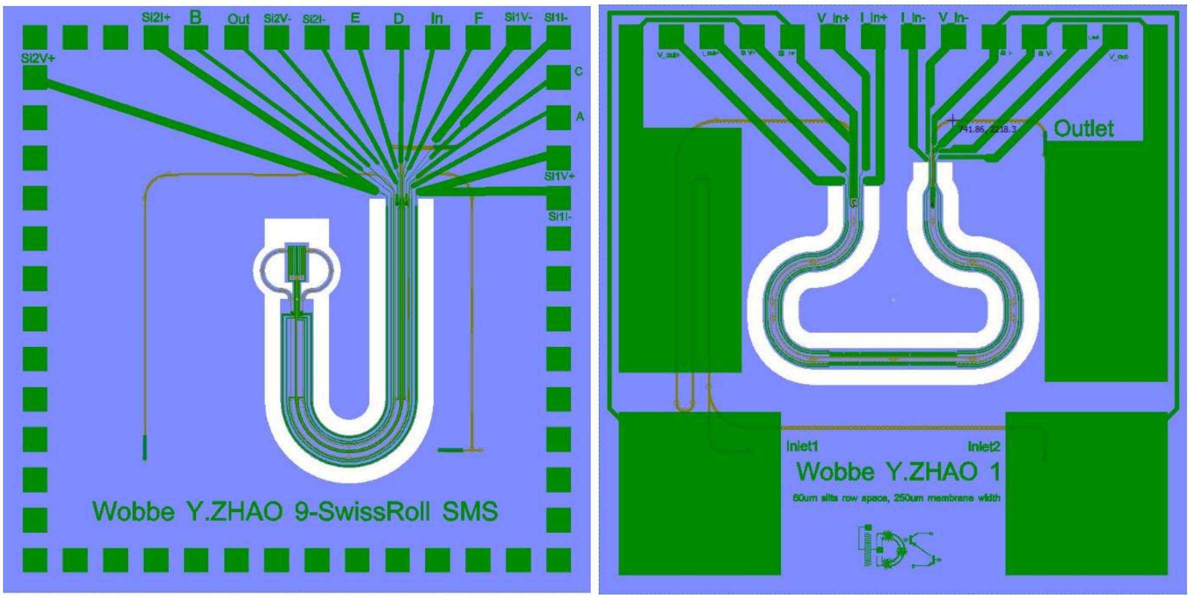


Figure (3) – Left – The Swissroll SMS chip blueprint; Right – The Corriolish SEM chip design blueprint

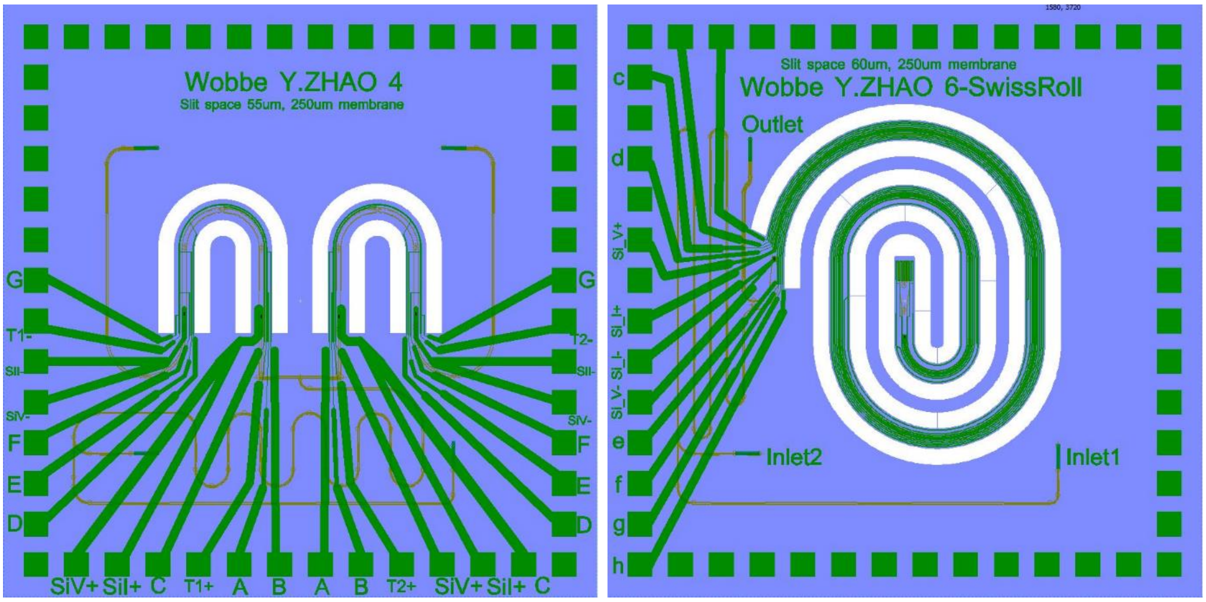


Figure (4) – Left – The U-shape SEM chip blueprint; Right – The Swissroll SEM chip design blueprint

Evaluation of the status of the chips

The first stage was to identify and make differences between chips of the same type. This was done by creating a coding. The names attached to the chips can be seen (see figure (1) and also in Appendix).

Photograph of the chips – microscope analysis

After the manufacturing process the chips will be checked using the Nikon microscope for minor imperfections and faults. The lenses used are 30mm and 40mm and the pictures are recorded digitally using the Nikon Digital Sight DS-L2. One common problem in the chips manufacturing is the discontinuity in the silicon strip – due to fact that during the etching process the SF6 gas is not accurately controlled. in this case the heater will not complete a circuit and joule heating is not possible, as the silicon will not conduct electricity.

This method is just the first step in a physical inspection, as the microscope is not able to see the profile of the silicon heater from a side or lateral view, but is a good first check that could separate bad chips from reaching further stages in the research.

Below a collection of images with a discontinuous silicon strips, chips damage and platinum delamination are presented. There is a list of imperfections that are harmless for the end result, as tiny particles and fragments that stand on top on the chips. The golden colour of the chips is due to the presence of gold on top of the platinum sensors.

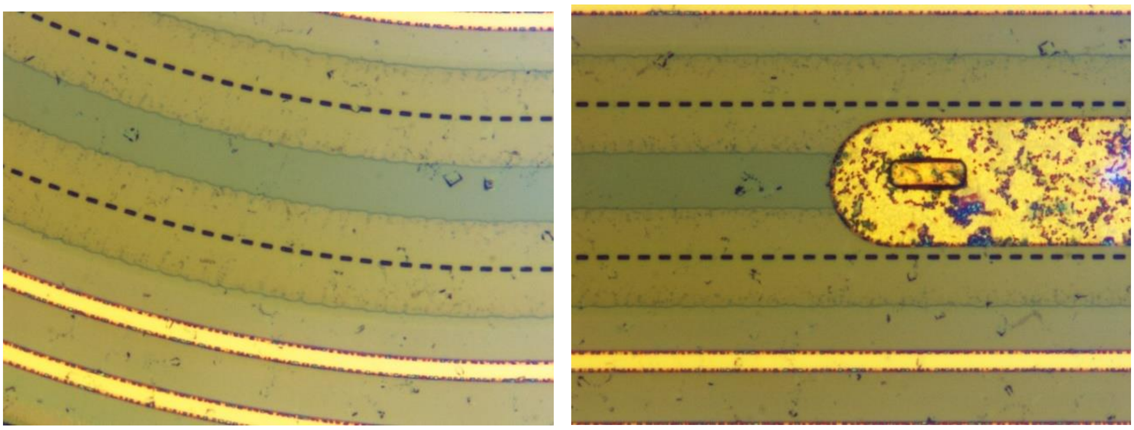


Figure (5) - Segments from a Swiss roll chip (C8)

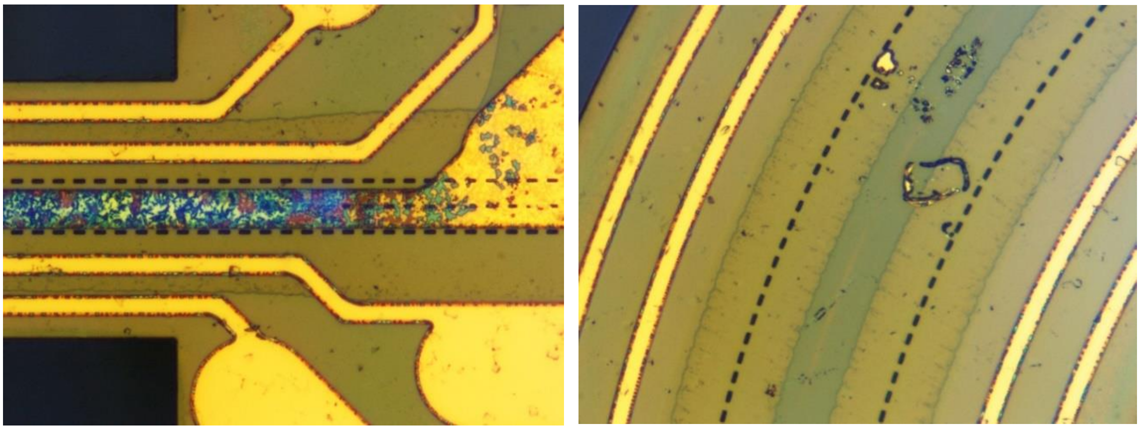


Figure (6) – Segments from a Swiss roll chip (C8)

Figure (7) – Segments from a Corriolish chip (C9)

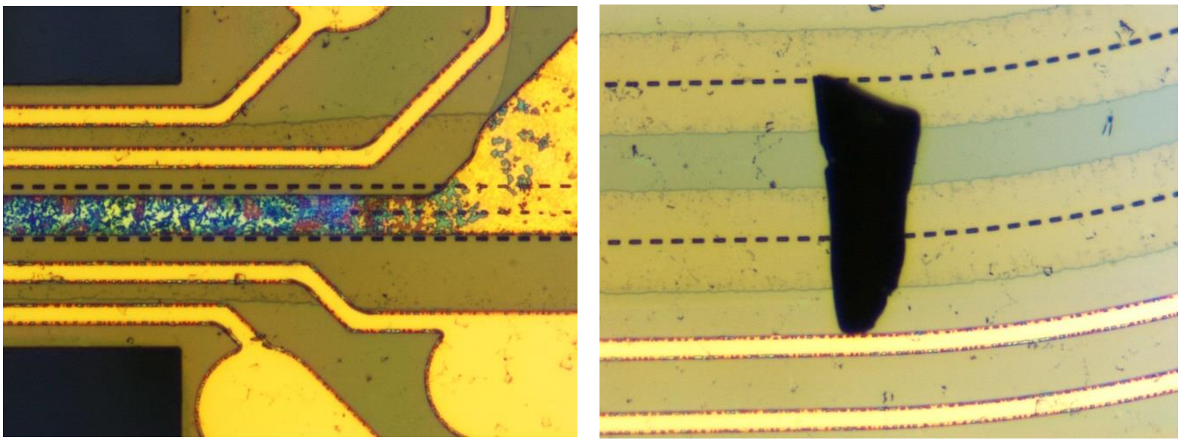


Figure (8) – Segments from a Corriolish chip (C9) Left – Platinum suffered some delamination, a thin layer phenomenon. This is harmless for the platinum.

Right – a silicon piece is on top of the channel – this has no effects on the working parameters of the chip.

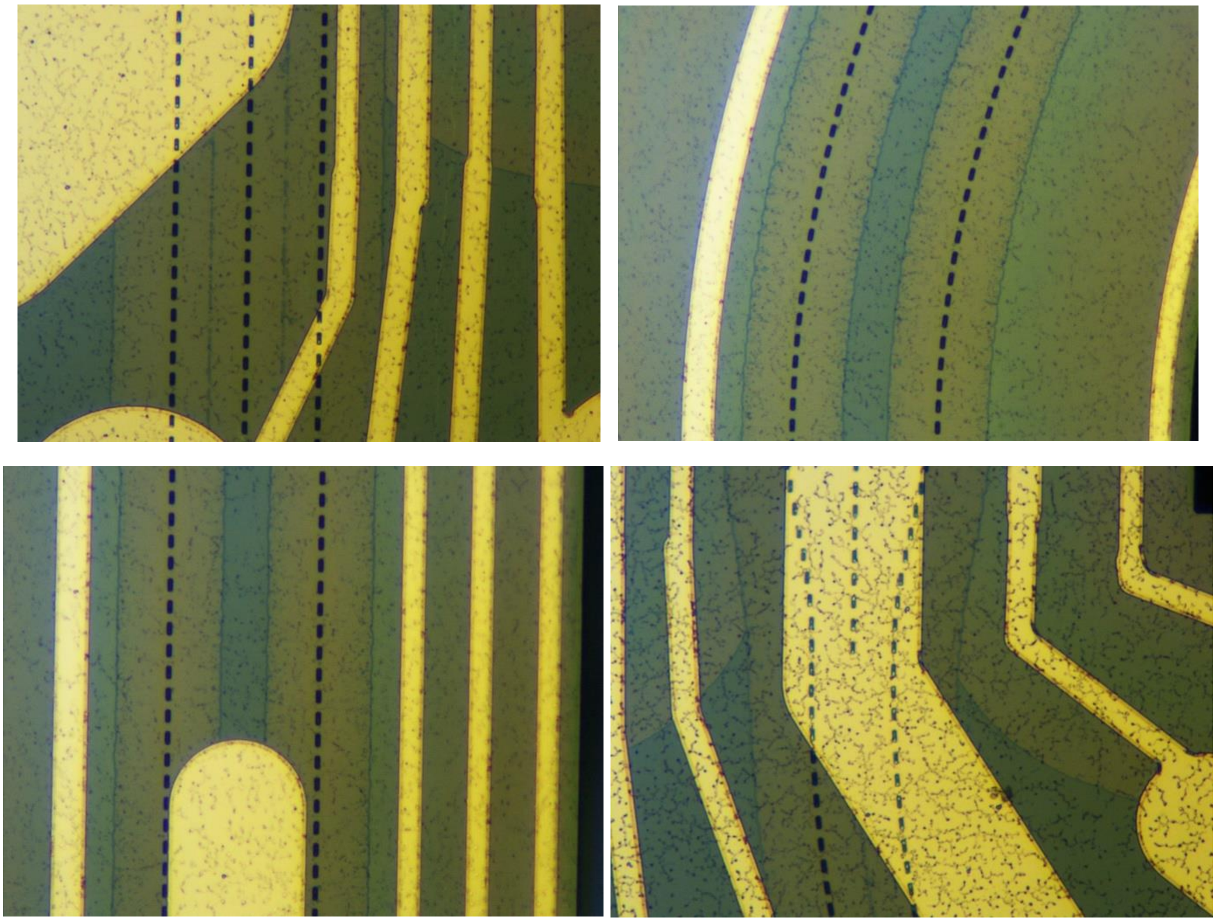


Figure (9) – A U-Shape SMS chip (Ce). The chip is very good condition, the Silicon can be very clearly seen to be continuous and there is a clear delimitation between the materials – platinum, silicon and the channels.

A lot of chips suffered damage or bad etching procedure, as some examples are showed below (figure 10 and 11):

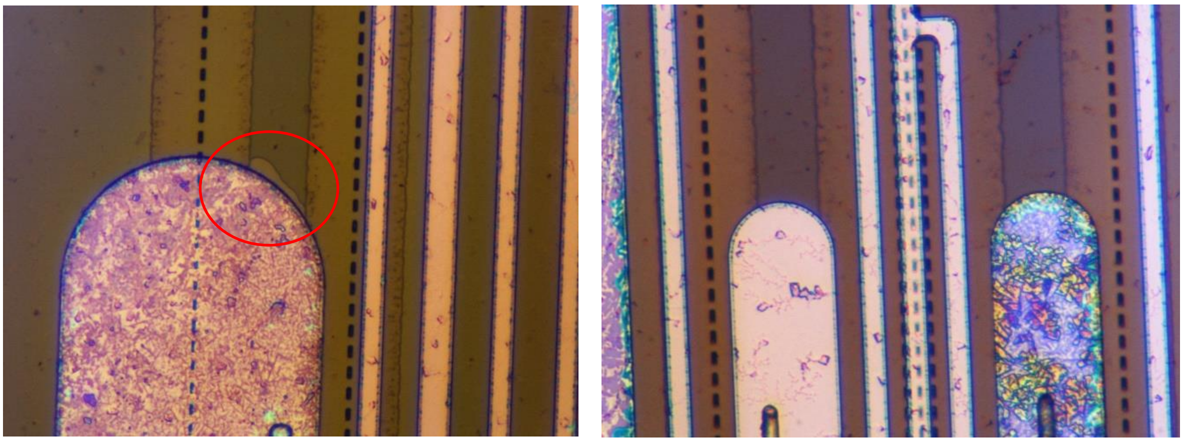


Figure (10) Left - Discontinuous silicon strip; Right - The purple platinum sensor is the effect of a delamination. The different colour of the images is due to the exposure settings of the camera

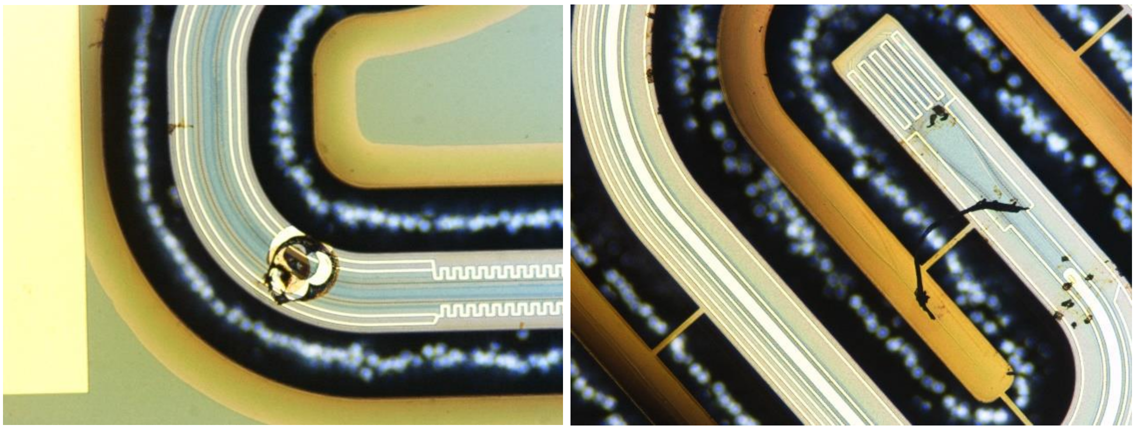


Figure (11) – Left - Damage made form an impact with an object, as a result the Pt sensor is broken (no resistance at the multimeter); Right – A broken wire and some dust particles are standing on top of the channels. This was proved to be harmless to the chip.

