

High Precision Flow Compensated Thermal Conductivity Detector for Gas Sensing with Read-out Circuit

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High Precision Flow Independent Thermal Conductivity Detector for Gas Sensing with Read-out Circuit

By

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Chapter 1	Introduction.....	1
1.1	Motivation.....	1
1.2	Objectives.....	1
1.3	Thesis Organization	2
Chapter 2	Gas Sensing	4
2.1	Overview of Gas Sensors	4
2.2	Basic Concepts of Heat Transfer	5
2.2.1	Heat Transfer	5
2.2.1.1	Conduction	5
2.2.1.2	Convection.....	5
2.2.1.3	Thermal Radiation.....	7
2.2.1.4	Heat Equation	7
2.2.2	The Seebeck Effect	7
2.2.2.1	Thermocouple.....	8
2.2.3	Reynolds Number	9
2.3	Summary of the Chapter	10
Chapter 3	Thermal Conductivity Detectors.....	11
3.1	Definition of Thermal Conductivity Detector.....	11
3.2	Applications of a Thermal Conductivity Detector	13
3.2.1	TCD as a Flow Sensor	13
3.2.2	TCD as a Pressure Sensor.....	13
3.3	State of Art of μ TCDs.....	14
3.3.1	A Micromachined Thermoelectric Sensor for Natural Gas Analysis.....	14
3.3.2	Surface-Micromachined Thermal Conductivity Gas Sensor for Hydrogen Detection.....	15
3.4	Summary of the Chapter	16
Chapter 4	Flow Compensated Thermal Conductivity Detector	18
4.1	State of Art of Flow Compensated Thermal Conductivity Detectors	18
4.1.1	A Micro-Thermoelectric Gas Sensor.....	18
4.1.2	A Thermal Conductivity Detector Composed of Four Heaters	19
4.1.3	A Flow Independent Multi-TCD for Gas Sensing.....	21
4.1.4	Performance Comparison	23
4.2	Dual-Thermal Conductivity Detector	23
4.2.1	Description of the Flow Compensation Method Qualitatively.....	24
4.2.2	Description of the Flow Compensation Method Quantitatively	25

4.3	Heat Transfer Analysis of the Dual-TCD	26
4.4	Summary of the Chapter	30
Chapter 5	Fabrication	32
5.1	Design Characteristics.....	32
5.2	Fabrication Process Steps.....	32
Chapter 6	Simulations	36
6.1	Static Simulations	37
6.2	Dynamic Simulations.....	41
6.3	Summary of the Chapter	50
Chapter 7	Encapsulation Module and Read-out Control Loop Circuit.....	51
7.1	Encapsulation Module.....	51
7.2	Read-out Circuit.....	54
7.2.1	DC System	54
7.2.2	AC System	55
7.2.3	Instrumentation Amplifier	56
7.2.4	Chopper.....	59
7.2.5	Phase Compensation.....	59
7.2.6	Difference Amplifier	60
7.2.7	Low Pass Filter Modules	61
7.2.7.1	Sallen-Key Low Pass Filter	61
7.2.7.2	Digital Filter	62
7.2.8	Non-Inverting Summing Amplifier	62
7.2.9	Voltage to Current Converter	63
7.3	PCBs.....	64
Chapter 8	Measurement Results.....	66
8.1	Resistance of the Heater and Thermopiles.....	66
8.2	Probe Station Cascade 33.....	66
8.3	Measurements Using an Infrared Camera.....	69
8.4	Measurements in the Vacuum Chamber	73
8.5	Measurements Using CO ₂ and He	74
8.6	Summary of the Chapter	78
Chapter 9	Conclusions and Future Work	79
9.1	Conclusions.....	79
9.2	Future work.....	82

Bibliography 83

Appendices 86

 A.1 Thermal Model of the Sensor 86

 A.2 Package Dimensions 91

Chapter 1 Introduction

1.1 Motivation

There are different types of gas sensors: Chemical and Physical. Although most common ones are based on chemical reactions, gas sensors based on physical properties, especially thermal conductivity detection, have gained popularity in the last decades.

Thermal conductivity detector (TCD) is classified as physical sensor because it measures the gas thermal conductivity. TCDs are important components of a gas chromatography (GC) system. In fact, research in TCD (especially μ TCD) began with miniaturization of GC systems. Applications where TCDs can be used are: flow sensing and pressure sensing.

One important disadvantage of both chemical sensors and thermal conductivity detectors is that they demonstrate flow dependence. Flow affects the measurement of the device, sometimes producing an important error. Methods to reduce flow dependence have been utilized. One method to reduce flow dependence is using another sensor as a reference placed in a different flow path. The measurements of this sensor are taken as a reference and are used to compensate the effect of flow on the sensing sensor. Another method is that to (re)calibrate the sensing sensor depending on the flow rate for every gas in the gas stream. These methods have had good results. However, it is necessary to have an additional sensor and additional flow path increasing efforts and cost.

The present thesis work is aiming to reduce flow dependence based on the in-line flow compensation using a dual-thermal conductivity detector (dual-TCD). The dual-TCD is placed in the gas sensing flow path suppressing the need of having an additional reference device and flow path. The dual-TCD is composed of two almost identical thermal conductivity detectors where one of them is very sensitive to the thermal conductivity of the gas while the other one is just a compensation device.

The TCDs of the dual-TCD are fabricated in the same membrane in parallel. The TCDs are laterally identically, but with a different depth of the detection chamber, resulting in different quasi-static sensitivities to the thermal conductivity of the sample gas. Both TCDs are differentially operated.

1.2 Objectives

The main objective of this work is to design, characterize and fabricate a flow independent thermal conductivity detector based on the concept of a dual-thermal conductivity detector MEMS device.

The development of the dual-TCD starts with a bibliographic research of the state of art of thermal conductivity detector as well as flow independent TCDs.

The design approach of the dual-TCD device starts from a previous TCD presented by [1]. The work of [1, 2] explains the fabrication and characterization of a single TCD where the gas is measured by means of a

sample chamber using heaters and thermopiles. From the heat transfer behaviour of the TCD of [2] it is possible to understand that if two similar TCDs work together, a flow compensated device might developed.

To verify whether the dual-TCD is flow independent, the device will be designed and simulated using Comsol Multiphysics v4.4. Using this software, simulations with and without a flow through the device will be performed.

A read-out circuit of the dual-TCD will be proposed and simulations of this circuit will be done using LTSpice IV. An encapsulation module will be designed to ensure laminar flow through the dual-TCD. Both the read-out circuit together the encapsulation module form the read-out control loop circuit.

Measurements will be made to compare simulations with real results.

1.3 Thesis Organization

This thesis begins with an introduction to the topic of thermal conductivity detectors where the motivation and the objective are stated. Then, the thesis is divided in the following chapters.

Chapter 2 Gas Sensing

In this chapter an overview of the most used gas sensors of nowadays is provided. These are based on chemical properties or physical properties. The advantages of using physical sensors instead of chemical sensors will be stated as well as the reason of why physical sensors are gained in popularity in the last decade. Also, in this chapter basic concepts of heat transfer will be provided.

Chapter 3 Thermal Conductivity Detectors

The principle of thermal conductivity detectors is defined in this chapter using the most simple device the so called hot-wire. Also, a comparison of the state of art of gas sensors is given. Two sensor based on thermal conductivity principle are compared. The thermal conductivity detector that will be used as starting point of this thesis work will be presented in this chapter.

Chapter 4 Flow Compensated Thermal Conductivity Detector

The state of art of different flow compensated TCDs will be discussed in this chapter. These devices will be compared accordingly to their performance. The flow compensated idea using a dual-Thermal conductivity detector is provided in this chapter. The heat transfer behaviour of the sensor is analysed by calculating the relation between the temperature and thermal conductivity.

Chapter 5 Fabrication

The fabrication process flow of the dual-thermal conductivity detector is given in this chapter.

Chapter 6 Simulations

In this chapter, simulations of the static and dynamic behaviour of the sensor are done. Static simulations show the sensitivity of the dual-thermal conductivity detector to thermal conductivity and dynamic simulations are performed to verify the flow compensation idea.

Chapter 7 Encapsulation Module and Read-out Control Loop Circuit

In this chapter, a measurement setup of the sensor is presented. This setup will be intended to be used in the real measurements of the sensor. This setup is composed of an encapsulation module and a read-out circuit. The encapsulation module has been designed in such a way to ensure laminar flow on the sensor. The read-out circuit has been thought to control the sensor while measuring different gases at different flow rates.

Chapter 8 Measurement Results

Gas measurements by using the dual-thermal conductivity detector are presented in this chapter. These measurements are intended to confirm simulations presented in chapter 6.

Chapter 9 Conclusions

The thesis work is concluded in this chapter.

Chapter 2 Gas Sensing

In this chapter an overview of the most important types of gas sensors will be provided as well as the concepts of heat transfer that will be useful when thermal conductivity detectors are defined. After defining the concepts of heat transfer, the principle of thermal conductivity detectors is given starting with the most basic TCD the so called “hot-wire” device. Then, the state of art of μ TCD is discussed. Finally, a comparison between the state of art μ TCDs is stated.

2.1 Overview of Gas Sensors

One of the most important areas of sensors is gas sensing. Basically, a gas sensor measures or detects the presence of a gas in some space. In the last years, gas sensors are increasingly used in the growing markets of automotive industry and environmental monitoring [3].

There are two main types of gas sensors based on the way of measuring the gas, these are: chemical and physical properties. Chemical sensors are the most common and widely used. These types of sensors are based on chemical interaction [4]. These sensors measure a change in the conductance or other physical properties between the gas and the sensing material during gas interaction [5]. One of the main advantages of chemical sensors is their high sensitivity to the measured gas [6]. However, they are not reliable in long-term use because they suffer from chemical contamination of the sensing part [5, 7]. Another problem of these sensors is their slow response time to changes in gas composition. One of the reason for the slow response time is that the chemical interaction highly depends on environmental temperature [8]. An example of this is the time response of the palladium-based chemical sensor for hydrogen. At low temperatures Pd-based gas sensor has a time response of several minutes detecting a concentration of 1% H in air, while at high temperature the time response is in the order of seconds [9, 10].

Sensors without chemical reaction are called physical sensors. These types of sensors measure a physical property of the gas like thermal conductivity. One of the major advantages of physical sensors is that as they measure a physical property they do not suffer from long term chemical contamination. This makes them much more reliable in long term use. Also, they have better response time in comparison to chemical sensors [8]. Within physical sensors, thermal conductivity detectors (TCDs) have become more and more important in the gas sensing area. One reason is that the use of gas sensors based on thermal conductivity detection has increased due to advances in the area of integrated circuit (IC) technology [11]. TCDs are widely utilized in control processes and especially in gas chromatography (GC) [12, 13]. In fact, miniaturization of GC devices has initiated research in micro-TCDs [14]. Besides, MEMS technology enables miniaturization at low cost [11]. TCDs do not suffer from chemical contamination in long-term use in comparison to chemical sensors, making them very reliable and robust. One of the areas where TCDs are very suitable is detecting hydrogen, because the thermal conductivity of hydrogen is approximately 7 times higher than that of air.

2.2 Basic Concepts of Heat Transfer

In this section the basic concepts of heat transfer will be defined. These concepts are useful for understanding the thermal conductivity detector.

2.2.1 Heat Transfer

Heat transfer is defined by the exchange of thermal energy between two or more physical systems depending on the temperature and pressure. The exchange of thermal energy always happens along a temperature gradient from the hottest region to the coldest region [15]. The three heat transfer mechanisms are: conduction, convection and radiation.

2.2.1.1 Conduction

The mechanism of conduction is one of the most important in micro-scale devices [1]. Heat is transferred in a body along a temperature gradient from the hottest part to the coldest part. This phenomenon also happens when two or more objects are in physical contact. The heat flows from the hottest object to the coldest one through the objects in between.

The law that models this phenomenon is called “Fourier’s Law” and it states that the heat flux ϕ_q is proportional to the negative temperature gradient $-\Delta T$ and the proportionality constant is thermal conductivity λ [2]:

$$\phi_q = -\lambda \cdot \Delta T \quad (1)$$

The sign of the above equation ensures that the heat flows down the temperature gradient [2]. Thermal conductivity λ is a property of the thermal transfer by conduction. It refers to the ability of a material to conduct heat through it.

2.2.1.2 Convection

Heat transfer by convection is produced by the movement of a mass of fluid from the heat source. There are two types of heat convection:

- Natural convection: This type of convection is not caused by any external source. Only the difference in density between the hot and the cold fluid causes the motion of fluid mass. When a fluid is subjected to a hot source becomes less dense and it is replaced by cold fluid generating a natural convection of the fluid surrounding the heat source.
- Forced convection: In this case, the convection is caused by an external source like a pump or a suction device. The effect of this external source causes an artificial convection current.

In the case of micro-scale devices natural convection is negligible [16], although in the presence of flow forced convection contributes to the heat transfer. The heat transfer by convection (natural and forced) is defined by Newton’s law of cooling:

$$P = hA(T_w - T_f) \quad (2)$$

where P is the rate of power transferred between the exposed surface of the body A and the fluid in. T_w is the temperature of the body, T_f is the temperature of the fluid and h is the convection coefficient. The convection coefficient is strongly related to the Nusselt number (Nu) which relates the convection to conduction heat transfer across a boundary [17]. The relation between Nu and h is defined as follows:

$$\overline{Nu} = \frac{\bar{h} \cdot L}{\lambda} \quad (3)$$

where L is the characteristic length and the bar above quantities signifies an average value. The characteristic length is defined as:

$$L = \frac{4A_p}{P_p} \quad (4)$$

where A_p is the area where the fluid flows and P_p is the perimeter where the fluid flows. Also, the Nusselt number is defined as a combination of physical parameters as follows[17]:

$$\overline{Nu} = C \left(\frac{\rho v L}{\mu} \right)^n \left(\frac{C_p \mu}{\lambda} \right)^m \quad (5)$$

where C is proportionality constant, ρ is the density of the fluid, v is the velocity of the fluid, μ is the dynamic viscosity of the fluid, C_p is the heat capacity of the fluid and n y m are coefficients.

The convection coefficient is not constant and its calculation is complex because it depends on the fluid flow, thermal properties of the fluid, the geometry of the stream and even the orientation in space of the system [17]. However, the calculation of h can be simplified defining different cases. The first step is to define if the convection is mainly natural or forced. Then, determine whether the flow is laminar or turbulent. Also, if the heat flux over the surface is uniform or not. Finally, if the flow is flowing parallel or perpendicular to the surface of the body. In our case, especially due to dimensions, we will have the following parameters: Forced convection, laminar flow, heat flux not uniform and parallel flow. For this especial case the Nusselt number and therefore the convection coefficient are [17]:

$$\overline{Nu} = 0.453 \left(\frac{\rho v L}{\mu} \right)^{1/2} \left(\frac{C_p \mu}{\lambda} \right)^{1/3} \quad (6)$$

$$\bar{h} = 0.453 \frac{\lambda}{L} \left(\frac{\rho v L}{\mu} \right)^{1/2} \left(\frac{C_p \mu}{\lambda} \right)^{1/3} \quad (7)$$

Replacing Equation (7) in (2) the heat transfer by forced convection is obtained.

2.2.1.3 Thermal Radiation

Thermal radiation is the energy emitted from a hot element in the form of electromagnetic radiation. Stefan-Boltzmann law states that the energy radiated by a body per second per unit area is proportional to the fourth power of the absolute temperature of the body:

$$P_{rad} = \varepsilon \sigma A T^4 \quad (8)$$

where ε is the emissivity factor, σ is the Stefan-Boltzmann constant equals to $5.67 \times 10^{-8} [\text{W} \cdot \text{m}^{-2} \cdot \text{K}^{-4}]$, and A is the area of the emitted surface.

2.2.1.4 Heat Equation

The distribution of heat in a certain region over time is described by the heat equation. This equation is a partial differential equation depending on the three spatial variables (x, y, z) and the time (t). The equation is given by [18]:

$$\frac{\partial T}{\partial t} = \alpha \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) + \frac{\Phi_s}{\rho C_p} \quad (9)$$

where Φ_s corresponds to the heat source power per volume V , ρ is the density and C_p is the specific heat capacity at a constant pressure. The thermal diffusivity α and the thermal conductivity λ can be related using the following equation:

$$\alpha = \frac{\lambda}{\rho C_p}. \quad (10)$$

If the system does not change in time, thus the temperature remains constant; the heat equation can be expressed as:

$$\frac{\partial T}{\partial t} = 0 \Rightarrow \alpha \left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) + \frac{\Phi_s}{\rho C_p} = 0 \quad (11)$$

and using Eq. (4) the heat equation turns in:

$$\left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2} \right) + \frac{\Phi_s}{\lambda} = 0 \quad (12)$$

2.2.2 The Seebeck Effect

Thermoelectricity is a phenomenon where thermal energy is converted into electrical energy and vice versa. It is a property of each conducting material. It was discovered in 1821 with the Seebeck effect [19]. The Seebeck effect is one of the thermoelectricity phenomenon, the other two are: the Peltier effect and the Thompson effect.

The Seebeck effect is the generation of a differential voltage when a temperature difference is applied between two points of a conductor or semiconductor. This effect was discovered by the German physicist Thomas Johann Seebeck in 1821 [19]. He realized that in a closed loop circuit consisting of two different conductors, subjected to a temperature difference between the junctions produced an electric current. The Seebeck coefficient is defined as the relation between a differential voltage (ΔV) divided by the temperature difference (ΔT) between two points of a material.

$$\alpha = \frac{\Delta V}{\Delta T} \quad (13)$$

This coefficient is also called the absolute Seebeck coefficient and depends on the material. The unit of α is $V \cdot K^{-1}$, but this value is normally very small, thus it is usually given in $\mu V \cdot K^{-1}$.

2.2.2.1 Thermocouple

The thermocouple is the most basic and common structure to observe the Seebeck effect. In fact, the physicist Thomas Seebeck discovered this effect using a thermocouple[19]. The thermocouple is composed by two dissimilar (semi)conductors joined at one end and leaving the other end as an open circuit. This will generate an open circuit voltage proportional to the temperature difference of both ends. Figure 1 shows a thermocouple structure.

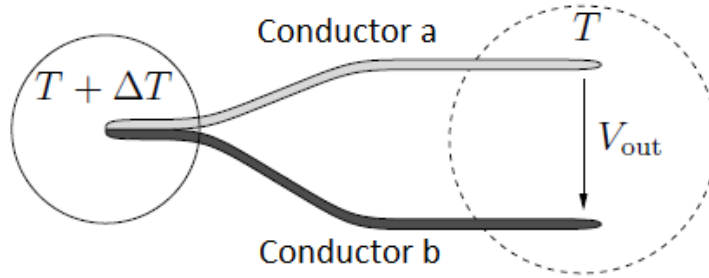


Figure 1: Representation of a thermocouple [20].

In this case V_{out} is expressed by:

$$V_{out} = \alpha_{ab} \Delta T \quad (14)$$

where α_{ab} ($\mu V \cdot K^{-1}$) is the Seebeck coefficient between conductors a y b, and ΔT is the temperature difference between both ends. Materials like Ni, Cu, Pb, N-type Polysilicon, between others are utilized to fabricate thermocouples. Table 1 shows the Seebeck coefficient of materials that are utilized to fabricate thermocouples.

Table 1: Seebeck coefficient of different materials [21].

Material	α [$\mu\text{V/K}$] @ 273 K	α [$\mu\text{V/K}$] @ 300 K
Pb	-0.995	-1.047
Cu	+1.70	+1.83
Au	+1.79	+1.94
Cr	+18.8	+17.3
Ni	-18	
Al		-1.7
Si		+435
P-type Polysilicon		+170
N-type Polysilicon		-120

From table above, silicon has the highest Seebeck coefficient, making it a very good candidate to fabricate a thermocouple. Another good material to fabricate thermocouples is Polysilicon. P-type and N-type Polysilicon have a high Seebeck coefficient and both are compatible integrated circuit processes with Silicon, making them very suitable to fabricate thermocouples (Table 1), especially when Si is utilized to design the main parts of an integrated circuit.

From Table 1 it can be seen that the Seebeck voltage is small, in the order of μV . This sometimes makes the read-out very complex. However, if several thermocouples are connected in series, the Seebeck voltage increases its value linearly with the number of thermocouples connected, leading to:

$$V_{out} = N\alpha_{ab}\Delta T \quad (15)$$

where N represents the number of thermocouples connected in series. The name of several thermocouples connected in series is thermopile.

2.2.3 Reynolds Number

The Reynolds number (Re) is a dimensionless number which states whether a fluid flow is laminar or turbulent depending on the physical properties of the fluid and the dimensions of the body where the fluid flows. This number is defined by the following equation [22]:

$$Re = \frac{4\rho Q}{\mu P} \quad (16)$$

where ρ is the density of the fluid, Q corresponds to the flow, μ is the dynamic viscosity of the fluid and P is the wetted perimeter. The wetted perimeter is defined as the actual perimeter where the fluid flows. If the path where the fluid flows is a rectangular section, thus the perimeter is the sum of all its sides.

To know if a flow is laminar or turbulent Re has the following rules: If $Re < 2100$ the flow is mainly laminar, while the flow becomes turbulent if $Re > 2100$ [22].

2.3 Summary of the Chapter

There are two main types of gas sensors: based on chemical reaction and based on physical properties. Chemical sensors have a high sensitivity to the gas, however suffer from chemical contamination in long term use. Thermal conductivity detectors are part of physical sensors. TCDs do not suffer from chemical contamination, thus, they are more robust and reliable than chemical sensors.

Basic concepts of heat transfer have been defined as well as Reynolds number which defines if the flow is laminar or turbulent. These concepts will be used during the development of this thesis.

Chapter 3 Thermal Conductivity Detectors

In this section the principle of thermal conductivity detection will be defined. Applications of thermal conductivity detectors will be presented as well as the state of art of TCDs. The TCD which will be used as the starting point of the flow compensated TCD is presented in section 3.3.2.

3.1 Definition of Thermal Conductivity Detector

A thermal conductivity detector is a physical sensor that measures the thermal conductivity of a gas by measuring heat flow and temperature by means of a heated element in the gas stream. In other words, a TCD measures the heat loss through the gas using a temperature sensor. There are two basic elements conforming a TCD, these are: a heater and a temperature sensor. The heater heats up the surrounding gas while the temperature sensor measures the heat transfer through the gas.

In terms of TCD devices, the most common TCD is the so called hot-wire device [12]. The hot-wire is basically a wire which acts as a heater and a temperature sensor at the same time, making it very simple to design. Usually, a simple resistor is used as a hot-wire. Figure 2 shows this basic device.

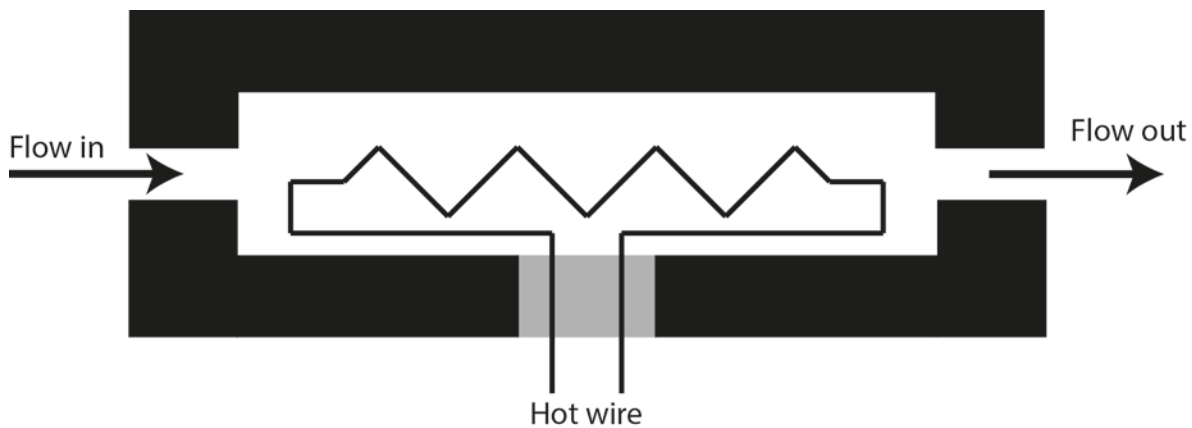


Figure 2: Hot wire.

The principle of the hot-wire is explained as follows. The hot-wire is fed by using an electrical current producing Joule heating effect and, depending on the surrounding gas (thermal conductivity), the temperature of the wire changes and can be detected as a change in resistance.

A usual read-out of this kind of TCD is the Wheatstone bridge, as shown in Figure 3 [23].

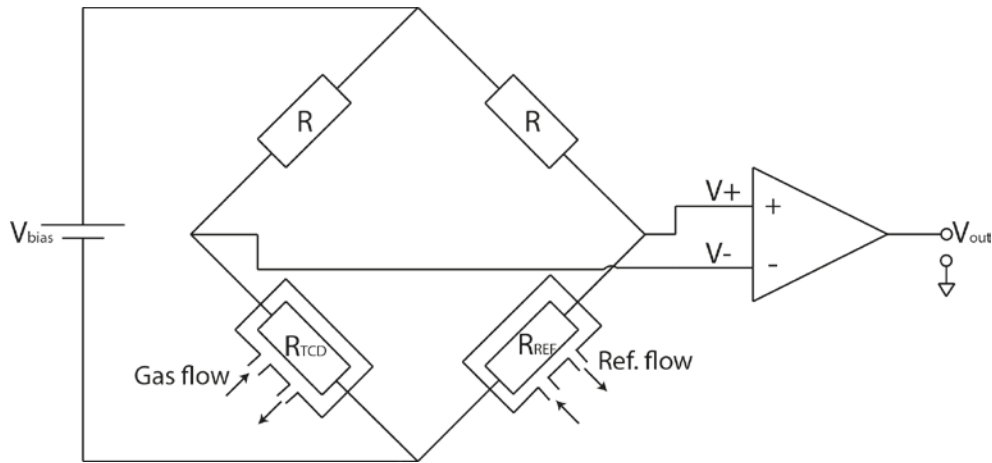


Figure 3: Circuit read-out of a TCD using a Wheatstone bridge.

One advantage of the TCDs in comparison to chemical sensors is their low cost. TCDs can be fabricated in batches by using integrated circuits. Another advantage is that the good sensitivity that TCDs can be achieved. In the case of TCDs where the heater and the temperature sensor are separated, the sensitivity can be increased by using large thermopile array. However, the thermal noise of the thermopile also increases.

One disadvantage of the TCDs is their low selectivity. Figure 4 shows the thermal conductivity of different gases compare to Air. Selectivity for CO_2 , Air, N_2 and CH_4 is low. However, selectivity for He and H_2 is high because these gases have a thermal conductivity approximately 6 times higher than Air. Thus, TCDs are suitable for He or H_2 measurement.

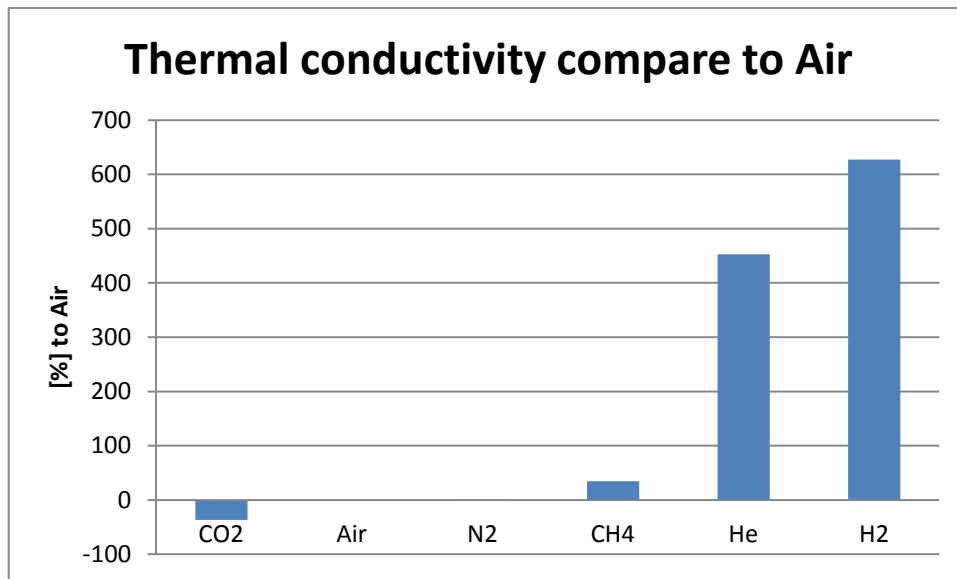


Figure 4: Comparison of thermal conductivity of different gases.

Detectivity of TCDs is low for gases with similar thermal conductivities than Air. The detector may confuse N_2 or CO_2 with Air because they have similar thermal conductivities. For He and H_2 detection should be more reliable.

3.2 Applications of a Thermal Conductivity Detector

The main application of TCDs is measuring a gas by means of its thermal conductivity. However, TCDs are also utilized in other applications. In the following sub-sections two applications will be presented.

3.2.1 TCD as a Flow Sensor

One application of TCDs is as flow sensor. The work presented by [24] shows that a TCD can be used as a flow sensor. The TCD is heated by a current and the change in its resistance is measured by using a Wheatstone bridge. If the flow through the TCD increases, the temperature of the TCD at a certain input current decreases. As the resistance of the TCD is temperature dependent, the flow effect on the TCD can be measured. Thus, a flow sensor is achieved. Figure 5 shows the results achieved by [24].

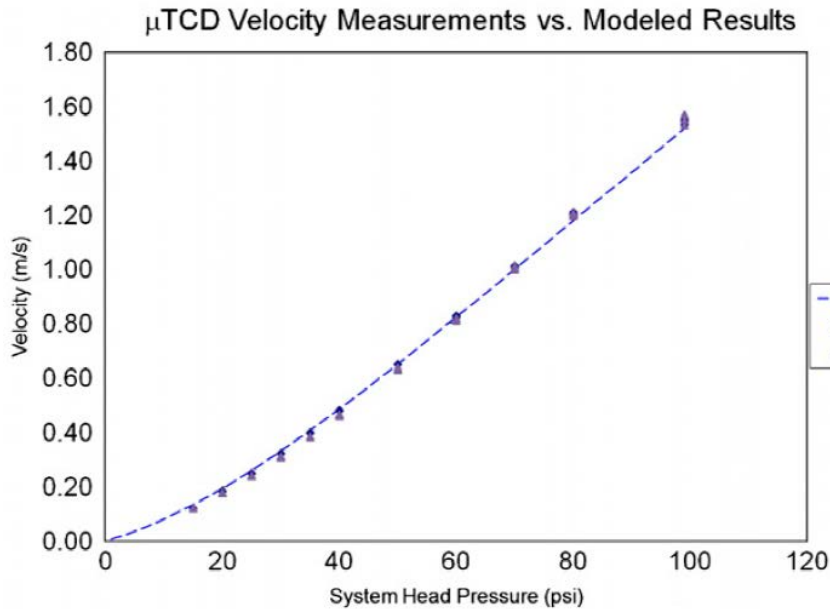


Figure 5: Flow rate measurements [24].