Gluing and Wire bonding

After the physical inspection, the broken chips will be excluded from further analysis and the ones that passed will move forward to the gluing phase. Gluing is an important stage because in order to complete the testing the chips need to be connected to a multimeter, a function generator and on a later stage to a fluid flow sensor. The IDS department has designed a rack (mask) in order to facilitate connectivity and manipulation throughout all the testing phases.

The gluing process is completed using epoxy adhesive and takes approximately 2 hours. A complete list of the tools needed to complete the gluing can be seen in figure (12) and the section dedicated in appendix.

The design of the rack and the correct connection will be presented below. The only alignment to be done is between the chip inlets and the rack's flow connections. There is a higher flexibility for all other connections, because the chips will be wire bonded at a next stage.

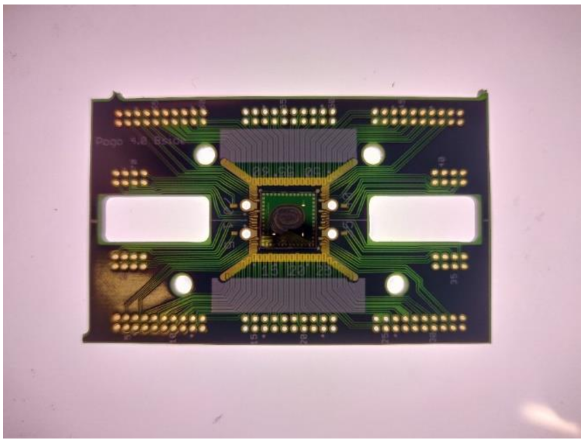


Figure (12) – A SEM Swissroll chip wireboned on the Coripogo v4.0 mask. The 3 inlets designed for the flow of the gas are visible. The Coripogo v4.0 mask is a mask designed by the IDS research group for domestic use

Afterwards, wirebonding process will take into account the space available on the chip, the length of the wire connection and the durability of the connection – any weak points as crossed connections must be avoided (figure 13). Furthermore, due to multiple chip designs, a general map of common connections will be made – ideally the silicon channels and the platinum segments should have the same numbers on the masks.

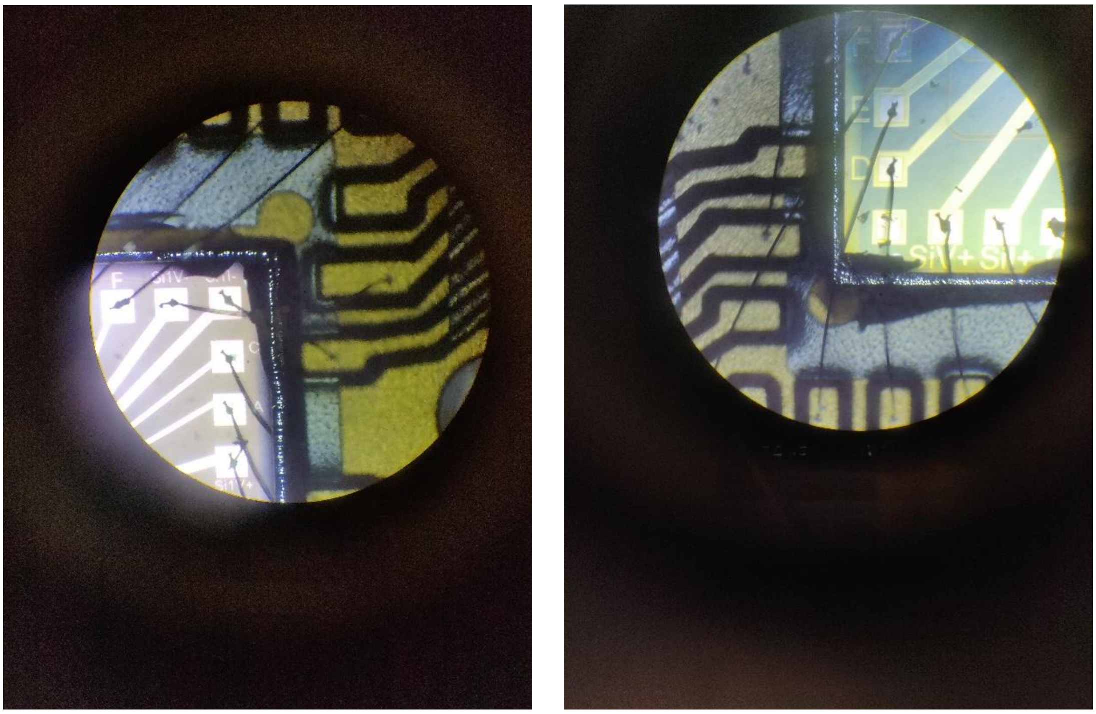


Figure (13) – A few wire bonds checked with the microscope.

Probe measurement

The probe measurement and the multimeter measurement aim the same goal: to check the shape of the silicon strips. Due to the fabrication process the silicon strip might have discontinuities or miss-connections with the wafer (figure()). The ideal shape of the silicon strip can be seen in figure ().

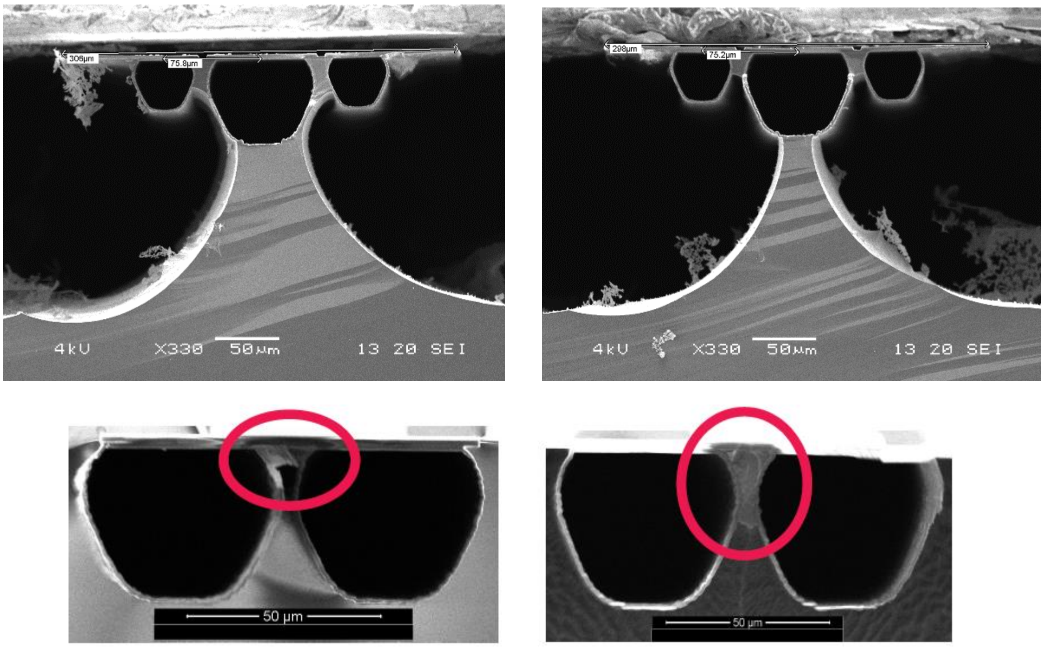


Figure (14) Top subfigures – SMS cross sections, corresponding to the chip C4 SMS. Left up corner– A bad connection, the silicon strip touches the bulk material. This situation usually gives a higher resistance than expected. Right up corner– The silicon strip is correctly connected Bottom figures – images of the cross section for normal SEM section – the highlighted circle represents the silicon heater. Figure retrieved from 2019 MFHS paper [4]

As an observation, there is no tool to check the in depth shape of the silicon strip along the chip, as the images above were obtained after the chips have been cross sectioned. The shape can only be expected by interpreting the measured resistance.

The connections for the platinum strip resistance measurements are presented below. Channels CH3 – CH9 are used to measure voltages, while channels CH221 and CH222 are used to measure the current applied trough (figure 15).

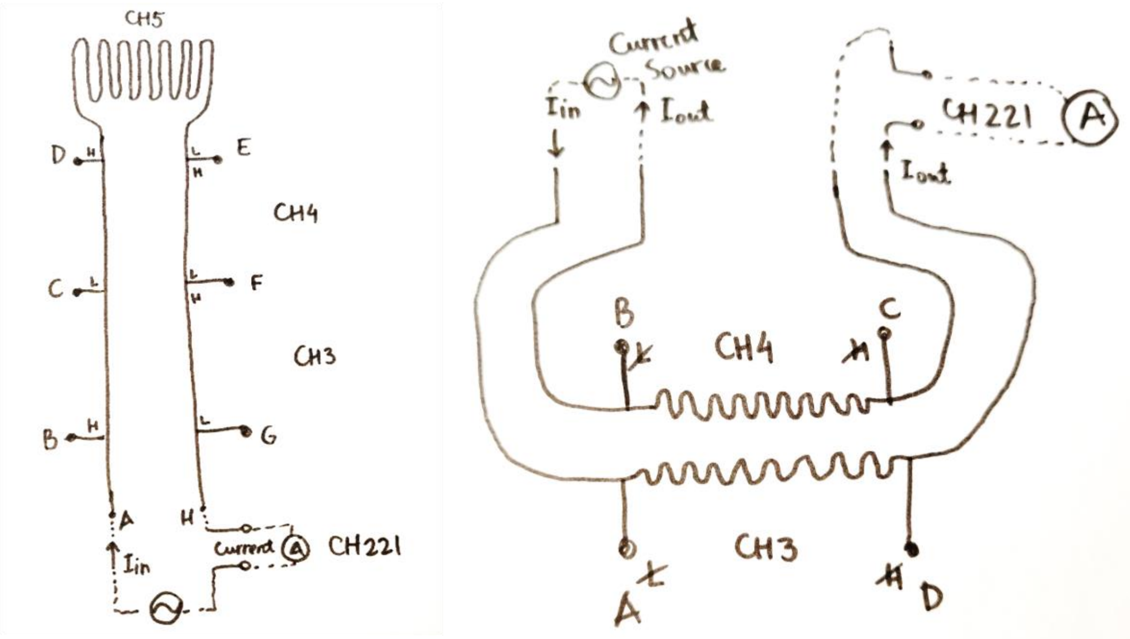


Figure (15) Left – The connections of the platinum strip for Chip SEM Swissroll; Right - The connections of the platinum strip for Chip SEM Corriolish

Calculations of the expected resistance for the silicon strip

Due to the uneven heat dissipation, the silicon heater will not have the same temperature along the whole length. In order to have an accurate measurement, the platinum sensor has been divided into sectors – there are connectors along the length and separate resistances can be measured. This method has it's own limitations, as there is not possible to measure the exact temperature at a certain point, but that will be covered by the theoretical aspects of the heat dissipation.

Ideally, the platinum sensor should be split into an infinitely small sectors, and each sector should be measured. In real life this is not possible so as a design choice, due to symmetry there have been decided to use a number between 5 to 7 sectors.

A few chips designs with the platinum sensors are presented in figure (16. In the chips blueprints, the platinum sectors are defined between to capital letters – for example A-B, B-C. The silicon connectors will have both a Voltage (VSi+, VSi-) and a Current connection (ISi+, ISi-).

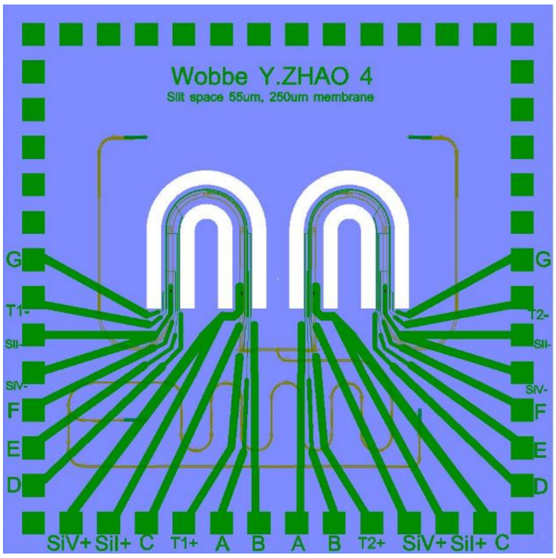
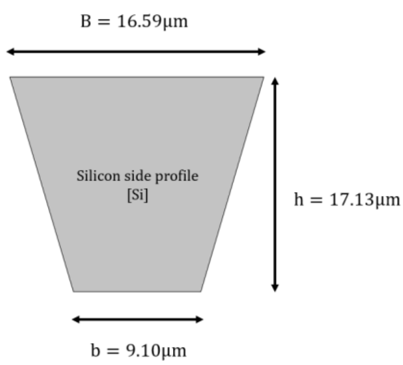


Figure (16) – The blueprint of a SEM U-shape microchip

Calculating the expected resistance of the silicon strip is a challenge, as at the moment in the resistivity formula there are 2 unknowns: the resistivity of the silicon and the cross sectional area of the strip. This issue can only let us make only estimations.

The cross sectional area of the silicon strip depends on the distance between the channels, the etching procedure and the chips design. The image of the cross sectional area is available in figure (14), where the cross area of an SMS and SEM is presented. The strip should be uniform along the whole length of the chip and ideally should have an area of approximately $220.034\mu m^2$.

The area was calculated by measuring the microscope images with a ruler. The side profile was estimated to be an isosceles trapezoid with the following dimensions:



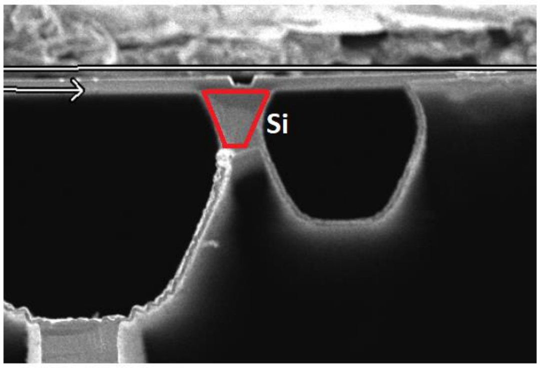


Figure (17) - The silicon side profile and the dimensions.

The calculation of the area of the trapezoid is:

$$\mathrm{A} = \frac{(\mathrm{B}+\mathrm{b}) \times \mathrm{h}}{2} = \frac{(16.59 + 9.10) \times 17.13}{2} = 220.034\mu\mathrm{m}^2$$

Due to the fact that in real life the silicon strip has imperfections along the length, some error margin has been calculated. 3 possible scenarios have been evaluated and the expected resistance for each case has been deduced. Similar to the MEMS paper [4] we have defined: $\mathrm{A_{small}} = 35\mu\mathrm{m}^2, \mathrm{A_{medium}} = 230\mu\mathrm{m}^2$ and $\mathrm{A_{high}} = 720\mu\mathrm{m}^2$. The resistivity of the silicon is another unknown in the equation. This value differs from paper to paper but for those calculations it was assumed to be between $\mathrm{R_{min}}$ when $\rho = 0.01 \times 10^4\ \Omega\ \mu\mathrm{m}$ and $\mathrm{R_{max}}$ when $\rho = 0.02 \times 10^4\ \Omega\ \mu\mathrm{m}$. [5]

The length of the silicon strip (approximated measurement using the blueprints) is calculated in the table below

Table (1) – The length of the silicon strip for 4 chips

The chip name	Length (μm)
Corriolish C9(SEM)	7500 μm
Swissroll C8(SEM)	28532 μm
U-shape Ce(SMS)	3144 μm
SMS swiss roll C2(SMS)	6142 μm

The resistance has been calculated using the formula:

$$R = \rho \frac{l}{A} \quad (2)$$

Table (2) – The resistance calculations for different cross sectional area of the silicon strip

	$\mathrm{R_{min}}$			$\mathrm{R_{max}}$		
	$\mathrm{A_{small}}$	$\mathrm{A_{medium}}$	$\mathrm{A_{high}}$	$\mathrm{A_{small}}$	$\mathrm{A_{medium}}$	$\mathrm{A_{high}}$
Corriolish(SEM)	21 428.5 Ω	3260.8 Ω	1041.6 Ω	44 285.71 Ω	6521.7 Ω	2083.3 Ω
Swissroll (SEM)	81520 Ω	12 405.2 Ω	3962.7 Ω	163 044 Ω	24 811.04 Ω	7925.75 Ω
U-shape (SMS)	8982.5Ω	1366.9 Ω	436.6Ω	17965.7 Ω	2733.9 Ω	873.3 Ω
SMS swiss roll (SMS)	17548 Ω	2670 Ω	853 Ω	35 099..1 Ω	5340.9 Ω	1706.1 Ω

Analysing this table with equation (2) it can be concluded that ideally the resistance of the silicon strip should lay in the grey boundaries from the table. Any value outside the boundaries mean a uneven shape of the silicon.

The width of the cross sectional area and implicitly the resistance measurement is crucial in the later stage of the research. The resistance directly influences the amount of power that the silicon strip can let trough, as the power is defined as:

$$P = \frac{V^2}{R}$$

The power formula proves that the higher the resistance the lower the maximum available power will be. Another important metric is the overall area of the silicon strip. A small area will result in a lower tolerance to heat and electric stress and the silicon strip may break down easily during joule heating.

The available voltage is limited by the function generator to a maximum value of 64V or 6A.

TCR of the platinum

The TCR (Temperature coefficient of resistance) is an important coefficient of measure for the thermal sensor, that can help us precisely estimate the temperature by knowing the resistance. The Platinum will be used as a thermistor. Using 2 of the 4 platinum strip connections, a current will be applied at the trough the sensor. A voltage meter will measure the voltage drop over the Pt sensor, and thus being able to deduct the resistance. The resistance changes with the temperature in a linear way, so the temperature can be approximated based on the measured resistance.

The formula of the TCR is defined by[6]:

$$\alpha = \frac{R - R_{ref}}{R_{ref}(T - T_{ref})}$$

Where $\alpha = \mathrm{TCR\ coefficient}$. The reference temperature (T_{ref}) will be around the value of 45ºC and the reference resistance (R_{ref}) will be the one corresponding to the 45ºC temperature.

The advantages of the materials used in the platinum sensor compared to other materials is that the TCR value of the alloy is constant up to temperatures up to 400ºC. As later measurements proved, the TCR of the sensor is not completely constant, but is rather linear with a very small slope. Other common materials as copper, aluminium or even silicon do not have a constant TCR at a higher temperatures. In order to prove the concept of this paper the platinum should be designed to resist temperatures up to 400ºC.

The platinum sensor as referred in the report so far is actually made of a mixture of materials: gold, platinum and tantalum (figure 18). The first layer is of tantalum (10nm), then on top a layer of platinum of 20nm and on top a layer of gold of 200nm. Although this composure, the sensor will be referred generically “platinum” throughout the report.

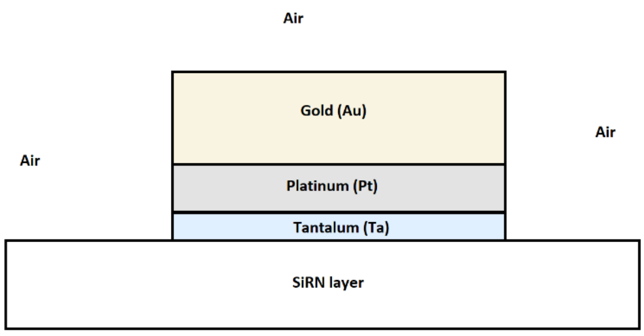


Figure (18) - The materials of the temperature sensor

TCR measurements procedure

The TCR can be calculated by measuring the resistance of the material at a different temperature than the reference temperature. In order to achieve a high accuracy, a set of 8 temperatures have been chosen and the TCR for each temperature was calculated (figure (19)). The start temperature and the reference in the measurements is approximately 45ºC and the other measurements will be done in steps of 5ºC up to 80ºC. The control of the temperature will be done using an oven. The choice of having the initial temperature of the measurements at 45ºC is due to the long temperature stabilisation time of the oven at low temperatures. In this way, choosing temperatures above 45ºC the experiment time decreases. The initial temperature was chosen to be 45ºC instead of the room temperature due to higher stability – the room temperature as recorded had variations between 20 and 24ºC and it was desired to have an uniform initial value for all measurements. The oven is controlled using LabVIEW.

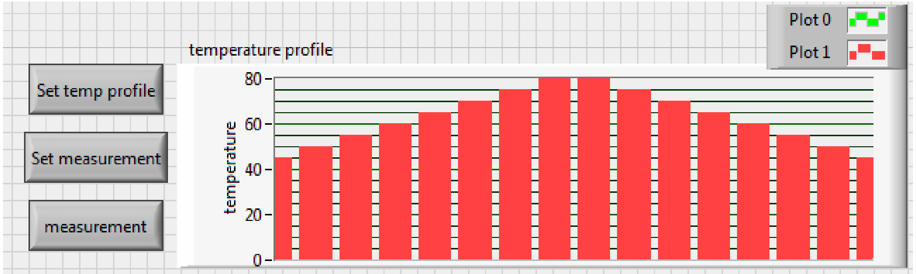


Figure (19) – The ramp up and down steps of the temperature, as it will be tested

The parameters of the multimeter measurements can be seen in the table 3 (stabilisation time, number of measurements and measurement time interval):

Table (3) – The parameters used in the heating with the oven

Number of measurements per temperature	300
Measurement time interval	1 sec
Max temp fluctuation	$\pm 0.4^{0}C$
Loop time stab settings	1 sec

The first step is when the oven is programmed to heat up to the first measurement temperature (45ºC). The temperature sensor inside records the temperature and sends a signal when the value is stable enough (it does not fluctuate more than ± 0.4ºC). The multimeter records the resistance 300 times, in intervals of a second.

Due to the accurate control of the temperature such a test lasts over 9 hours – figure (19) presents the temperature raise as measured in the oven. The time is measured in seconds.

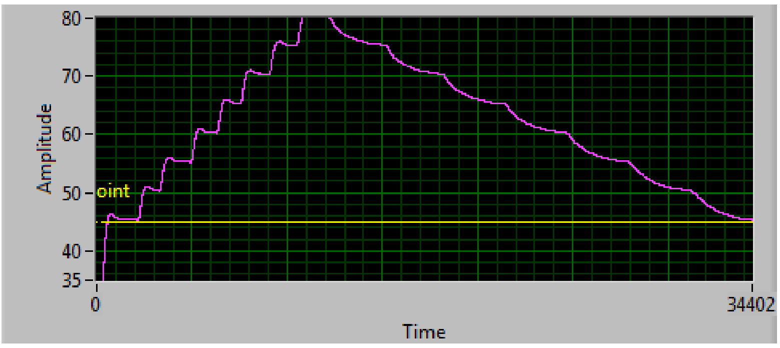


Figure (20) – The control of the temperature while the oven ramps up and down

For every applied temperature from the oven, the same current will be inputted along the Pt sensor. The value of the current is known (around 0.5 mA). The value of the current was chosen conveniently so that the power drop would not exceed 0.1W. Every platinum section will be connected to a voltage meter. Using LabVIEW, 300 values of the voltage meter will be recorder for each temperature step, and then the resistance will be calculated.

The TCR of the silicon

Unlike the platinum, the TCR of the silicon cannot be used as a constant in the temperature calculations, so the temperature of the heater may be harder to estimate. For this, 2 methods have been proposed:

- First, for any given temperature, the measured resistance of the platinum sensors will be converted into a temperature using the material's TCR formula. It is known that the TCR of the platinum is constant, so the temperature only depends on the resistance. Due to the fact that the Platinum is not touching the silicon, having a layer of SiRN in between, there are heat losses around the sensor. The true temperature of the silicon can be approximated by calculating the heat loss with the environment (presented in the subsection below).

- The temperature estimated above can be compared with another approximation: the TCR of the silicon. At any given moment we know the resistance of the silicon: the division of the Voltage applied over the generated current. The behaviour of the TCR of the silicon is not documented extensively (up to 200C) and cannot be accurately predicted at high temperature. By experimentation, the TCR of the silicon is rising linearly at low temperatures and behaves logarithmically at high temperatures. Thus, for low temperatures (up to 80C) the TCR values can be approximated by using a linear function approximation.

The formula for calculating the temperature of the silicon:

$$T = ax + b$$

Where a and b are the line coefficients, T is the temperature and x is the TCR of the silicon. Previously, the line was calculated by choosing 2 points of the silicon TCR and finding the slope a.

Heat dissipation along the length of the silicon strip

In the measurements above, the temperature of the platinum was assumed to be the same on every segment along the silicon heater. In reality, this is not the case, as the silicon heater dissipates heat unevenly and trough the surrounding materials. This difference of temperature can be calculated and added as a correction.

The silicon heater's temperature will be uneven along the length. This is due to the physical properties of the material and the heat dissipation of the strip – there will be higher dissipation on the edges and smaller dissipation in the middle – this translated into a lower temperature on the edges and higher in the middle.

The measurements will be proved with a theoretical calculation, using the paper of John van Baar [7].

The heat transfer from the silicon heater to the air in the x-direction

$$-\frac{1}{R'_b}\frac{\partial^2 T(y)}{\partial y^2} + G'_f T(y) = P'$$

The value of the temperature along the Pt sensor can be estimated accurately by replacing the position on the bar in the 2nd order DE.

The solution of the differential equation is:

$$T(y_n) = \frac{P'}{G'_f}\left(1 - \frac{\cosh\left(y_n \cdot l\sqrt{R'_b G'_f}\right)}{\cosh\left(\frac{1}{2}\, l\sqrt{R'_b G'_f}\right)}\right)$$

The most important variables in the 2nd order DE are: l – the length of the silicon strip, P' – the power dissipated along the bar, G_f' line conductance trough the gas (in our case the air) and R_b' - the thermal line resistance of the silicon.

Where $T(y_n)$ is the temperature at a defined position along the length of the bar. The solution function has been plotted on figure (21), the lines have the shape of a hyperbolic cosine.

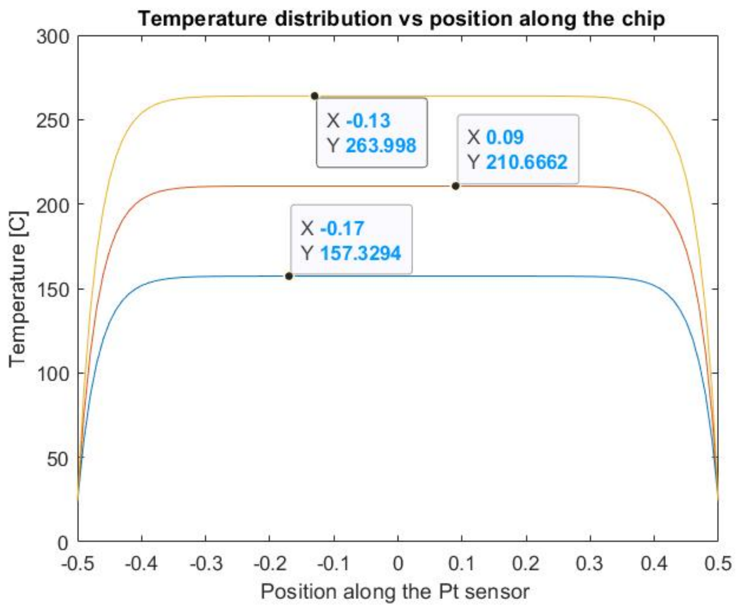


Figure (21) – The temperature distribution along the length of the silicon strip with the coefficients in the actual model

The figure above presents the temperature for different powers: 0.25W the blue line, 0.35W the red line and 0.45W the yellow line. The temperature calculated for a power of 0.35W is 260ºC. The x axis numbering indicates the position along the sensor -0.5 meaning the leftmost point and 0.5 the rightmost point.

Heat transfer calculations

The calculations of the difference of temperature between the platinum and the silicon strip combine multiple heat transfer concepts. The calculations are based on a model that has certain limitations (they are presented in the section below “Limitations”). The first step in the calculations was to analyse the silicon strip at the molecular level and understand what phenomenon creates the increase of temperature in the silicon strip.

When the material rests at the room temperature (24ºC), the molecules inside the material move freely as they are in thermal motion [8]. The speed and the direction of the moving molecules define the temperature of the material – for a higher molecular motion there is also a higher temperature and vice versa. Over this molecular motion an electric current is applied. The electrical current is defined by a flow of electrons inside the silicon strip from one side to the other. When a high electrical current is applied, the electrons collide with the molecules, creating more movement that translates into a higher thermal motion. In this way, the increase of the electrical current translates into an increase of temperature. The molecular level analysis also states that the energy from the electrical domain is transferred to the thermal domain.

In the process of the joule heating there are 2 types of heating that are happening: the conduction and the convection.
The conduction is defined as a transfer of energy from a more energetic to the less energic particles along the material without the transfer of any substance [9]. In addition, the thermal energy stored in the silicon also produces a convection effect. The convection is defined as the random molecular motion that moves the energy from one place to another [10]. Inside the silicon the diffusion effect occurs, the molecules of silicon are trying to spread uniformly throughout the material.

The proposed model presented in figure (22) uses the concepts defined above to make an estimation of the heat transferred between the silicon and the platinum.

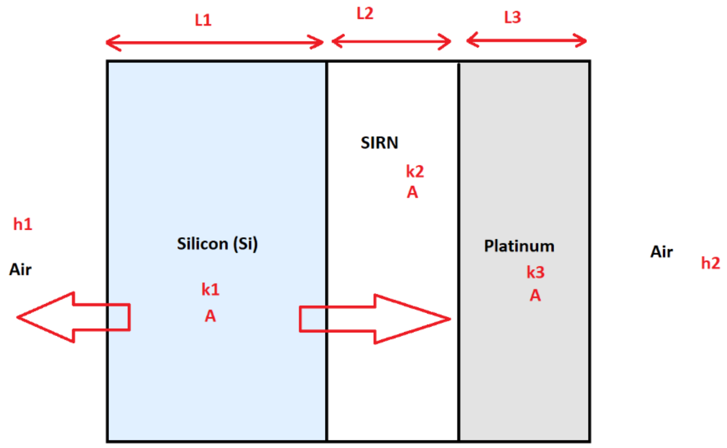


Figure (22) - The model of the parallel plate approximation with the 3 elements. The silicon heater is shown as the heat source

The formula that will integrated the phenomenon of conduction and convection is the Newton law of cooling [11], defined by:

$$\Delta T = \dot{Q} \times R_T$$

ΔT - the change of temperature between 2 points (in this case the change between Silicon and Platinum)
$\dot{Q}$ – the heat change per unit time
R_T – the thermal resistance
This law is an equivalent of the electrical domain, where the change of temperature ΔT is the correspondence of the voltage, the heat $\dot{Q}$ is the current and instead of the electrical resistance now it is thermal resistance.
In this particular case the heat change per unit time is can be seen as the total heat per unit time. The heat in principle is the energy so, it can be replaced by the generic energy. During the joule heating, the only energy applied to the circuit is the electrical energy, which is just the multiplication between the electrical power and the time the power was applied. Making this approximations, the formula for the heat transfer is equivalent to the electrical power applied to the silicon.

$$\dot{Q} = \frac{Q}{dt} = \frac{\text{Energy}}{dt} = \frac{P \times t}{dt}$$

$$\dot{Q} \cong P$$

So, it can be concluded that:

$$\Delta T = P \times R_T$$

The thermal resistance can be calculated by using the model presented above, using parallel plated approximation (Introduction to engineering part 3 transfer paper assignment figure 2.7). The silicon heater is surrounded by materials on all sides. The calculations below will show the difference of temperature calculated only in the vertical direction (from silicon to the platinum). Furthermore, the calculation of the temperature difference is possible in the assumption of convection inside the materials, so that the temperature at the surface of every material is constant.
The thermal resistance of a material is defined by:

$$R_{Th} = \frac{L}{k \times A}$$

Where L is the length of the material in the heat transfer direction, A is the area of the cross section surface that makes contact with the other materials in the direction of the heat transfer and k is the thermal conductivity. The value for the thermal conductivity for the materials are taken from Fundamentals of Heat and Mass Transfer book. [12]

Table (4) – The thermal conductivity of the 3 materials used in the model

Material	Value $k[W/mK]$
Silicon	130
SiRN	25
Platinum	77.8

The cross sectional area is assumed to be the same in all situations – all 3 materials are present along the length of the chip and they have approximately the same width. The 3 materials have a series configuration, so the overall resistance is just the summation of the 3 individual resistances.
The lengths of the materials were calculated using the chips cross-section images in figure (14) and they have the values:
Table (4) – The length of the 3 materials used in the model

Material	Length (μm)
Silicon	$L_1 = 17.13$
SiRN	$L_2 = 2$
Platinum (thin layer)	$L_3 = 0.2$

The last parameter that influences the calculations is the h parameter (free convection with air). On the top part of the chip, the platinum is connected free to the outside, the value was approximated to be 10. On the left had side, the air that communicates with the silicon is trapped between the bulk and the silicon strip so the convection ratio will be approximated to 2. The values were used using the tables in the book [13].

$$R_T = R_{Si} + R_{SiRN} + R_{Pt}$$

$$R_T = \frac{L_1}{k_1 \times A} + \frac{L_2}{k_2 \times A} + \frac{L_3}{k_3 \times A}$$

$$\Delta T = P \times \left(\frac{L_1}{k_1 \times A} + \frac{L_2}{k_2 \times A} + \frac{L_3}{k_3 \times A} + \frac{1}{h_1} + \frac{1}{h_2} \right)$$

The function was plotted using MATLAB and is presented in figure ():

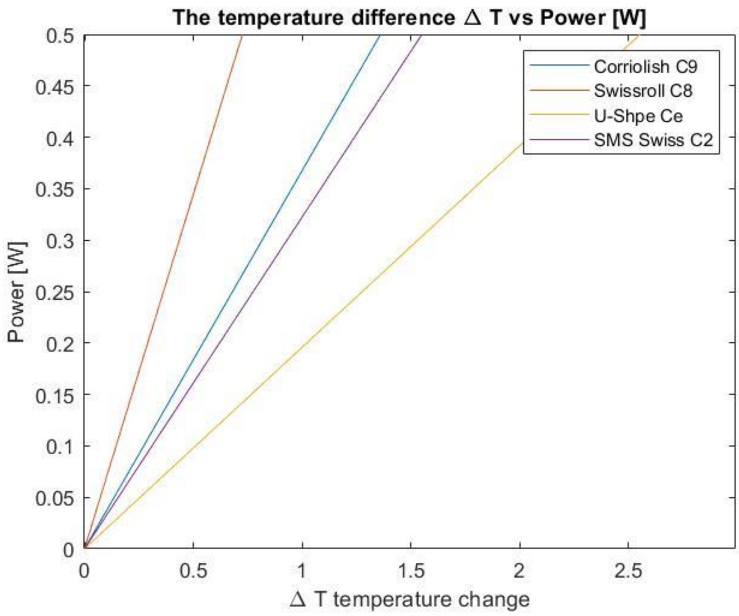


Figure (23) - The vertical heat transfer between the silicon and the platinum
Due to the very thin layer of SiRN and platinum compared to the silicon, in the vertical direction the heat transfer is almost complete. The graph above shows that the temperature of the platinum deviates with a maximum of 0.5ºC at a power of 0.35W. This result is expected due to the fact that the SiRN material has a calculated thermal resistance of only 0.643 K/W.