3.2.2 TCD as a Pressure Sensor

Another application of TCDs is as pressure sensor. The work of presented by [25] shows that the thermal conductivity of a gas is pressure dependent in a certain pressure range. Using the same principle to measure gas, thermopiles measure the temperature of the heater which depends on the thermal conductivity of the gas. As the thermal conductivity is pressure dependent, the temperature measured by the thermopiles is also pressure dependent. The graph of Figure 6 shows the pressure dependence of thermal conductivity of air at room temperature.

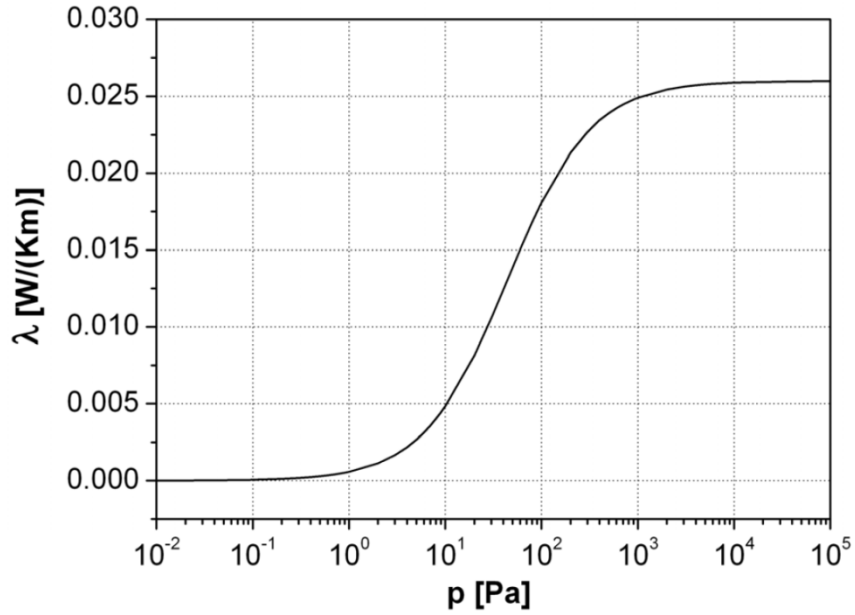


Figure 6: Application of a TCD as a pressure sensor. Measuring air at room temperature [25].

Figure 6 clearly shows that the TCD can be used as a pressure sensor in the range of 10 to 100 Pa.

3.3 State of Art of μ TCDs

In this section the state of art of different μ TCDs will be presented.

3.3.1 A Micromachined Thermoelectric Sensor for Natural Gas Analysis

A micromachined TCD is presented by [26]. This TCD is used to measure natural gas. The sensor is composed of a central heater and 40 aluminium-N type polysilicon thermocouples. The heater is fed by a current producing heat in the membrane which is measured by the thermocouples. The temperature measured by the thermocouples is proportional to the thermal conductivity of the gas.

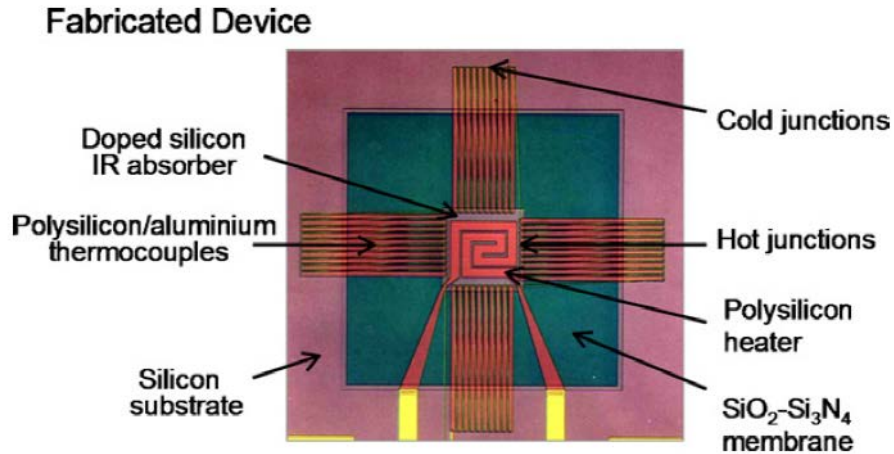


Figure 7: TCD for natural gas analysis [26].

Advantages of this TCD are: long term use, low cost, high sensitivity and fast response time.

Disadvantages of this TCD are: the fabrication of this device is complex because it uses bulk-micromachining which results in lower performance of the device. Also, the large quantity of thermocouples increases the thermal noise of the output.

3.3.2 Surface-Micromachined Thermal Conductivity Gas Sensor for Hydrogen Detection

The TCD presented in this section is the one used to design the dual-TCD to achieve a flow compensated device. This surface-micromachined TCD has been designed by [1] and also presented in [27]. The TCD is composed of a heated element and two thermopiles used as temperature sensors. The heater and the thermopiles are placed on top of a thin membrane made of silicon nitride. The heater is placed in the center of the membrane while the thermopiles are positioned at both sides of the heater. The structure of the membrane can be a bridge or cantilever. These structures provide the highest thermal isolation when using thermopiles as temperature sensors [27]. Underneath of the membrane a TEOS sacrificial layer is placed on top of the silicon substrate. The sacrificial layer is etched and a sample chamber is fabricated. The sample chamber has a depth of 4 μm . The sample chamber is reached by holes or slits made on the membrane. The TCD is shown in Figure 8: TCD using separated hot element and temperature sensor [27].

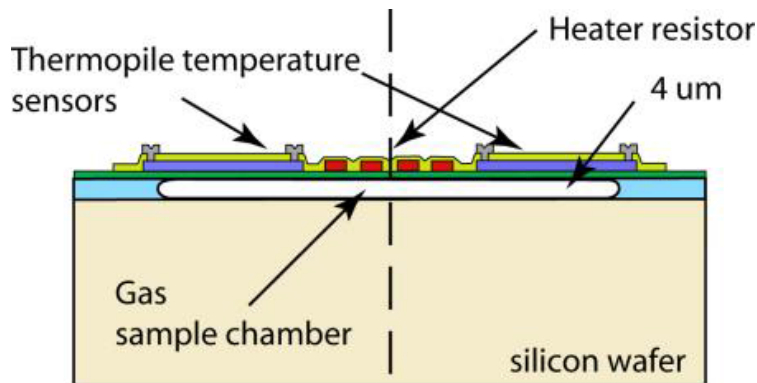


Figure 8: TCD using separated hot element and temperature sensor [27].

The heater is fed by using an electrical current producing Joule heating effect. The heat transfers through the membrane and through the sample chamber which is filled with the gas. An important heat loss is produced in the sample chamber which depends on the gas inside. In this way the temperature measured by the thermopiles depends on the gas inside of the sample chamber. Thus, depending on the thermal conductivity of the gas, certain temperature is measured by the thermopiles.

The sensitivity of the sensor to thermal conductivity mainly depends on the ratio between the heat flow through the gas and the heat loss through the supporting structure [27]. The long supporting beams have a much lower heat conduction than the sample chamber because of the large area and thin layer of gas of the sample chamber, even though the thermal conductivity of the beams are much higher than the gas. This has as a consequence that a low proportion of heat is lost in the beam and a high proportion of heat is lost in the sample chamber. This implies a high sensitivity for the thermal conductivity of the gas in the sample chamber.

Simulations made by [1] shows that the sensitivity highly depends on the depth of the sample chamber. Also,[1] has found optimal dimensions of the TCD to achieve maximal sensitivity to thermal conductivity of the gas. The optimal sensor sensitivity for hydrogen detection of 568.8 mV is achieved for 1.2 mW input power by utilizing a beam length of 280 μm and a gas path length of 20 μm . Also, the sensitivity can be improved by using a simple-clamped bridge structure with large gas openings. This increases the sensitivity by 5 times. Large openings allow to fill the sample chamber easily and increases the thermal isolation of the membrane[1]. The performance of this work is presented in Table 2 and it is compared to the sensor of section 3.3.1.

Table 2: Performance comparison of the TCD developed by [1].

Device	Sensitivity [mV]	Input Power [mW]	Power Sensitivity [V/W]	Detection limit	Stability
[26]	250 (Ethane in methane)	59.50	4.20 (Ethane in methane)	-	Good
[1]	648.8 (Hydrogen in air)	1.2	1.40 (Hydrogen in air)	0.0000214% (CO ₂ in air)	Good

3.4 Summary of the Chapter

The principle of thermal conductivity detector has been presented utilizing the simplest TCD: the hot-wire. The principle states that a thermal conductivity detector measures the thermal conductivity of the gas by means of a heated element in the gas path. The main components of a TCD are: heater, temperature sensor and (in some cases) a sample chamber.

The main application of a TCD is as gas sensor but it has also the potential to be used as flow sensor as well as pressure sensor.

The state of art of TCD has been presented in section 3.3 where two different TCDs have been explained. Both TCDs are composed of a resistor (heated element), thermopiles (temperature sensor) and a sample chamber. The device of [1, 27] has a better performance than [26] in terms of sensitivity and input power, for example. The device of [1, 27] is the starting point in the development of the flow compensated TCD, which will be explained in the following section.

Chapter 4 Flow Compensated Thermal Conductivity Detector

In this section the state of art of different devices and techniques to compensate the flow dependence will be presented. Then, our approach to obtain a flow compensated device using a dual-thermal conductivity detector will be defined. The approach starts presenting the topology of the dual-TCD, following by a qualitative description of the method as well as a quantitative description. Also, the heat transfer model of the dual-TCD is stated using a single TCD in section 4.3. Simulations using the heat transfer model are performed to analyse the behaviour of the dual-TCD. Finally, a summary of this section will be provided.

4.1 State of Art of Flow Compensated Thermal Conductivity Detectors

4.1.1 A Micro-Thermoelectric Gas Sensor

A micro-thermoelectric chemical sensor for hydrogen and atomic oxygen is proposed by [28]. The device was fabricated by using surface micromachining and is shown in Figure 9.

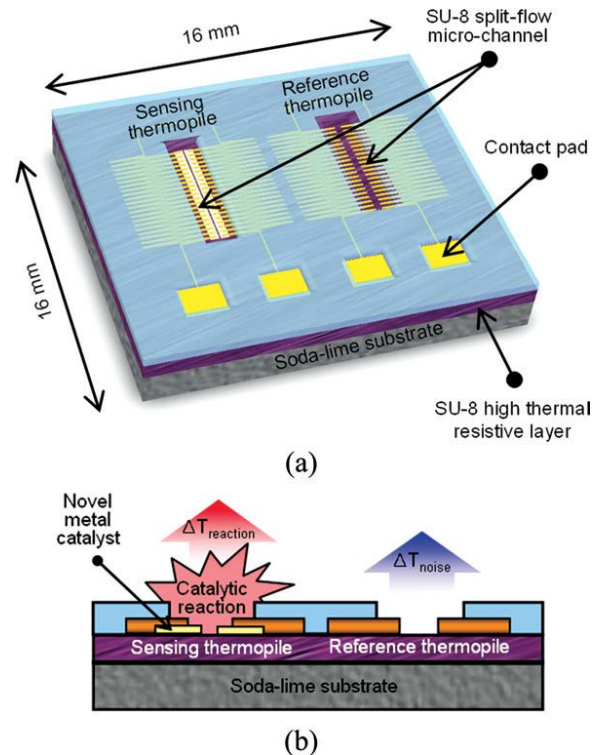


Figure 9: Proposed micro-thermoelectric gas sensor by [28]. (a) Topology of the gas sensor. (b) Simplified principle of the compensation method for reducing external thermal noise.

A thermoelectric material and a novel metal catalyst are fabricated on a layer of SU-8 which is highly thermally resistive to reduce heat transfer to the substrate [28]. The gas sensor detects a gas by measuring the

heat produced by the catalytic reaction between the gas and a novel gas-sensitive palladium catalyst. The difference in temperature is measured by thermopiles composed of copper (Cu) and bismuth (Bi).

As the sensor is vulnerable to temperature change due to temperature variations produced by the injected gas, a reference thermopile is placed on the same chip to compensate this temperature noise. The sensor is composed of two thermopiles: the sensing thermopile and the reference thermopile. The sensing thermopile is placed on top of the palladium catalyst measuring the temperature reaction while the reference thermopile does not have any reactive material underneath thus it measures the temperature of the fluid (temperature noise). Then, both voltage signal produced by the thermopiles are subtracted using a differential amplifier where the temperature noise is reduced.

Advantages and Disadvantages

This device does not need any heater because it measures the catalytic reaction between hydrogen and the palladium. Also, a simple read-out composed of thermopiles and a differential amplifier is used. The thermal noise is reduced by using a reference thermopile in the same chip.

However, to measure other gases the reactive material must be changed. Also, it has a slow response time due to the required temperature for palladium catalyst interaction needs to be constant. Moreover, as the device is a chemical sensor, it suffers from chemical contamination in long term use.

4.1.2 A Thermal Conductivity Detector Composed of Four Heaters

The approach proposed by [29] presents a μ TCD which has been designed for a μ GC. The μ TCD is composed of four thermistor thermal conductivity cells. Usually, a TCD is composed of two heaters, one is used as a reference and the other is used as the sensing element. However, [30] has demonstrated that using four heaters, two in the reference column and two in the sensing part, the sensitivity of the TCD is twice as sensitive as the one with two heaters. The μ TCD proposed by [29] has four thermistors suspended in the air flow channel covered by a glass wafer. The width and the depth of the heaters cells were the same as the flow channel, implying that the dead volume of the thermal conductivity cell was very small [29]. The four thermistor μ TCD is read using a Wheatstone bridge configuration as shown in Figure 10.

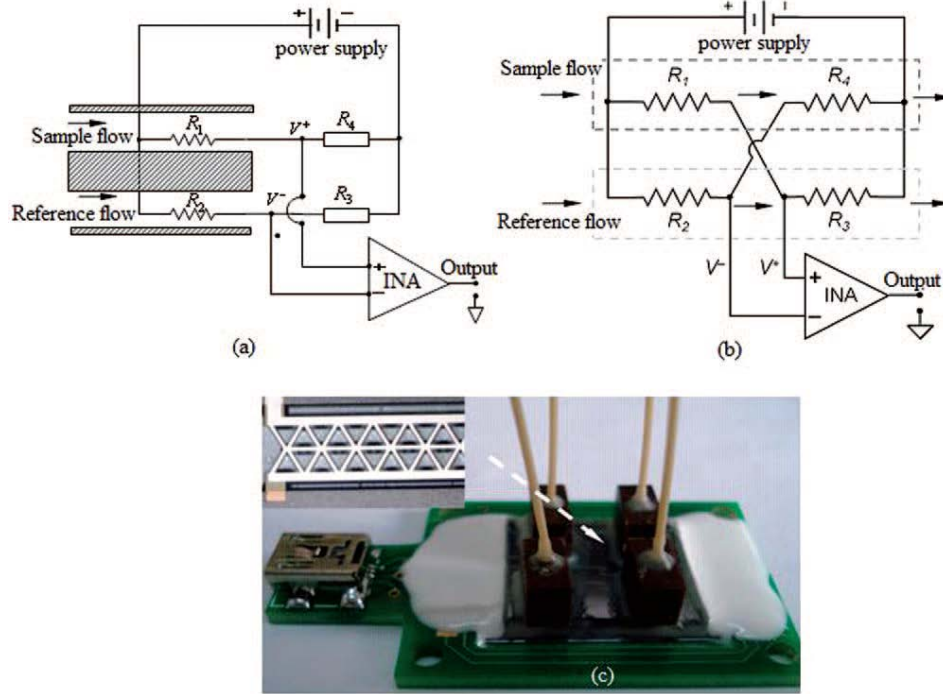


Figure 10: (a) Typical two heater TCD configuration. (b) The proposed topology using four heaters. (c) Photograph of the proposed μ TCD [29].

As shown in Figure 10 two different flow paths have been used, one as a reference and another where the sample gas flows. The output is read by an instrumentation amplifier. This configuration is typical to reduce the thermal noise produced by the fluid flow. An interesting simulation made by [29] shows the temperature response of each heater for different velocities of the flow. This simulation is shown in Figure 11.

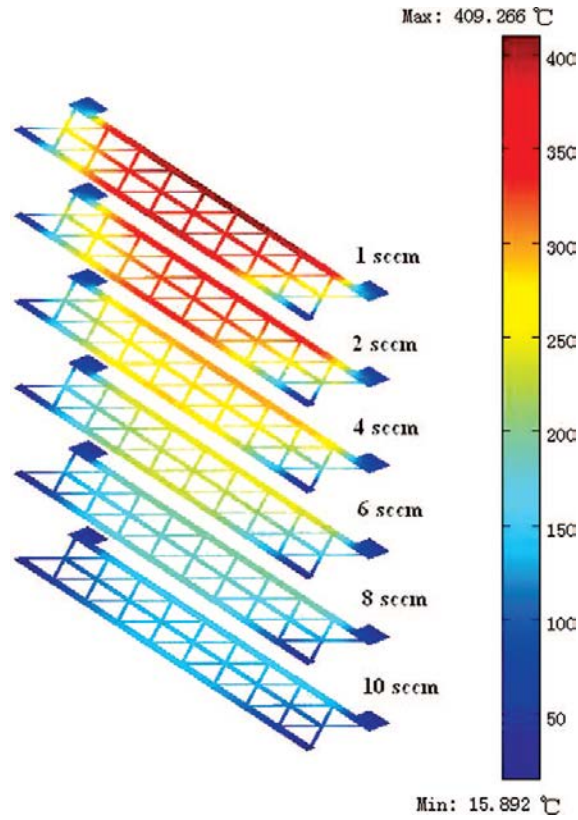


Figure 11: Temperature distribution on the thermistor when using a 500 ppm benzene mixed in H_2 with different velocities [29].

The temperature distribution of the thermistor for different flows rate shows that the temperature decreases when the flow increases. However, for flows in the range of 1 to 3 sccm the temperature variation seems to have a lower impact due to the flow. The work of [29] suggests that the velocity of the carrier gas should be below 3 sccm to avoid thermal noise.

Advantages and Disadvantages

One advantage of this design is the use of four thermistors instead of two, and by doing so, increasing the sensitivity of the TCD. Also, the glass wafer which covers the thermistors seems to be useful to have a good thermal isolation as well as laminar flow through the device. This could avoid any inconvenience caused by turbulences.

However, the use of four thermistor instead of two increases the power supply of the system. The proposed design still use a reference gas path to reduce the thermal noise produced by the flow.

4.1.3 A Flow Independent Multi-TCD for Gas Sensing

The approach proposed by [31] presents a TCD that is formed by several regions where each region is a TCD itself. Figure 12 shows this approach.



Figure 12: A flow insensitive TCD using several TCD regions [31].

Each region is mechanically and electrically independent, each capable of operating at a different temperature. The idea is that the flow is pre-heated in the first TCD region S1 which implies that the thermal gradient is fully developed in S2 and S3, as shown in Figure 13.

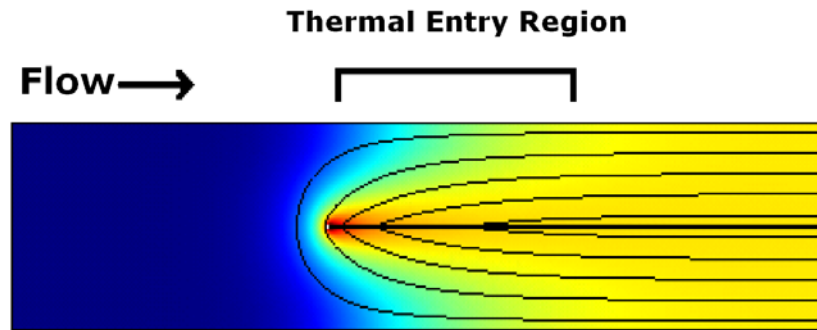


Figure 13: Temperature isotherm development. The flow is coming from left to right. The thermal entry region shows where the isotherms are not parallel to flow implying that the heat loss by convection cannot be dismissed [31].

In the regions where the isotherms are parallel with the heated element, out of the thermal entry region, the heat is transported to the surrounding gas as if it were stationary [31], making these regions mostly flow independent.

A model of the device has been done using COMSOL Multiphysics. The device is composed of four TCDs in series, each one having a heater pad of $500\ \mu\text{m}$, separated by $20\ \mu\text{m}$ and the flow channel has a height of $100\ \mu\text{m}$. The four TCDs are exposed to different flow rates (from left to right) in a range of $[0.25 - 3]\ \text{m/s}$. Finite element model (FEM) simulations were made by [31] and are shown in Figure 14.

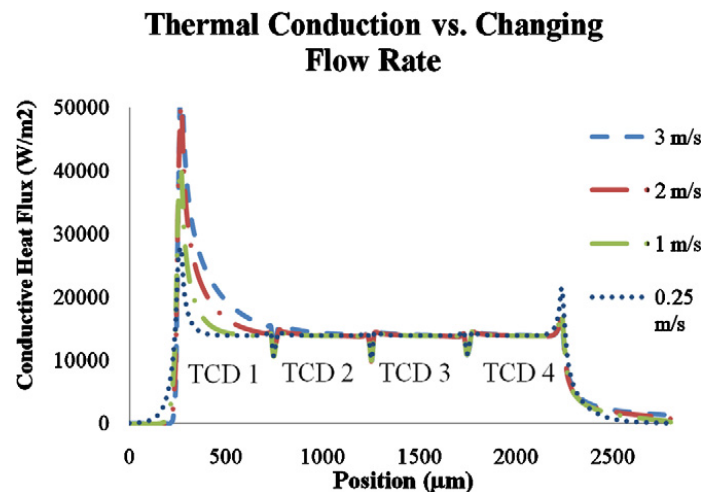


Figure 14: Effect of thermal heat flux of a TCD device when applying different flow rates [31].

Figure 14 shows that the central TCDs suffer from less flow dependence, especially TCD 3, in comparison to the TCDs at both extreme positions. The first TCD (TCD 1) is the most affected by the different flows, requiring more and more power to maintain a constant temperature. Also, TCD 4 suffers from some tailing effect at lower flow rates [31]. Table 3 shows the voltage response using two different sizes of the heater pad.

Table 3: Maximum voltage response of each TCD as a percentage of TCD 1 [31].

Device	TCD 2 [%]	TCD 3 [%]	TCD 4 [%]
500 μm TCDs	14.61	3.22	4.25
1000 μm TCDs	5.75	0.27	1.40

TCD 3 shows lowest flow dependence and also the size of the heater pad affects the flow dependence.

Advantages and Disadvantages

The main advantage of this design is that the design itself can reduce the impact of thermal noise without using a reference flow path. The TCD is placed in the sensing flow path and the flow has a small impact on the central regions. This is a big advance in comparison to the usual designs where two flow paths (reference and sensing) are used to reduce the effect of thermal noise. Also, the read-out of this design seems to be simple by using a Wheatstone bridge configuration.

However, the design uses at least four independent TCDs which implies a higher power consumption in comparison to usual designs where two TCDs are utilized. Also, as each TCD is independent of each other the electronic to control the temperature of each TCD is also multiplied by four implying more power consumption as well as the necessity of having four different controls.

4.1.4 Performance Comparison

The three TCDs presented in the previous sections are compared in terms of their main characteristics.

Table 4: Performance comparison of state of art flow compensated TCDs.

	Sensitivity [mV]	Power [mW]	Time response [s]	Sensitivity P/λ [W/W/m $\cdot$ K]	Flow Independent
[28]	160 (H ₂ in air)	Catalytic activation	7	-	Using a reference device in a reference flow path
[29]	20 (toluene)	12500	20	-	Until a flow of 3 sccm.
[31]	15	12	150 μs	-	Using a multiple TCD region, central regions flow independent

4.2 Dual-Thermal Conductivity Detector

The dual-TCD is composed of two quasi-identical TCDs fabricated in the same wafer. Each of these devices is a TCD itself but they have different functions in the entire structure. Each TCD is composed of a heater which is placed in the middle of its structure and thermopiles are placed to each side of the heater. Both the

heater and the thermopiles are put on top of a thin membrane. The heater acts as a heated element while the thermopiles as temperature sensors. Both TCDs have the same structure seeing from the top, as shown in Figure 15 (a). The thin membrane separates the substrate from the heaters and thermopiles. Each TCD has a sample chamber which is placed just underneath of the membrane, as shown in Figure 15 (b).

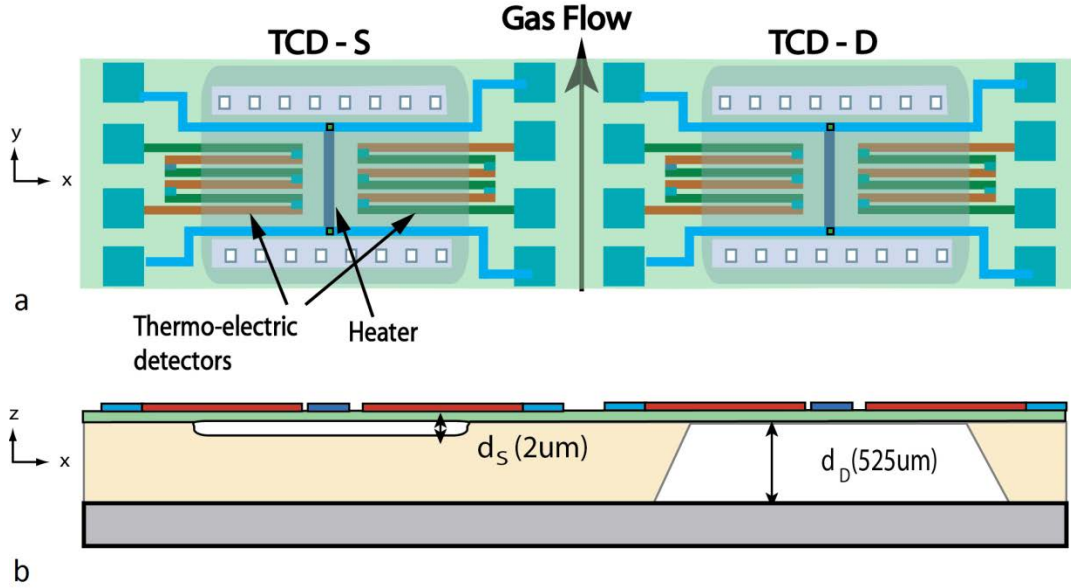


Figure 15: Dual-TCD device. (a) Top view of the device. (b) Cross section of the device.

These cavities have different depths and they define the functionality of each device in the dual-TCD. Each cavity is placed right underneath each heater (and also part of the thermopiles). These sample chambers are filled by the gas using holes or slits in the membrane. One of the sample chamber is very shallow while the other one is considerably deeper. In this case, the shallow cavity has a depth of $2\mu m$ and the deep cavity has a depth of $525\mu m$. The difference in the depth of the chambers affects the sensitivity of each device to the thermal conductivity of the gas. The shallow cavity TCD (TCD-S) is supposed to be more sensitive to the thermal conductivity than the deep cavity TCD (TCD-D) because the heat loss in the TCD-S is mainly in the cavity, through the gas, in comparison to the TCD-D where the heat is lost in the deep chamber as well as the membrane. In this way, the TCD-S is the one which actually measures the gas while the TCD-D is a compensation device.

In sum, the dual-TCD is composed of two identical TCDs next to each other, but with the only difference in the depth of the sample chamber which defines the function of each device in the dual-TCD. The TCD-S is more sensitive to the thermal conductivity of the gas than the TCD-D.

4.2.1 Description of the Flow Compensation Method Qualitatively

The fluid flows on top of the dual-TCD filling both chambers by means of slits or holes on top of the membrane. The heat in the entire structure is lost in three ways: by convection, by conduction on the membrane and by conduction through the sample chambers. Radiation is also a way to lose heat, however, this effect is negligible at this dimensions [1]. As the dual-TCD is composed of two identical devices viewing from the top, thus the heat loss by convection is the same on both TCDs. Besides, the holes or slits are made in such a way that the flow velocity inside of the chambers is very slow (close to zero), thus the heat loss by convection inside of the chambers is practically negligible.

On the other hand, the heat loss in the membrane must be very similar in both TCDs, as both devices are quasi-identical. Therefore only the heat loss by conduction through the gas remains.

Table 5: Comparison of both TCDs in terms of sensitivity, flow and structure.

Temperature of the membrane 310 K	TCD 2 μ m	TCD 525 μ m
Sensitivity (gas)	+	-
Flow on top of the membrane	Identical	
Flow on bottom of the membrane	Low	Very low
Structure	Identical	

Thus, if both devices are operated differentially with respect to the input power, a flow independent device might be achieved.

4.2.2 Description of the Flow Compensation Method Quantitatively

If both heaters are connected to a current source, they will produce heat by Joule heating effect. The heat spreads over the thin membrane where the thermopiles measure the temperature difference between the heater (via the membrane) and the substrate. The temperature of the membrane, as measured by the thermopiles, is kept constant at a certain value above ambient by controlling the current supply of the heaters. In this way, the heat loss of the TCD-S device and the TCD-D device is given by the total thermal conductance G which is proportional to the total power of each device. The total power of each TCD is given by:

$$P = \Delta T \cdot G \quad (17)$$

where ΔT is the temperature difference between the heater and substrate (ambient). Neglecting radiation effects, G can be split as follows:

$$G = G_b + G_g + G_c \quad (18)$$

with G_b the thermal conductance of the beam, G_g the thermal conductance through the gas, and G_c corresponds to the heat loss by convection. Replacing Eq. (18) in (17) the total power of each TCD is as follows:

$$P_S = \Delta T \cdot (G_{bS} + G_{gS} + G_{cS}) \quad (19)$$

$$P_D = \Delta T \cdot (G_{bD} + G_{gD} + G_{cD}) \quad (20)$$

where the subscript S corresponds to the TCD-S and D corresponds to the TCD-D.

Keeping both sensors at the same temperature and since the beam conductivity and the heat loss due to convection are identical, the terms G_c and G_b are equal and the power difference can be written as

$$\Delta P = P_S - P_D = \Delta T(G_{gS} - G_{gD}) \quad (21)$$

where G_{gS} corresponds to the TCD-S and G_{gD} corresponds to the TCD-D. The power difference depends on the difference in the thermal conductance in the sample chamber G_g of the TCD-S and the TCD-D device. At the same time, the thermal conductance of the gas G_g can be approximated by:

$$G_g = \frac{\lambda_g A}{d} \quad (22)$$

where λ_g is the thermal conductivity of the gas, A is the area of the membrane and d is the depth of the cavity. Substituting Eq. (22) in (21) results in the power difference, which is expressed as:

$$\Delta P = \Delta T \left(\frac{\lambda_g A}{d_S} - \frac{\lambda_g A}{d_D} \right) = \Delta T \lambda_g A \left(\frac{1}{d_S} - \frac{1}{d_D} \right) \quad (23)$$

Since the depth of the cavity d_D is much larger than d_S , the second term of Eq. (23) can be almost neglected. Operation at a constant temperature difference ΔT results in:

$$\Delta P \approx \Delta T \frac{\lambda_g A}{d_S} \quad (24)$$

Equation (24) shows that the power difference of both devices is proportional to the thermal conductivity of the gas, independent on the gas flow and the thermal conduction of the supporting structure. Equation (24) also shows that TCD-S is very sensitive to the heat loss through the gas while the TCD-D acts as a compensation device of the entire structure.