The temperature difference is not close to reality as the SiRN material also dissipates heat along the walls of the channels. The heat transfer with the sides will not be discussed in this report.

Limitations

The theoretical calculations are based on a model, thus they cannot describe in detail the real life behaviour. The parallel plate approach used above only considers the heat transfer in the vertical direction, from the silicon heater to the platinum sensor. There is also a substantial heat transfer in the horizontal direction, but those were not discussed in the paper. By evaluating on the scope of this calculation and the assumptions made – the temperature difference between the layer of the silicon and the layer of the platinum, the limitation is not considered to have a considerable impact. If the target would have been to calculate the temperature that the silicon would have reached at a certain temperature, then the heat transfer with the sides would have been crucial.

The calculations do not take into account the shape of the chips – each design was created to achieve a different performance – the swiss roll should have smaller lateral heat losses that a straight line SMS chip. In the calculations, the shape of the silicon heater was not taken into account, as the length "L" was considered to be a straight line.

Results and measurements

Resistance measurements

The resistance of the silicon strip has been measured using the probe for each chip. In the rightmost column there is a short observation about how the value compares to the expected value.

Table (5) – The measured resistance of the silicon strip compared to the ideal value calculated on the theory

	Measured resistance (4 point multimeter)	Verdict
Corriolish(SEM)	$7032\ \Omega$	*The value is slightly higher – that means the area of the strip is smaller.* ***Higher*** *change of breakdown at high powers*
Swissroll (SEM)	$15\,870\ \Omega$	*The value is under* ***normal*** *parameters. Expected to have* ***good*** *performance*
U-shape (SMS) – left channel	$7124\ \Omega$	*The value is very high – that means the area of the strip is small.* ***Very vulnerable*** *at high powers*
SMS swiss roll (SMS) - left channel	$1895\ \Omega$	*The value is under* ***normal*** *parameters. The silicon strip may have a thicker are than average. Expected to have* ***good*** *performance*

As a general observation, the verdicts presented above are not evaluating the possibility that the silicon strip is uneven along the length. The silicon bar could have variations in the cross sectional area. This translates to a different resistance when compared to an ideal straight line.

TCR of the Platinum

The TCR of the platinum (α) will be measured using the formula below:

$$\alpha = \frac{R - R_{ref}}{R_{ref}(T - T_{ref})} \qquad (4)$$

$$R = R_{ref}\left(1 + \alpha(T - T_{ref})\right)$$

$$R = R_{ref} + \alpha(T - T_{ref})R_{ref}$$

Out of the 14 chips given initially for measurements, only 7 have had their TCR calculated. The status for every chip is in the appendix.

The method for calculating the TCR of a chip is by averaging the 300 values recorded for every temperature step and then applying formula (4) to find α. In theory, the TCR of the platinum should be a constant, so after the calculation of the TCR for each step (7 values), the overall values will be averaged.

Below there are the tables with the value of the TCR for each chip. Because there are multiple Platinum sections, those are the values for the platinum segments that are central in the chip (for example for circuit C8 the Pt section is the one connected to CH4 and for chip C9 the Pt channel is CH5).

Table (6) – The TCR calculated for every chip

Temperature	TCR C8 Corriolish	TCR Ce U-Shape	TCR C9 Swiss roll	TCR C2 SMS Swiss roll
50.39	0.0012885	0.0012435	0.001175	0.001216382
55.40	0.001187152	0.0011963	0.001184	0.001188973
60.44	0.001222316	0.0011884	0.001191	0.001229826
65.40	0.001190742	0.0011849	0.001191	0.001235372
70.40	0.001213126	0.0011877	0.001191	0.001210311
75.34	0.001194036	0.0011845	0.001193	0.001223116
80.40	0.001207576	0.0011846	0.001194	0.001237178
Average TCR	0.001214778	0.0011957	0.001188	0.00121733

After observing variations from the general average TCR and the individual values it was decided to make a plot for each chip with the TCR value vs the temperature. The plots concluded against the theory because the TCR of the platinum strips is a linear function with a small slope. The plots for each individual chip can be seen below. Attached to the points, the trendline function of Microsoft Excel was used, for a period of extra 100 points. Furthermore, the function of the line of the TCR was also calculated.

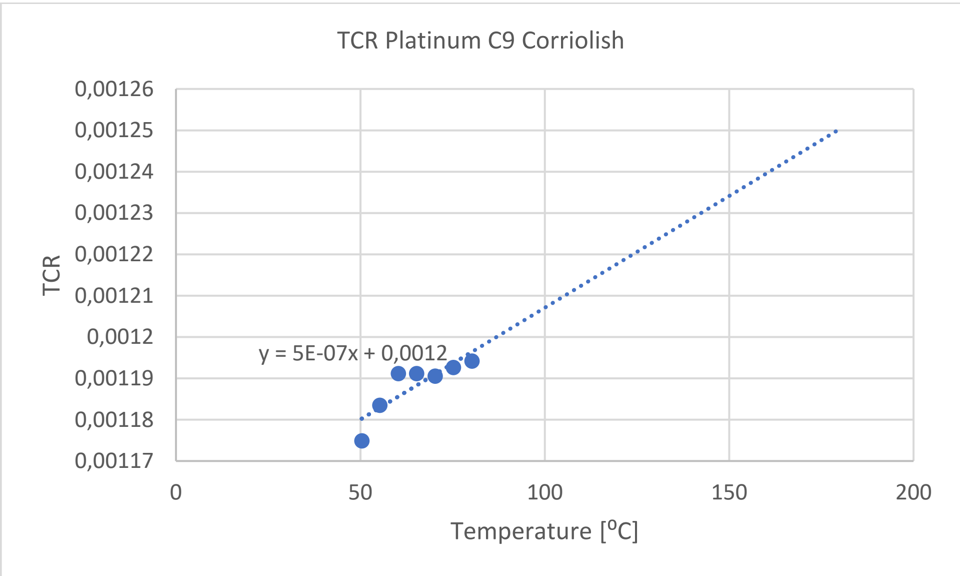


Figure (24) The TCR of the Platinum for a segment of the C9 Corriolish Chip. The line shows a linear fit line to the points, so overall there is a slight increase of the TCR. The value is small though and is kept in the bounds.

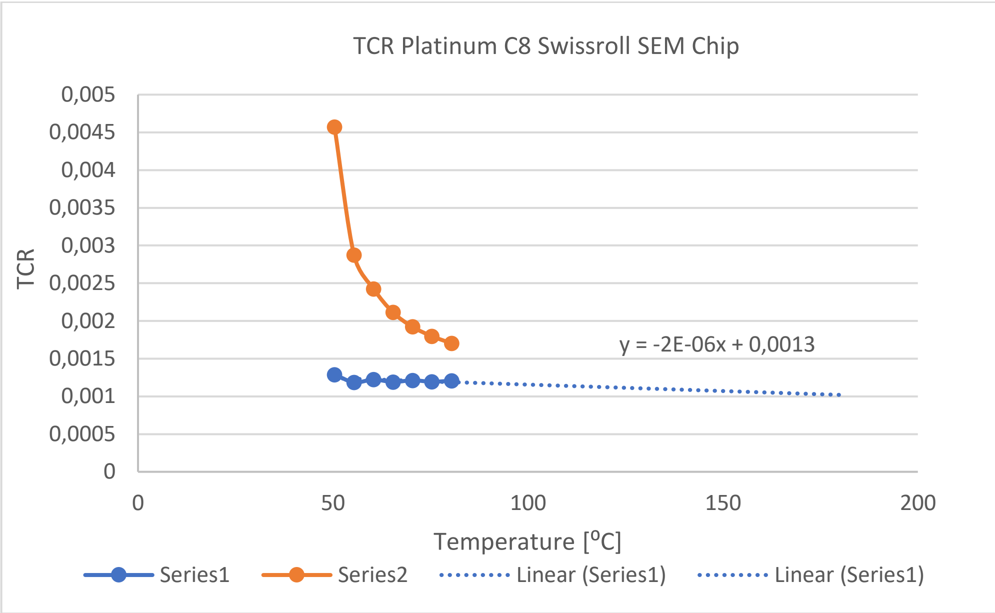


Figure (25) – The TCR of the Platinum for a segment of the C8 Swiss roll Chip. The line shows a linear fit line to the points, so overall there is a slight increase of the TCR. The value is small though and is kept in the bounds.

The orange line represent the results of a segment of the sensor situated on the edge – it was connected to the multimeter to CH203. The exponential behaviour could be the cause of a bad connection.

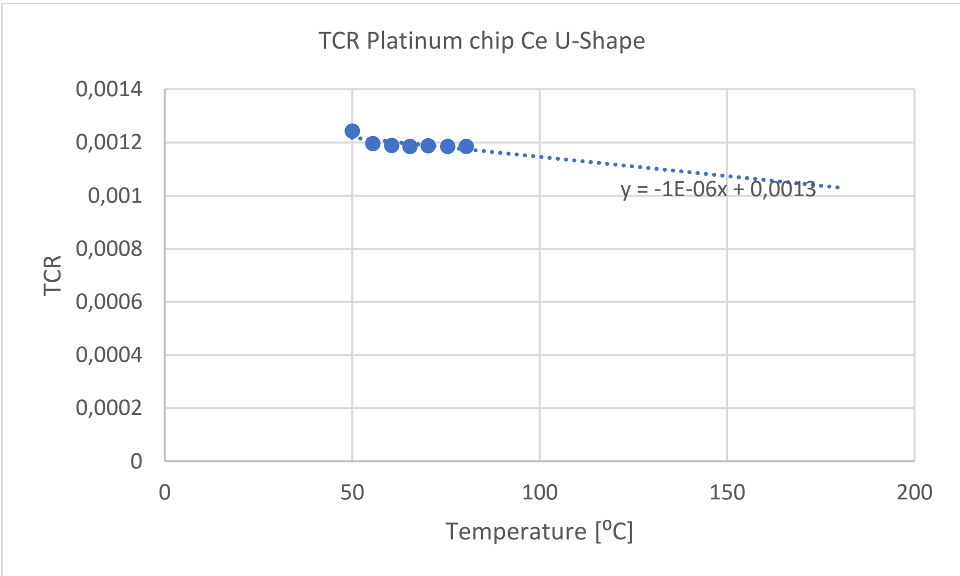


Figure (26) – The TCR of the platinum sensor for the chip Ce Ushape. The platinum segment used is the centre one

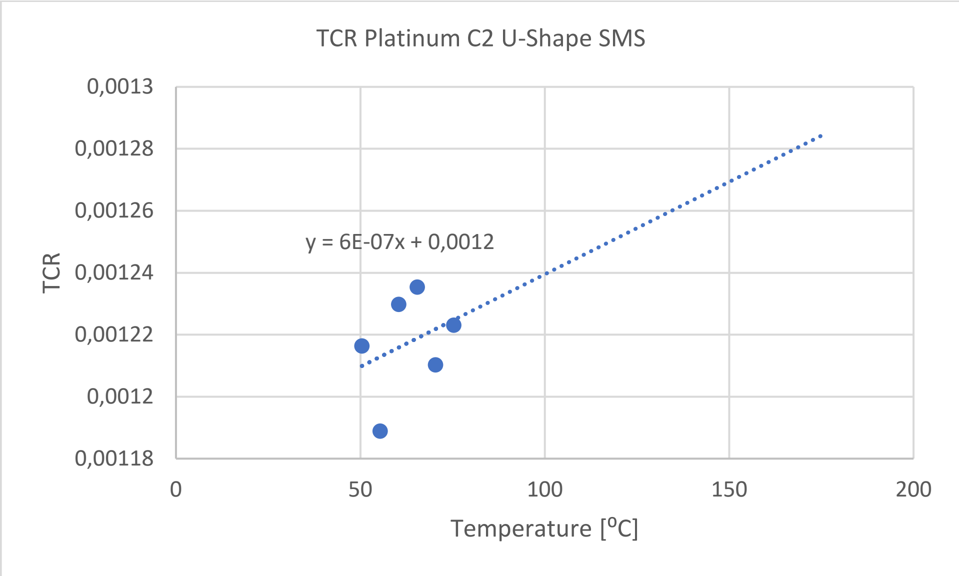


Figure (27) - TCR of the chip C2 U-shape

Another important mention is that the TCR of the platinum is not constant over a large temperature. The TCR value can be approximated roughly to a value of 0.0012 but the values is to increasing with respect to temperature.

Another observation is that the TCR value of the platinum, gold and tantalum varies a lot in multiple sources. Below the default values for the TCR of the materials are presented:

Table (7): The TCR values for the 3 materials that compose the sensor

Physical properties of the sensor alloy	
Ideal TCR (theoretical value) []	$0.0039\ K^{-1}$
Ideal TCR Gold []	$0.0034\ K^{-1}$
Ideal TCR Tantalum []	$0.0033\ K^{-1}$
2018 MEMS paper thin film platinum TCR []	$0.0024\ \mathrm{K}^{-1}$

Table (8): Actual measurements - average values

Chip	Average TCR of the platinum – calculated (table 6)
C2 – SMS	0.0012303
C8 – Swiss roll	0.0012147
C9 – Corriolish	0.0011900
Ce – U-Shape	0.0011957

The deviations between the MEMS paper and the calculation presented in this paper can be justified by thin film effects such as grain boundaries, defects and displacement on the electrical transport in thin films. One of the reason to those deviations could be in the structure of the platinum sensor, as the physical structure is made of multiple materials that can contribute differently to the overall TCR.

TCR of the silicon

The TCR of the silicon will be calculated with the same method as the platinum. The measurements fit the expectations, the TCR is rising with the temperature. In order to predict the TCR at higher temperatures, the same excel linear approximation was used (the trendline).