A flow independent thermal conductivity detector could be achieved by operating the dual-TCD differentially, without the necessity of having two different flow paths where the reference path is used to reduce the effect of flow.

4.3 Heat Transfer Analysis of the Dual-TCD

The heat transfer of the dual-thermal conductivity detector can be analysed by using the work of [2]. In [2] the absorption of the detector was calculated, but reversing this idea, it is possible to obtain the heat transfer of the detector.

In our case, as both TCDs of the dual-TCD have the same topology (with the only difference in the depth of the sample chamber), the heat transfer analysis can be done for only one TCD. Thus, the structure that will be analysed is shown in Figure 16.

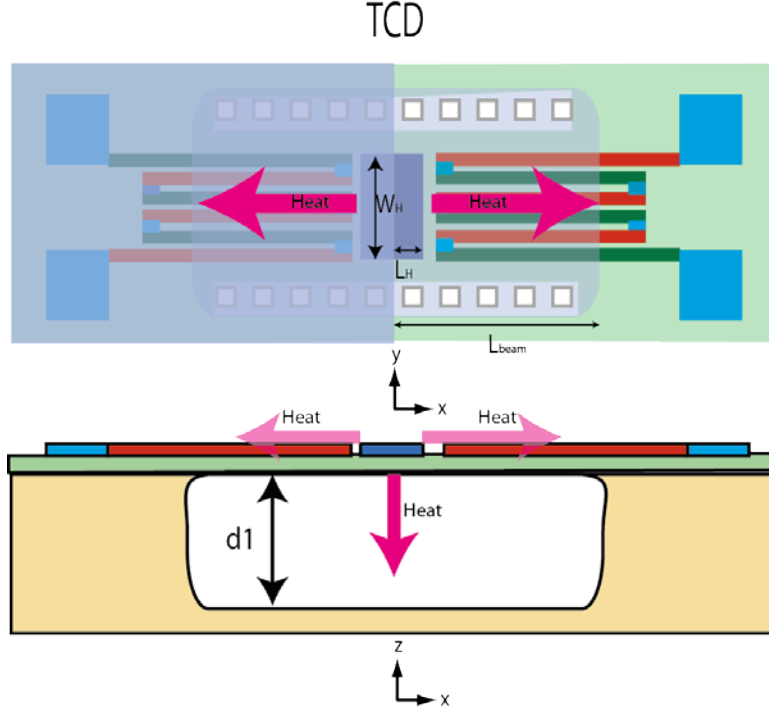


Figure 16: Heat transfer analysis of the dual-TCD by using half of the structure.

As the TCD of Figure 16 is symmetrical, the heat transfer analysis can be done by using the half of the structure[2]. The goal of this heat transfer analysis is to find the temperature distribution along the membrane. The total heat transfer depends on radiation, convection and conduction. Conduction is the most important heat transfer method at these micro-dimensions; radiation and convection only contribute in a small proportion to the total heat transfer[2]. The steps to calculate the temperature distribution along the membrane are explained in A.1. Thus, the analytical solution of the temperature distribution is expressed as:

$$T(x) = \left(\frac{P_{in}}{\beta \cdot W \sum \lambda_s t_s (\beta \cdot L_{beam} + \coth(\beta(L_{beam} - L_H)))} \right) \frac{\sinh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} + T_0. \quad (25)$$

Where P_{in} is the input power to the heater, W is width of the heater, $\sum \lambda_s t_s$ is summation of the product of the thermal conductivity and thickness of each thin film layer in the membrane (membrane + thermopile), L_{beam} is the length of the membrane, L_H is the length of the heater and T_0 is the ambient temperature. The term β refers to the heat transfer coefficient including radiation, conduction and convection terms and it is defined in Eq. (26).

$$\beta = \left(\frac{(4\varepsilon\sigma T_0^3 + \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) + h)}{\sum \lambda_s t_s} \right)^{1/2}. \quad (26)$$

Where d_1 is the depth of the sample chamber and d_2 is the distance between the top of the TCD and the encapsulation module.

Combining Equations (25) and (15) the output Seebeck voltage generated by the thermopile can be obtained. The output Seebeck voltage is as follows:

$$V_{out} = N\alpha_{ab} \left(\frac{P_{in}}{\beta \cdot W \sum \lambda_s t_s (\beta \cdot L_{beam} + \coth(\beta(L_{beam} - L_H)))} \right) \frac{\sinh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} \quad (27)$$

Equation above shows that the heat produced by the heater is totally coupled to the voltage produced by the thermopiles.

Using Equation (25) simulations of the temperature distribution along the membrane as well as the sensitivity to thermal conductivity of the gas can be done for the dual-TCD. For those simulations parameters of Table 6 are utilized.

Table 6: Parameters used to simulate temperature vs. d_1 for different gases.

Parameter	Value	Gas	Thermal conductivity λ [W/m·K]
P_{in} [mW]	5	CO ₂	0.01622
W_H [μ m]	154	Air	0.02570
L_H [μ m]	14	CH ₄	0.03458
L_{beam} [μ m]	106	He	0.14200
t_s [μ m]	0.6	H ₂	0.18690
x [μ m]	40		
d_2 [mm]	1		
T_0 [K]	300		
λ_s [W/m·K]	20		

The first simulation is to realize the sensitivity to thermal conductivity of both TCDs of the dual structure for a constant temperature at 40 μ m from the center of the heater. Results are shown in Figure 17.

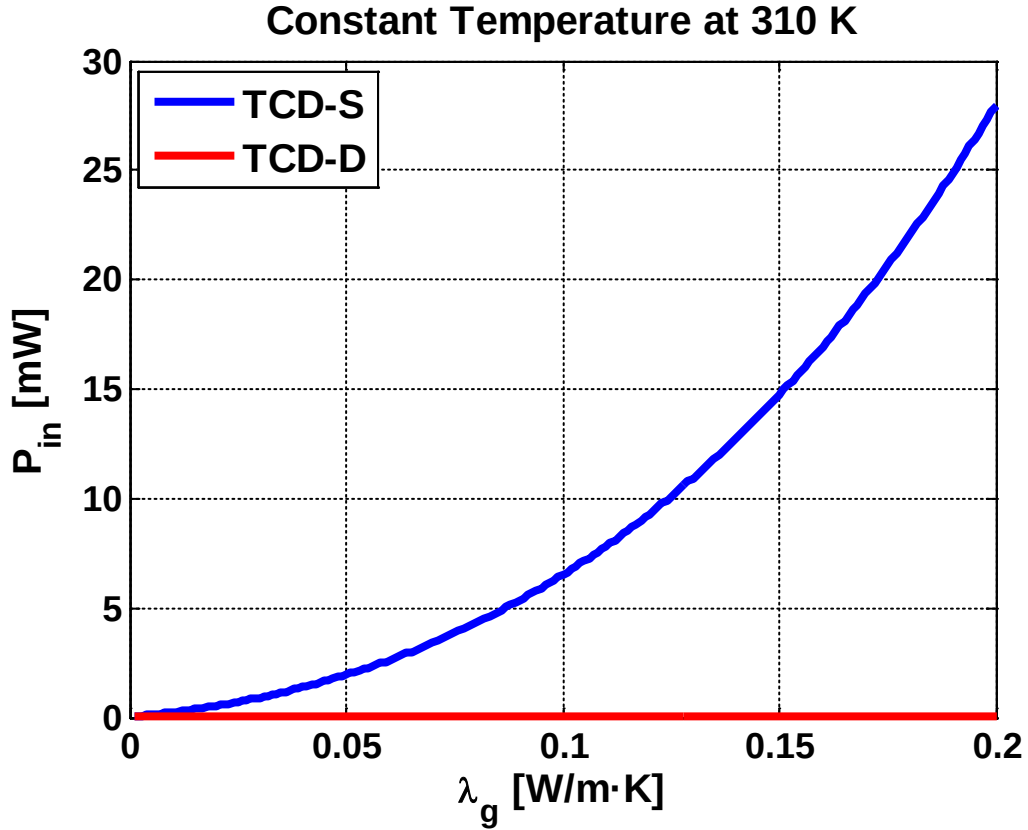


Figure 17: Simulation of P_{in} vs. λ_g maintaining a constant temperature of 310 K at 40 μm from the center of the heater.

Simulation shows that the TCD-S needs more power to maintain a constant temperature than the TCD-D. This means that the TCD-S is much more sensitive to thermal conductivity changes than the TCD-D. The approximate sensitivity of the TCD-S S_S is equal to 0.133 [W/W/m·K] while the sensitivity of the TCD-D is $S_D = 0.001$ [W/W/m·K]. This confirms the idea of using a TCD with a shallow cavity as sensing element and a TCD with a depth cavity as a compensation device.

Another simulation that can be done using Equation (25) is to see the temperature behavior at 40 μm from the center of the heater varying the depth of the sample chamber for different gases for a constant power. Results are shown in Figure 18.

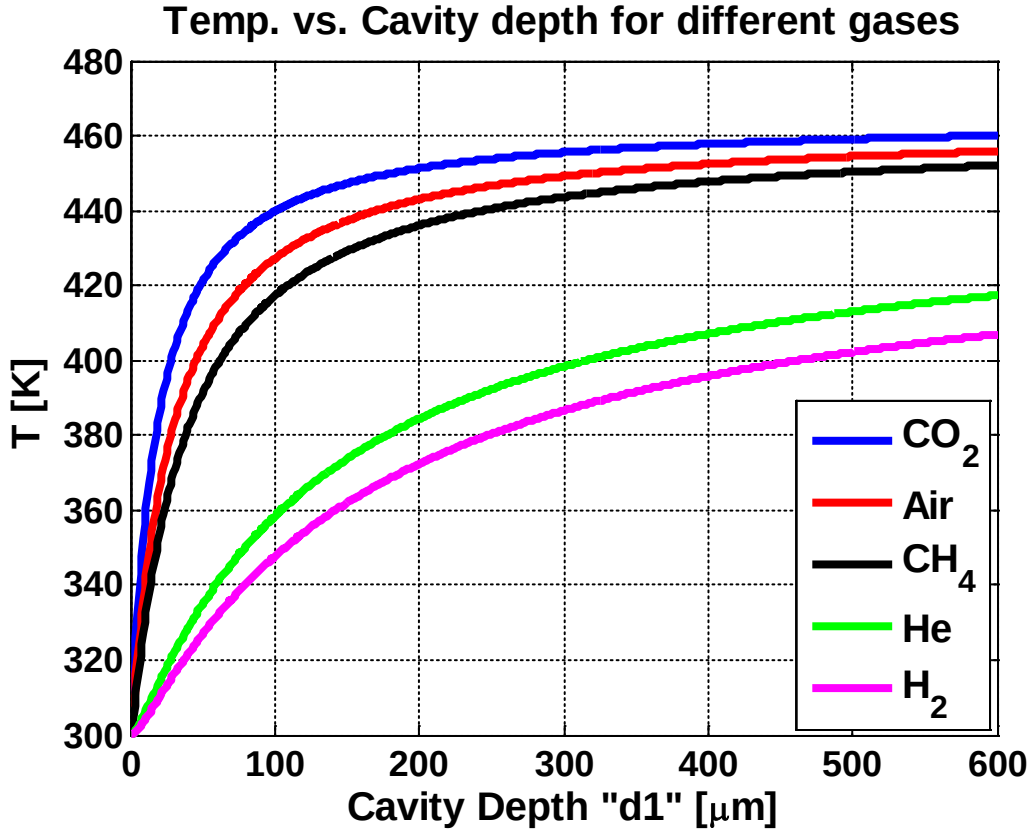


Figure 18: Simulations of the temperature of the membrane vs. cavity depth for different gases without convection.

Simulations show that the temperature behaviour for all gases depends strongly on the depth of the sample chamber. The temperature on the membrane is very sensitive to d_1 smaller than $50 \mu\text{m}$. The temperature starts achieving a constant value for values of d_1 higher than $400 \mu\text{m}$. The same behaviour is seen for all gases, but especially for CO_2 , Air and CH_4 , gases with lower λ than He and H_2 .

Gases with higher λ have a higher heat loss. This has as a consequence that more power is required to reach the same temperature level than gases with a lower λ .

4.4 Summary of the Chapter

The state of art of flow compensated thermal conductivity detectors has been presented. Two TCDs use one TCD in the gas stream and another one in a reference path. The TCD in the reference path is used to measure the effect of flow on the TCD. Then, this effect is utilized to compensate the device on the gas stream. In this way it is possible to reduce the effect of flow on the measurements.

On the other hand, [31] presented a novel TCD for gas sensing using multi-region TCDs to reduce the effect of thermal noise. In this case the entire sensor is placed in the gas stream, without the necessity to use a reference path. The effect of flow is reduced especially in the central regions where the TCDs are almost flow independent. This device has good features like a power consumption of 12 mW and a time response of $150 \mu\text{s}$.

A novel flow compensated idea using a dual-TCD topology has been presented. The dual-TCD is composed of two quasi-identical TCDs with the only difference in their sample chamber depth. One of the sample chamber is very shallow while the other is very deep. As both TCDs are almost identical, when they are differentially operated the heat transfer only depends on the conduction through the chambers.

The heat transfer over the membrane of the dual-TCD has been shown. Simulations of this relation show that for a cavity deeper than 400 μm the temperature tends to stabilize to a constant value while for cavities narrower than 200 μm the temperature is very sensitive to the deep of the cavity.

In this way the flow compensated dual-TCD fulfills the following conditions to achieve the flow independence:

- **Both TCDs have a different static sensitivity to the thermal conductivity of the gas (due to different gas sample chambers).**
- **The flow inside of the chamber is negligible.**
- **Both devices are exposed to the same gas stream (not using two different gas stream).**
- **TCDs operate at the same membrane temperature.**

Chapter 5 Fabrication

5.1 Design Characteristics

In micro thermal conductivity detectors is very important to achieve a high sensitivity to thermal conductivity of the gas. Thus, reducing the heat loss in the membrane is important. Floating membrane structures have shown a good performance in terms of heat loss. These structures can be made by using bulk-micromachining process or surface-micromachining process. However, bulk-micromachining is a more complicated process than surface-micromachining, lowering the yield of devices. On the other hand, surface-micromachining can make more complex devices in comparison to bulk-micromachining by using cheaper mechanical elements [1]. Thus, to reduce the heat loss of the structure a floating membrane is chosen by using surface-micromachining.

The dual-TCD is composed of two heaters, four thermopiles and one membrane. Both heaters are made of P-type polysilicon because it has good linearity to temperature variation and low thermal conductivity. Moreover, it is compatible with surface-micromachining process.

The thermopiles are made combining P-type polysilicon and N-type polysilicon. Both types of polysilicon have a relatively high Seebeck coefficient: $+170 \mu\text{V/K}$ for P-type and $-120 \mu\text{V/K}$ for N-type. If both materials are used to make a thermocouple a total Seebeck coefficient of $+290 \mu\text{V/K}$ is achieved. Each thermopile is composed of 12 thermocouples connected in series. Thus, the total Seebeck coefficient of each thermopile is $3480 \mu\text{V/K}$.

As it was stated before a floating membrane is chosen for the dual-TCD. The membrane is made using silicon nitride due to its good thermal conductivity properties.

5.2 Fabrication Process Steps

The dual-TCD has been made in DIMES facility at TU-Delft. The sensor has been fabricated using surface-micromachined MEMS technology. The sensor is fabricated on a $525 \mu\text{m}$ silicon wafer. On top of the Si wafer a $2 \mu\text{m}$ layer of silicon-dioxide is deposited and reflowed. This TEOS is used as a sacrificial layer [27]. The membrane is made by using a layer of 700 nm of low-stress silicon nitride and is deposited on top of the sacrificial layer. Then, a $0.3 \mu\text{m}$ thickness layer of polysilicon is placed on top of the SiN. In the polysilicon layer the heaters and thermopiles are patterned. After that, the structure is protected by a $0.3 \mu\text{m}$ layer low stress of SiN. After patterning, the N-type polysilicon and the P-type polysilicon are doped and the dopants are activated by annealing. The contacts are opened and a deposition of Al/Si 1% is made to create the contacts of the device in step number 7. Holes of $20 \mu\text{m}$ by $20 \mu\text{m}$ are etched in the membrane using vapour HF. Then the deep cavity is made by using backside DRIE etch. In the final step the shallow cavity is created using HF at 75% freeze dried or supper critical dried. The fabrication flow is shown in Figure 19.

1. TEOS deposition 2 μm & Reflow



2. Membrane layer low stress: Silicon Nitride 700 nm



3. Conductive layers: PolySilicon 300 nm



4. Patterning & Doping PolySilicon



5. Protective Membrane layer low stress: Silicon Nitride 300 nm



6. CoN + doping, CoP - doping and annealing to activate dopants



7. Contacts openings, deposition of Al/Si 1%



8. Access holes etching



9. Backside DRIE etch: Deep cavity



10. HF 75% freeze dried or supper critical dried: Shallow cavity



Figure 19: Fabrication flow of the dual-TCD.

Two types of dual-TCD have been fabricated: with a short thermopile and with a long thermopile. In the case of the short device the dimension of the TCD is $330 \times 350 \mu\text{m}$ (Figure 20). In the case of the long device the dimension of the TCD is $600 \times 350 \mu\text{m}$ (Figure 21).

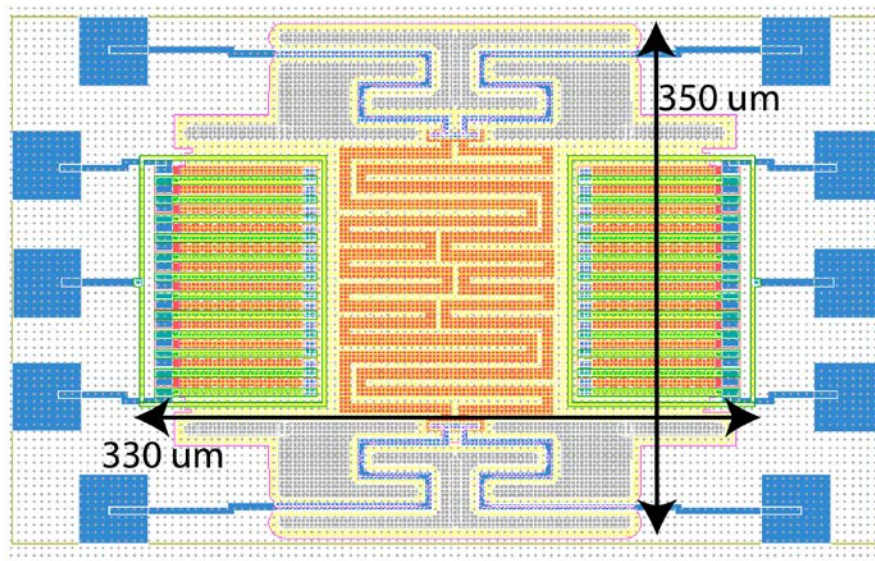


Figure 20: Width and length dimensions of the short TCD.

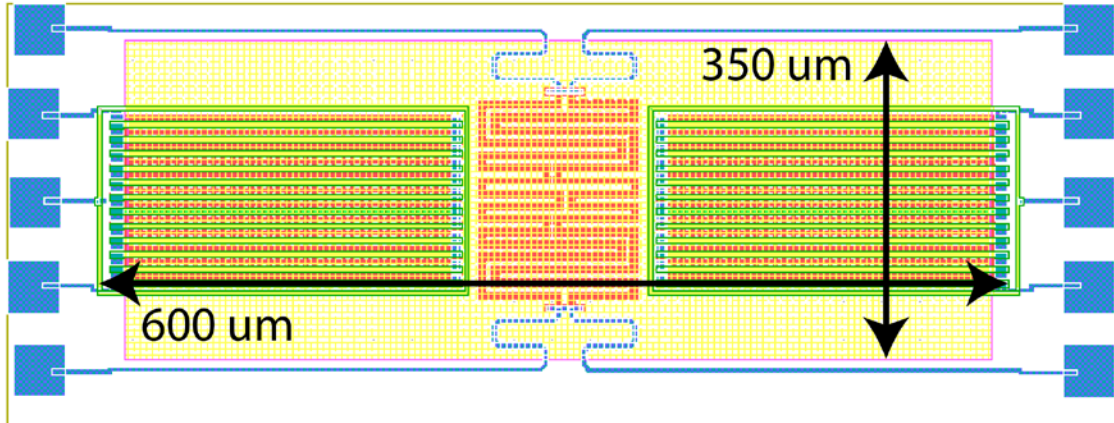


Figure 21: Width and length dimensions of the long TCD.

Different topologies of the dual-TCD have been designed using a bridge or a membrane, as shown in Figure 22 and Figure 23.

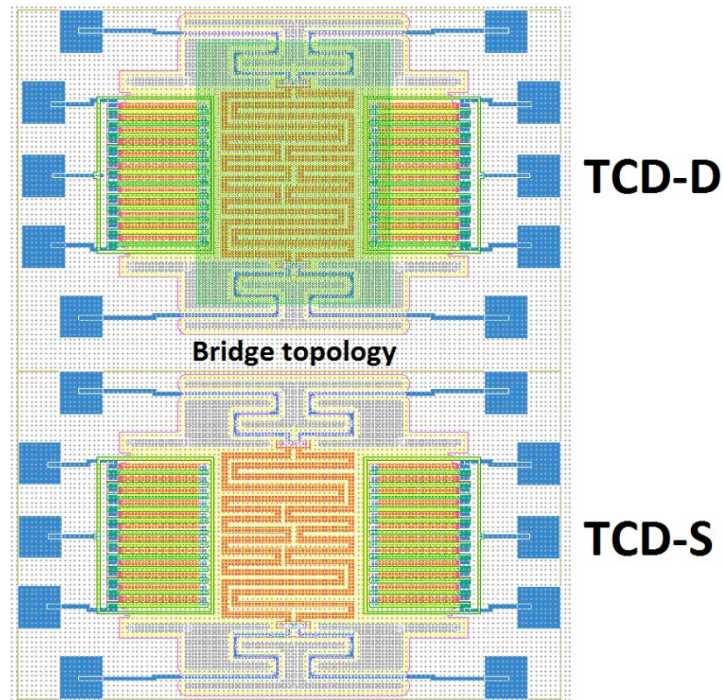


Figure 22: Bridge configuration of the TCDs.

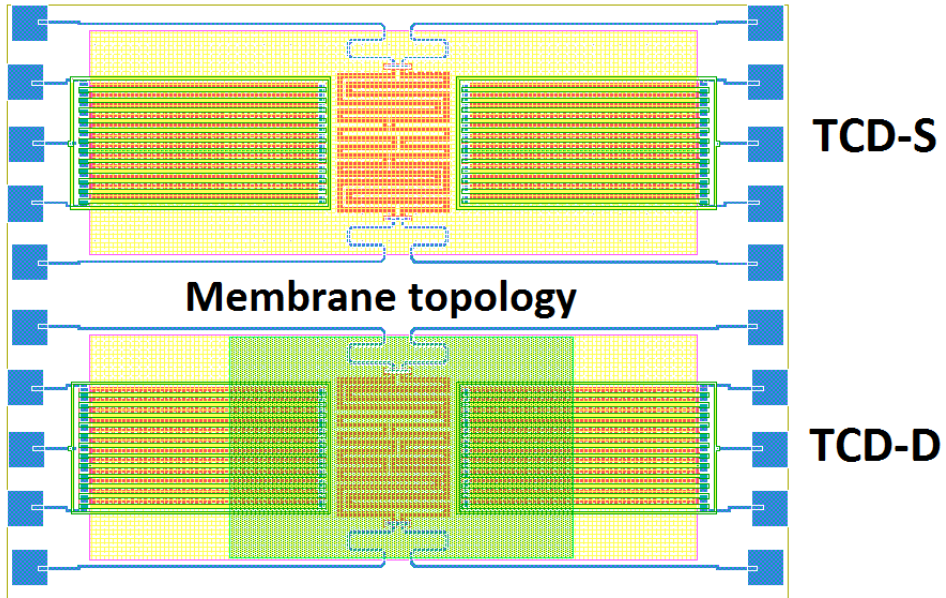


Figure 23: Long dual-TCD using a membrane topology.

In total 7 short dual-TCD and 5 long dual-TCD has been fabricated.

Chapter 6 Simulations

Simulations have been performed to characterize and verify the behavior of the flow independent dual-TCD device. The dual-TCD has been designed and simulated using Comsol Multiphysics v4.4. Comsol is a powerful software where different kinds of physical domains can be simulated using finite element method (FEM). Electrical, mechanical, fluid and chemical domains can be simulated; even they can be combined if it is necessary. More information about Comsol can be found in [32].

The dual-TCD has been designed in Comsol following the same logic “layer by layer” presented in section “Fabrication”. The dimensions of the dual-TCD designed in Comsol are: width of 154 μm , length of the membrane 464 μm , width of the heater 28 μm , shallow cavity 2 μm and deep cavity 525 μm . The physical properties of each material used in Comsol are:

Table 7: Physical properties of the materials used in Comsol.

Parameter	Thermal conductivity λ [W/m·K]	Density ρ [kg/m ³]	Heat capacity C_p [J/kg·K]
Silicon	131	2329	700
Silicon-dioxide	1.4	2200	730
Silicon nitride	20	3100	700
PolySilicon	34	2320	678

The model of the dual-TCD designed in Comsol is shown in Figure 24.

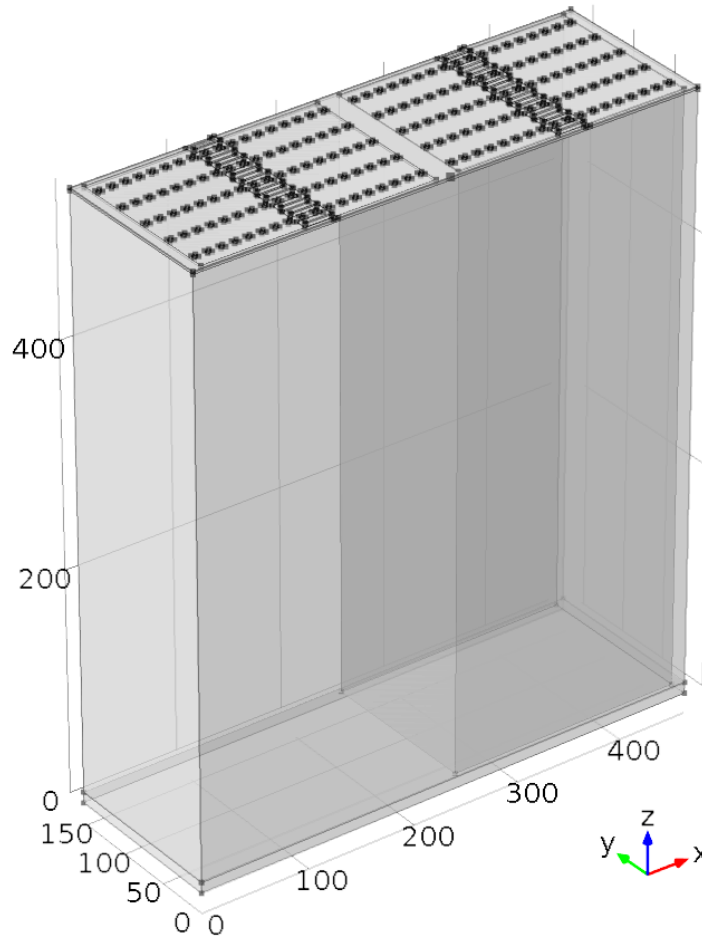


Figure 24: Dual-TCD designed in Comsol. The left TCD corresponds to the shallow cavity device while the right TCD is the deep cavity device.

Simulations have been divided into two groups: Static and Dynamic. The difference between them lies in the use of an input gas flow passing through, an especially, on top the sensor. Static simulations were made without an input flow while dynamic simulations were made using different input flows for different gases. In the following sections static and dynamic simulations are presented, explaining the modules that have been utilized and results.

6.1 Static Simulations

Static simulations have been made to verify the response of the dual-TCD for different gases. As it was stated in 4.2 the TCD with the shallow cavity should be more sensitive to different gases than the TCD with the deep cavity. In other words, the goal of these simulations is to verify the sensitivity to thermal conductivity of each TCD.

To verify the sensitivity to thermal conductivity of the dual-TCD it is necessary to simulate the heat transfer behavior in combination with the electrical behavior of the device. Comsol provides a module which combines electric and heat transfer behaviors, this module is called “Joule Heating”. The controlled variable of this module is the current density (or current) or voltage. Utilizing one of these variables the Joule heating effect of structure is simulated.

Static simulations were performed using the method described in the following paragraph. The controlled variable of the static simulations is the current. This is used to feed both heaters. The heat produced by the heaters spreads through the membrane where the temperature difference between the heater and the substrate is measured at the same position where thermopiles are placed (44 μm from the center of the heater). In the case of the model made in Comsol, thermopiles are not designed, but using properties of the software, the temperature is measured in the positions where the thermopiles are supposed to be placed. The idea of these simulations is to keep the temperature at the position of the thermopiles at 310 K by means of the controlled current for different gases. Gases fill the sample chambers of both TCD and surround the entire sensor as well. Table 8 shows the physical properties of the gases that have been used.

Table 8: Physical properties of the gases used in simulations.

Gas	Thermal conductivity λ [W/m·K]	Density ρ [kg/m³]	Heat capacity C_p [J/kg·K]	Dynamic viscosity μ [μPa·s]
CO2	0.0162	845.7	1.8150	0.151
Air	0.0257	1005	1.2050	0.180
N2	0.0261	1041	1.1382	0.179
CH4	0.0346	2226	0.6568	0.111
He	0.1420	5193	0.1785	0.190
H2	0.1869	14310	0.0825	8.890

A simulation of the dual-TCD has been made. This simulation is to verify heat behavior over the membrane. Both heaters are fed by the same current of 0.4 mA and the gas is Air.

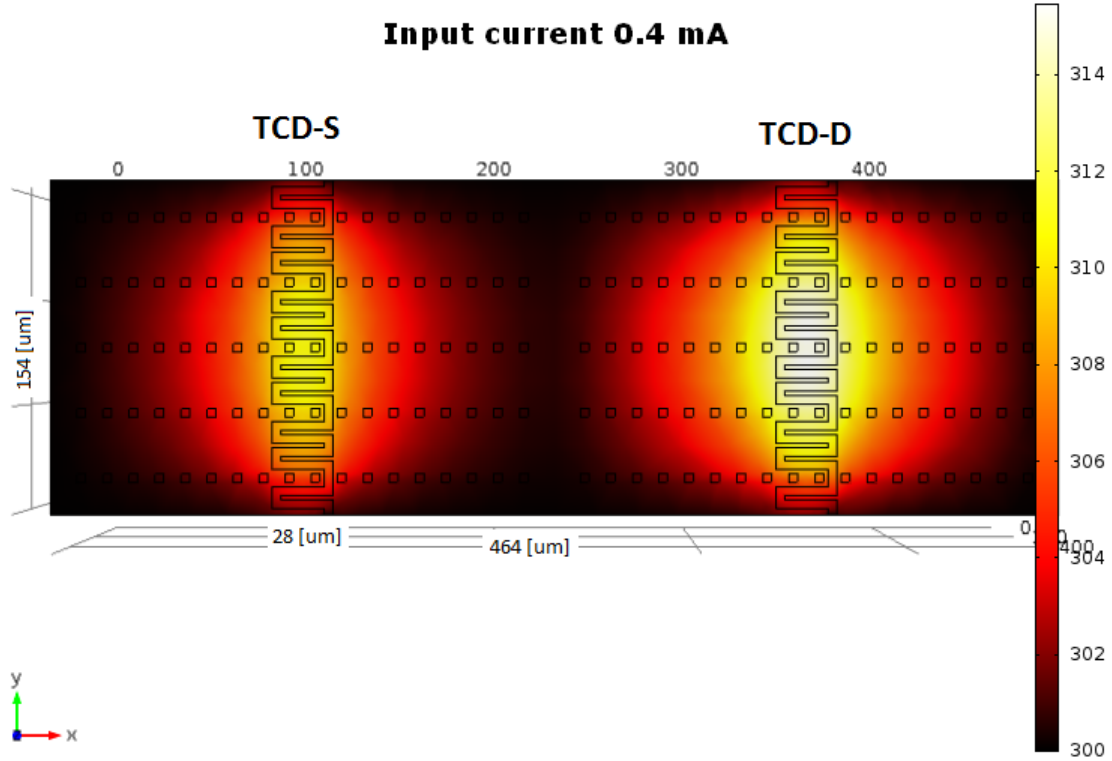


Figure 25: Heat behavior of the dual-TCD. Both heaters have been fed using an input current of 0.4 mA.

Figure 25 show that both heaters achieve different temperature using the same input current. The heater corresponding to the deep cavity device (right side of Figure 25) achieves a higher temperature in comparison to the shallow cavity device (left side). This difference in the behavior is mainly due to the different depth of the cavities. In the case of the TCD-S there is a huge heat loss through the gas in the cavity, while in the TCD-D an important part of the heat is lost in the membrane itself. This behavior of the dual-TCD was predicted in section 4.3 using Equation (25) and shown in Figure 18. In that case, by using a constant input power the temperature achieved by shallow cavities is lower than temperatures achieved by deep cavities. One important deduction of this result is that the TCD-D needs to be fed by a smaller current than the TCD-S to reach the same temperature.

Another important result of this simulation is that, the heat over the membrane spreads in a different way on both TCDs. The oval shape around the heater of the TCD-S is narrower than the oval around the heater of the TCD-D. The reason is also due to the way in how the heat is lost in the entire structure. As it was stated previously, the heat loss in the TCD_S occurs mainly though the gas in the cavity instead of the membrane where a small part of the heat is lost. This makes that the heat transfer profile on the membrane becomes narrower in comparison to the TCD-D device. One important consequence of these different profiles is that, probably the sensitivity of both TCDs to a flow on top of the structure might be different and the temperature should be measured closer to the heater.

On the other hand, if both cavities of the dual-TCD and the surroundings are filled with different gases, the total power of each TCD for maintaining a constant temperature of 310 K at 44μm from the heaters is shown in Table 9 and the results are plotted in Figure 26.

Table 9: Static response of the dual-TCD. Required heater power for different gases for maintaining a constant temperature of the thermopile of 310 K.

Gas	Thermal conductivity λ [W/m·K]	SC TCD P [mW]	DC TCD P [mW]
Vacuum	0	5.29	5.29
CO ₂	0.01622	7.81	5.38
N ₂	0.02604	9.34	5.50
CH ₄	0.03458	10.67	5.60
He	0.14200	25.71	6.89
H ₂	0.18690	30.69	7.44

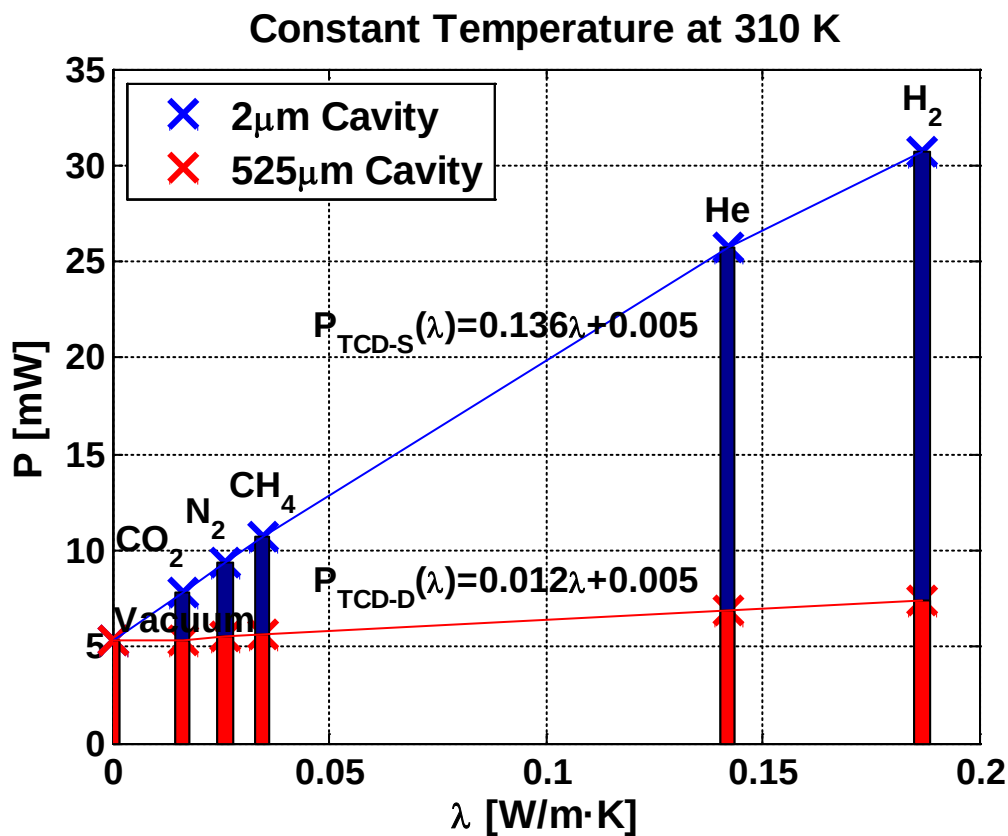


Figure 26: Simulation of the static response of the dual-TCD. Total power of each TCD vs. thermal conductivity.

The results presented in Table 9 show that there is a power consumption in vacuum ($\lambda=0$) which is due to the effect of the conductivity of the suspension of the membranes (G_b). On the other hand, using different gases, the results show that much more power is required to maintain a constant temperature in the TCD-S than in the TCD-D. This is due to the large heat loss through the thin layer gas in the shallow cavity. As a result the TCD-S is much more sensitive to the gas thermal conductivity than the TCD-D, which basically acts as a compensation device to keep the membrane at certain temperature. These results confirm the ideas concluded in 4.2.

Figure 26 plots the results of Table 9 and shows the relation between the power dissipation and the gas thermal conductivity of both devices. This relation is almost linear, in both cases, having the following relations:

$P_{TCD-S}(\lambda) = 0.136 \cdot \lambda + 0.005$ [W] with a correlation coefficient $R^2 = 0.998$ and $P_{TCD-D}(\lambda) = 0.012 \cdot \lambda + 0.005$ [W] with $R^2 = 0.998$. The sensitivity of the SC device is $S_\lambda = 0.136$ W/(W/m·K), while the sensitivity of the DC device is only $S_\lambda = 0.012$ W/(W/m·K).

The TCD-S has a sensitivity 11 times higher than the TCD-D to the thermal conductivity of different gases.

The power obtained in the static simulations can be used as a starting point of the dynamic simulations.

6.2 Dynamic Simulations

Dynamic simulations have been performed to verify the flow compensation idea of the dual-TCD as well as the time response of the sensor. These simulations were made using the “Joule Heating” and the “Laminar Flow” modules in Comsol Multiphysics V4.4. Both modules were coupled by simulating first the laminar flow and then using these results to simulate the heat transfer of the sensor.

In this kind of simulations there are two controlled variables: the current density and the flow. As in the case of static simulations, both heaters are fed by different currents keeping the temperature at the thermopile position at a constant temperature of 310 K, but in this case there is also a gas flow passing through the sensor. As a feeling one expects that the gas flow decreases the temperature of the structure due to forced convection. This should have as a consequence that if the flow speed increases, then the current density (power) should also increase to keep the membrane at a constant temperature. The range of the flow is from 1 mL/min to 10 mL/min.

Before presenting the power behavior of the dual-TCD, the flow inside of the cavities has been simulated to verify if the flow is negligible or not.

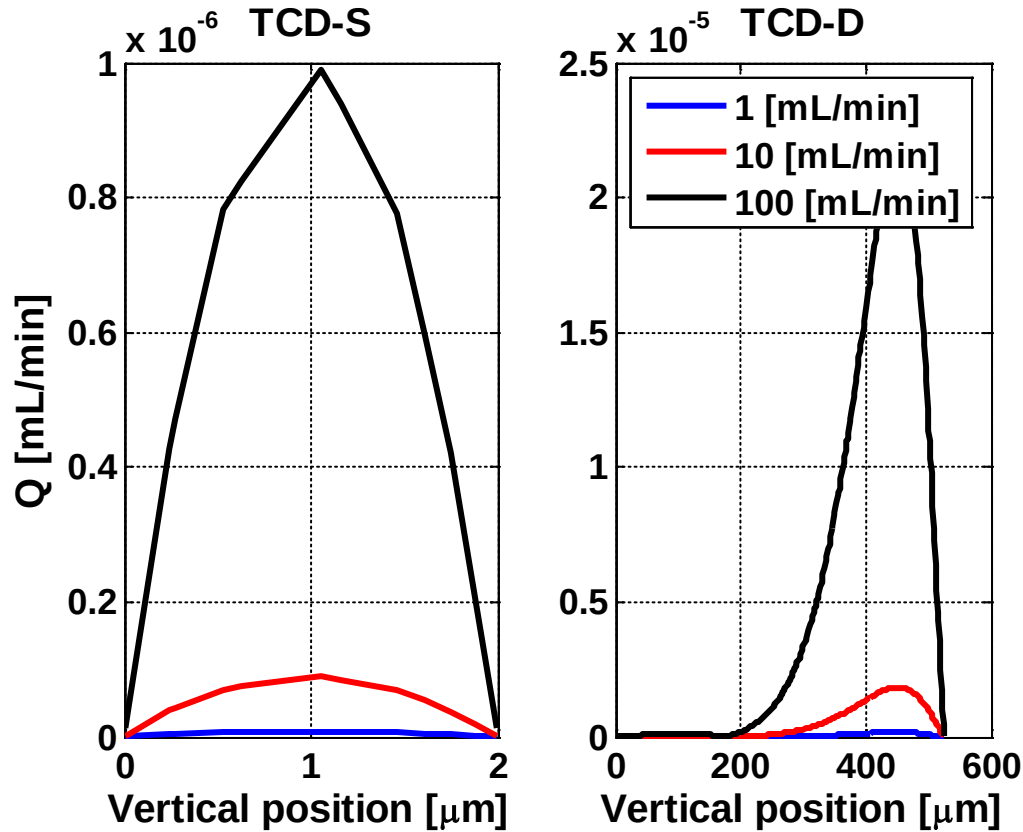


Figure 27: Flow inside of the cavities. The flow in both cases is very low in the order of nL/min.

Figure 27 shows that the flow inside of both cavities is very low in the order of nL/min. The flow in the deep cavity reaches a higher value than in the shallow cavity, as it was expected. This simulation confirms that the dimensions of the holes ensure that the heat loss by convection inside of the cavities is negligible.

Another simulation is the time response to fill both sample chambers of the dual-TCD. This time has been calculated as the time needed to reach a constant flow inside of the cavities. As the holes for both TCDs have the same size, the deep cavity should require more time to reach a constant flow than the shallow cavity due to it takes more time to fill it. Thus, the time response of the system is calculated for the TCD-D device. The input flow to the system is 10 mL/min and simulations have been done for different sizes of the holes.

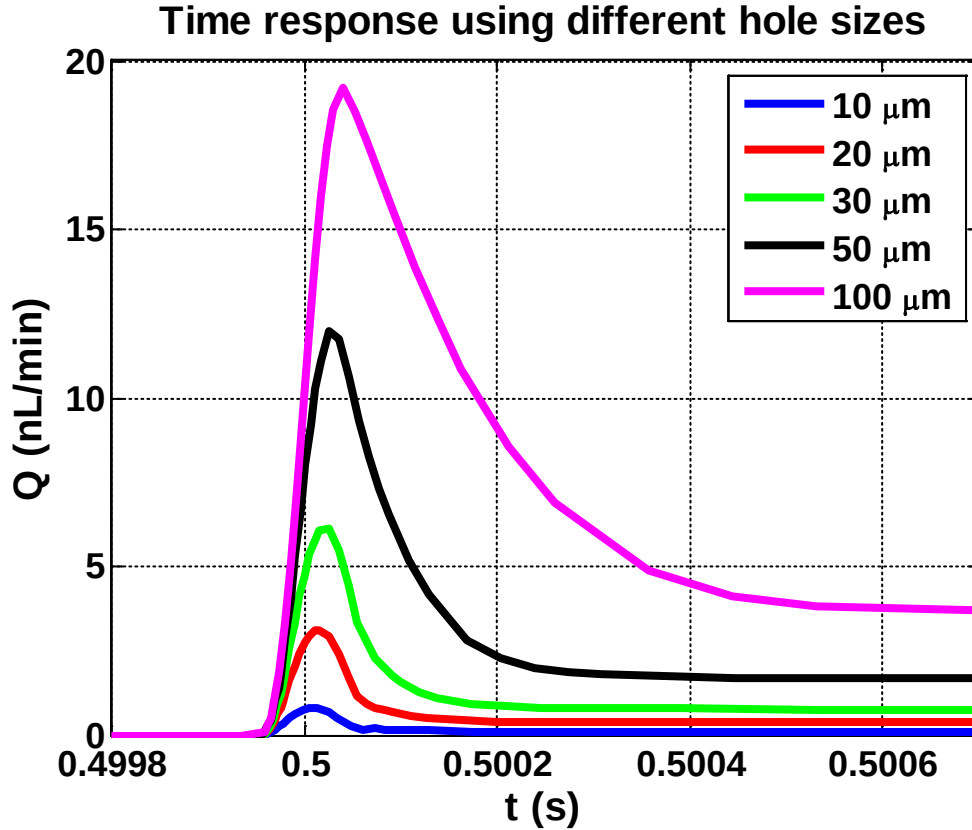


Figure 28: Time response of the dual-TCD device for different holes sizes for an input flow of 10 ml/min to the system.

As Figure 28 shows, different time responses have been obtained depending on the size of the hole. It shows that for bigger hole sizes it takes more time to reach a constant flow in the cavity, which makes sense because bigger holes allows more gas to flow inside of the cavities. The time response in the case of 20 μm hole size is $t_G = 0.4$ ms, which corresponds to the time response of the dual-TCD.

On the other hand, results of the power behavior of the sensor when it is exposed to different flows are presented. In the case of the TDC-S results are shown in Table 10 and Figure 29; and the results concerning to the TDC-D are tabulated in

Table 11 and Figure 30.

Table 10: Power of the SC TDC to keep a constant temperature of 310 K for different input flows and for different gases.

Gas	0 ml/min P [mW]	1 ml/min P [mW]	4 ml/min P [mW]	7 ml/min P [mW]	10 ml/min P [mW]
CO ₂	7,81	7,86	7,93	7,98	8,01
Air	9,29	9,32	9,43	9,49	9,53
CH ₄	10,67	10,72	10,86	10,94	11,00
He	25,71	25,72	25,86	26,05	26,25
H ₂	30,69	30,71	30,89	31,17	31,45

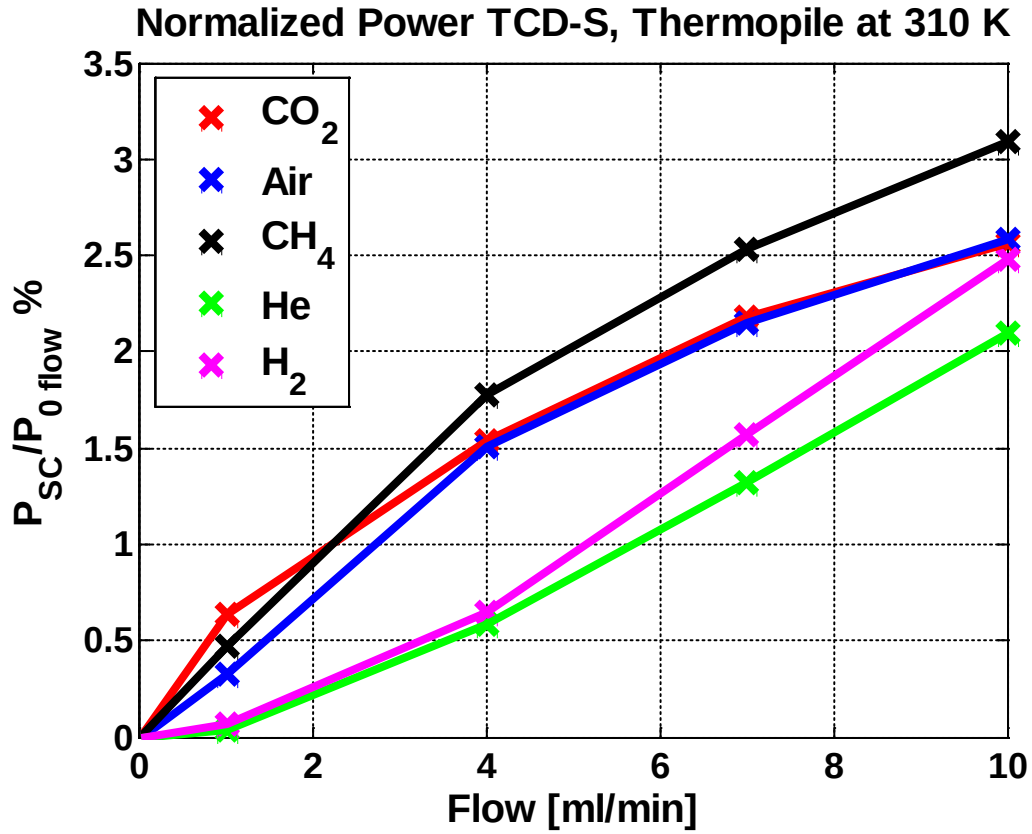


Figure 29: Relative change of the heating power of the SC-TCD vs. flow for different gases. Normalized to zero flow.

Table 11: Power of the DC TDC to keep a constant temperature of 310 K for different input flows and for different gases.

Gas	0 ml/min P [mW]	1 ml/min P [mW]	4 ml/min P [mW]	7 ml/min P [mW]	10 ml/min P [mW]
CO ₂	5,38	5,43	5,50	5,53	5,56
Air	5,50	5,53	5,61	5,66	5,70
CH ₄	5,60	5,64	5,75	5,81	5,86
He	6,89	6,90	6,97	7,08	7,19
H ₂	7,44	7,45	7,54	7,68	7,82

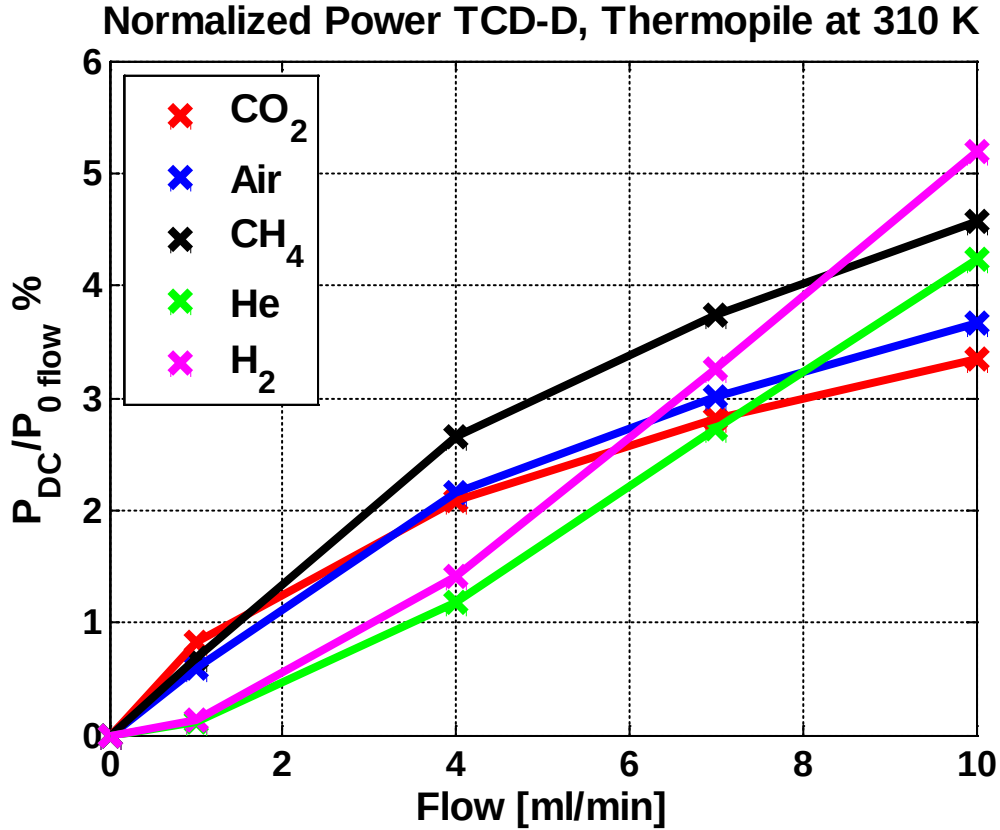


Figure 30: Relative change of the heating power of the DC-TCD vs. flow for different gases. Normalized to zero flow.

The first impression analyzing Figure 29 and Figure 30 is that each TCD is flow dependent. They need to increase the power to maintain a constant temperature of 310 K. This flow dependence was expected; however, the sensitivity of the relative power vs flow presented in Figure 29 and Figure 30 are not the same for both TCDs. It was expected that both TCDs had the same sensitivity to flow variations, but sensitivities are different, especially in the case of He and H₂. Thus, the flow compensation idea of the dual-TCD principle will not work in the case of He and H₂. Table 12 shows the sensitivity of each TCD and difference between sensitivities in terms of percentage.

Table 12: Sensitivity of each TCD with respect to flow variations for different gases.

Gas	SC-TDC S_{QSC} [mW/ml/min]	DC-TCD S_{QDC} [mW/ml/min]	Difference [%]
CO ₂	0.0209	0.0180	13.50
Air	0.0244	0.0201	17.59
CH ₄	0.0334	0.0256	23.26
He	0.0543	0.0292	46.27
H ₂	0.0758	0.0381	49.80

It can be seen in Table 12 that the difference in sensitivity between both TCDs is approximately 20% for CO₂, Air and CH₄. However, in the case of He and H₂ this value increases considerably achieving a

difference of almost 50%. One of the reasons is that the sensitivity of each TCD is different because the heat transfer profile on the membrane is different for each TCD. This was shown in section 6.1 Figure 25. Also, this might be because the TCD-D is not compensating enough to the TCD-S in the case of gases with high thermal conductivity. In fact, Figure 18 shows that for He and H₂ the temperature does not reach a constant value for a cavity deeper than 400 μm . Simulating Equation (25) for higher values of the depth of the cavity, results show that the temperature for He and H₂ starts reaching a constant value for values deeper than 1000 μm .

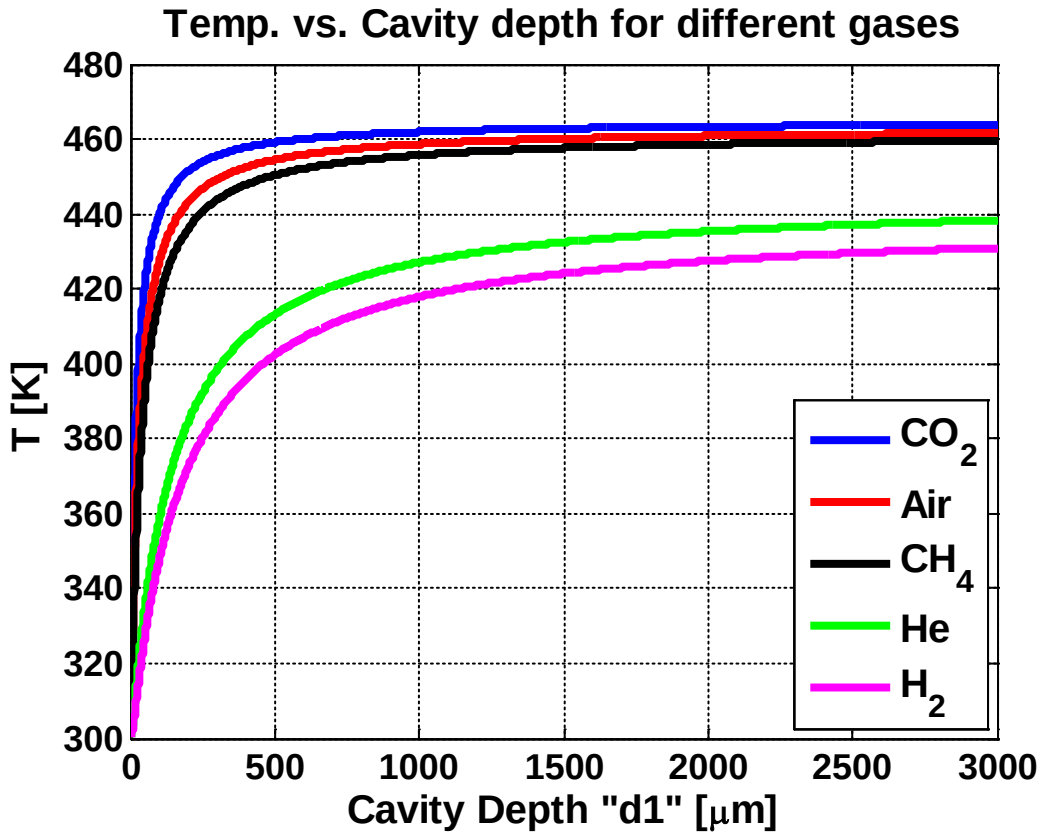


Figure 31: Temperature of the membrane increasing the value of the deep of the cavity.

In the case of He the temperature changes 21 degrees between 250 to 500 μm of d_1 . Between 500 and 1000 μm the temperature difference is 14 K. Now, between 1000 and 1500 μm the temperature difference is 5 degrees. Thus, in the case of He and H₂ TCD-D will compensate in a better way using a deep cavity higher than 1500 μm .

Another reason is that the temperature is measured at a certain distance from the heater. Possibly if the temperature were measured on the heater itself, the sensitivity might be equal for both devices. The last statement leads to simulate the sensitivity in terms of distance from the heater. These results are shown in Table 13 and Figure 32.

Table 13: Impact on the sensitivity of each TCD on measuring the temperature closer to the heater. The distances are from the center of the heater, thus the edge of the heater is at 14 μm .

	42 μm	30 μm	18 μm	14 μm
Gas	$S_{QSC} - S_{QDC}$ [%]	$S_{QSC} - S_{QDC}$ [%]	$S_{QSC} - S_{QDC}$ [%]	$S_{QSC} - S_{QDC}$ [%]
CO ₂	13.50	7.86	1.37	0.51
Air	17.59	8.94	3.08	0.19
CH ₄	23.26	13.03	4.40	0.99
He	46.27	23.78	11.46	3.96
H ₂	49.80	26.87	10.81	3.17

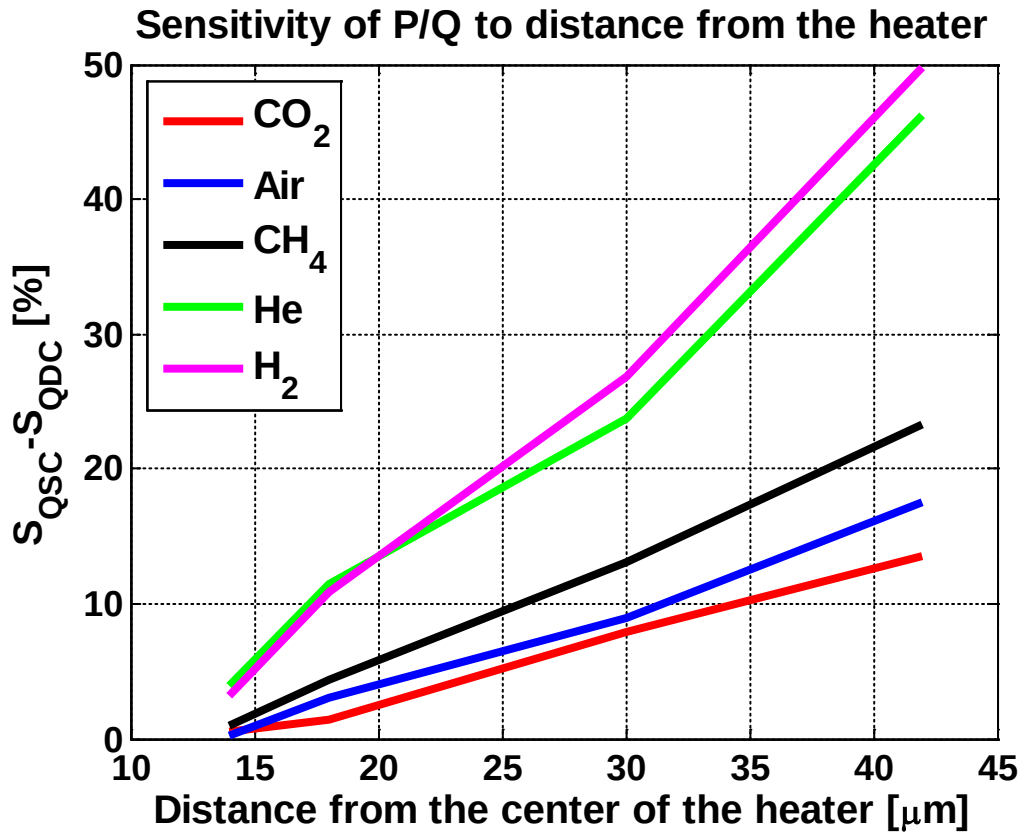


Figure 32: Differential sensitivity of the dual-TCD to distance from the center of the heater.

From Figure 32 is easy to realize that distance from the center of the heater has a great impact on measuring the temperature on the membrane. Closer to the heater the sensitivity of the TCDs is almost the same while farther from the heater the sensitivity of the TCDs becomes quite different, especially for He and H₂.

In sum, to have a flow compensated device in the case of He and H₂ the TCD-D must deeper than 1500 μm or the temperature on the membrane should be measured closer to the heater. In the case of CO₂, air and CH₄ the compensation device seems to work fine but to have a better compensation the temperature on the membrane should be measured closer to the heater. Having this in mind, we can apply a compensation factor to the TCD-S to reduce the effect of measuring at a certain distance from the heater. From Table 12, the maximum difference in the sensitivities of the TCDs is almost 50%, so applying a 50% of compensation to the Power/Flow sensitivity of the TCD-S, the result is shown in Figure 33.