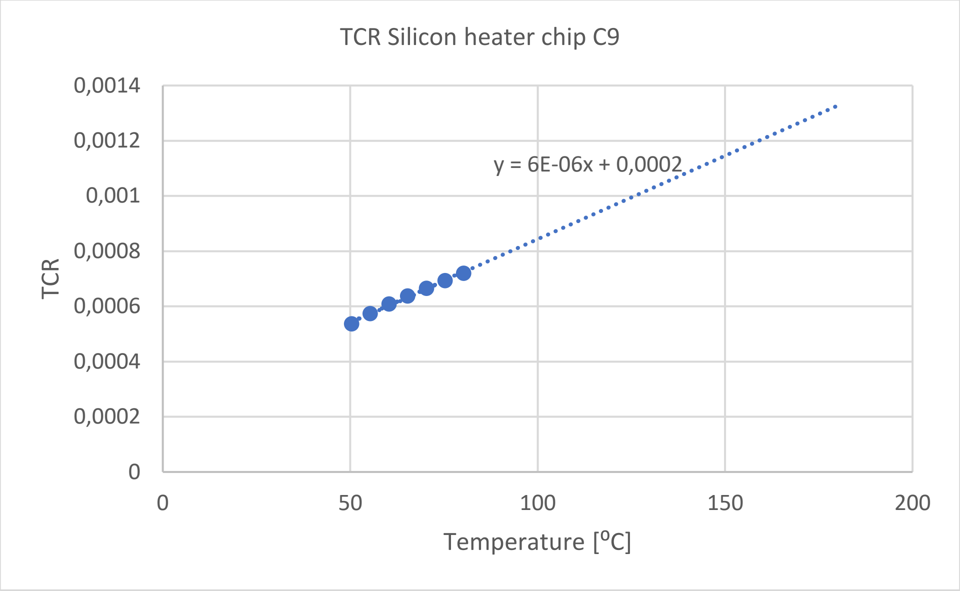


Figure (28) – TCR line of the chip C9 Corriolosh

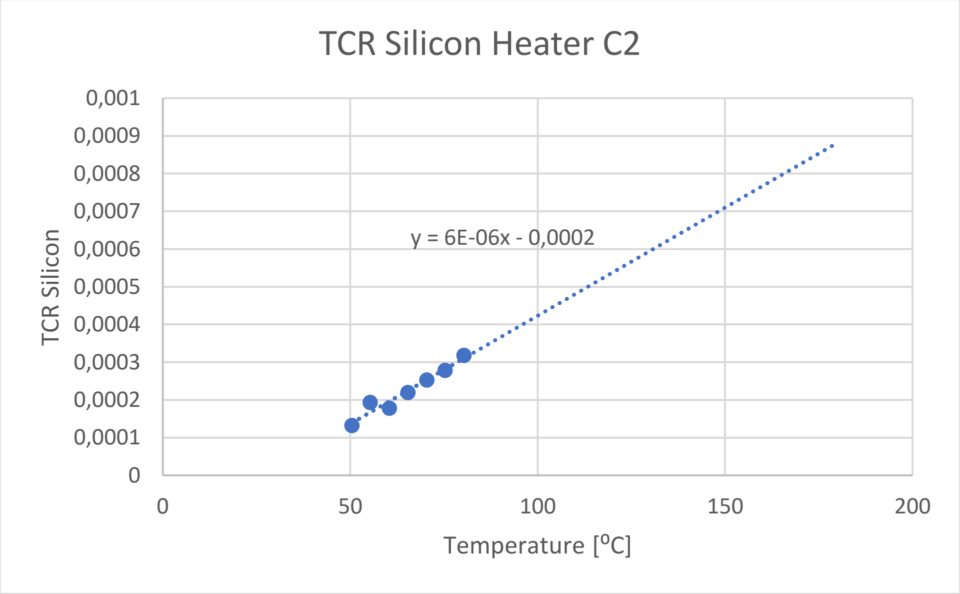


Figure (29) - TCR line of the chip C2 SMS Swiss roll

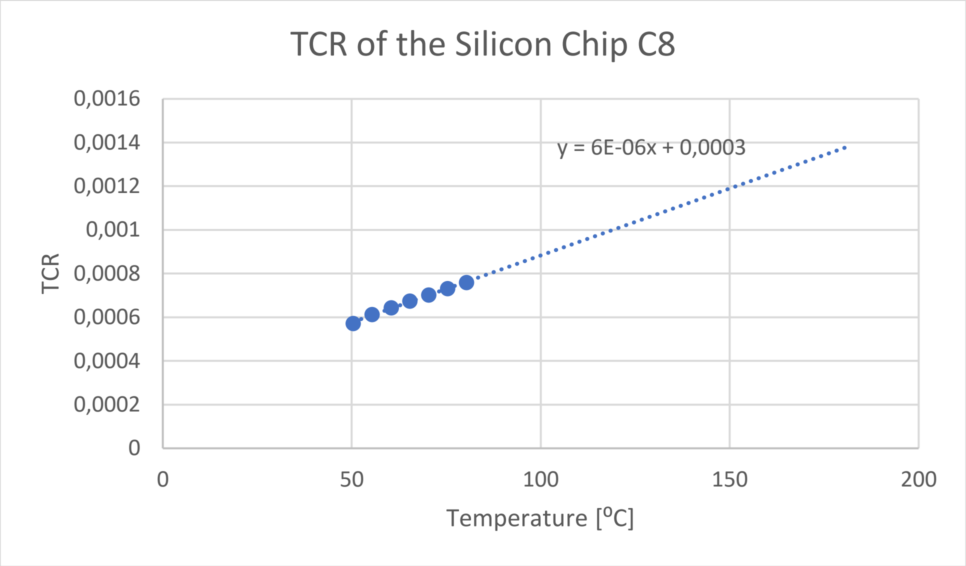


Figure (30) - TCR of the silicon strip Chip C8 Swiss roll

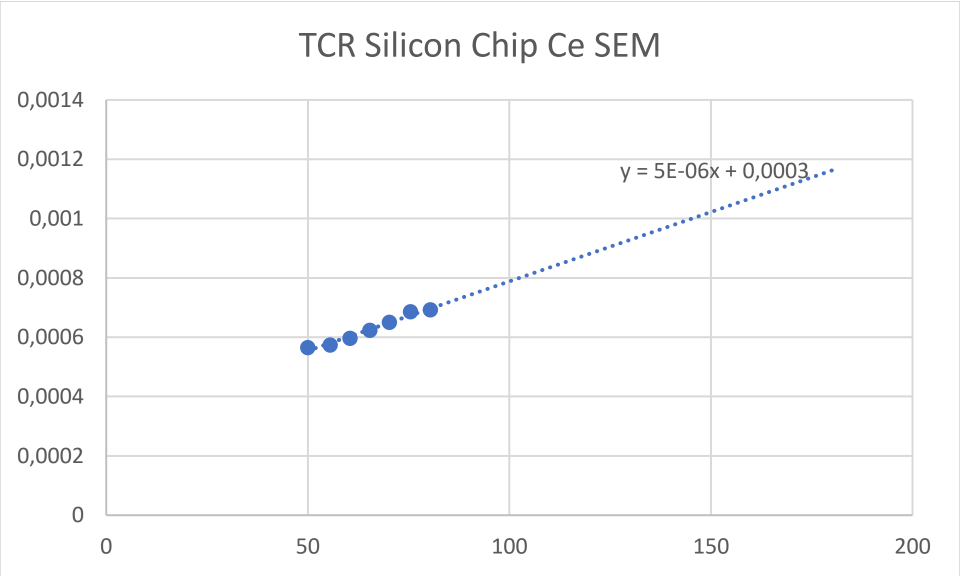


Figure (31) - TCR of the silicon strip Chip Ce SEM

Table (9) – The TCR of the silicon for each temperature for each of the 4 chips

Temperature	TCR C8 Corriolish	TCR Ce	TCR C9	TCR C2
50.39	0.000572	0.000564	0.000537	0.000132
55.40	0.000613	0.000573	0.000574	0.000193
60.44	0.000644	0.000596	0.000608	0.000178
65.40	0.000674	0.000623	0.000637	0.000219
70.40	0.000703	0.000649	0.000665	0.000252
75.34	0.000732	0.000685	0.000693	0.000278
80.40	0.000759	0.000692	0.00072	0.000318

As a general observation, the TCR of the silicon is similar for all chips with the exception of the chip C2 (highlighted in grey). One possible explanation could be in the very small resistance (512 Ω) that signifies a possible uneven shape of the silicon. The small resistance could mean a ununiform distribution of the material resulting in connection between the heater and the layer or a very thin section.

Furthermore, the trendline formula specific to each chip will be used later in the estimation of the temperature based on the resistance.

Joule heating

The last step in the proof of design is to use the joule heating method to heat up the silicon heater and prove the capability to withstand high temperatures (over 200C). Also the capacity to reach a high temperature with the least amount of power will be evaluated.

The heat produced is directly proportional with the power applied trough the material. The experiment is limited by the power source – the maximum voltage that can be applied is 64V and 6A. In real life only one metric can be used: either voltage or current, but not both – the other metric will be generated depending the internal resistance of the silicon strip. So, in order to achieve a high temperature, the resistance of the silicon strip should be as small as possible.

$$P = \frac{V^2}{R}$$

Due to the possibility of a silicon strip to break down, it was decided to increase the voltage. This ensures a slow increase of power (the power increases exponentially by a factor of ½ while the voltage):

$$V = \sqrt{P \times R}$$

During the experiment LabVIEW was used to record the progress. The data presented in the plots of LabVIEW is slightly inaccurate, due to the fact that the formulas of for the graphs of the plots are not accurate. The most important readings are the ones of the resistances for the platinum sensor and the silicon saved in the text document.

The last step before joule heating, a high level design of the methods to calculate the temperature of the silicon are made:

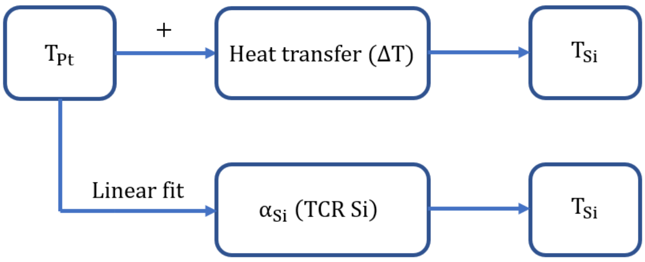


Figure (32) - The high level design of the 2 methods used in the calculation of the silicon temperature

The formulas used for the calculations of the parameters, also used in tables 10 and 11 are presented are presented in the table below:

Table (9.1): The formulas used to calculate α_{Si} and $T_{Si,\alpha}$ in tables 10 and 11

Chip C8 formulas in table (10)	
α_{Si}	$\alpha_{Si} = 4.930681722 * 10^{-7} \times R_{Si} - 0.007466$
$T_{Si,\alpha}$	$T_{Si} = \frac{R - R_0}{\alpha_{Si} R_0} + T_0$
Chip C9 formulas in the table (11)	
α_{Si}	$\alpha_{Si} = 6.12 * 10^{-6} \times T_{Pt} + 0.0002288$
$T_{Si,\alpha}$	$T_{Si} = \frac{R - R_0}{\alpha_{Si} R_0} + T_0$

For the linear approximation method 2 pairs of values are taken, and the line that they create is calculated. For example for circuit C9 – resistance and temperature: (15983, 45.339) and (16408, 80.406)

Table 10: Joule heating of chip C9 Corriolish

Voltage [V]	Power dissipated [W]	SI resistance [Ω]	Pt ch4 resistance [Ω]	α_{Si} calculated based on T_{Pt}	α_{Pt}	$\mathbf{T_{Pt}[^0C]}$	ΔT correction $[^0C]$	$\mathbf{T_{Si} = T_{Pt} + \Delta T}$ $[^0\mathbf{C}]$	$T_{Si,\alpha}$ – based on α_{Si} $[^0\mathbf{C}]$	Temp difference
2.09	0.0006	7032.12	169.87	0.000333	0.001172	**17.08**	0.00	**17.08**	20.86	3.79
6.00	0.0051	7044.75	171.09	0.000370	0.001174	**23.00**	0.01	**23.01**	28.56	5.57
16.00	0.0354	7232.01	178.85	0.000598	0.001187	**60.30**	0.05	**60.35**	80.99	20.69
20.99	0.0591	7459.92	184.50	0.000761	0.001197	**86.92**	0.09	**87.00**	116.62	29.70
21.00	0.0591	7460.78	184.53	0.000762	0.001197	**87.07**	0.09	**87.16**	116.69	29.62
30.99	0.1176	8168.75	197.37	0.001122	0.001219	**146.02**	0.17	**146.19**	184.18	38.16
35.99	0.1502	8622.78	204.04	0.001305	0.001230	**175.83**	0.22	**176.05**	214.47	38.64
40.99	0.1840	9131.47	210.68	0.001483	0.001241	**204.95**	0.27	**205.22**	243.04	38.09
45.99	0.2180	9703.44	216.85	0.001646	0.001252	**231.51**	0.32	**231.83**	272.95	41.44
50.59	0.2484	10302.31	221.54	0.001768	0.001260	**251.46**	0.36	**251.82**	305.27	53.81
55.99	0.2876	10898.22	228.57	0.001948	0.001272	**280.84**	0.42	**281.26**	324.83	43.99
60.99	0.3247	11455.21	234.73	0.002102	0.001282	**306.11**	0.47	**306.58**	341.94	35.84
63.98	0.3481	11759.50	238.55	0.002197	0.001289	**321.59**	0.51	**322.10**	348.87	27.27
63.98	0.3483	11753.16	239.34	0.002216	0.001290	**324.79**	0.51	**325.29**	345.83	21.05

Table 11: Joule heating of the chip C8 Swiss roll

Voltage [V]	Power dissipated [W]	SI resistance [Ω]	Pt1 resistance (ch3)[Ω]	α_{Si} calculated based on R_{Si}	α_{Pt}	$\mathbf{T_{Pt}[^0C]}$	ΔT correction $[^0\mathbf{C}]$	$\mathbf{T_{Si} = T_{Pt} + \Delta T}$ $[^0\mathbf{C}]$	$T_{Si,\alpha}$ – based on α_{Si} $[^0\mathbf{C}]$	Temp difference
2.00	0.0002	15901.83	179.42	0.000375	0.001207	**23.54**	0.00	**23.54**	27.56	4.02
7.00	0.0030	15930.58	179.77	0.000389	0.001207	**25.12**	0.01	**25.13**	32.82	7.70
11.00	0.0076	15956.69	180.41	0.000402	0.001207	**28.05**	0.02	**28.07**	37.28	9.23
12.00	0.0090	15963.70	180.47	0.000405	0.001207	**28.33**	0.02	**28.35**	38.43	10.10
17.00	0.0180	16019.15	181.57	0.000433	0.001208	**33.33**	0.05	**33.38**	46.86	13.54
22.00	0.0301	16098.35	183.01	0.000472	0.001208	**39.90**	0.08	**39.99**	57.22	17.31
27.00	0.0448	16252.01	184.73	0.000547	0.001209	**47.75**	0.12	**47.87**	73.09	25.34
27.00	0.0449	16239.95	184.69	0.000541	0.001209	**47.57**	0.12	**47.69**	72.01	24.44
31.99	0.0624	16415.09	186.61	0.000628	0.001210	**56.33**	0.17	**56.49**	85.75	29.42
36.99	0.0822	16655.28	188.67	0.000746	0.001211	**65.75**	0.22	**65.98**	99.42	33.67
41.89	0.1007	16855.33	190.46	0.000845	0.001212	**73.92**	0.27	**74.19**	107.88	33.97
46.99	0.1278	17285.15	193.26	0.001057	0.001213	**86.65**	0.35	**87.00**	120.72	34.07
51.99	0.1529	17682.15	195.56	0.001253	0.001214	**97.18**	0.42	**97.59**	128.72	31.54
56.99	0.1794	18106.40	197.94	0.001462	0.001216	**108.03**	0.49	**108.51**	134.90	26.87
61.99	0.2069	18575.64	200.18	0.001693	0.001217	**118.23**	0.56	**118.79**	139.95	21.73

Table 10 - Data presentation

Above, the recorded data for the first Joule heating experiment is presented (the only raw data presented are the Voltage, Si Resistance and Pt resistance). In addition, here have been calculated the Power dissipated trough the silicon strip as well the TCR coefficients needed for the temperature calculation of each material. The table calculates the temperature of the silicon using two methods: using the TCR of the silicon and using the temperature of the platinum. It is assumed that the second method gives a more accurate result, and the huge difference will be explained in the error analysis.

The Silicon strip reached a maximum temperature of 325ºC and tolerated a resistance increase of over 67.3%, at a dissipated power of 0.348W. The chip reached the best performance out of the 4 joule heated chips, confirming the expectations done in the resistance calculations section.

The correct value of the temperature of the silicon is considered to be the orange column, as the accuracy and method of calculation depends on less variables. The value of the temperature of the silicon is strictly depended on alfa: the TCR coefficient, which is erroneous for the first method.

The platinum strip reached a temperature of 324ºC. The TCR of the platinum was calculated using a linear approximation, not a constant value (at the maximum temperature the TCR is 0.00129006).

Table 11 - Data presentation

The temperature of the silicon reached a value of 118.79ºC, at a dissipated power of 0.2069W and tolerated a resistance change of 16.8%. Unlike the previous chip, the difference between the temperature of the silicon based on the temperature of the platinum and the one based on resistance is lower. This proves the observation that the first method of calculation is accurate only at a small temperature. In terms of performance, the high resistance of the silicon (15901.83 Ω at room temperature) was not able to produce too much power – only 0.2069W at 62V. Furthermore, the long silicon strip was proved to be not effective, managing to heat up only to 118.79ºC. The experience with the previous chip, that tolerated a much higher temperature and is assumed to have a thinner cross section suggests that this chip could go under further experiments, by applying even more voltage and rising the temperature even higher.

The platinum reached a temperature of 118ºC. The TCR of the platinum was also calculated by a linear approximation function based on the change of resistance.

The results for chips Ce and C2 are presented separately due the unusual behaviour or the silicon strips and the remarkable events that followed the experiment.

Table 12: The chip C2 Joule heating

Power Dissipation	Voltage [V]	Si resistance	CH203 Pt R	Ch205 Pt R
0.000	0.00	280.54	68.38	155.20
0.006	1.30	285.67	68.40	155.24
0.073	4.68	298.81	68.55	155.57
0.221	7.89	282.08	68.85	156.33
0.126	8.17	528.29	68.83	156.13
0.000	8.40	-130260206.20	68.62	155.71
0.000	8.40	130259865.12	68.56	155.59
0.000	7.50	-34859276.49	68.49	155.43
0.000	7.50	-43589287.04	68.46	155.37

In the table above, the results of the joule heating of the chip C2 – U-shape are presented. The first observation is at the resistance of the silicon strip itself, that is very different than the one measured at the TCR calculation – instead of 528,758 Ω at room temperature now the value is around 280 Ω.

The experiment was done is same parameters as the above 2, but due to unforeseen circumstances, the platinum resistance did not change along the time of the experiment. The values presented above correspond to the values of the strips at room temperature. The possible causes could be in the bad functioning of the chip itself – the silicon heaters did not heat up the platinum as designed or a malfunction of the multimeter.

Breakdown of the chip C2

It was expected that the silicon heater may have an uneven shape, which translate into weak points, places where the material is very thin and cannot tolerate big power dissipation.