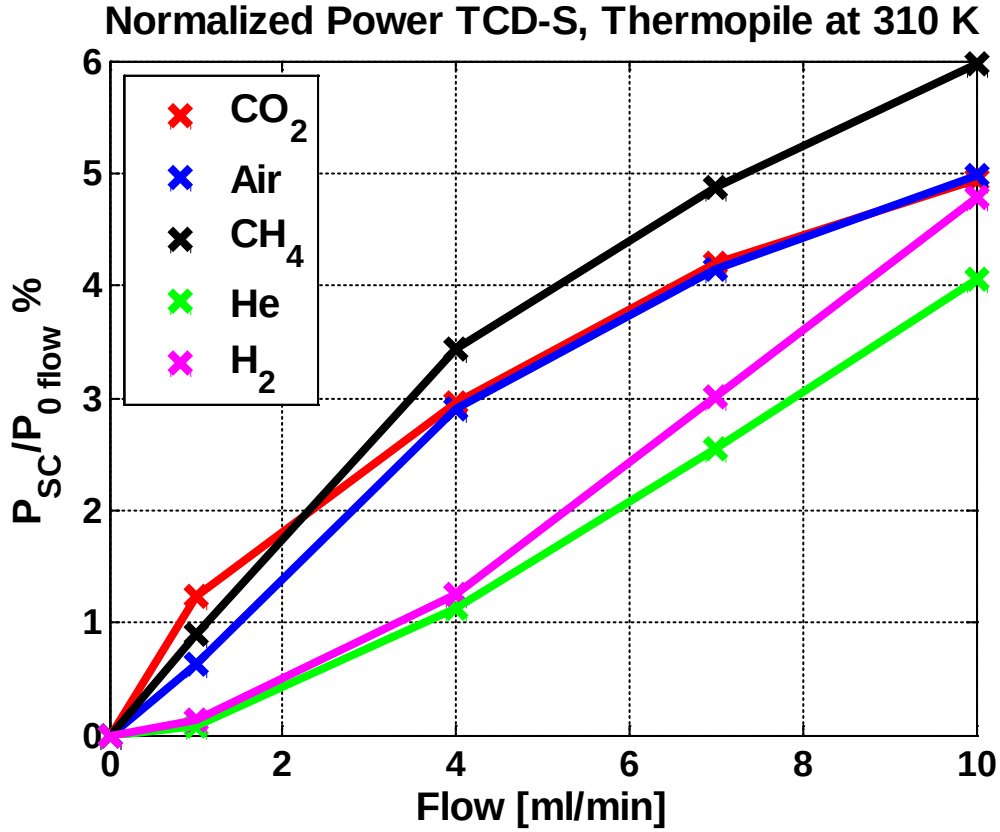


Figure 33: Normalized power of the SC-TCD using a compensation of 50%.

Using this compensation factor to supply the effect of not measuring on the same position of the heaters, the normalized power of the TCD-S (Figure 33) is much more similar to the normalized power of the TCD-D of Figure 30. Now, if the power difference of both TCDs is calculated, the results are as follows:

Table 14: Required heater power difference of the dual-TCD to maintain a constant temperature of the thermopile at 310 K using a compensation factor of 2 in the SC-TCD.

Gas	0 ml/min ΔP [mW]	1 ml/min ΔP [mW]	4 ml/min ΔP [mW]	7 ml/min ΔP [mW]	10 ml/min ΔP [mW]
CO ₂	2.43	2.44	2.45	2.46	2.47
Air	3.79	3.80	3.82	3.84	3.84
CH ₄	5.07	5.08	5.11	5.13	5.14
He	18.82	18.82	18.83	18.85	18.85
H ₂	23.25	23.25	23.29	23.31	23.35

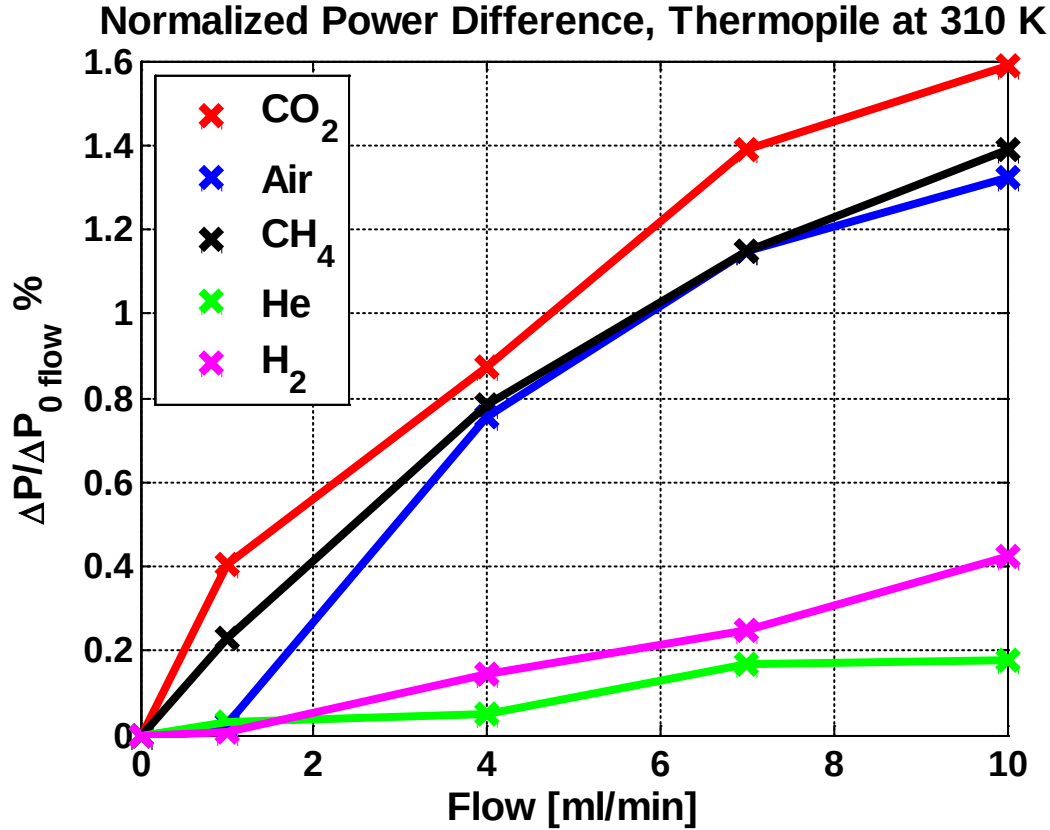


Figure 34: Power difference of the dual-TCD using a compensation factor of 2 in the SC-TCD.

Figure 34 shows that the flow dependence of the dual-TCD decreases considerably in comparison to each TCD itself. Especially, He and H₂ reduce dramatically their flow dependence, around 12 times. Also, it is possible to realize that for gases with a higher thermal conductivity (in this case CO₂, air and CH₄) the power profile tends to saturate for flows higher than 10 mL/min. These results confirm the concept of the flow compensated dual-TCD principle. Table 15 shows the improvement of using the dual-TCD in comparison to the TCD-S and the TCD-D.

Table 15: Improvement of using the dual-TCD in comparison to the SC-TCD and DC-TCD.

Gas	Dual-TCD in comparison to TCD-S [%]	Dual-TCD in comparison to TCD-D [%]
CO ₂	3.11	2.11
Air	3.76	2.76
CH ₄	4.30	3.30
He	22.67	23.67
H ₂	11.34	12.34

The power supplies for each gas to maintain the temperature at 310 K are shown in Table 16 and Table 17.

Table 16: Supply of the heater for different gases and flows using a voltage reference.

Gas	0 ml/min V_{ref} [V]	1 ml/min V_{ref} [V]	4 ml/min V_{ref} [V]	7 ml/min V_{ref} [V]	10 ml/min V_{ref} [V]
CO ₂	12.91	12.95	13.02	13.05	13.08
Air	14.09	14.12	14.20	14.25	14.28
CH ₄	15.11	15.15	15.25	15.31	15.35
He	23.56	23.57	23.63	23.72	23.82
H ₂	25.77	25.78	25.86	25.98	26.09

Table 17: Supply of the heater for different gases and flows using a current reference.

Gas	0 ml/min I_{ref} [μ A]	1 ml/min I_{ref} [μ A]	4 ml/min I_{ref} [μ A]	7 ml/min I_{ref} [μ A]	10 ml/min I_{ref} [μ A]
CO ₂	614.79	616.77	619.91	621.67	623.03
Air	671.12	672.35	676.27	678.35	679.96
CH ₄	719.64	721.21	726.21	728.89	730.95
He	1122.10	1122.36	1125.34	1129.74	1134.11
H ₂	1227.18	1227.67	1231.40	1236.96	1242.59

6.3 Summary of the Chapter

Static simulations have shown that the shallow cavity device is 11 times more sensitive than the deep cavity device. To maintain both devices at the same temperature, less power is needed for the TCD-D than the TCD-S.

Dynamic simulations have demonstrated that the dual-TCD principle can be used to reduce flow dependence. However, some problems have been found when measuring He and H₂. For these gases the power/flow sensitivity for both TCDs is different having as a consequence that the flow compensation principle does not work. One solution is that the cavity of the TCD-D should be deeper than 525 μ m. Simulations have shown that a cavity deeper than 1500 μ m should compensate better than a cavity of 525 μ m (Figure 31). Another solution is to measure the temperature of the membrane closer to the heater. If the temperature is measured on the edge of the heater, the difference between the power/flow sensitivity reduces to 3%.

Compensation of 23 times has been achieved and a time response of 0.4 ms has been found. The sensitivity of sensor is 0.136 W/W/m $\cdot$ K. The highest power consumption of the dual-TCD is 40 mW when measuring H₂.

Chapter 7 Encapsulation Module and Read-out Control Loop Circuit

An encapsulation module and a read-out control loop circuit have been designed and built to test the dual-TCD device. This measurement setup has been made to verify and compare the real function of the dual-TCD with simulations made in Comsol Multiphysics. In the following sections both the encapsulation and the read-out circuit will be explained.

7.1 Encapsulation Module

The encapsulation module is used to conduct the flow from the gas flow generator to the dual-TCD. The idea is to design a box where the package of the dual-TCD fits with two holes where the gas generator can be connected. There is one constraint in the design of the encapsulation module which is to ensure that the gas flow will be laminar on top of the dual-TCD. One of the reason is that laminar flow on top of the device ensures that the heat loss by convection of each device will be identical. If turbulences were present, the heat loss by convection would be different in each device. This would lead to the consequence that the differential operation of the flow compensation dual-TCD might not work.

Reynolds number is used to verify if a gas flow through a certain body is laminar or turbulent (2.2.3). Reynolds number depends strongly on the dimensions of the path where the gas is flowing and also on the physical parameters of the gases. The idea is to design a box which covers the package where the dual-TCD is placed and conduct the gas to the place where the sensor is. A duct is designed to conduct the gas only where the sensor is placed and not to other parts of the package. This duct is the main constraint to have laminar flow on top of the sensor and its surroundings. The main idea of the encapsulation module is shown in Figure 35.

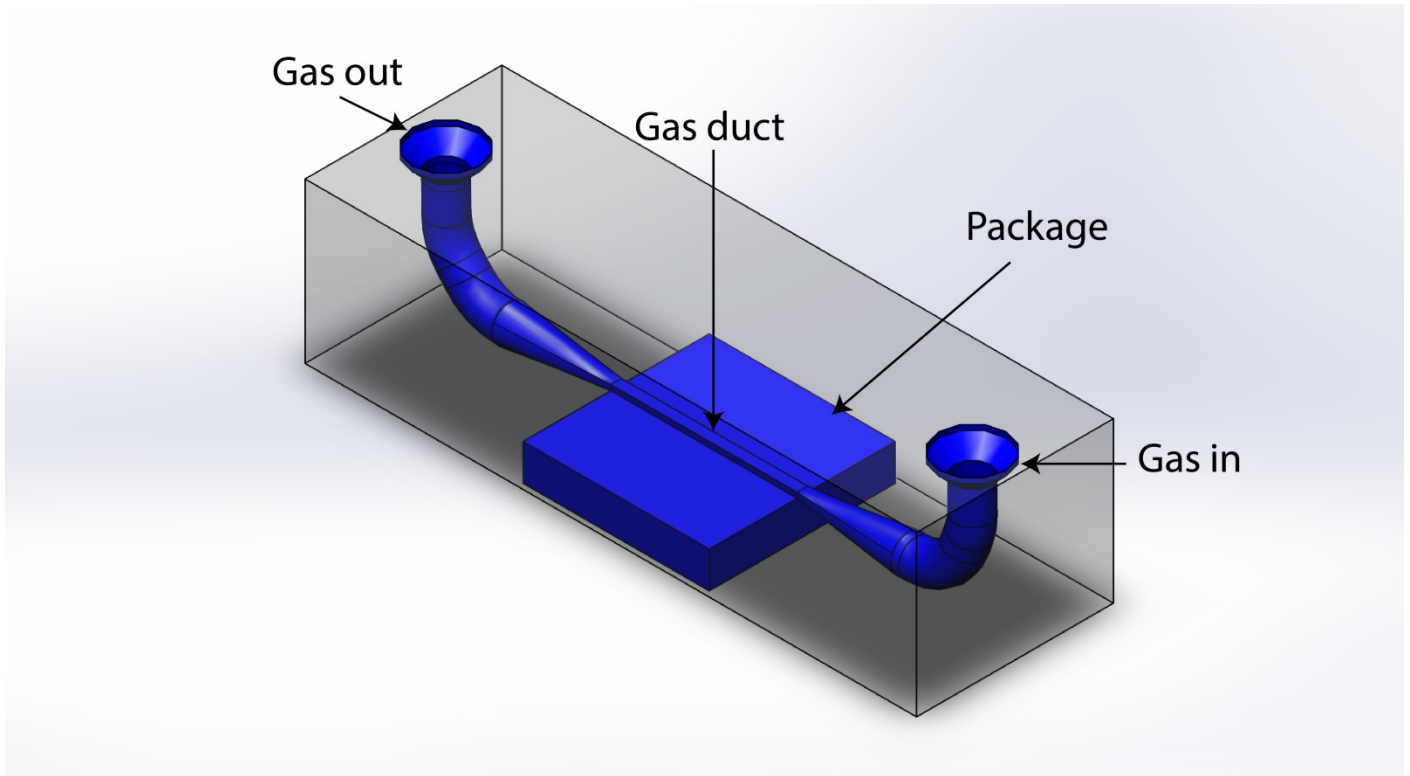


Figure 35: Idea of the encapsulation module.

The package should fit exactly in the encapsulation module and on top of it a small duct is placed. Package dimensions are presented in A.2. This duct conducts the gas which is connected from outside. The dimensions of the duct should be chosen accordingly to have laminar flow through the sensor for all gases. If the dimensions of the duct are: height 0.5 mm and width 7 mm, Reynolds numbers for different gases and different flows are shown in Table 18.

Table 18: Reynolds numbers for different gases and flows.

Gas	Re for 1 ml/min	Re for 4 ml/min	Re for 7 ml/min	Re for 10 ml/min
CO ₂	0,54	2,14	3,75	5,35
Air	0,30	1,19	2,09	2,98
CH ₄	0,26	1,05	1,84	2,62
He	0,04	0,17	0,29	0,42
H ₂	0,04	0,17	0,29	0,41

From Table 18 is possible to realize that the highest Reynolds number is when CO₂ is used at a flow rate of 10 ml/min. This number is 5.35 which is smaller than 2000, number when the flow changes from laminar to turbulent. This means that the chosen dimensions of the duct are correct. To ensure that these dimensions are correct, flow simulations have been done in Comsol Multiphysics.

To simulate flow, the module “Laminar Flow” has been used. This module allows simulating the flow impact on a certain structure. In this case, the controlled variables are the physical parameters of the gases and the input flow. If CO₂ at 10 ml/min is simulated, the result is shown in Figure 36.

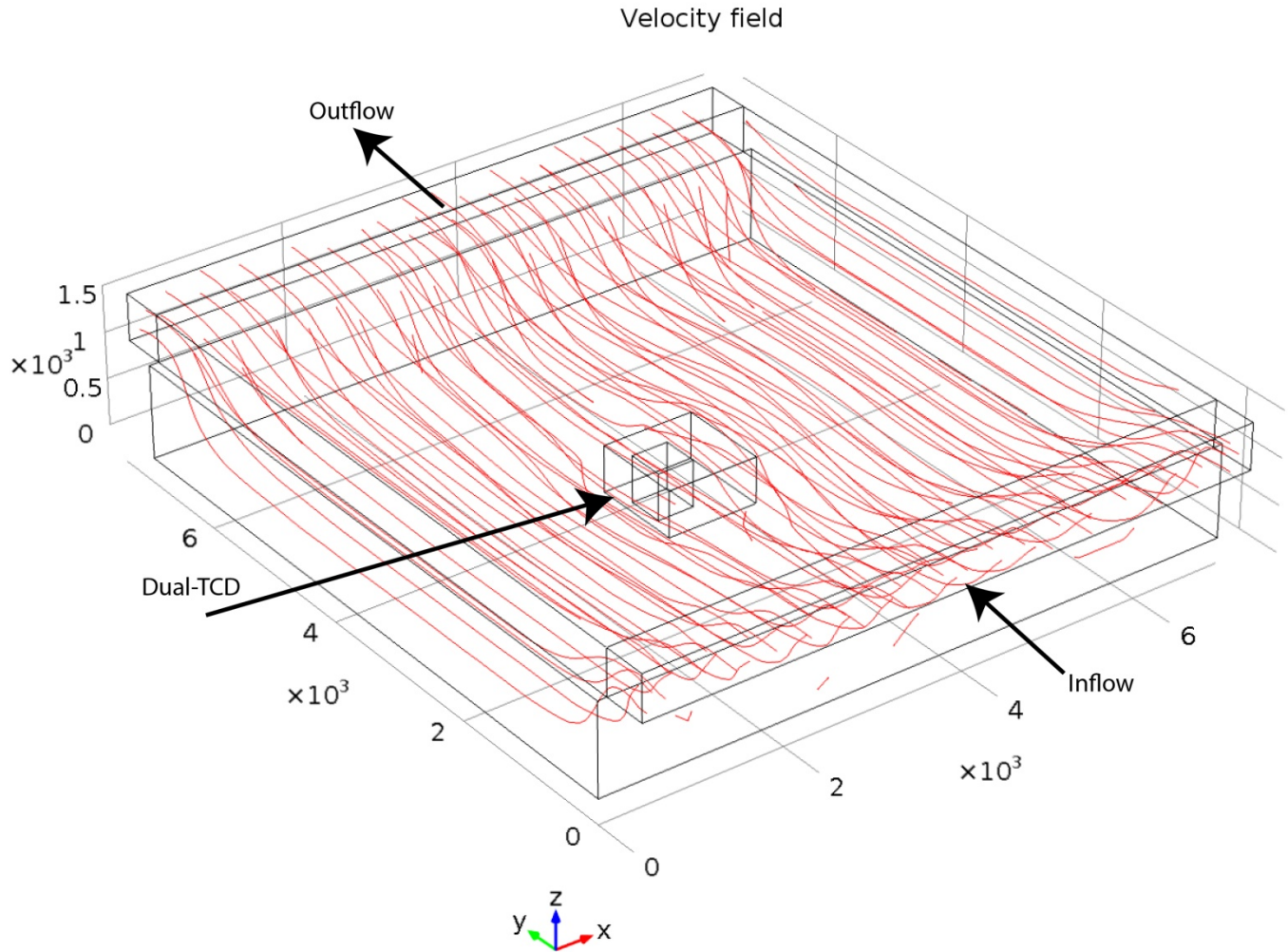


Figure 36: Flow simulation using Comsol Multiphysics.

In Figure 36 the dual-TCD has been placed as if it were in the package. Then, the package is placed inside of the encapsulation module of Figure 35. An input flow is simulated and the behavior shows that the flow inside of the encapsulation module is laminar even in the edges of the dual-TCD. Also, it shows that the flow is laminar on top of the dual-TCD.

Reynolds numbers and simulation show that the flow is laminar using a duct of 0.5mm by 7mm. Thus, a duct with these dimensions has been chosen.

The encapsulation module is designed using SolidWorks 2014 in EWI faculty at TU-Delft. The final model of the encapsulation is shown in Figure 37.

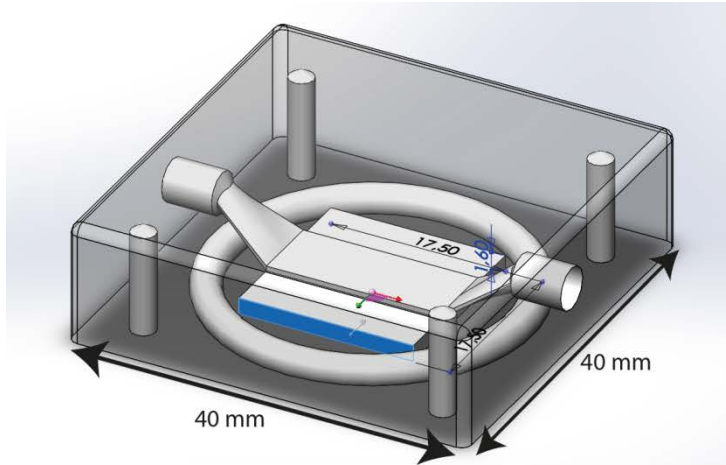


Figure 37: Final model of the encapsulation module, designed in SolidWorks. The module was fabricated in EWI at TU-Delft.

The duct is designed in such a way that ensures that the gas only flows where the dual-TCD is placed. Also, the ring around the “package place” is made to put a plastic o-ring to avoid any kind of leakage. This encapsulation module has four holes to mount and screw it on a PCB where the dual-TCD is placed.

7.2 Read-out Circuit

7.2.1 DC System

A read-out control loop circuit has been designed to read the Seebeck voltage generated by the thermopiles as well as to control the power supply of the heaters. This voltage is directly related to the measured gas (Equation (27)). The idea of this read-out circuit is to control and maintain the temperature of the thermopiles constant at a certain value reading the output Seebeck voltage.

The DC circuit is shown in Figure 38.

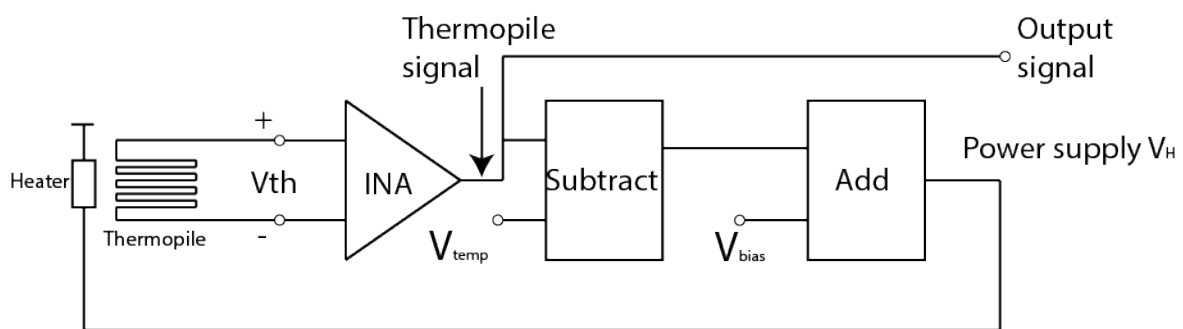


Figure 38: DC read-out control loop circuit.

A bias voltage (V_{bias}) provides the power to the heater to maintain a constant temperature at thermopile position. The thermopile produces an output voltage (V_{th}) proportional to the temperature of the thermopile. At the same time the temperature is proportional to the thermal conductivity of the gas. V_{th} is amplified by

using an instrumentation amplifier (INA) and its output corresponds to the measured gas. Then V_{th} is applied to a differential amplifier where V_{th} is compared to V_{temp} . The voltage V_{temp} is proportional to the operating temperature of the TCD (310 K). The difference between V_{temp} and V_{th} is calculated to see if the thermopile is hotter or colder than the operating temperature. The differential amplifier is followed by a summing amplifier where the difference $V_{temp}-V_{th}$ is added to V_{bias} . The reason for adding these two signals is to supply the adequate amount of power to the heater in the case that the temperature of the thermopile is different than the reference. The output of this stage supplies the heater maintaining the temperature of the thermopile constant. Thus, the control loop keeps the supply of the heater approximately at V_{bias} .

However, this circuit suffers from noise and offsets due to the different components, especially the INA. The INA is the stage which has the higher noise and offset due to its configuration. To avoid most of the noise and offset produced by the INA a modulation and demodulation stage is added to the circuit.

This circuit controls only one of the TCDs. Thus, two of these circuits have to be fabricated to control both TCDs of the dual-TCD.

7.2.2 AC System

The modulation and demodulation is made by a complementary CMOS chopper [33]. Besides the chopper device, an analog and a digital low pass filter have been added to the circuit as well as a voltage to current converter. Also, a phase compensation technique is used to synchronize the choppers. The final circuit is shown in Figure 39.

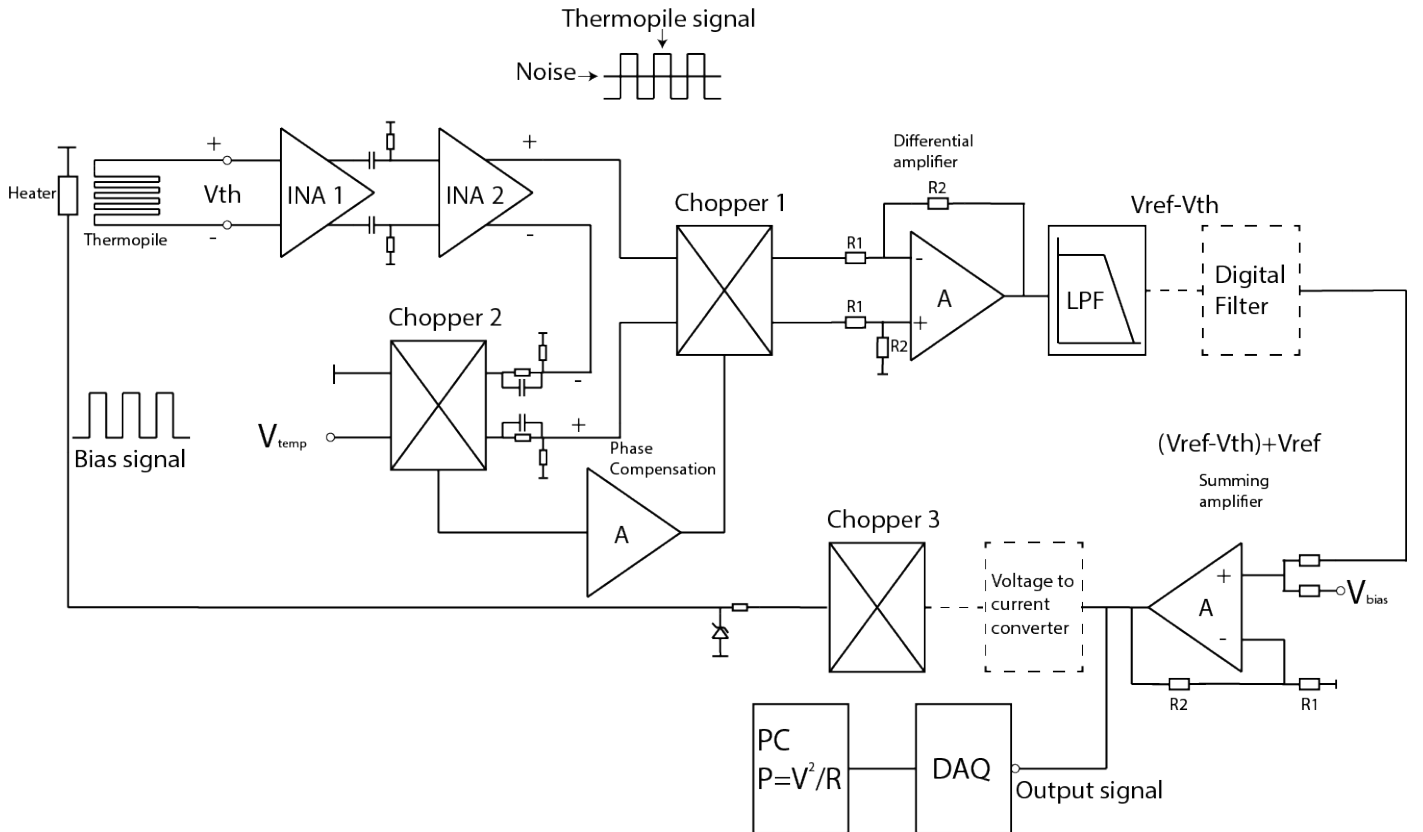


Figure 39: Final circuit of the read-out of the dual-TCD.

A bias voltage (V_{bias}) supplies the heater. This voltage is modulated by Chopper 3. Between Chopper 3 and the heater there is a protection circuit to avoid overload of the heater. The heat produced by the heater is measured by the thermopile as temperature. This temperature depends on the thermal conductivity of the gas. The thermopile produces an output Seebeck voltage (V_{th}) proportional to the temperature. V_{th} is a differential voltage and it is applied to INA 1. INA 1 amplifies V_{th} from the millivolt level to volt level. After INA 1 there is a passive filter to cancel any kind of high frequency distortion. In case that the amplification provided by INA 1 is not enough, there is another INA which can provide more amplification. The output from INA 1 (or INA 2) is the input of Chopper 1. At the same time, V_{temp} is also modulated by Chopper 2 and this output is the other input of Chopper 1. Next to Chopper 2 there is a passive filter of 50Hz to avoid high frequency noise. The negative output of INA 1 (or INA 2) is tied to the negative output of Chopper 2 which is usually ground. Then, both inputs to Chopper 1 are demodulated and at the same time the offset and noise of the INA are modulated, thus offset and noise do not affect the signals. The outputs of Chopper 1 are the inputs of the differential amplifier where $V_{\text{temp}} - V_{\text{th}}$ is obtained. This difference is filter using a 2nd order Sallen-Key low pass filter (LPF). In addition to the Sallen-Key LPF there is the chance to use a digital filter, in case higher selectivity is required. This digital filter is an 8th order LPF. Once the signal ($V_{\text{temp}} - V_{\text{th}}$) has been filtered, it is applied to a summing amplifier where it is added to V_{bias} . Depending on the value of $V_{\text{temp}} - V_{\text{th}}$ the summing amplifier provides the necessary power to the heater to maintain a constant temperature of the thermopile. In this way is possible to control the temperature of the thermopile depending on the gas.

This circuit controls only one of the TCDs. Thus, two of these circuits have to be fabricated to control both TCDs of the dual-TCD.

In the following sections every block of the circuit of Figure 39 will be explained as well as their noise and offset contribution.

7.2.3 Instrumentation Amplifier

The instrumentation amplifier is a common block to amplify a differential signal. The output of the INA can be differential or single; in this case a differential output is used. One of the most important features of the INA is the improvement of the common mode rejection ratio (CMRR). Using the INA topology of Figure 40 the differential signal is amplified but the common mode signal no. In this way the CMRR is improved notably. The topology of the INA is shown in Figure 40.

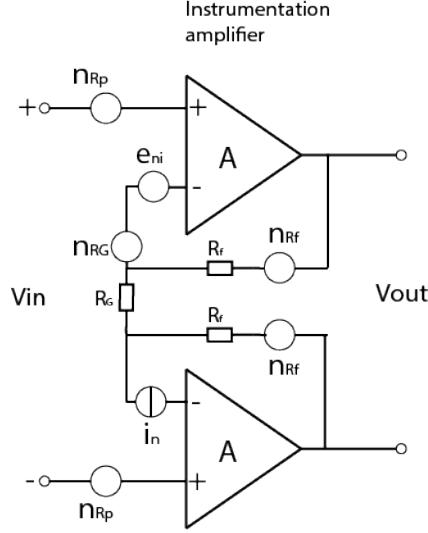


Figure 40: Instrumentation amplifier topology.

The gain of the INA depends on the resistors R_G and R_f as Equation (28) shows:

$$\frac{V_{out}}{V_{in}} = \left(1 + \frac{2R_f}{R_G}\right) \quad (28)$$

In this design a variable gain with a maximum value of 100 is stated. To do this, R_f is implemented using a potentiometer and R_G is set to a value of 1 k Ω . Thus, the potentiometer R_f is equal to 49.5 k Ω ~ 50 k Ω .

The noise and offset of the INA depends strongly on the opamps used. However, most of the noise and offset is canceled by using the chopper modulator after the INA. Still, the total input referred noise of the INA has been calculated and is presented in Equation (29).

$$n_{INA} = \sqrt{8kTR_p + 4kTR_G + 8kT \frac{R_f^2}{R_G} + 2e_{ni}^2 + 2(i_n R_p)^2} \left[nV / \sqrt{Hz} \right] \quad (29)$$

where k is the Boltzmann constant, T is the temperature, R_p is the resistance of the thermopile wiring, e_{ni} is the noise voltage of the opamp and i_n is the noise current of the opamp. To have a very low noise and offset two low-noise low-offset opamps are used. The model of the opamp is the LT1037 [34] of Linear Technologies. The main characteristics of this opamp are compared with simulations in LTSpice and are tabulated in Table 19.

Table 19: Main characteristics of the LT1037 opamp compared to simulations in LTSpice.

Opamp	CMRR [dB]	Noise [nV/ $\sqrt{Hz}$]	Offset Max. [uV]	BW [MHz]
LT1037 [31]	126	4.5 @ 10 Hz	25	45
LT1037 simulations	127	12 @ 10 Hz	20	0.02

Table 19 shows that most of the simulated characteristics are compared to those provided by the manufacturer. However, there is a considerable difference in the BW. This might be due to the model of the opamp is not updated in the software. However, this is not a problem for our system because the maximum modulation of the signals is 100 Hz.

The equivalent input noise of the INA can be calculated by using Eq. (29) and the values of R_G and R_f . The value of R_p has been calculated and its value is 100 k Ω . The values of e_{ni} and i_n are taken from [34]. The value of the input referred noise of the INA is $n_{INA}=0.646 \mu\text{V}/\sqrt{\text{Hz}}$.

Using the input referred noise of the INA is possible to calculate the equivalent noise in terms of thermal conductivity. The equivalent λ noise is calculated by using Eq. (24). The input referred noise of the INA is 2.041 μV @ 10 Hz. Using n_{INA} the equivalent noise of λ can be calculated. The equivalent noise of λ is $\lambda_n=9.369 \times 10^{-16} \text{ W/W/m}\cdot\text{K}$ @ 10 Hz @ 310 K. The equivalent noise of λ is negligible.

Using the LT1037 opamp to design the INA, simulations in LTSpice have been done to obtain the main characteristics of the INA. Figure 41 shows the CMRR and BW simulation of the INA and these results are tabulated in Table 20.

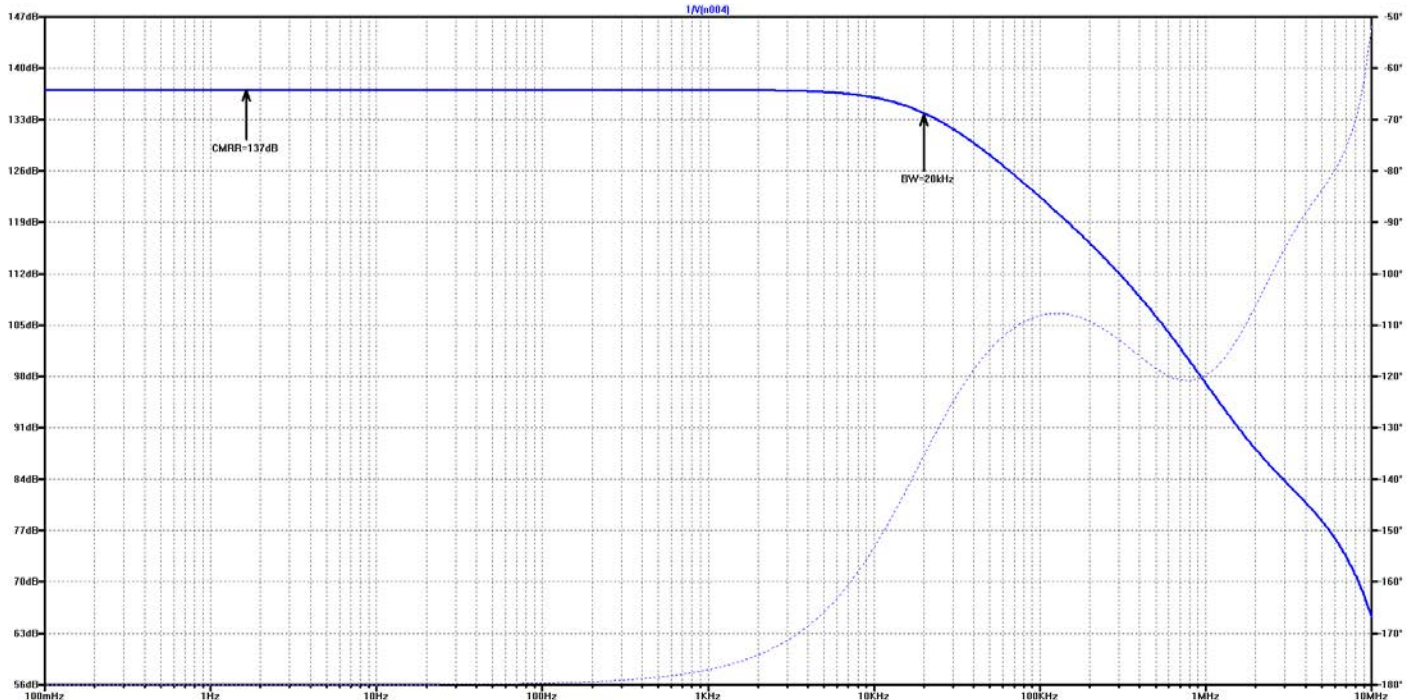


Figure 41: Simulation of the INA using LTSpice.

Table 20: Main characteristics of the INA.

Opamp	CMRR [dB]	Noise [$\text{nV}/\sqrt{\text{Hz}}$]	Offset Max. [μV]	BW [kHz]
INA using LT1037	137	9 @ 10 Hz	25	20

Simulations show that the noise of the INA is 9 nV @ 10 Hz. Using the input referred noise of the INA the value is 2 μ V @ 10 Hz. The difference between these two values is because the noise of the resistors has not been included in simulations.

7.2.4 Chopper

A complementary CMOS chopper [33] has been used as the modulator and demodulator in the read-out circuit. Its function is to cancel the noise and offset of the INA stage as well as the noise of other components of the read-out circuit. The model of the chopper corresponds to a MAX4526 of Maxim Integrated[33] which is optimized for high-speed applications, such as chopper amplifiers. It is an almost ideal floating ± 1 phase-reversing switch having a very low leakage current and a large on-off current ratio. As the modulation of the read-out circuit is no more than 100 Hz, the differential charge injection, typically 10pC, does not contribute to the offset. The typical configuration of this device when it is using as a modulator-demodulator is shown in Figure 42.

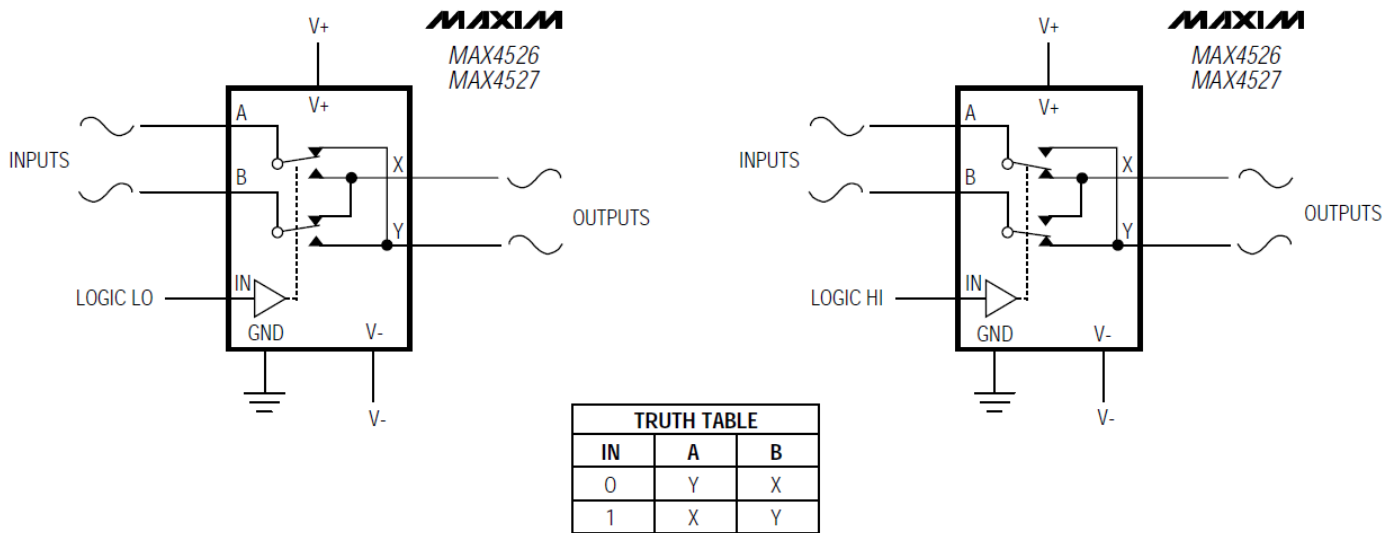


Figure 42: Chopper stage implemented using a phase-reversing switch model MAX4528 Maxim Integrated[33].

The main characteristics of the MAX4528 are tabulated in Table 21.

Table 21: Main characteristics of the chopper device [33].

Characteristic	MIN	TYP	MAX
Single supply [V]	+4.5		+36
Dual supply [V]	± 4.5		± 18
Max. input current [mA]			± 20
Typical leakage current [nA]	-0.5	0.01	0.5
Charge injection [pC]		1	10

7.2.5 Phase Compensation

Between Chopper 1 (demodulator) and Chopper 2 (modulator) there is a phase compensation circuit (Figure 39). The input signals of Chopper 1 are not in phase due to the different electric paths that they have to

cover. Thus, this phase compensation circuit is necessary to compensate both choppers to avoid any kind of spikes that can be produced in the following subtract stage.

The phase compensation is implemented by using an adjustable phase-shifting all-pass network [35]. A circuit consists of a single opamp with resistors and capacitors. The circuit is shown in Figure 43.

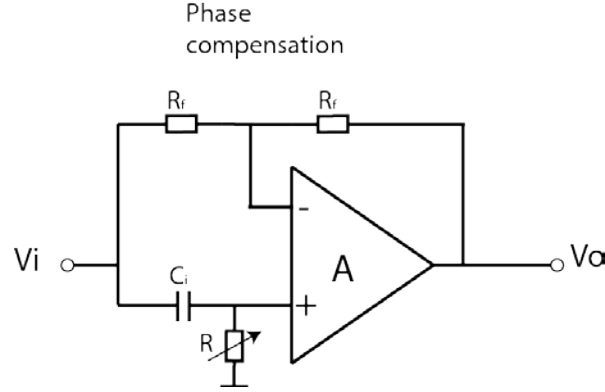


Figure 43: Phase compensation circuit.

The measured input of the sensor (x_i) is usually amplified obtaining $H(x)$ and/or its phase changes due to modulation $\Phi(x)$. The transfer function of the phase compensation circuit is given by [35]:

$$|H(j\omega)| = 1 \text{ and } \angle H(j\omega) = \phi = \pi - \arctan(2\pi fRC_i). \quad (30)$$

From Equation (30), the phase delay ϕ is controlled by using a potentiometer R for a given frequency ω .

This circuit is connected in the LOGIC channel of the choppers 1 and 2. In this way both choppers are kept in phase avoiding spikes in the following stages.

7.2.6 Difference Amplifier

The difference amplifier is used to compare the thermopile voltage with the reference voltage. Then, this difference is added to subtracted to from the reference voltage in case of more or less power is required to feed the heater. The difference amplifier is composed of an opamp and resistors. The topology of this block is shown in Figure 44.

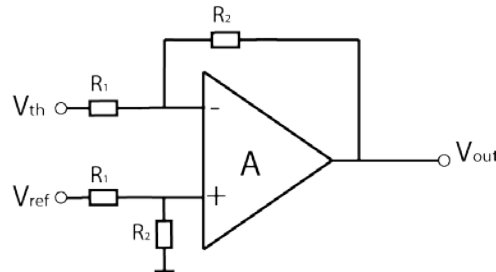


Figure 44: Difference amplifier.

The output voltage of the difference amplifier is as follows:

$$V_{out} = \frac{R_2}{R_1} (V_{ref} - V_{th}) \quad (31)$$

A unity gain is set for this block, thus $R_1 = R_2 = 1 \text{ k}\Omega$.

Table 22: Components and values of the difference amplifier.

Component	Value
Opamp	LT1037
R_1	1 k Ω
R_2	1 k Ω

7.2.7 Low Pass Filter Modules

The read-out circuit has two low pass filters, one is a 2nd order Sallen-Key and the other is an 8th order digital filter. Both filters are explained in the following sub-sections.

7.2.7.1 Sallen-Key Low Pass Filter

The Sallen-Key 2nd order low pass filter is one of the most widely used filter in electronics due to its simplicity and the high performance that offers. Its topology is shown in Figure 45.

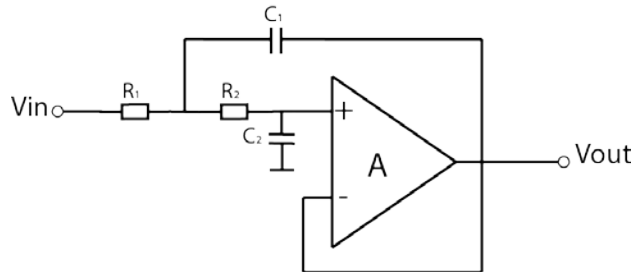


Figure 45: Sallen-Key topology.

The transfer function ($H_{SK}(s)$) and its natural frequency (f_0) are as follows:

$$H_{SK}(s) = \frac{1}{1 + C_2(R_1 + R_2)s + C_1C_2R_1R_2s^2} \quad (32)$$

$$f_0 = \frac{1}{2\pi\sqrt{C_1C_2R_1R_2}} \quad (33)$$

As the range of the modulation frequency is 20 - 100 Hz, f_0 should have a lower value than 20 Hz. Also, the frequency of the network 50 Hz sometimes does not filter by the power supply, thus the LPF should also filter this frequency. Therefore, the natural frequency of the filter is set to $f_0 = 1 \text{ Hz}$ to avoid any inconvenience. The values of the resistors and capacitors are chosen as follows: for simplicity $R_1 = R_2$ and

$C_1 = C_2$. If $R_1 = 100\text{k}\Omega$, then $C_1 = 1.59\text{ }\mu\text{F}$. However, this is not a usual value of a capacitor, thus a value of $1\text{ }\mu\text{F}$ is chosen. Using $C_1 = 1\text{ }\mu\text{F}$ and $R_1 = 100\text{ k}\Omega$, $f_0 = 1.59\text{ Hz}$. In sum, the Sallen-Key LPF is composed of the following components and values.

Table 23: Components and values of the Sallen-Key filter.

Component	Value
Opamp	LT1037
R_1	$100\text{ k}\Omega$
R_2	$100\text{ k}\Omega$
C_1	$1\text{ }\mu\text{F}$
C_2	$1\text{ }\mu\text{F}$

7.2.7.2 Digital Filter

A digital filter is placed after the Sallen-Key filter in case a higher selectivity is needed. This filter is implemented using the 8th order low pass switched-capacitor filter model MAX291 of Maxim Integrated[36]. The filter can be set up with corner frequencies from 0.1 Hz to 25 kHz. This corner frequency is tunable by a clock signal. The power supply of the filter is up to 11 V in single operation and $\pm 5.5\text{ V}$ in dual operation. The pin configuration is shown in Figure 46.

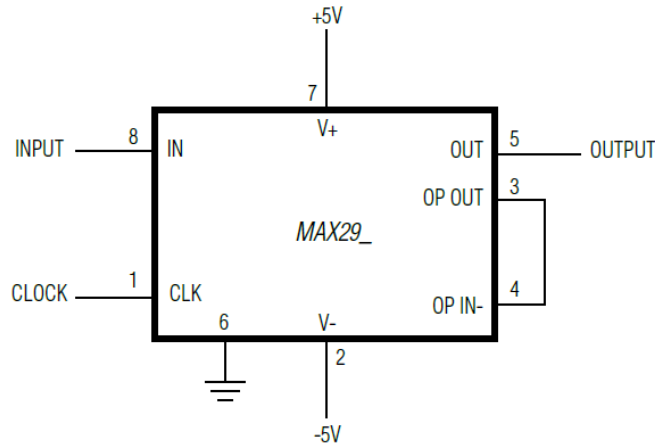


Figure 46: Pin configuration of the digital filter MAX291 [36].

The clock signal to the corner frequency has a ratio of 100:1. Thus, if a corner frequency of 1 Hz wants to be set, the clock signal has a value of 100 Hz.

7.2.8 Non-Inverting Summing Amplifier

The summing amplifier is used to supply more or less power to the heater around a voltage reference. The non-inverting summing amplifier is composed of a single opamp and four resistors. Its topology is shown in Figure 47.

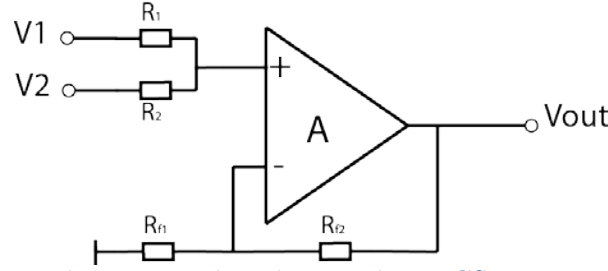


Figure 47: Non-inverting summing amplifier.

The output voltage of the non-inverting summing amplifier is as follows:

$$V_{out} = \left(1 + \frac{R_{f2}}{R_{f1}}\right) \left(V_1 \frac{R_2}{R_1 + R_2} + V_2 \frac{R_1}{R_1 + R_2}\right). \quad (34)$$

For simplicity $R_1 = R_2 = 1 \text{ k}\Omega$, thus Equation (34) reduces to:

$$V_{out} = \left(0.5 + \frac{R_{f2}}{2R_{f1}}\right) (V_1 + V_2). \quad (35)$$

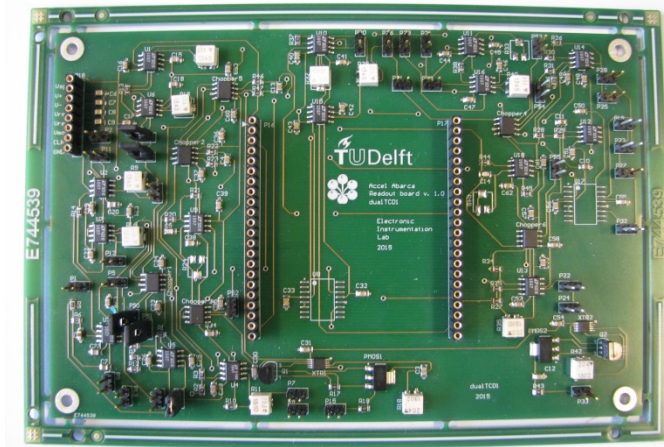
The main function of this block is to add both input signals, thus amplification is not the most important part of this block. However, it might be useful to have the chance to change the amplification. If an amplification in the range of 1 to 10 is chosen and fixing $R_{f1} = 1 \text{ k}\Omega$, therefore the value of R_{f2} should vary from $1 \text{ k}\Omega$ to $20 \text{ k}\Omega$. In sum, the components and values of the non-inverting summing amplifier are tabulated in Table 24.

Table 24: Components and values of the non-inverting summing amplifier.

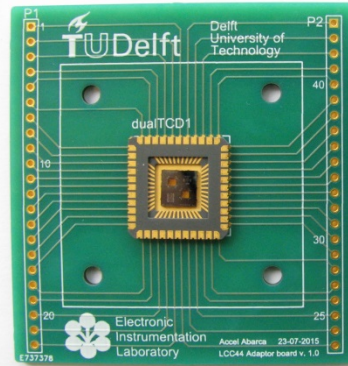
Component	Value
Opamp	LT1037
R_1	$1 \text{ k}\Omega$
R_2	$1 \text{ k}\Omega$
R_{f1}	$1 \text{ k}\Omega$
R_{f2} potentiometer	$1 - 20 \text{ k}\Omega$

7.2.9 Voltage to Current Converter

The voltage to current (V-I) converter block is placed next to the non-inverting summing amplifier. The reason of having a V-I is that, when measuring certain gases like He and H₂, the voltage reference to supply the heaters of the dual-TCD is higher than the voltage supply of most of the components of the read-out circuit. Thus, a V-I converter is used in those cases. The V-I converter is implemented by using the model XTR111 of Texas Instruments [37]. This device allows converting a voltage to a current and there is the chance to give some amplification to the current. The circuit of this device is shown in Figure 48.



Read-out control loop circuit



PCB dual-TCD



Encapsulation module

Figure 49: PCB of the read-out control loop circuit, PCB of the dual-TCD and encapsulation module.

The PCB of the read-out control loop has been partially tested. The INAs and the 2nd order Sallen-Key filter have been tested. The INAs achieve a gain of 10. This gain is 10 times lower than the expected gain of 100. This difference could be because the potentiometers are not accurate enough to achieve the expected gain of 100. In fact, during measurements, it has been found that some potentiometers do not reach the default value of 50 k Ω (at 50 k Ω the gain should be approximately 100). The noise of the INAs have been measured and it has a value of 3 μ V @ 10 Hz. This value is comparable to the calculated noise of 2 μ V @ 10Hz. It is possible that the difference of 1 μ V in the noise could be because of the inner resistance of the (wiring) paths of the PCB. The bandwidth of the INAs results in 15 kHz. The bandwidth is in the order of the simulated value of 20 kHz.

Chapter 8 Measurement Results

In Chapter 8 the measurement results of the dual-TCD will be presented. Different types of measurements have been done by using probe station, infrared camera and vacuum chamber. Measurements have been done in vacuum, air and CO₂.

8.1 Resistance of the Heater and Thermopiles

The first measurements were to calculate the resistance of the heaters and thermopiles of the dual-TCD. These measurements were done for both types of dual-TCD: short and long. The method utilized to measure the resistance was the four point test. Results of this test are shown in Table 25.

Table 25: Four point measurements.

Component	Heater resistance [k Ω]	Thermopile resistance [k Ω]
Short dual-TCD	7.78	101.6
Long dual-TCD	7.68	30.9

The heater resistance has a value between 7.68 k Ω and 7.78 k Ω . In the following sections an average value of 7.73 k Ω will be used when it is necessary.

8.2 Probe Station Cascade 33

The probe station Cascade 33 was used to test different samples of the dual-TCD. These measurements have been done to choose the best samples that will be bonded. The probe station Cascade 33 is part of the measurement laboratory in DIMES facility at TU-Delft. The probe station Cascade 33 allows to use an input current or voltage and to measure output current or voltage. The measurement setup is shown in Figure 50.

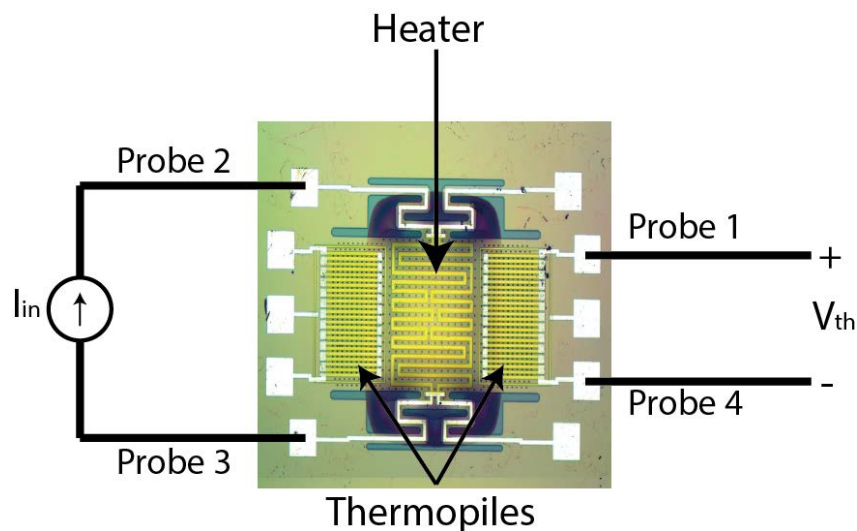


Figure 50: Measurement setup using the probe station Cascade 33.

An input current supplies the heater producing heat in the membrane (probe 2 and probe 3). The heat in the membrane is measured by thermopiles as a temperature difference between the substrate (ambient) and the membrane. This temperature difference (ΔT) will produce an output Seebeck voltage (V_{th}) proportional to ΔT . In these measurements the input current of the TCD-D is swept in the range [0 - 1] mA and in the range of [0 - 2] mA in the case of TCD-S. These ranges have been chosen by using the results of simulations of Chapter 6 (section 6.1). The value of the resistance of the heater (7.73 k Ω) has been used to convert power to current. Simulations have shown that for air an input power close to 5 mW (0.8 mA) is required to heat the membrane of the TCD-D to achieve a temperature of 310 K in the thermopiles. In the case of the TCD-S an input power close to 10 mW (1.1 mA) is required. To compare these measurements to static simulations of section 6.1, the results are presented in terms of input power and temperature of the thermopile. The temperature of the thermopile is calculated by using Eq. (15) and the total Seebeck coefficient 3480 $\mu V/K$ (section 5.1). Results are shown in the following Figures.

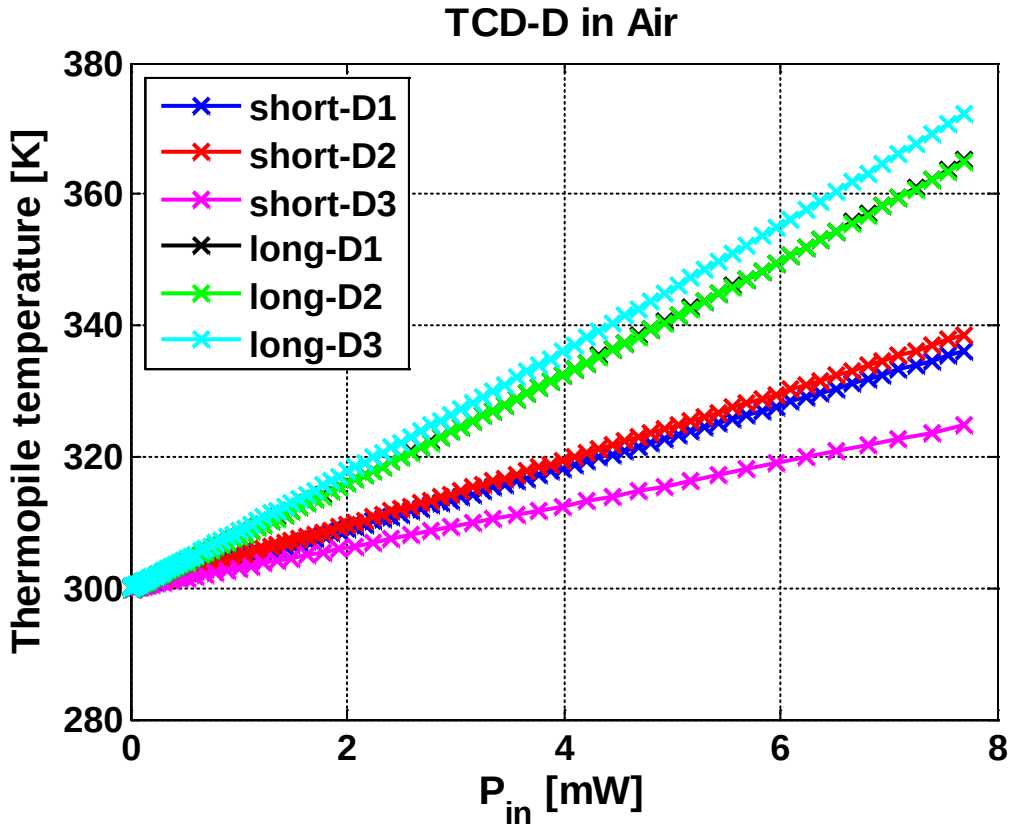


Figure 51: Measurements of the input power vs. thermopile temperature of the deep cavity TCD in air. Three different short TCD devices have been tested as well as three different long TCD devices.

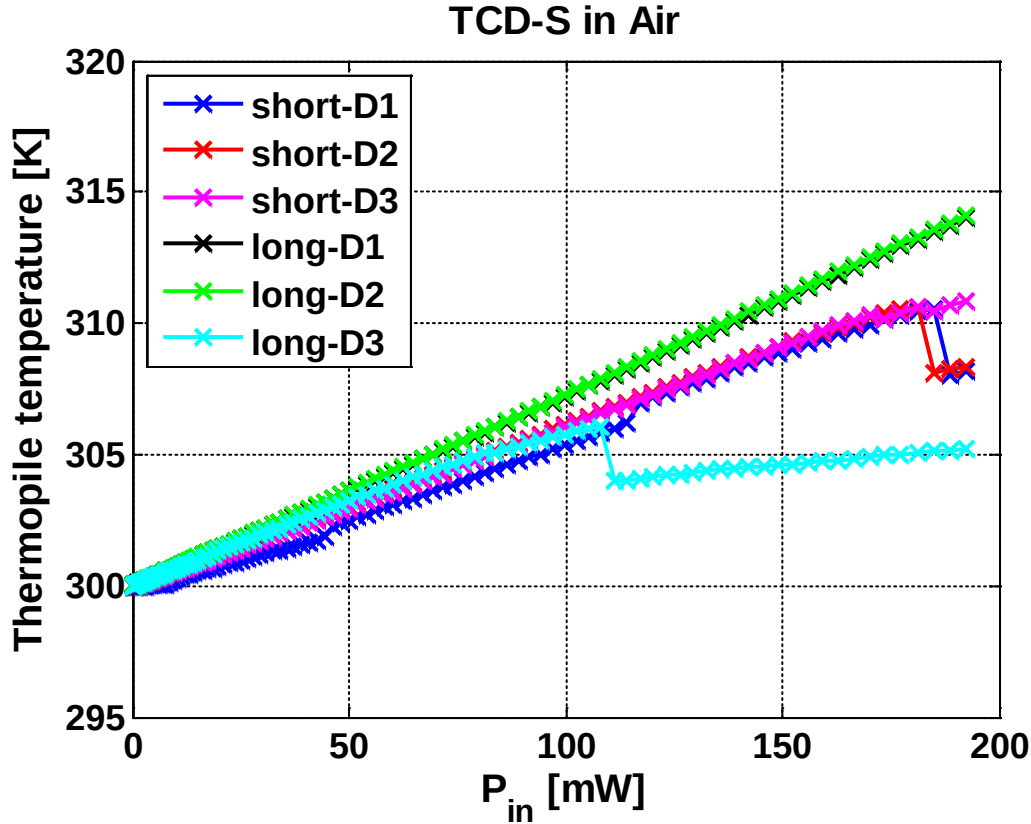


Figure 52: Measurements of the input power vs. thermopile temperature of the shallow cavity TCD in air. Three different short TCD devices have been tested as well as three different long TCD devices.