While doing joule heating over the C2 SMS Swissroll chip, at just a power of 0.2W, the silicon heater stopped drawing current (column 6 table 12). This translates in a breakdown in the material shape and a disconnection in the circuit. In theory, this breakdown could be seen using the microscope, as the silicon strip is openly visible. Unfortunately, after investigating the chip after the breakdown no evidence was found that the silicon strip could have any discontinuities (figure 33, 33.1 and 34).

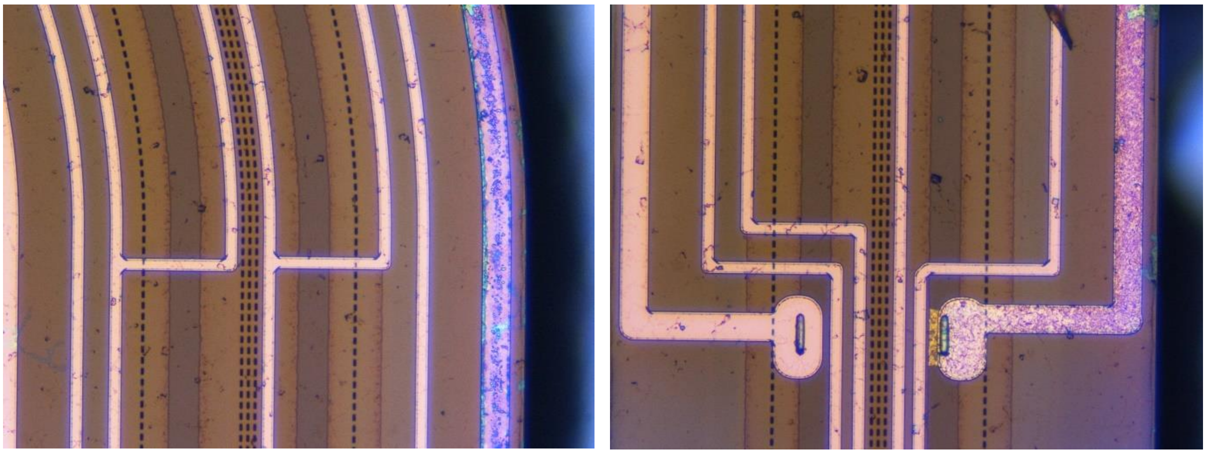


Figure (33) – Samples of the chip C2

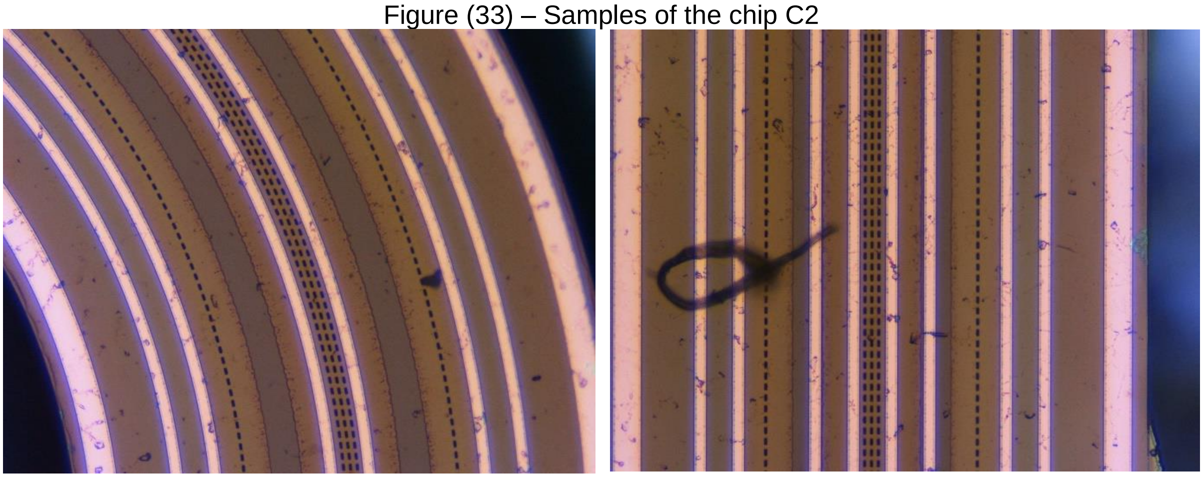


Figure (33.1) - The silicon strip of the chip C2 – The dark brown material is continuous along the way and has no sign of damage

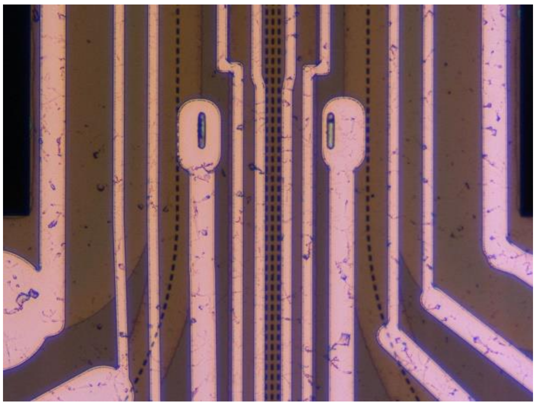


Figure (34) - The connection of the begging of the chip C2

The relation between the power and temperature of the materials is expected to be exponential. Figure (35 and 36) plot the temperature of the silicon vs the power dissipation during the joule heating process, confirming the expectations. At high powers, the silicon strip heats up slower and caps at the maximum possible value. If the power will increase even further the possibility of the silicon strip to break down increases.

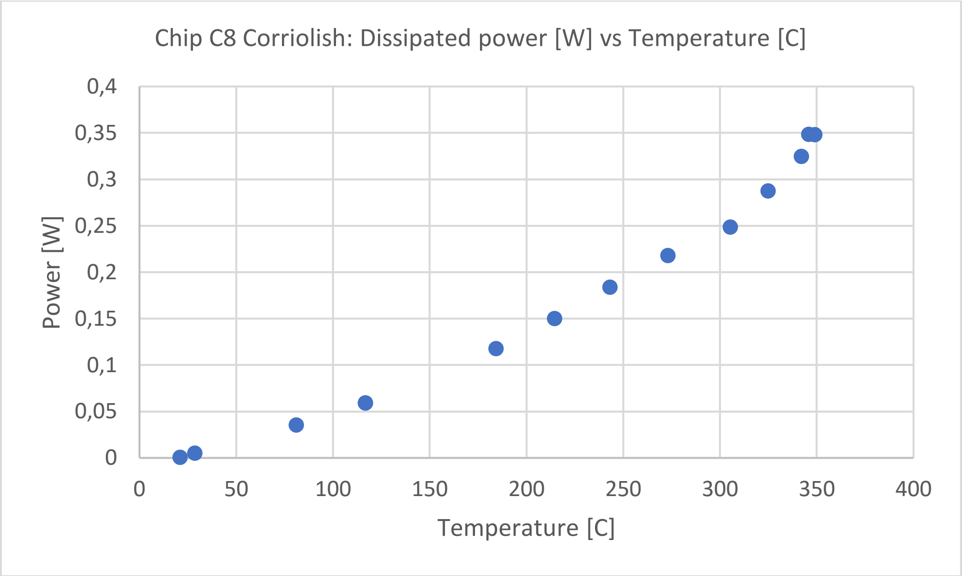


Figure (35) – The plot of the temperature of the silicon vs the increase of power for chip C8 – it can be observed the exponential behaviour

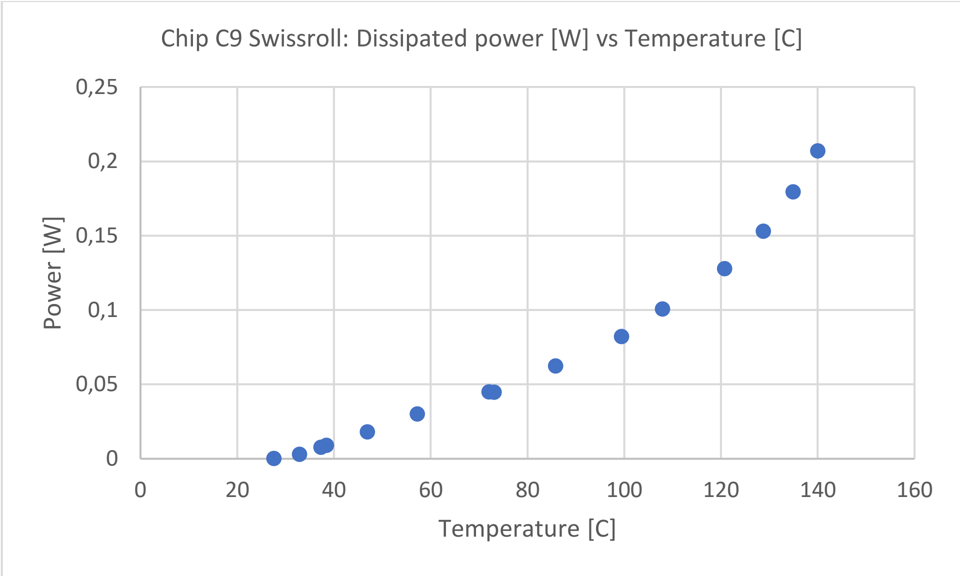


Figure (36) – The plot of the temperature of the silicon vs the increase of power for chip C9

The temperature change between the platinum and the silicon was not similar in the 2 methods used. For the second method (the calculation based on T_{Pt}) for Corriolish chip, the difference reached 34ºC at a power of 0.1W and for the chip C9 it reached a difference of 53ºC at 0.25W (figure 37). The decreasing difference of temperature at high powers proves the plots above (figure (35) and (36)), the temperature gets saturated at high powers, so the materials tend to have the same temperature.

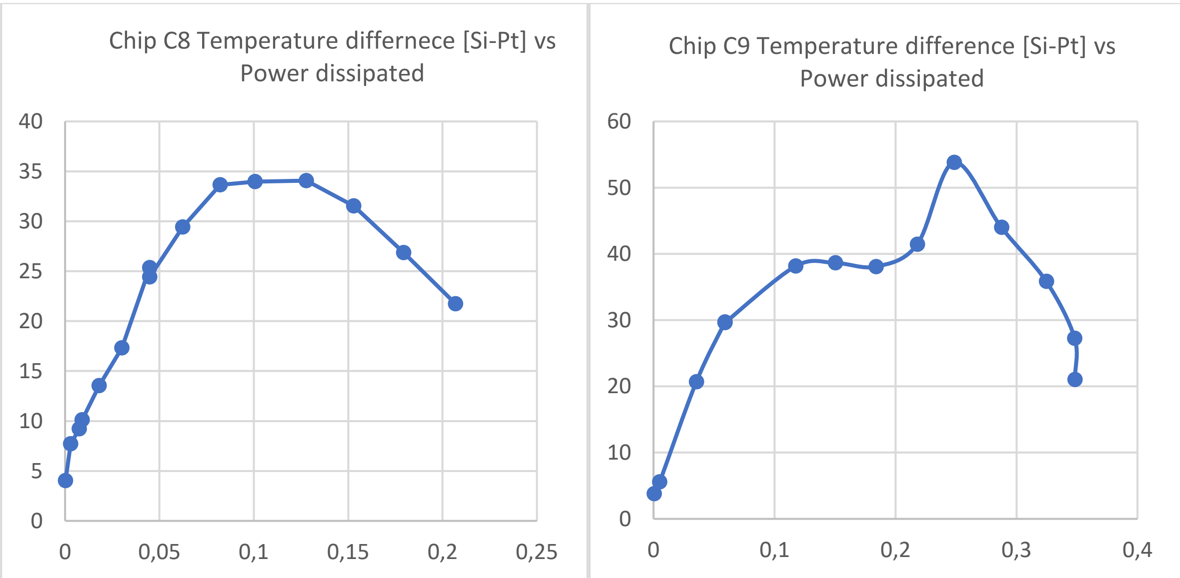


Figure (37) – The temperature difference between the silicon and the platinum sensors for chips C8 Swissroll and chip C9 Corriolish

This temperature difference could be explained by the error in the linear approximation of the TCR for the silicon. It can be seen that for very low powers, the difference is small, but as the temperature increases the difference becomes larger. Eventually the difference decreases at high powers, when the temperature of the silicon reaches the saturation value.

This difference could also address another issue: a bad model for the TCR of the Silicon. Throughout the report, it was assumed that the TCR of the silicon behaves linearly. According to figure 37, it can be concluded that the silicon does not gain temperature that fast, following a logarithmic shape. A plot of a possible shape for the silicon TCR is presented in the error analysis.

Limitations

During the joule heating, the chips C8 and C9 were not tested to the maximum limit. In this case, the experiment was limited only to the maximum output value of the power source – 64V and 6A. This limitation could have been solved by choosing to change the current instead of voltage.

For 64V of applied voltage the peak current was 0.0033A for chip c8 and 0.0054A for chip C9, less than 0.1% of the power source capacity. Although, as figure (35 and 36) show, there is a high chance that the power applied was pushing the silicon strips to the limit. The silicon strips reached almost the saturation temperature, the last step before a break down.

Error analysis

TCR calculations

Platinum

The process of data gathering for the TCR calculations is presented in detail in section (Method – TCR calculations). This process involves approximations and averaged values, so all the errors will be discussed below. Also the standard deviations will be calculated.

First, the TCR of every platinum strip is calculated as the average value of the TCR for each temperature step. In order to see how inaccurate the average value is, the standard deviation of all the TCRs has been calculated using the excel function STDV.P. The formula is presented below (5) and calculated the square root of the average deviation between a value and the mean of all values.

$$STDV.P = \sqrt{\frac{\sum(TCR - \mu)^2}{N}} \quad (5)$$

Table (13) – The standard deviation for each chip

	TCR C8 Corriolish	TCR Ce	TCR C9	TCR C2
Standard Deviation	0.00003233	0.00001988	0.00000623	0.00001562

The highest standard deviation was obtained at the Corriolish chip, C8 with a value of 0.000032. This is proven also by Figure (25), which shows an exponential behaviour for the TCR. Even though the error was more prominent for the leftmost channel, the TCR measurement for this chip was considered to be the most accurate due to possible errors at the multimeter.

Silicon

For the calculation of the error of the silicon TCR a much more in depth discussion will be made. The TCR of the silicon was estimated as a straight line, but it was not proven by any measurements for all temperatures (the linear behaviour was proven up to a maximum temperature of 80ºC). The linear approximation method has a series of weak points and multiple situations where errors could be propagated. First, the line equation of the TCR has been done using reference values from the first stage of the measurements. After the chips were exposed to heat from the oven and to current from the multimeter, the reference resistance has changed. This new resistance value could add an error to the calculation of the temperature. As figure (40) shows, after the joule heating experiment, the silicon heated up to 118ºC. For the reference temperature of 50.4ºC the TCR of the silicon is now 0.00043 instead of 0.00057.

Furthermore, the TCR line may behave logarithmically at high temperatures. Using the same formula (3), the TCR of the silicon may be calculated - based on $T_{si,\alpha}$(the temperature of the silicon closest to the platinum):

$$\alpha_{Si} = \frac{R - R_{ref}}{R_{ref}\left(T_{si,\alpha} - T_{ref}\right)}$$

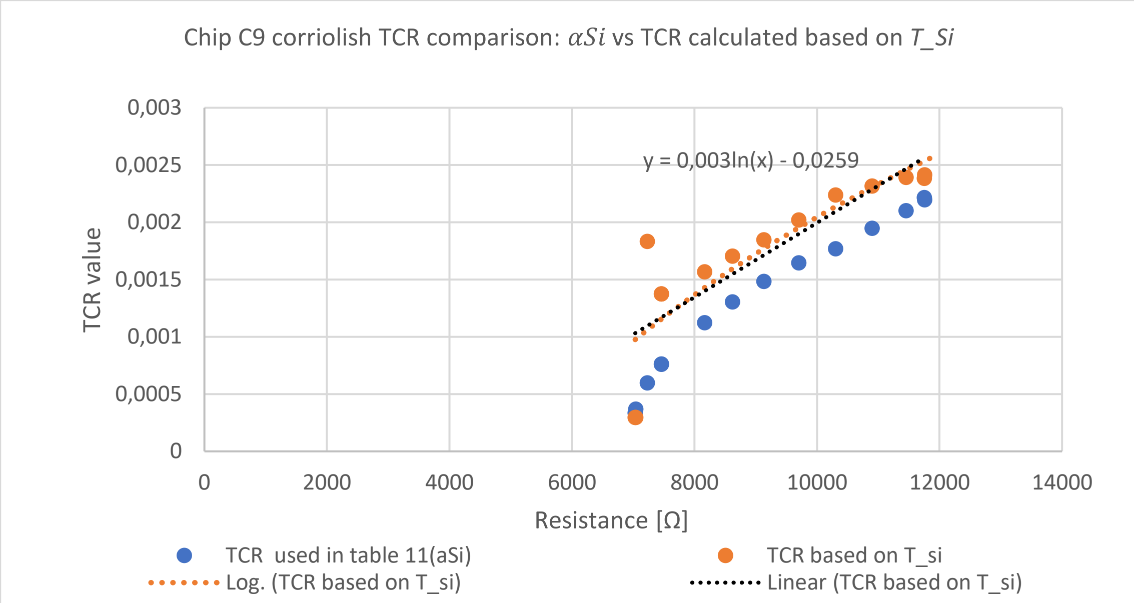


Figure (38) – The difference between the TCR calculated based on T_{Si} from table (10) and α_{Si} (table 10)

In the figure above there is a comparison between the TCR used to calculate the temperature of the silicon through second method and a TCR derived from the T_{si}. As figure (38) shows, for the orange points there are both a shift in value and a trend to a logarithmic shape (logarithmic trendline approximated the points better than a linear trendline). As a conclusion, the change of temperature described above is due to both a bad linear approximation of the function of the TCR with regard to T_{Pt} and the limitations of the linear model.