From Figures above, it can be seen that there are three short dual-TCDs (out of 7) and three long dual-TCDs (out of 5) that are working. The other fabricated devices showed very high output thermopile voltages in the order of 50 V. It is possible that the contacts of these devices are connected to the substrate increasing their electrical resistance and as a consequence very high voltages are achieved.

Both results show the linear relation between the input power and the thermopile temperature (thermopile voltage). Another important result is that long devices require less power than short devices to achieve the same temperature. This implies that long devices are more power efficient and have a higher T/P_{in} sensitivity than short devices.

Comparing Figure 51 to Figure 52, it is clear that deep cavity TCDs require less power than shallow cavity TCDs to achieve the same temperature. This confirms that the TCD-S is much more sensitive than the TCD-D losing more heat in the cavity through the gas (in this case air).

Figure 51 shows the results of the deep cavity TCDs. In the case of short TCDs for an input power in the range of $[0 - 7.5]$ mW temperature increases almost 40 degrees. In the case of long TCDs temperature increases almost 70 degrees. To achieve the temperature of 310 K in short devices, an input power of 3 mW is required. In the case of long devices an input power of 1.3 mW is necessary. These results differ from simulations where an input power of 5 mW is used. Comparing the model used in simulations to the long TCD, there is an important difference in dimensions between both models which can explain the difference

in the input power. In the case of short TCD, the dimensions are also distinct, especially the length of the heater. The heater of the short TCD is almost 200 μm longer than the model in Comsol. Another reason is that, the physical properties of the materials might change during the fabrication process and the values may not be the same as the ones used in simulations.

Figure 52 shows the results of the shallow cavity TCDs. The first impression of these results is the high power consumption of the TCDs. These results are unexpected because simulations have shown that an input power in the order of 10 mW is required to achieve 310 K in the thermopile. Figure 52 shows input powers in the order of 100 mW to achieve a thermopile temperature of 310 K, 10 times higher than simulations. One possible reason of this behavior is that the membrane could be stuck to the substrate, in consequence, high power is required to heat up a bigger area with different thermal conductivities. Another reason is that the shallow cavity may not have been etched properly during the fabrication process. Thus, the cavity could be thinner than the expected 2 μm . Also, it is possible that the heater of the shallow cavity device is not heating the membrane. Also, the thermopiles of the TCD-S may not work properly because some parts of them are stuck to the substrate. However, this last reason is the less probable because the thermopiles of the other TCD-D are working properly and the thermopiles of both devices have been fabricated by using the same process. Besides, there is an output signal from the thermopiles of the TCD-S; a small but it is possible to see changes.

8.3 Measurements Using an Infrared Camera

Measurements using an infrared camera have been done to find a reason of the high power consumption of the TCD-S. The idea of using the IR camera is to see if the heater of the TCD-S is heating the membrane. The infrared camera is placed on top of the heaters of the dual-TCD to measure the temperature of the heaters while applying an input power. Also, the output thermopile voltage is measured by using a nano/micro voltmeter. The measurement setup consists of a power supply, a nano/micro voltmeter and the infrared camera, as shown in Figure 53.

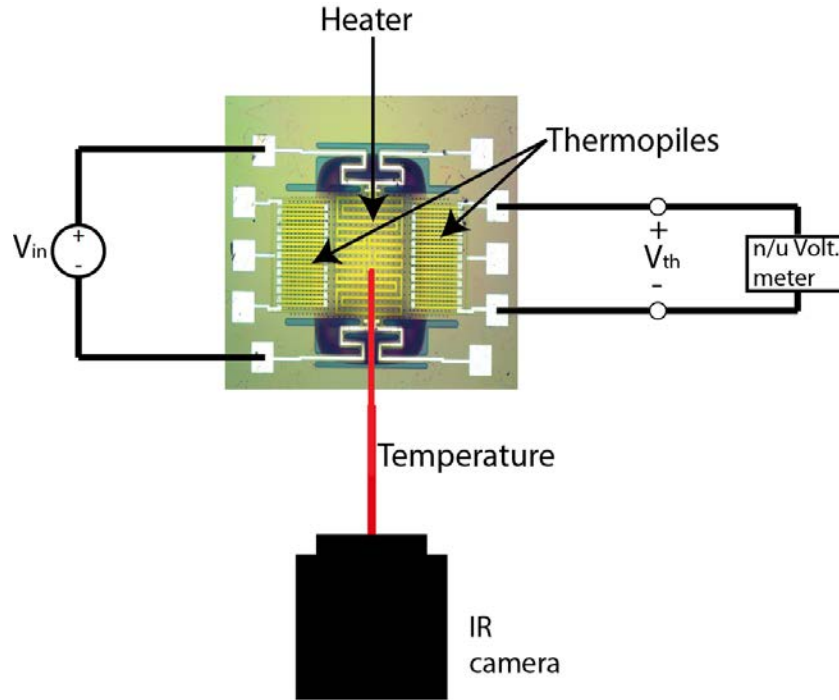


Figure 53: Measurement setup by using an IR camera to measure the temperature of the TCDs.

Measurement of the input power vs. the temperature measured by using the IR camera are shown in Figure 54 and Figure 55.

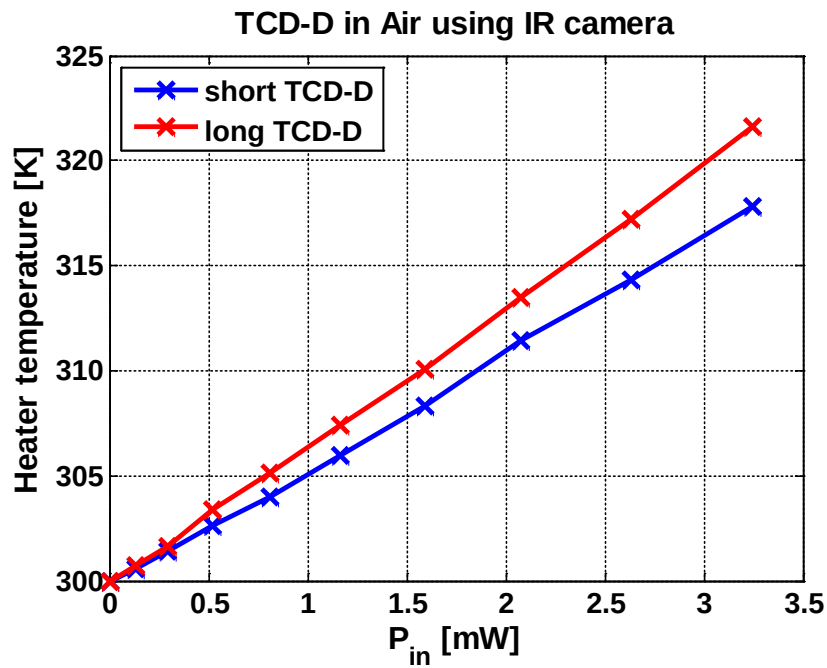


Figure 54: Measurements of the input power vs. heater temperature of the deep cavity TCD in air by using the IR camera.

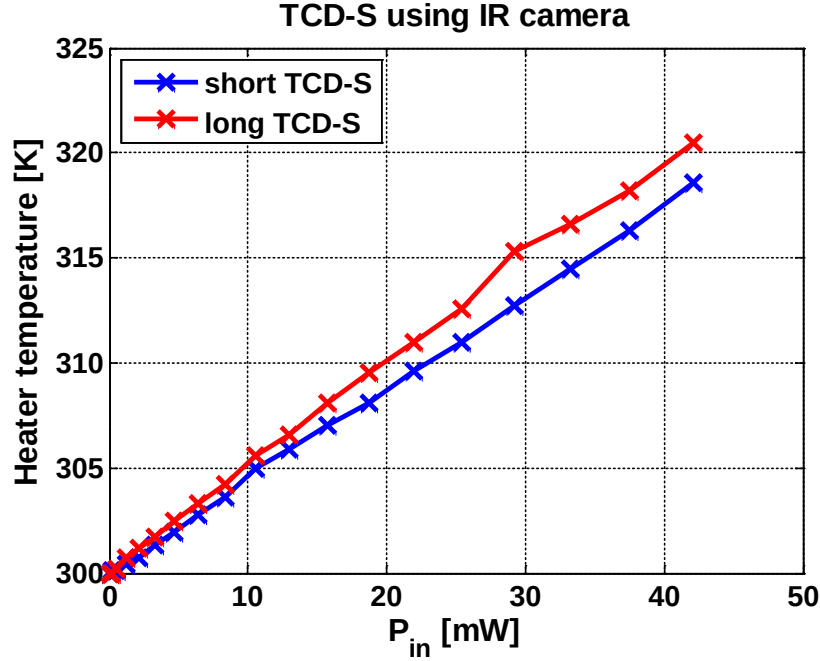


Figure 55: Measurements of the input power vs. heater temperature of the shallow cavity TCD in air by using the IR camera.

The results corresponding to the deep cavity TCDs are shown in Figure 54. These results are in line with the results obtained using the probe station Cascade 33 (Figure 51). It shows that the long TCD is more sensitive than the short TCD to input power, confirming this behavior.

On the other hand, Figure 55 shows the results corresponding to the shallow cavity TCD. The temperatures measured by the IR camera show a linear increment proportional to the input power. This confirms that the heater of the TCD-S works properly. Moreover, these measurements are in accordance with simulations. However, these differ from those obtained by using the probe station in terms of input power required to reach certain temperature. For example, to achieve a temperature of 310 K, the IR camera shows that an input power of 19 mW is required, but in the case of the probe station the input power is 130 mW for the same temperature.

The output thermopile voltages of the TCD-D and TCD-S have been measured as well. The thermopile voltages are converted to temperature by using the total thermopile Seebeck coefficient of 3480 $\mu\text{V/K}$. Temperatures measured by the IR camera and by the thermopiles are compared in Figure 56 and Figure 57.

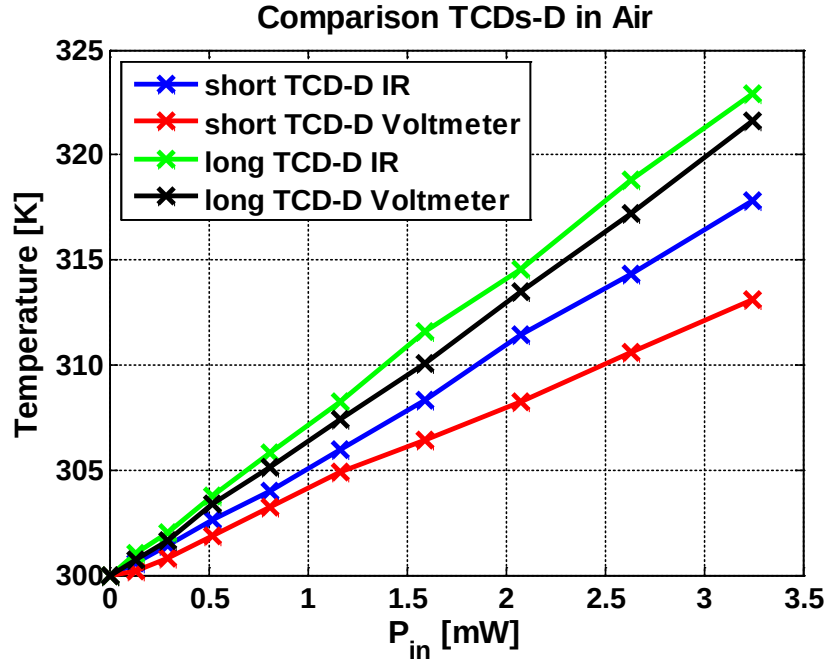


Figure 56: Temperature measurements of the short TCD-D and long TCD-D. Comparison of the temperature measured by the IR camera to the temperature (thermopile voltage) measured by the nano/micro voltmeter.

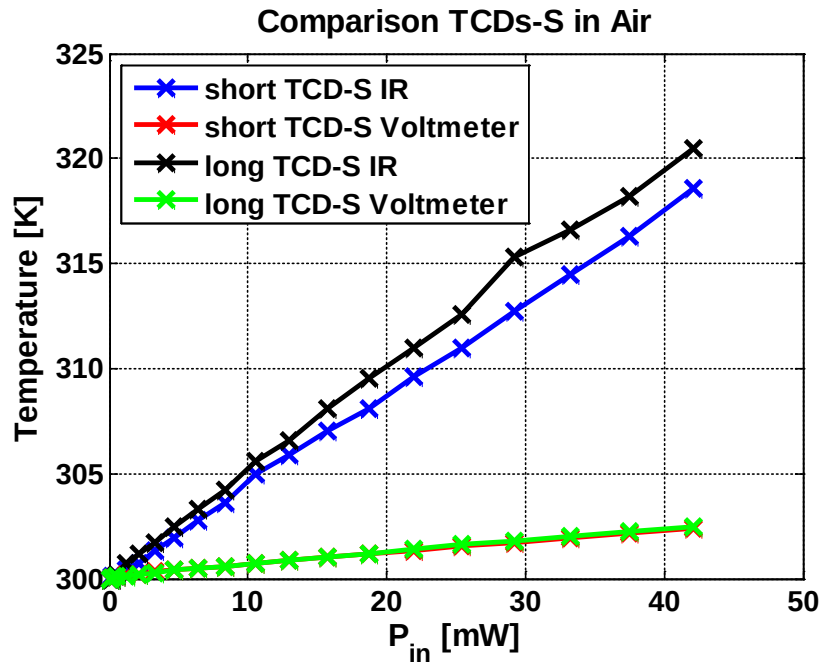


Figure 57: Temperature measurements of the short TCD-S and long TCD-S. Comparison of the temperature measured by the IR camera to the temperature (thermopile voltage) measured by the nano/micro voltmeter.

Figure 56 shows the difference in the temperature of the deep cavity devices measured by the IR camera and the thermopile (by using the nano/micro voltmeter). It can be seen that the temperature measured by the IR camera is higher than the temperature measured by the thermopiles. The difference between these two values

is approximately 1.5 % in the case of short TCD-D and approximately 0.5 % in the case of long TCD-D. This small difference can be explained because the temperature of the membrane is measured at a certain distance from the center of the heater. If the thermopiles were close to the heater, the temperature measured by the IR camera and the thermopiles were almost the same.

Figure 57 shows the difference in the temperature of the shallow cavity devices measured by the IR camera and the thermopile. In the case of the TCD-S devices, there is a big difference in the temperature measured by the IR camera and the temperature measured by the thermopiles. The temperature measured by the IR camera is approximately 7 times higher than the measured by the thermopiles.

These measurements discard the possibility that the heater was not operating as expected. This gives the idea that the membrane could be stuck to the substrate or the sample chamber does not have the expected depth of 2 μm . To verify if there is a problem with the sample chamber underneath of the membrane, measurements in vacuum were proposed.

8.4 Measurements in the Vacuum Chamber

Measurements in the vacuum chamber has been done to verify if there is sample chamber in TCD-S. In the case that there is sample chamber both devices of the dual-TCD should have the same thermopile output voltages. If there is no sample chamber these voltages should be very different. The measurement setup of the measurements in vacuum is composed of a power supply, the nano/micro voltmeter and the vacuum chamber. The output thermopile voltage is measured by using the nano/micro voltmeter and it is converted to temperature dividing it by the total Seebeck coefficient 3480 $\mu\text{V/K}$. Figure 58 shows the measurements in vacuum.

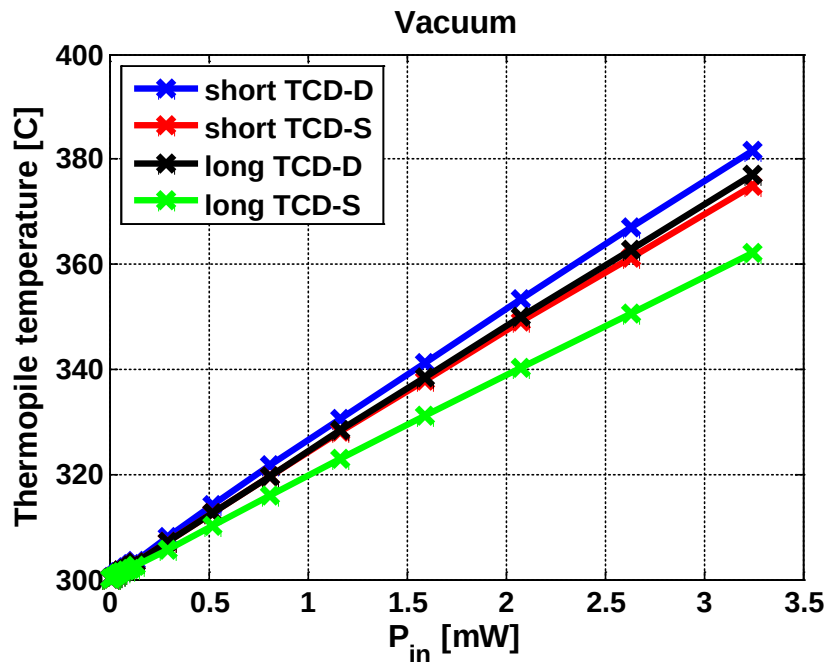


Figure 58: Measurements of the input power vs. thermopile temperature of the short dual-TCD and long dual-TCD in vacuum.

Measurements in vacuum show that the long dual-TCD and the short dual-TCD achieve a similar thermopile temperature (output thermopile voltage) applying an input power in the range of [0 – 3.5] mW. This confirms that there is shallow cavity underneath of the membrane. However, the problem of the low output thermopile voltage persists. One reason of the low sensitivity is that the cavity could be shallower than the expected 2 μm . To see if this is the reason simulations of the dual-TCD in Comsol for cavities narrower than 2 μm have been done. Simulations have been compared to measurements of the short TCD-S and the long TCD-S. Results are shown in Figure 59.

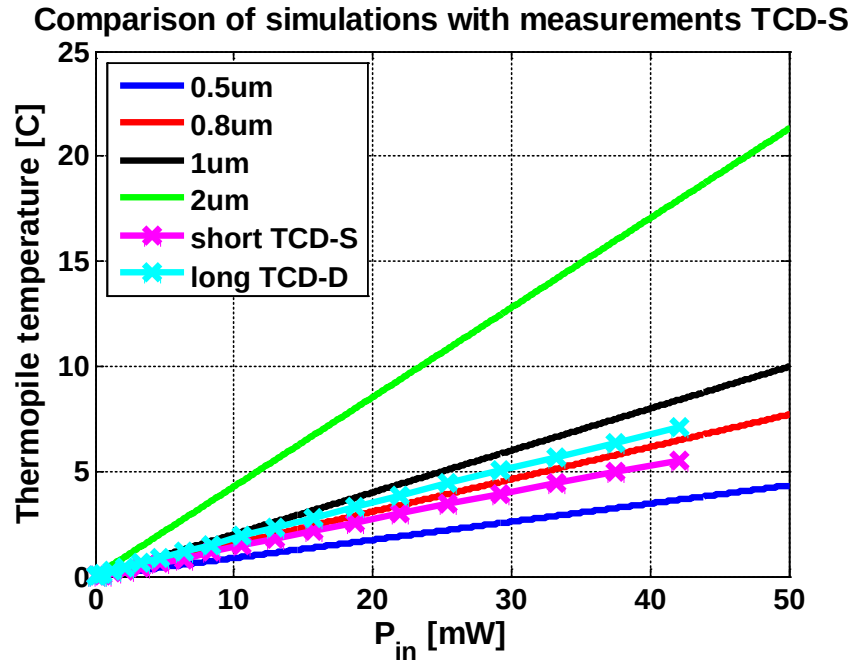


Figure 59: Comparison of simulations with measurements of the TCD-S device.

From Figure 59 is possible to see that measurements of the TCD-S devices are similar to those simulations of the thermopile temperature for a cavity depth of 0.8 μm . These results confirm that the cavity is shallower than 2 μm . It is also possible that the membrane could be bended in direction to the cavity at the position of the thermopiles, reducing the cavity gap. The difference between the simulated results and the measurement results is approximately 7 times for a cavity depth of 2 μm .

8.5 Measurements Using CO₂ and He

Measurements using CO₂ and He have been done. These measurements are compared to measurements in vacuum and Air. In the case of the shallow cavity TCD a compensation factor of 7 has been applied to the measured temperature. This compensation factor has been found in the previous section to compensate the fact that the shallow cavity is not exactly 2 μm depth. Figure 60 and Figure 61 show these results.

Measurements in vacuum, CO₂, Air and He; short TCD-D

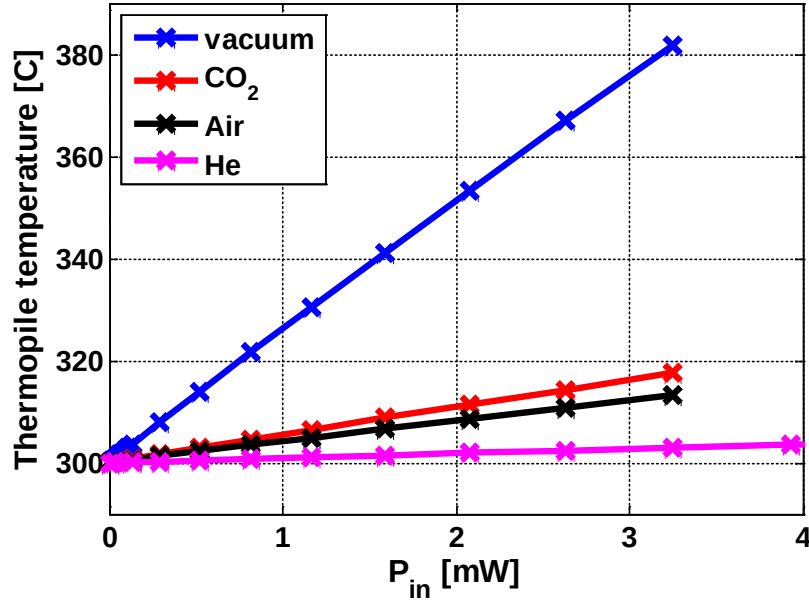


Figure 60: Input power vs. thermopile temperature for different gases using the short TCD-D.

Measurements in vacuum, CO₂, Air; long TCD-S

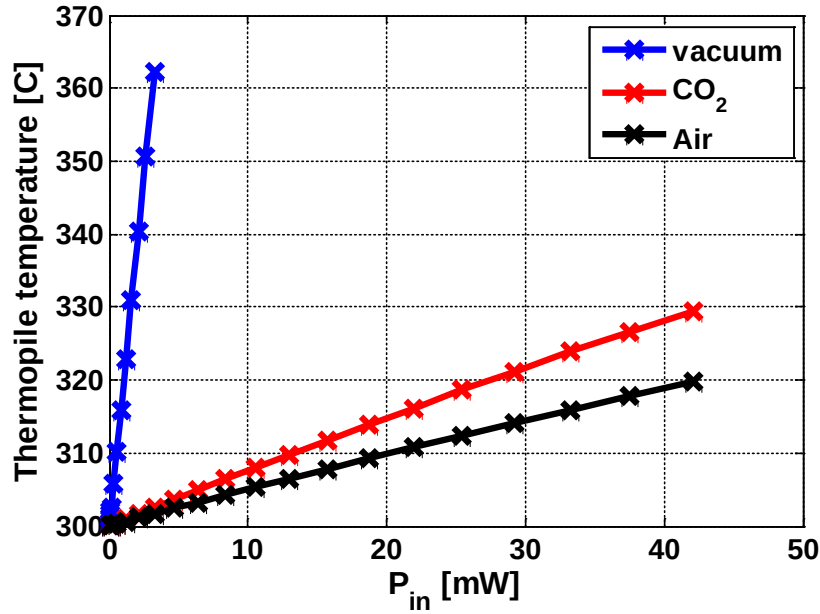


Figure 61: Input power vs. thermopile temperature for different gases using the short TCD-S.

From Figure 60 and Figure 61 it can be seen that both TCDs of the dual-TCD are sensitive to measure different gases. Also, the TCD-D requires less power than TCD-S to achieve the same temperatures at the position of the thermopiles.

The T/P_{in} sensitivity and the V_{th}/P_{in} sensitivity for the TCD-S and TCD-D are shown in Table 26.

Table 26: T/P_{in} sensitivity and the V_{th}/P_{in} sensitivity for the TCD-S and TCD-D for different gases.

Gas	TCD-S $S_{T/P_{in}}$ [K/W]	TCD-D $S_{T/P_{in}}$ [K/W]	TCD-D $S_{V_{th}/P_{in}}$ [V/W]	TCD-D $S_{V_{th}/P_{in}}$ [V/W]
Vacuum	19156	25130	66,67	87,45
CO ₂	701	5482	2,44	19,08
Air	473	4065	1,65	14,15
He	-	908	-	3,16

Time responses to a step of 1 V have been done for different gases. The time responses for different gases are shown in Table 27.

Table 27: Time responses for different gases of the TCD-S and TCD-D.

Gas	TCD-S t_r [ms]	TCD-D t_r [ms]
Vacuum	22	22
CO ₂	15	15
Air	12	12
He	-	5

Table 27 shows that the timer responses depends on the thermal conductivity of the gas. For higher thermal conductivity the time response decreases.

If these results are combined in a thermal conductivity vs. input power plot, it is possible to find the P_{in}/λ sensitivity to compare it to simulations. Table 28 and Figure 62 show the measurement results.

Table 28: Measurements of the static sensitivity of the dual-TCD. The value of He for TCD-S has been calculated by using the obtained sensitivity.

Gas	Thermal conductivity λ [W/m·K]	TCD-S P [mW]	TCD-D P [mW]
Vacuum	0	0.507	0.370
CO ₂	0.01622	13.25	1.81
Air	0.02604	20.25	2.43
CH ₄	0.03458	-	-
He	0.14200	109.97	10.95
H ₂	0.18690	-	-

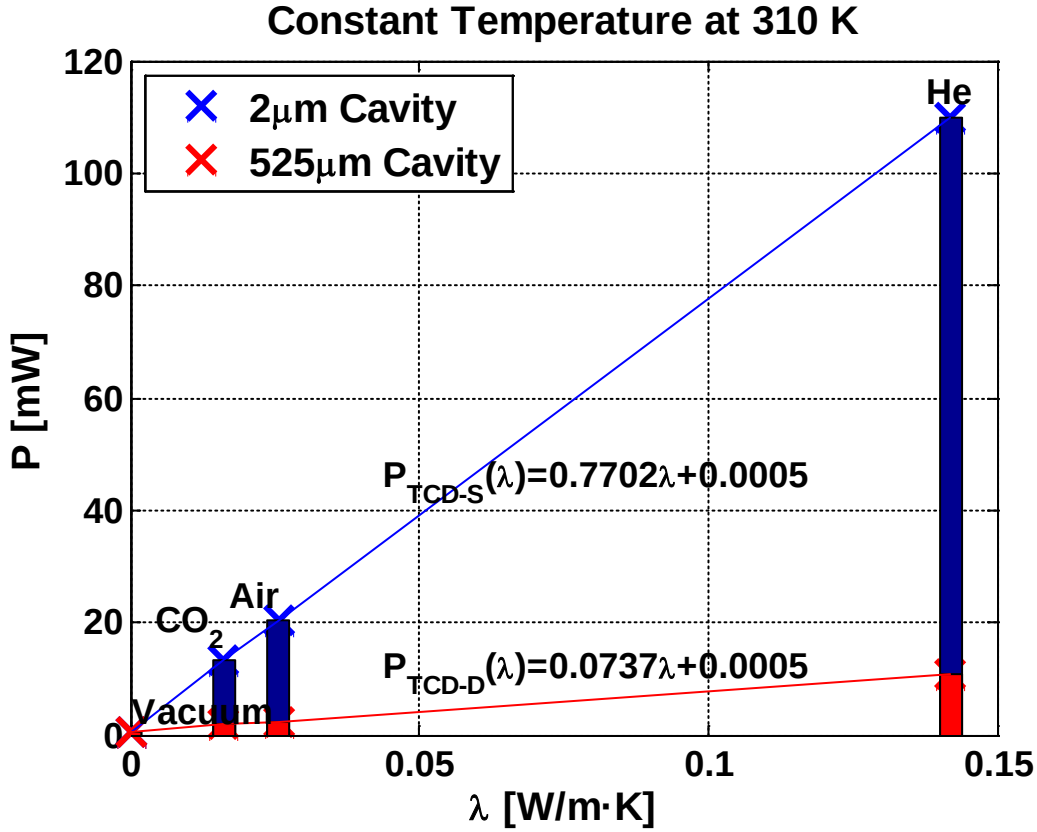


Figure 62: Measurement of the static response of the dual-TCD. Total power of each TCD vs. thermal conductivity.

Figure 62 shows the measurement of the static response of the dual-TCD for different gases. It can be seen that there is a power consumption in vacuum which is due to the effect of the conductivity of the suspension of the membrane. The TCD-S is much more sensitive than TCD-D requiring much more power to achieve a temperature of 310 K in the position of the thermopiles. As a result the TCD-S is much more sensitive to the gas thermal conductivity than TCD-D. This result confirms that the TCD-S is the sensor used to measure the gas while TCD-D acts as a compensation device.

Figure 62 shows that the relation between the power consumption and the gas thermal conductivity is almost linear. These relations are as follows:

$P_{TCD-S}(\lambda) = 0.7702 \cdot \lambda + 0.0005$ [W] with a correlation coefficient $R^2 = 0.9997$ and $P_{TCD-D}(\lambda) = 0.0737 \cdot \lambda + 0.0005$ [W] with $R^2 = 0.9995$. The sensitivity of the TCD-S device is $S_\lambda = 0.7702$ W/(W/m·K), while the sensitivity of the TCD-D device is only $S_\lambda = 0.0737$ W/(W/m·K). The sensitivity of the TCD-S is 10 times higher than the sensitivity of the TCD-D.

Table 29: Comparison of measurements with simulations.

Device	Measurements S_λ [W/W/m·K]	Simulations S_λ [W/W/m·K]	Times higher
TCD-S	0.7702	0.136	5.7
TCD-D	0.0737	0.012	6.1

Table 29 shows that the sensitivity of measurements is approximately 6 times higher than simulations. This difference could be because the dimensions of the fabricated dual-TCD are not the same as the simulated dual-TCD. Another possibility is that the physical properties of the materials of the fabricated dual-TCD might suffer from variations during the fabrication process. However, the difference in sensitivity between both TCDs for real measurements and simulations is approximately 10 times.

8.6 Summary of the Chapter

Different dual-TCD devices have been measured. First using the probe station Cascade 33 devices have been tested to see which of the fabricated devices work properly. These first measurements have demonstrated that the TCD-S is more sensitive than TCD-D when measuring Air. Also, these measurements have shown that the TCD-S requires much more power than simulations. Different reasons for this difference in power have been proposed.

A second measurement using an infrared camera has been done. The results of these measurements have demonstrated that the heaters of the dual-TCD are working properly, heating the membranes.

Measurements in vacuum have been done. These measurements demonstrated that there is shallow cavity underneath of the membrane for TCD-S. Also, these measurements have shown that there is no problem with the thermopiles of the TCD-S. From these results it has been thought that the shallow cavity is not deep enough as expected. Simulations for different depth of the cavity have been redone and compared to measurements results. This comparison has shown that the cavity of the real TCD-S is approximately $0.8\text{ }\mu\text{m}$ instead of $2\text{ }\mu\text{m}$. A compensation factor of 7 times has been used to compensate this difference.

Measurements using Air, CO_2 and He have been done. The results have demonstrated that TCD-S is much more sensitive than TCD-D to different gases. Thus, TCD-S is the device used to measure gases while TCD-D is used as a compensation device. The P_{in}/λ sensitivity of the TCD-S is 10 times higher than the P_{in}/λ sensitivity of the TCD-D. This result is in agreement with simulations.

Chapter 9 Conclusions and Future Work

9.1 Conclusions

Principle

A flow compensated gas sensor using a dual-thermal conductivity detector has been designed, simulated and fabricated. The dual-TCD gas sensor uses two almost identical TCDs to compensate the flow. Both TCDs are composed of independent components. These are: a heater, thermopiles and a sample chamber. The heaters and thermopiles are placed on top of a low thermal conductivity thin membrane which is placed on top of the sample chambers. The difference between both TCDs is the depth of the sample chamber, one of them is very shallow while the other is very deep. The shallow device is very sensitive to thermal conductivity of the gas while the deep device acts as a compensation device only. Working both devices in differential mode a heat transfer by convection and heat transfer by conduction of the membrane are canceled and only heat transfer by conduction through the gas remains. Thus, a flow compensated gas sensor is achieved. One of the main advantages of the dual-TCD gas sensors is that, it is placed in the sensing gas path without the need to use two different gas paths to reduce the effect of thermal noise produced by flow. Also, it does not need to be calibrated for different gas depending on the flow. The dual-TCD compensates the thermal noise of the flow in-line without the need of a reference.

The heat transfer behavior of the sensor shows that the TCD-S is much more sensitive than the TCD-D to thermal conductivity.

Simulations

Two different simulations have been performed: statics and dynamics. Static simulations are performed without using a flow while dynamic simulations use different flows through the dual-TCD.

Static simulations have shown that the TCD-S needs more power than TCD-D to achieve the same temperature at thermopiles positions. Moreover, TCD-S has shown having a higher sensitivity than TCD-D to thermal conductivity of the gas. This confirms the idea that the shallow TCD can be used as a sensing device while the deep TCD is used as a compensation device. Simulations have shown that the dual-TCD has a power consumption in vacuum ($\lambda=0$) which is due to the effect of the conductivity of the suspension of the membranes (G_b). The sensitivity achieved by the TCD-S is $S_\lambda = 0.136 \text{ W}/(\text{W}/\text{m}\cdot\text{K})$, while the sensitivity of the TCD-D is only $S_\lambda = 0.012 \text{ W}/(\text{W}/\text{m}\cdot\text{K})$. Therefore, the sensitivity of the shallow device is 11 times higher than the sensitivity of the deep device.

Different dynamic simulations have been performed. The first one is to realize if the flow inside of the cavities is low enough to neglect the convection inside. Figure 27 shows that the flow inside of the cavities is very low in comparison to the input flow, this confirms that the dimensions of the holes ensure that the convection in the cavities can be neglected.

Also, the time response of the sensor has been calculated for different sizes of the holes in the range of [10 - 100] μm . Simulations show that the time response of the sensor has very small variation depending on the size and for a size of 20 μm the time response is $t_G = 0.4 \text{ ms}$.

The flow response of the dual-TCD has been performed changing the power supply of the heaters to maintain a constant temperature of 310 K at 44 μm from the center of the heater (thermopile position). The range of the flow is [0 - 10] ml/min. First simulations show that the power to flow sensitivity of each TCD is different in the case of He and H₂ (50% difference). As a consequence that the differential power principle of the dual-TCD does not work for these gases. The reasons for this sensitivity difference have been found. Simulating the temperature distribution of Equation (25), it shows that the TCD-D is not deep enough to compensate the whole sensor. In the case of He and H₂ a deep cavity deeper than 1500 μm is recommended (Figure 31). Another reason is that the heat profile of the TCDs is different as shown in of Figure 25. This is due to the fact that the heat in the TCD-S is mainly through the gas in the cavity while in the case of the TCD-D there is an important loss in the membrane. Based on this, the P/flow sensitivity has been simulated regarding the temperature measurement from the heater. Figure 32 have shown that closer to the heater the power to flow sensitivity difference is considerably reduced from 50% to 3% for He and H₂. Having this in mind, a 50% compensation has been applied to the dual-TCD and results show that the P/flow sensitivity of both TCDs is almost the same (Figure 30 and Figure 33). Calculating the power difference between the two TCDs, results show that flow dependence is reduced notably to 23 times in the case of He (Figure 34 and Table 15).

Thus, a flow compensated device can be achieved by using the dual-TCD principle.

Fabrication

Devices have been fabricated by using surface-micromachining MEMS process. Two types of dual-TCD have been fabricated: short dual-TCD and long dual-TCD. The difference between them is the length of the thermopiles. The thermopiles of the short dual-TCD have a length of 165 μm while the thermopiles of the long dual-TCD have a length of 600 μm . Seven short dual-TCDs and five long dual-TCDs have been fabricated.

Encapsulation Module and Read-out Control Loop Circuit

A novel measurement setup has been designed and fabricated. It is composed of an encapsulation module and a read-out circuit. The encapsulation module has been designed in such a way to ensure laminar flow on top of the dual-TCD and its surroundings. Calculations and simulations of the encapsulation module confirm that the dimensions of it ensure laminar flow. The highest Reynolds number is 5.35 for CO₂ @ 10 mL/min.

The read-out circuit is supposed to control the temperature of the thermopile by using a voltage or current reference. The values of voltage and current references for different gases has been found in the simulations section (Table 16 and Table 17). The read-out circuit measures the output Seebeck voltage of the thermopiles and compares it to the reference. Depending on the value of the comparison, then, this signal is added to or subtracted from the reference. Thus, depending on the gas the control loop provides more or less power to the heater to maintain the temperature of the thermopile. The block that contributes with the higher amount of noise is the instrumentation amplifier. The noise of the INA has been calculated and it is $n_{INA} = 0.646 \mu\text{V}/\sqrt{\text{Hz}}$. This input referred noise contributes a noise in terms of thermal conductivity of $\lambda_n =$

$9.369 \times 10^{-16} \text{ W/W/m}\cdot\text{K @ 10 Hz @ 310 K}$. This value is negligible and it should not affect the measurements of thermal conductivity.

An AC system has been proposed to reduce the noise and offset of the INAs by using modulation-demodulation stages.

The read out control loop circuit has been built by using discrete components. These components have been chosen in such a way to have the less amount of noise.

Measurements of the INAs have shown that a gain of 10 is achieved. This gain is 10 times lower than the expected gain of 100. This difference is because the potentiometers do not reach the default value. The noise of the INAs have been measured and it has a value of $3 \mu\text{V @ 10 Hz}$. This value is comparable to the calculated noise of $2 \mu\text{V @ 10Hz}$. It is possible that the difference of $1 \mu\text{V}$ in the noise could be because of the inner resistance of the (wiring) paths of the PCB. The bandwidth of the INAs results in 15 kHz. The bandwidth is in the order of the simulated value of 20 kHz.

Measurements

Short and Long dual-TCDs have been tested by using the probe station Cascade 33. These measurements have done to choose the best samples for the following measurements. These test have demonstrated that the TCD-S a requires much more power than TCD-D to achieve the same temperature. Also, measurements show that the relation between the input power and the thermopiles temperature (thermopile voltage) is linear. A problem with the TCD-S have been found. These devices require much more power than those of simulations. Different measurements have been done to see the reason for this problem.

Measurements using an infrared camera have indicated that both heaters increase their temperature while supplying them with an input power. This concludes that there is no problem with the heaters and they are reacting to different input powers. Also, these measurements confirm that measurements at a certain distance from the heater changes the temperature measured by the thermopiles. After these measurements the problem has been focused on the depth of the shallow cavity and the sensitivity of the thermopiles of the TCD-S.

Measurements in vacuum have been done to see if there is shallow cavity as well as to test the sensitivity of the thermopiles of the TCD-S. These measurements have shown that both TCDs have almost the same thermopile temperature – input power behavior. This concludes that there is shallow cavity as well as the thermopiles are sensitive to temperature changes. Simulations of the thermopile temperature for different depth of the shallow cavity have been done. These simulations have been compared to measurements of the TCD-S in Air. These comparisons have shown that the cavity has a different depth than the expected. The real value of the shallow cavity might be $0.8 \mu\text{m}$ instead of the expected $2 \mu\text{m}$. From this result a compensation factor of 7 should be used.

Different gases have been tested. These measurements have shown that the sensitivity of the TCD-S is 10 times higher than the sensitivity of the TCD-D. This result is in agreement with simulations. However, the real devices require much power than the simulated dual-TCD to reach a temperature of 310 K. This difference in the P_{in}/λ is probably because the real dual-TCD has different dimensions than the simulated

device. The real dual-TCD has a higher P_{in}/λ sensitivity than the simulated dual-TCD but the simulated device is more power efficient than the real one.

Performance comparison

	Sensitivity [mV]	Power [mW]	Time response [s]	Sensitivity P/λ [W/W/m·K]	Flow compensation
[28]	160 (H ₂ in air)	Catalytic activation	7	-	Using a reference device in a reference flow path
[29]	20 (toluene)	12500	20	-	Until a flow of 3 sccm.
[31]	15	12	150 μ s	-	Using a multiple TCD region, central regions flow independent
Dual-TCD	34.8	120.92 (for He)	5 ms (for He)	0.7702	Using one TCD to measure the gas and another as compensated device. Both devices are placed in the same gas stream.

Simulations and measurements have demonstrated that the flow compensated principle by using a dual-TCD works and it could be a solution to cancel the effect of flow while measuring gases.

9.2 Future work

The dual-TCD performance has to be tested by using other gases like CH₄ and H₂. Also, the flow compensation performance of the dual-TCD has to be tested by using a mass flow controller. This will completely characterize the device.

The read-out control loop circuit has to be used together with the dual-TCD. Also, the INAs stages should be redesigned to compensate the lower sensitivity of the shallow cavity device due to the narrower cavity.

Bibliography

- [1] S. Chen, “Surface-micromachined thermal conductivity gas sensors for hydrogen detection”, M.Sc. dissertation, Faculty of Electrical Engineering, Mathematics and Computer Science, Delft University of Technology, Delft, The Netherlands, October 2010.
- [2] H. Wu, “MEMS-Based linear thermopile detector arrays for IR microspectrometers”, Ph.D. dissertation, Faculty of Electrical Engineering, Mathematics and Computer Science, Delft University of Technology, Delft, The Netherlands, August 2011.
- [3] S. Ohira, and K. Toda, “Micro gas analyzers for environmental and medical applications”, *AbalyticaChimicaActa* 619 (2008), pp. 143-156.
- [4] L. Francioso, R. Rella, P. Siciliano, J. Spadavecchia, D. Presicce, A.M. Taurino, S. Capone, and A. _Forleo, “Solid state gas sensors: state of art and future activities”, *Journal of Optoelectronics and Advanced Materials*, vol. 5, pp. 1335-1348, 2003.
- [5] P. Tardy, J.-R. Coulon, C. Lucat, and F. Menil, “Dynamic thermal conductivity sensor for gas detection”, *Sensors and Actuators B*, vol. 98, pp. 63-68, 2004.
- [6] P.T. Moseley, “Solid state gas sensors”, *Meas. Sci. Technol.*, vol.8, No 3, pp. 223-237, 1997.
- [7] S.J. Kim, “Gas sensors based on Paschen’s law using carbon nanotubes as electron emitters”, *Journal of Physics D: Applied Physics*, vol. 39, pp. 3026-3029, 2006.
- [8] S. Chen, “Surface-micromachined thermal conductivity gas sensors for hydrogen detection”, M.Sc. dissertation, Faculty of Electrical Engineering, Mathematics and Computer Science, Delft University of Technology, Delft, The Netherlands, October 2010.
- [9] J.L. Cruz D. Zalvidea, A. Diez and M.V. Andres. Hydrogen sensor based on apalladium-coated fibre-taper with improved time-response. *Sensors and Actuators*, 114:268–274, 2006.
- [10] H. Xia Z. Zhao, M.A. Carpenter and D. Welch. All-optical hydrogen sensor based on a high alloy content palladium thin film. *Sensors and Actuators*, 113:532–538, 2006.
- [11] R.P. Manginell, J.H. Smith, and A.J. Ricco, “An overview of micromachined platforms for thermal sensing and gas detection”, *Proceedings of SPIE on International Society of Optical Engineering*, pp. 273–284, 1997.
- [12] H.A. Daynes, *Gas Analysis by Measurement of Thermal Conductivity*, Cambridge University Press, Cambridge, Great Britain, 1933, pp. 1–302.
- [13] W. Jennings, E. Mittlefehldt and P. Stremple, *Analytical Gas Chromatography*, Second Edition, Elsevier Inc., ISBN-13: 978- 0-12-384357-9, 389 pages. Sep. 1997.
- [14] S.C. Terry, J.H. Jerman (1981): A miniature gas chromatograph for atmospheric monitoring. *Environ. Intl.* **5**, pp. 77–83.
- [15] J. H. Lienhard IV and J. H. Lienhard V, “A heat transfer textbook third edition”, Phlogiston press, Cambridge, Massachusetts, United States, 2008.
- [16] X. J. Hu, A. Jain, and K. E. Goodson, “Investigation of the natural convection boundary condition in microfabricated structures”, *International Journal of Thermal Sciences*, vol. 47, pp. 820-824, 2008.

- [17] De-Yi Shang, "Theory of heat transfer with forced convection film flows", Springer-Verlag Berlin Heidelberg, DOI: 10.1007/978-3-642-12581-2, 2011. <http://www.springer.com/us/book/9783642125805>
- [18] T. Elbel, S. Poser, and H. Fischer, "Thermoelectric radiation microsensors", *Sensors and Actuators A: Physical*, vol. 42, pp. 493-496, 1994.
- [19] Seebeck, T. J., "Magnetische Polarisation der Metalle und Erze durch Temperatur-Differenz," *Abhandlung der deutschen akademie der Wissenschaften zu Berlin*, pp. 265-373, 1822.
- [20] Graf, A., Arndt, M., Sauer, M., and Gerlach, G., "Review of Micromachined Thermopiles for Infrared Detection," *Measurement Science and Technology*, vol. 18, pp. 59-75, 2007.
- [21] van Herwaarden, A. W., and Sarro, P. M., "Thermal Sensors Based on the Seebeck Effect," *Sensors and Actuators*, vol. 10, pp. 321-346, 1986.
- [22] O. Reynolds. 1883. An experimental investigation of the circumstances which determine whether the motion of water shall be direct or sinous, and of the law of resistances in parallel channels. *Phil. Trans. Roy. Soc. London*, 174, 935-982.
- [23] R. L. Grob, and E. F. Barry, "Modern Practice of Gas Chromatography", John Wiley & Sons, Inc., Hoboken, New Jersey, 2004.
- [24] B. C. Kaanta, H. Chen, and X. Zhang, "Novel Device for Calibration-free Flow Rate Measurements in Micro Gas Chromatography Systems," *J. Micromech. Microeng.* 20 (2010) 095034 (7pp), doi:10.1088/0960-1317/20/9/095034, August, 2010.
- [25] D. Randjelović, V. Jovanov, Ž. Djurić, and M. Matić, "Vacuum MEMS Sensor Based on Thermopiles-Simple Model and Experimental Results," *Proc. 26th International Conference on Microelectronics (MIEL 2008)*, Niš, Serbia, 11-14 May, 2008.
- [26] S. Udina, M. Carmona, G. Carles, J. Santander, L. Fonseca, and S. Marco, "A Micromachined Thermoelectric sensor for Natural Gas Analysis: Thermal Model and Experimental Results," *Sensors and Actuators B: Chemical*, vol. 134, pp. 551-558, 2008.
- [27] de Graaf, G., and Wolffenbuttel, R., "Surface-micromachined Thermal Conductivity Detectors for Gas Sensing," *I2MTC 2012 Conf.*, pp. 1861-1864, 2012.
- [28] Chung-il Lee Yong-Jun Kim Se-Chul Park, Seung-Il Yoon and Soonho Song. A micro-thermoelectric gas sensor for detection of hydrogen and atomic oxygen. *Analyst*, 134:236-242, 2009.
- [29] J. H. Sun, D. F. Cui, X. Chen, L. L. Zhang, H. Y. Cai, and H. Li, "A micro gas chromatography column with a micro thermal conductivity detector for volatile organic compound analysis", *Review of Scientific Instruments* 84, 025001, 2013.
- [30] D. Cruz, J. P. Chang, S. K. Showalter, F. Gelbard, R. P. Manginell, and M. G. Blain, "Microfabricated thermal conductivity detector for the micro-ChemLab", *Sensors and Actuators B: Chemical*, vol. 121, pp. 414-422, 2007.
- [31] B. C. Kaanta, H. Chen and X. Zhang, "Flow Rate Insensitive Thermal Conductivity Detector", *Transducers 2011*, Beijing, China, June 5-9, 2011.
- [32] <http://www.comsol.com/>
- [33] <http://datasheets.maximintegrated.com/en/ds/MAX4526-MAX4527.pdf>
- [34] <http://cds.linear.com/docs/en/datasheet/100737fbs.pdf>

- [35] D. Jurisic and N. Mijat, "Low-sensitivity, low-noise, band-rejection and all-pass active-RC filters", Proceedings of the IEEE International Symposium on Industrial Electronics, ISIE 2005, vol. 3, pp. 1175-1178, 2005.
- [36] <http://datasheets.maximintegrated.com/en/ds/MAX291-MAX296.pdf>
- [37] <http://www.ti.com/lit/ds/symlink/xtr111.pdf>
- [38] <http://www.ti.com/>

Appendices

A.1 Thermal Model of the Sensor

Figure 63 shows the schematic of one of the TCD. The top view shows the dimensions of the sensor in the XY axis and the cross section shows the dimensions in the ZX axis. The temperature along the beam will be derived in the following sections assuming a uniform temperature distribution in the y-direction and a uniform thermal conductivity of the beam[2].

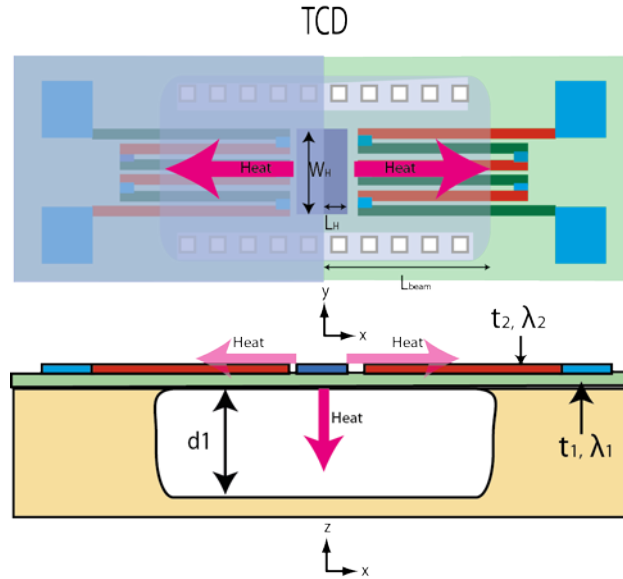


Figure 63: Heat transfer in the thermal conductivity detector.

Heat Loss of the Heater

The heat loss of the heaters is mainly due to radiation and conduction through the gas in the cavity. In terms of radiation, the heat loss P_{rad-H} is given by:

$$P_{gas-H} = \varepsilon \sigma A_H (T_H^4 - T_0^4) \quad (37)$$

where A_H is the area of the beam $A_H = W \cdot L_H$, T_H corresponds to the temperature of the heater and T_0 is the ambient temperature.

Heat conduction P_{cond-H} from the heater to the membrane through the gas in the sample chamber is given by [18]:

$$P_{gas-H} = \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) A_H (T_H - T_0) \quad (38)$$

Where λ_{gas} is the thermal conductivity of the gas in the sample chamber, d_1 is the depth of the cavity and d_2 is the distance from the top of the sensor to the encapsulation.

The temperature of the heater is moderately higher than the temperature of the ambient implying that [2]:

$$T_H > T_0 > 0 \text{ and } \frac{T_H - T_0}{T_0} \ll 1 \Rightarrow \frac{T_H}{T_0} \approx 1. \quad (39)$$

Therefore, the heat radiation can be written as follows:

$$\begin{aligned} P_{\text{rad-H}} &= \varepsilon \sigma A_H (T_H^4 - T_0^4) = \varepsilon \sigma A_H (T_H^2 + T_0^2)(T_H^2 - T_0^2) \\ &= \varepsilon \sigma A_H (T_H^2 + T_0^2)(T_H + T_0)(T_H - T_0) \\ &= \varepsilon \sigma A_H T_0^3 \left(\left(\frac{T_H}{T_0} \right)^2 + 1 \right) \left(\frac{T_H}{T_0} + 1 \right) (T_H - T_0) \\ &\approx 4 \varepsilon \sigma A_H T_0^3 (T_H - T_0). \end{aligned} \quad (40)$$

Thus, the total heat power from the heater to the beam is given by:

$$\begin{aligned} P_H &= P_{\text{in}} - (P_{\text{rad-H}} - P_{\text{gas-H}}) \\ &= P_{\text{in}} - \left(4 \varepsilon \sigma T_0^3 + \lambda_{\text{gas}} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) \right) (T_H - T_0) A_H \end{aligned} \quad (41)$$

First Boundary Condition: Temperature between the Heater and the Beam

The first boundary condition is established in the interface between the heater and the membrane in $x=L_H$. In this position the heat flux is conducted in two ways [2]:

- 1) The heat flux in one dimension can be expressed using Eq. (1):

$$\phi_q = -\lambda_{eq} \frac{dT(x)}{dx} \quad (42)$$

and the thermal conductivity of the beam is equal to:

$$\lambda_{eq} = \frac{\sum \lambda_s \cdot t_s}{\sum t_s} \quad (43)$$

where t_s corresponds to film thickness ($s = 1, 2, \dots$) and the λ_s is the thermal conductivity of each film ($s = 1, 2, \dots$).

- 2) Using the definition of the heat flux which states that it is equal to the amount of energy flowing through a certain surface per unit area per unit time resulting in:

$$\phi_q = \frac{P_H}{A_c} = \frac{P_{in} - \left(4\varepsilon\sigma T_0^3 + \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) \right) (T_H - T_0) A_H}{A_c} \quad (44)$$

where A_c represents the cross sectional area $A_c = W \sum t_s$.

Combining both equations of the heat flux (12) and (14) results in:

$$\phi_q = \frac{P_{in} - \left(4\varepsilon\sigma T_0^3 + \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) \right) (T_H - T_0) A_H}{A_c} = -\lambda_{eq} \frac{dT(x)}{dx}. \quad (45)$$

Using equation above a first order differential solution of the temperature along the interface between the heater and the beam is obtained:

$$\left. \frac{dT(x)}{dx} \right|_{x=L_H} = - \frac{\frac{P_{in}}{W} - \left(4\varepsilon\sigma T_0^3 + \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) \right) (T_H - T_0) L_H}{\sum \lambda_s t_s}. \quad (46)$$

Heat loss of the Beam

The heat loss of the beam is, as well as the heat loss of the heater, due to conduction through the gas in the cavity and radiation along the surface. Using the same equations as in the case of the heater but utilizing the parameters of the beam the heat loss is given by:

$$P_{gas-b} = \lambda_{gas} \left(\frac{1}{d_1} + \frac{1}{d_2} \right) A_{beam} (T(x) - T_0) \quad (47)$$

$$P_{rad-b} \approx 4\varepsilon\sigma A_{beam} T_0^3 (T(x) - T_0) \quad (48)$$

where the surface of the beam is $A_{beam} = W \cdot L_{beam}$ and the temperature of the beam $T(x)$ is in the range $x = [L_H - L_{beam}]$.

Second Boundary Condition: Temperature along the Beam

Rearranging equation (6) the differential equation results in:

$$\frac{\partial(T(x) - T_0)}{\partial x^2} + \frac{\Phi_s}{\lambda_{eq}} = 0 \quad (49)$$

If Φ_s is represented by the relation between the power of the heater per volume $V(V = A_{beam} \sum t_s)$, results in the following relation:

$$\Phi_s = -\frac{P_{rad-b} + P_{gas-b}}{V} = \frac{\left(4\varepsilon\sigma T_0^3 + \lambda_{gas}\left(\frac{1}{d_1} + \frac{1}{d_2}\right)\right)A_{beam}}{A_{beam} \sum t_s}(T(x) - T_0). \quad (50)$$

Combining equations (19), (20) and using (13), the second order differential equation of the temperature along the beam is given by:

$$\frac{\partial(T(x) - T_0)}{\partial x^2} - \frac{\left(4\varepsilon\sigma T_0^3 + \lambda_{gas}\left(\frac{1}{d_1} + \frac{1}{d_2}\right)\right)}{\sum \lambda_s t_s}(T(x) - T_0) = 0. \quad (51)$$

Defining the heat transfer coefficient β by:

$$\beta = \left(\frac{\left(4\varepsilon\sigma T_0^3 + \lambda_{gas}\left(\frac{1}{d_1} + \frac{1}{d_2}\right)\right)}{\lambda_{eq} t_{eq}} \right)^{1/2}. \quad (52)$$

The general solution of the second order differential equation of the temperature of the beam is given by:

$$T(x) - T_0 = Ae^{-\beta x} + Be^{+\beta x}. \quad (53)$$

To find coefficients A and B two boundary conditions are stated:

- At hot edge the temperature is: $T(L_H) = T_H$.
- At cold edge the temperature is: $T(L_{beam}) = T_0$.

Applying both boundary conditions the coefficients of equation (23) are given by:

$$A = \frac{(T_H - T_0)e^{2\beta L_{beam}}}{e^{\beta L_{beam}}(e^{\beta(L_{beam}-L_H)} - e^{-\beta(L_{beam}-L_H)})} \quad (54)$$

$$B = -\frac{(T_H - T_0)}{e^{\beta L_{beam}}(e^{\beta(L_{beam}-L_H)} - e^{-\beta(L_{beam}-L_H)})}. \quad (55)$$

Replacing both coefficient in equation (23) and rearranging the temperature along the beam is given by:

$$\begin{aligned}
T(x) - T_0 &= \frac{(T_H - T_0)e^{2\beta L_{beam}}e^{-\beta x}}{e^{\beta L_{beam}}(e^{\beta(L_{beam}-L_H)} - e^{-\beta(L_{beam}-L_H)})} - \frac{(T_H - T_0)e^{\beta x}}{e^{\beta L_{beam}}(e^{\beta(L_{beam}-L_H)} - e^{-\beta(L_{beam}-L_H)})} \\
&= \frac{e^{\beta L_{beam}}(e^{\beta(L_{beam}-x)} - e^{-\beta(L_{beam}-x)})}{e^{\beta L_{beam}}(e^{\beta(L_{beam}-L_H)} - e^{-\beta(L_{beam}-L_H)})} (T_H - T_0) \\
T(x) - T_0 &= \frac{\sinh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} (T_H - T_0).
\end{aligned} \tag{56}$$

The last step is to calculate the temperature of the heater T_H at the position $x = L_H$. To do this, equation (16) is used. Thus, T_H is given by:

$$\begin{aligned}
\frac{dT(x)}{dx} &= \frac{d\left(\frac{\sinh(\beta(L_{beam}-x))}{\sinh(\beta(L_{beam}-L_H))}\right)}{dx} (T_H - T_0) \\
&= \frac{-\beta \cosh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} (T_H - T_0)
\end{aligned} \tag{57}$$

and combining equations (16) and (27), the temperature of the heater is equal to:

$$\begin{aligned}
\frac{\beta \cosh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} (T_H - T_0) &= \frac{\frac{P_{in}}{W} - \left(4\varepsilon\sigma T_0^3 + \lambda_{gas}\left(\frac{1}{d_1} - \frac{1}{d_2}\right)\right) (T_H - T_0)L_H}{\sum \lambda_s t_s} \\
T_H &= \left(\frac{P_{in}}{\beta \cdot W \sum \lambda_s t_s (\beta \cdot L_{beam} + \coth(\beta(L_{beam} - L_H)))} \right) + T_0.
\end{aligned} \tag{58}$$

Finally, replacing equation (58) in (56) the temperature in x direction along the beam is given by:

$$T(x) = \left(\frac{P_{in}}{\beta \cdot W \sum \lambda_s t_s (\beta \cdot L_{beam} + \coth(\beta(L_{beam} - L_H)))} \right) \frac{\sinh(\beta(L_{beam} - x))}{\sinh(\beta(L_{beam} - L_H))} + T_0. \tag{59}$$

A.2 Package Dimensions

The package that has been used to place the dual-TCD is a LCC44. The dimensions of this package are shown in Figure 64[38].

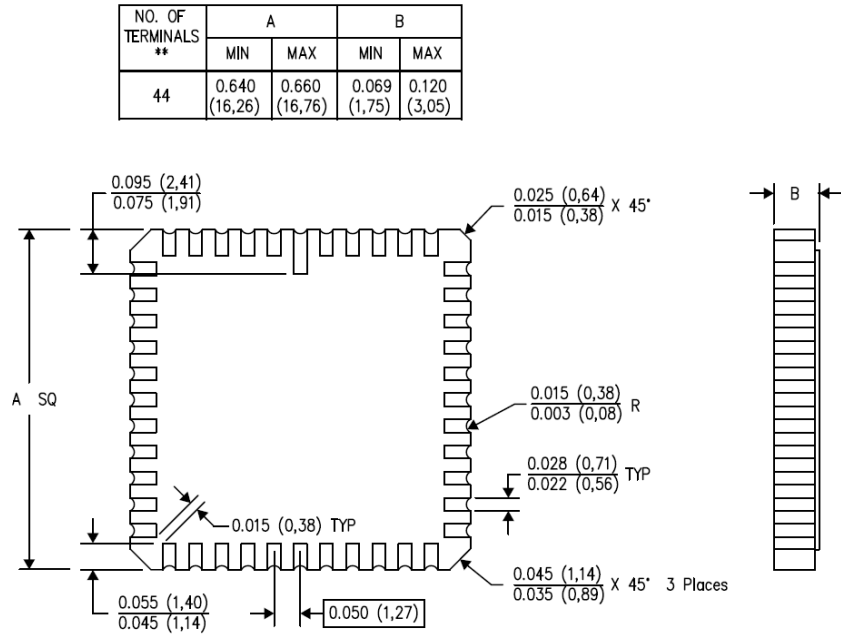


Figure 64: LCC44 package dimensions [38].