As final observation, in this report the temperature of the silicon based on the theoretical temperature difference is considered to be a more accurate value. First, in theory, the TCR of the platinum should respect the linear behaviour calculated above up to very high temperatures, so the temperature of the platinum leaves less room for errors. Also, from the physical point of view, the temperature of the silicon should be very close to the temperature of the platinum due to the chips design and the very small distance between them. The heat transfer between the 2 materials is almost perfect.

TCR Measurements after the joule heating

The Joule heating process changes the molecular structure of the materials, effecting the repeatability of the results. After the joule heating, the TCR measurements will be repeated, in order to observe the changes made to the materials. The TCR of the silicon and platinum will be redone.

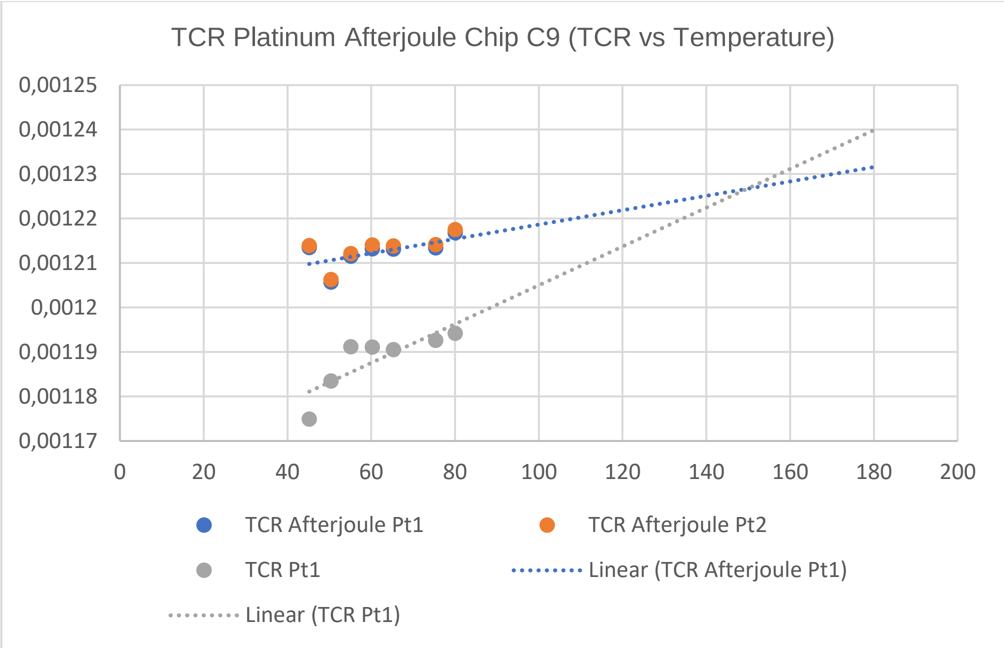


Figure (39) – A comparison between the TCR before the joule heating and after the joule heating.
The increase in the value of the TCR can be explained by the fact that the increase of temperature changed the molecular structure of the platinum strip, creating a much more concentrated, dense material.

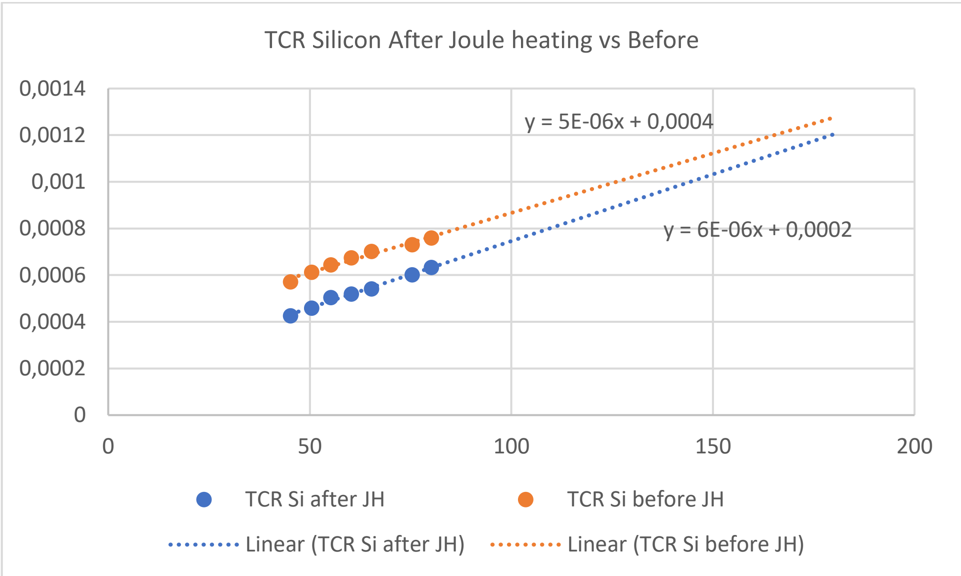


Figure (40) - The temperature of the silicon based on the TCR of the silicon.
The TCR of the silicon decreased after the joule heating and the overall resistance of the strip increased. This could be an effect of the high temperatures applied to the chip, when the discontinuities and imperfections of the material tend to uniform itself, altering the overall resistance.

Conclusion

As a conclusion this report proved the enhanced performance of the silicon used as a heater. This report presents in detail the progress of the measurements starting from microscope analysis of the chips, gluing procedure, calculations regarding the expected resistance of the silicon heater and finally results after the Joule Heating procedure. Under the parameters described in this report the silicon heater of Chip C9 Corriolish achieved a temperature of 325ºC at a dissipated power of only 0.35W. The performance of the silicon is strictly related to multiple criteria as the physical properties (width, length and height) and the shape of the design of the chip.

The physical properties influence the resistance or the maximum possible dissipated power and it could create weak sports where the silicon strip could break up. Multiple designs of chips have been used – so far the most effective was chip C9 Corriolish and chip C8 Swiss roll (both of them in 2 channel configuration SEM). Out of a wafer of 14 chips, only 4 chips fulfilled the physical requirements to finish the experiment, so the calculated production reliability is 29%.Out of the 4 only the 2 enumerated above managed to produce noticeable results. The other 2 SMS chips suffered from silicon strip breakdown or poor heating performance.

Finally this report showed the proof of concept for a MEMS chip used in microfluidic applications with a silicon heater and managed to describe the behaviour both in theory and simulations.

References

[1] – Dennis Snow - Plant Engineer's reference Book, Second edition, Section 13.9.2 - Wobbe number

[2] - Biomass Gasification and Pyrolysis Practical Design, 2010, Pages 27-63 section HHV

[3] – 2019 MFHS Paper - HIGHLY-DOPED BULK SILICON MICROHEATERS AND ELECTRODES EMBEDDED BETWEEN FREE-HANGING MICROFLUIDIC CHANNELS BY SURFACE CHANNEL TECHNOLOGY byY. Zhao, H.-W. Veltkamp, T.V.P. Schut, page 2 figure 1

[4] – 2019 MFHS Paper - HIGHLY-DOPED BULK SILICON MICROHEATERS AND ELECTRODES EMBEDDED BETWEEN FREE-HANGING MICROFLUIDIC CHANNELS BY SURFACE CHANNEL TECHNOLOGY byY. Zhao, H.-W. Veltkamp, T.V.P. Schut, page 3 figure 2

[5] – 2019 MFHS Paper - HIGHLY-DOPED BULK SILICON MICROHEATERS AND ELECTRODES EMBEDDED BETWEEN FREE-HANGING MICROFLUIDIC CHANNELS BY SURFACE CHANNEL TECHNOLOGY byY. Zhao, H.-W. Veltkamp, T.V.P. Schut, page 3 - RESULTS AND DISCUSSIONS

[6] – 2019 MEMS paper - HIGH POWER SI SIDEWALL HEATERS FOR FLUIDIC APPLICATIONS FABRICATED BY TRENCH-ASSISTED SURFACE CHANNELTECHNOLOGY - Henk-Willem Veltkamp , Yiyuan Zhao , Meint J. de Boer equation 1, page 1

[7] – Distributed thermal micro sensors for fluid flow – John van Baar chapter 4 – V-grooved Pirani pressure sensor with distributed temperature sensing

[8] – The Feynman Lectures of Physics Vol. I Ch. 44: The Laws of Thermodynamics

[9] – Fundamentals of Heat and Mass Transfer – Incopera/DeWitt/Bergman/Lavine – page 3 Conductionn

[10] – Heat transfer - Chris Long & Nasser Sema - chapter 1 section 1.1

[11] – R. H. S. Winterton (1999) Newton's law of cooling, Contemporary Physics – equation 1 page 206

[12] – Fundamentals of Heat and Mass Transfer – Incopera/DeWitt/Bergman/Lavine – page 62 figure 2.5

[13] – Fundamentals of Heat and Mass Transfer – page 8 table 1.1

Future research

This paper could be improved by further calculations and follow up experiments:

- For a better temperature calculation a curve of the TCR of the Silicon should be calculated. The temperature range the material should be evaluated from room temperature up to 200C
- Appling fluid through the most performant chip C9 Coriolish to prove the application it was designed for
- Theoretical calculations for the heat transfer for the sides of the silicon heater.

Appendix 1: Simulations

In order to prove the theory and the measurements, the have also been done software simulations in comsol. The physics added have been electromagnetic heat source and temperature coupling.
Due to the lack of the chips blueprints, the COMSOL simulation will focus on a very basic phenomenon – the heat dissipation and the temperature change of a system of Silicon – SiRN and Platinum surrounded by air. The parameters of the simulation will be presented below as well as a figure with the model.

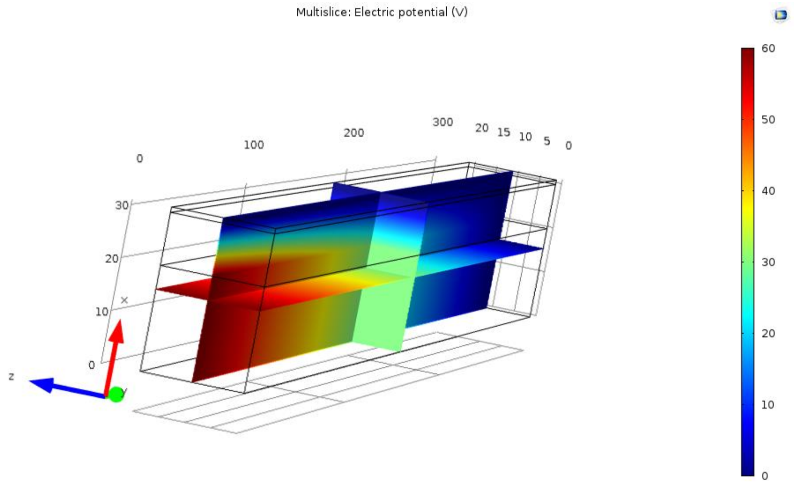


Figure (41) – The voltage drop over the Si-SiRN-Pt system – length $300\ \mu m$

The first step was to create a geometry that resembles the configuration of the silicon heater. The dimensions of the silicon as presented in figure() are $20\ \times 20 \times 300\ \mu m$. For the simulations there have been done 2 geometries: one where the length of the bar is $300\ \mu m$ and one when the length is $7500\ \mu m$. The first length was chosen in order to visualise better the physical behaviour of the system. The second length tries to make the comparison between the theory calculation for the heat transfer between the silicon and the platinum in section (). For the second length ($7500\ \mu m$), the platinum sensor thickness was changed to $10\ \mu m$ due to very long computational time as a result of building a very fine mesh.

As design choices a few decisions were made. Due to errors regarding the distribution of voltage, the SiRN layer was simulated to have a thickness of $10\ \mu m$. For lower thickness, the SiRN does not behave as a dielectric, and the voltage drop is conducted to the platinum as well. This phenomenon of conducting partially the electricity and having a voltage drop over the SIRN can be seen in figure (41). The platinum layer was set to a thickness of $1\ \mu m$

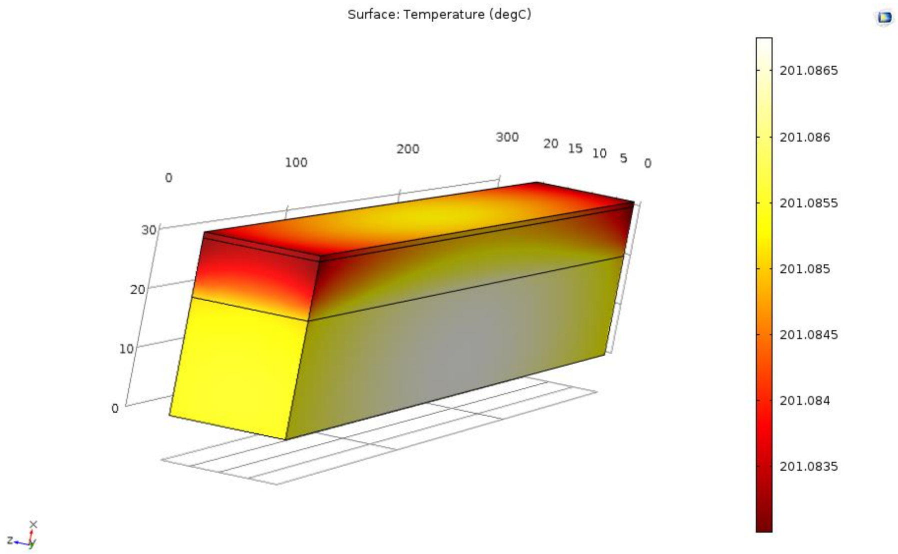


Figure (42) – The temperature distribution of a silicon bar with a voltage drop of 60V - length $300\ \mu m$. The temperature of the bar reached over 200ºC

Table (14) - The variables used in the experiment

	Value
Electrical conductivity silicon	$0.015\ \ [\mathrm{S/m}]$
Heat flux heat transfer coefficient	$10\ \ [\mathrm{W/m^2K}]$
Initial temperature (room temperature)	$293.15\ \ [\mathrm{K}]$

Once the length of the bar increases, the temperature decreases and the distribution becomes even along the length of the bar. This can be seen in figure (43) down, at a length of $1000\ \mu\mathrm{m}$. This phenomenon could be explained by the fact that the transfer between the silicon to the platinum is almost complete – for a very short bar the temperature change is in the range of 0.001C, so when the length of the bar increased the temperature difference present along the bar become dominant in front of the vertical distribution.

The simulations prove the theory in the sense that the heat transfer through the SiRN is very high, and the temperature difference between the silicon and the platinum are very small.

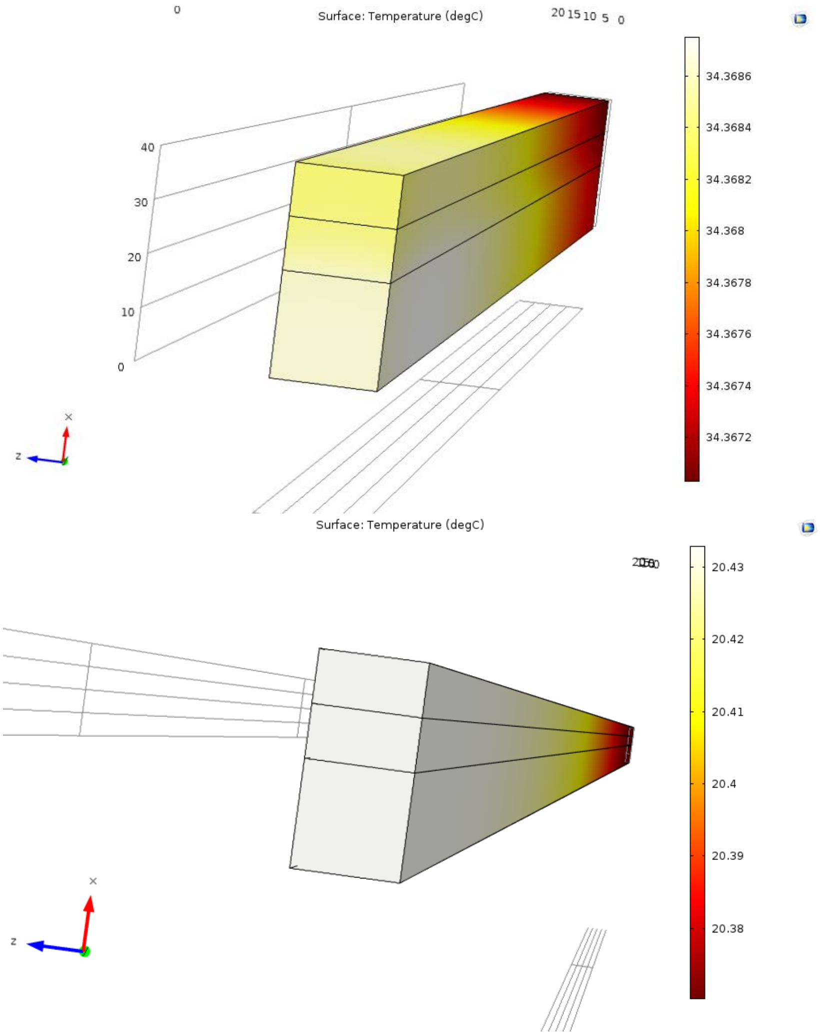


Figure (43) Top – The bar with a length of 1000um – the temperature distribution is spread more along the length rather than height – the edge of the platinum has the same temperature as the silicon heater.

Appendix 2

Project planning

Before the start of this project, the project planning has been agreed. All the sections have been completed with the respect of the last one, the test of the chips with the real fluid

Table 1: The timetable agreed at the beginning of the project

Calendar		Assistant
1 week 11-17Nov	Collect the chips Learn to use the microscope and take pictures of the chips under the microscope – check whether the Si is continuous – chips are described in MFHS)* Glue chips on the holder and wire bonding	Thomas
1 week 17-24 Nov	Glue chips on the holder and wire bonding (7 chips glued and wire bonded, 4 ready for the Si Heating, 1 ready for the fluid measurement)	Thomas
1 week 25-1 Dec	Experiment on TCR of Pt (Platinum) and silicon wires – oven MATLAB analysis	Remco Yiyuan
1 week 1Dec – 7 Dec	Heating the Si heater and measure with the Pt T-sensor (according to the MEMS paper results) Joule heating	Remco
1-2 weeks 7 Dec – 20Dec	COMSOL Simulations of the circuit and system TCR experiment after joule heating	
5Jan – 29 Jan	Report writing and finishing paper	Yiyuan Remco
Extra	Insert H2 in the channels and measure them (optionally)	

Table 1: Conclusion and status of the measured chips

		Multimeter reading			
		Normal OHM	OHM 4W	Verdict	TCR
C2 SMS U-Shape	Left channel – SI1	Overload	Overload	Open circuit – broken	Ready
	Right channel – SI2	0.528,758 kΩ	0.517,137 kΩ	OK	
Ce SMS 2 U-shape	Left channel – SI1	7.1247 kΩ	Overload	OK	Ready
	Right channel - SI2	6.900 kΩ	0.3 MΩ	OK	
C4 SMS	Left channel – SI1	1.895,1 kΩ	1.895,6 kΩ	OK	Not Ready
	Right channel - SI2	Overload	Overload	Open circuit - broken	
C6 –SMS*	Left channel – SI1	Overload	0.006 Ω	Open circuit - broken	Ready
	Right channel - SI2	Overload	-	Open circuit - broken	

C5 – 1 SI strip Swiss roll	SI1	Overload 0.300 MΩ	Overload	Open circuit - broken	Ready

*Platinum is damaged

Extra chips after probe measurements

		Probe measurement	OHM 4W	Verdict	TCR
C8 Swiss roll	SI1	15.87 kΩ		OK	Not ready*
C9 Coriolish	SI1	7.03 kΩ	7.032,4 kΩ	OK	Ready 06/12/2019

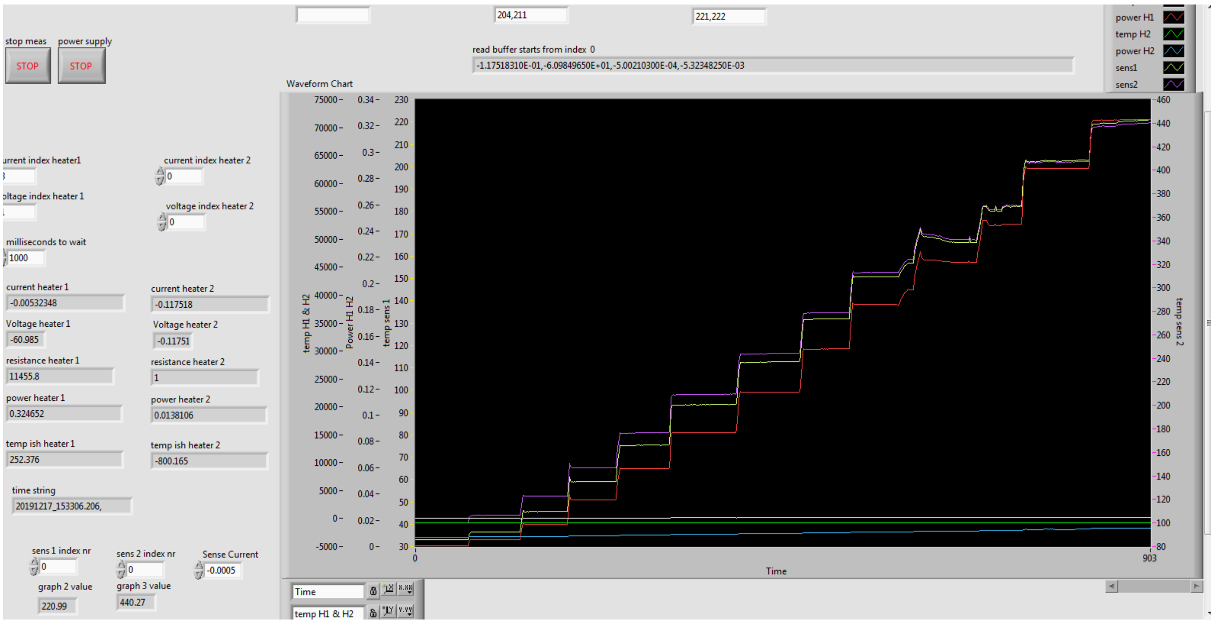
The code for the calculation of the heat transfer formulas implemented in Matlab (figures 21 and 23)

```
syms y(x)
Dy = diff(y);
Rb = 4.5*10^5 %1/148; %thermal line resistance of the beam in [K/W*m]
Gf = 0.001875; %line conductance through the gas in [W/(K*m)]
P1 = 0.25;  %electrical line power [W/m]
P2 = 0.35;
P3 =0.45;
l = 0.075;  % the lenght of the beam [m]
yn = -0.5:0.01:0.5; %normalised position along the si beam (-0.5 to 0.5)
z = l* (Rb*Gf)^1/2;

ode = (-1/(Rb*l*l))*diff(y,x,2) + Gf*y == P1;
cond1 = y(0) == 24;
cond2 = Dy(0) == 0;

conds = [cond1 cond2];
ySol(x) = dsolve(ode,conds);
ySol = simplify(ySol);

figure (1);
fplot (ySol); % plot a symbolic function
title("The solution of the DE 'cosh' function");

%%J van baar function plots
figure (2);

Tn = 24 + (P1/Gf)*(1 - cosh(yn*l*((Rb*Gf)^1/2))/cosh((1/2)*l*((Rb*Gf)^1/2)));
Tn2 = 24 + (P2/Gf)*(1 - cosh(yn*l*((Rb*Gf)^1/2))/cosh((1/2)*l*((Rb*Gf)^1/2)));
Tn3 = 24 + (P3/Gf)*(1 - cosh(yn*l*((Rb*Gf)^1/2))/cosh((1/2)*l*((Rb*Gf)^1/2)));

plot(yn,Tn, yn, Tn2, yn, Tn3);
xlabel('Lenght of the Pt sensor [mm]');
ylabel('Temperature [C]');
title(" Temperature distribution vs position along the chip");

%% The temperature difference formula
L1 = 17.13*10^-6;                   %Silicon layer lenght
L2 = 2*10^-6;                       %SiRN layer lenght
L3 = 0.2*10^-6;                     %Platinum lenght
L4 = 100*10^-6;
l = 7500*10^-6;                     %C9 Corriolish SEM
l1 = 28532 * 10^-6;                 %C8 Swissroll SEM
l2 = 3144*10^-6;                    % U shpae Ce
l3 = 6142*10^-6;                    % C2 Swiss roll SMS
w = 16.59*10^-6;
A = l* w;
A1 = l1*w;
A2 = l2*w;
```

```
A3 = 13*w;
k1 = 130;
k2 = 25;
k3 = 77.8; %(refference Fundamentals of heat)
h1 = 2;
h2 = 10;
%h_air = 0.024;
P_swipe = 0:0.01:0.5;
R_SIRN = L2/(k2*A);
R_SI = L1/(k1*A);
R_PT = L3/(k3*A);
%R_SIRNside = L4/(k2*1*4.5*10^-6);
%R_par = 1/(1/R_SIRNside + 1/R_SI + 1/R_SIRNside);
%R_air = L4/(h_air*A);

Rth = L1/(k1*A) + L2/(k2*A) + L3/(k3*A) + 1;          %C9 Corriolish SEM
Rth1 = L1/(k1*A1) + L2/(k2*A1) + L3/(k3*A1) + 1;      %C8 Swissroll SEM
Rth2 = L1/(k1*A2) + L2/(k2*A2) + L3/(k3*A2) + 1;      %U shpae Ce
Rth3 = L1/(k1*A3) + L2/(k2*A3) + L3/(k3*A3) + 1;      %C2 Swiss roll SMS
%R_SIRNside/(R_SIRNside + R_SI)
DT = P_swipe * Rth;
DT2 = P_swipe * Rth2;
DT3 = P_swipe * Rth3;
DT1 = P_swipe * Rth1;
figure (3);
plot (DT, P_swipe, DT1, P_swipe, DT2, P_swipe, DT3, P_swipe);
xlabel('\Delta T temperature change');
ylabel('Power [W]');
title("The temperature difference \Delta T vs Power [W]");
```

LabVIEW plots:

During the measurements, LabVIEW was a great tool to visualise the progress. During the joule heating, the real time values for the resistance of platinum and silicon temperature proved to be great feedback.



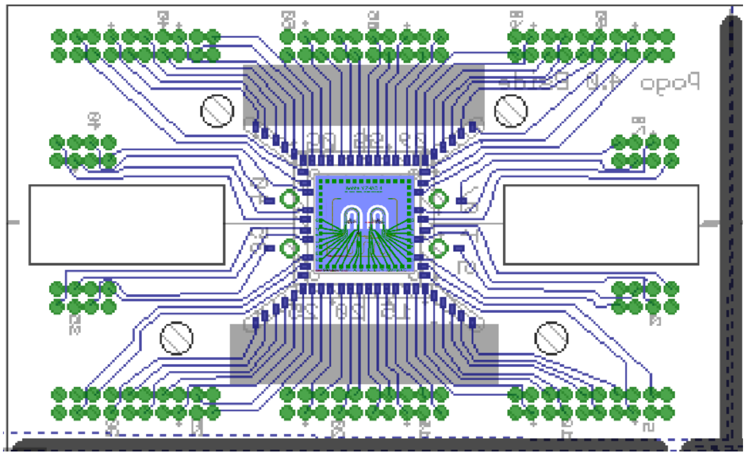
Appendix: Figure (1) - The visual interface of Labview during the joule heating of chip C9 Corriolish. On the left axis the resistance of the silicon, the power dissipated and the temperature of the platinum are displayed.

The convection coefficient table - Fundamentals of Heat and Mass Transfer – page 8 table 1.1

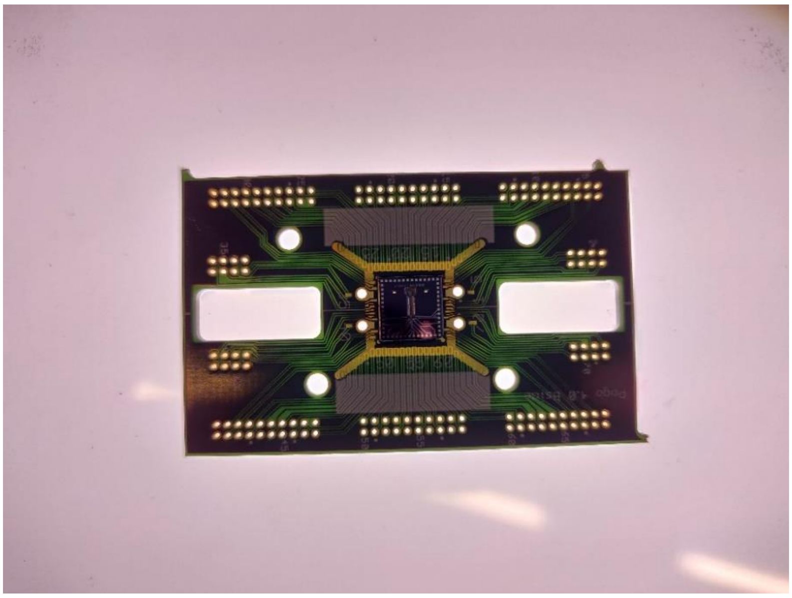
TABLE 1.1 Typical values of the convection heat transfer coefficient

Process	h $(W/m^2 \cdot K)$
Free convection	
Gases	2–25
Liquids	50–1000
Forced convection	
Gases	25–250
Liquids	100–20,000
Convection with phase change	
Boiling or condensation	2500–100,000

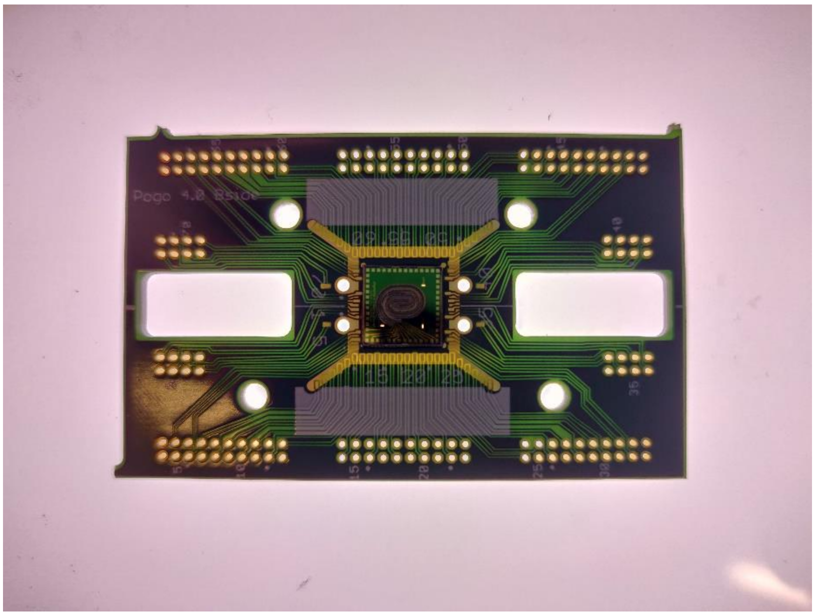
Appendix: Figure (2) – The free air transfer values



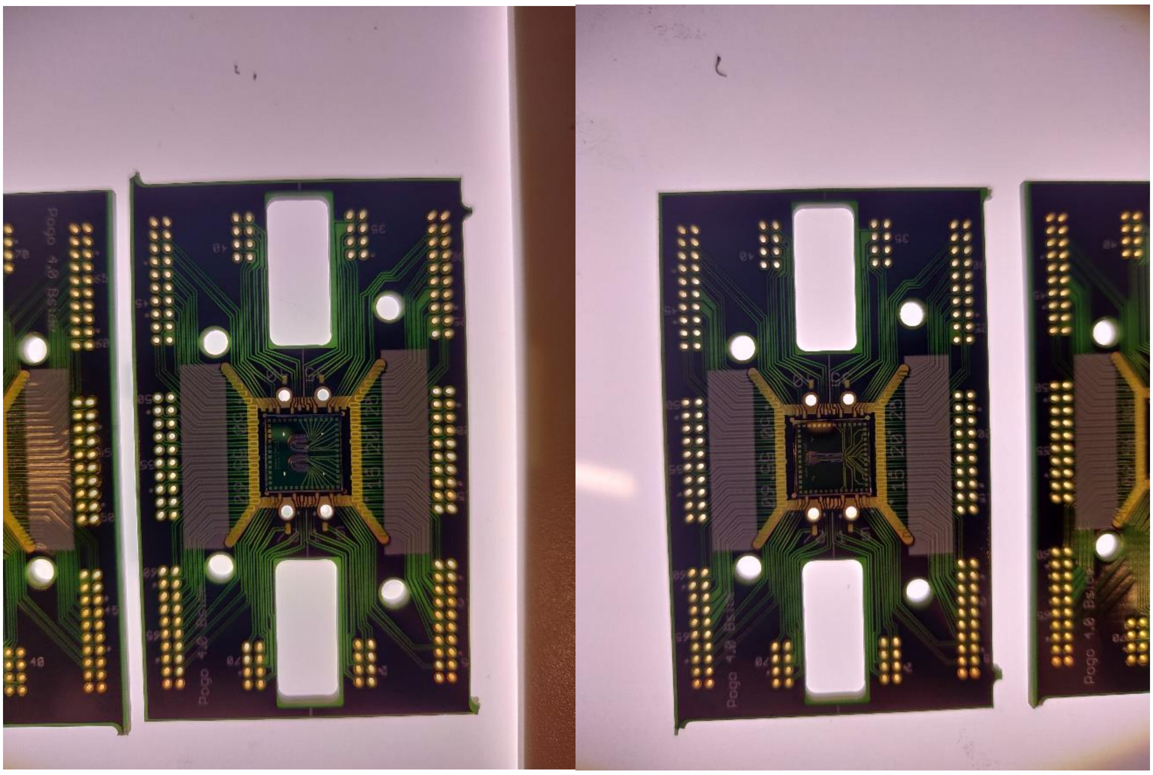
Appendix: Figure (3) - The coripogo mask and the U-shape chip connected in the middle



Appendix: Figure (4) Gluing - Chip C6 – SMS – and the inlets for the gas are visible



Appendix: Figure (5) – Glueing - Chip C5 – the Swiss roll – the 3 places for the gas testing can also be visible



Appendix: Figure (6) - Chips Ce and C3 – Right - The gluing process failed as the inlets are not visible

Appendix: Figure (7) - The tools needed for glueing: epoxy, alcohol, a piece of paper, a towel and wooden sticks. Optionally the chips could be placed in a lamp for better alignment with the inlets.