University of Colorado at Boulder

Department of Mechanical Engineering

Nano-enabled Energy Conversion, Storage and Thermal Management Systems

Measurement of the In-Plane Thermal Conductivity by the Parallel Thermal Conductance Technique

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Introduction

When a temperature gradient exists in a body, it has been shown that there is an energy transfer from the high temperature region to the low temperature region. In this case, the energy is transferred by conduction and the heat transfer per unit area is proportional to the temperature gradient:

$$\frac{q}{A} \sim \frac{\partial T}{\partial x} \tag{1.1}$$

If the proportionality constant is inserted,

$$q = -kA\frac{\partial T}{\partial x} \tag{1.2}$$

The positive constant k is called the thermal conductivity of the material, and the minus signed is inserted to satisfy the second principle of the thermodynamics. Equation 1.2 is called Fourier's law of heat conduction after the French mathematical physicist Joseph Fourier, who made very significant contributions to the analytical treatment of conduction heat transfer. It is important to note that Equation 1.2 is the defining equation for the thermal conductivity and that k has the units of watts per meter per Celsius degree in a typical system of units in which the heat flow is expressed in watts.

1.1 Thermal Conductivity

Equation 1.2 is the defining equation for thermal conductivity. Experimental measurements may be made in order to determine the thermal conductivity of different materials.

The mechanism of thermal conduction in a gas is a very simple one. The kinetic energy of a molecule depends on its temperature. Thus, in a high-temperature region the molecules have higher velocities than in lower-temperature regions. When a molecule moves from a high-temperature region to a region of lower temperature, it transports kinetic energy and gives it up through collisions with lower-energy molecules.

The mechanism of thermal energy conduction in liquids is considerably more complex because the molecules are more close from each other and molecular force fields have a strong influence on the energy exchange in the collision process.

Thermal energy in solids is conducted by to models: lattice vibration and transport by free electrons. In good electrical conductors, a large number of free electrons move across the lattice structure. These electrons transport electric charge as well as they may carry thermal energy from the high-temperature regions to lower temperature regions. The same energy may also be transmitted as vibrational energy in the lattice structure, although this latter mode of energy transfer is not as large as the electron transport, and for this reason good electrical conductors are almost always good thermal conductors.

Chapter 2

Thermal Conductivity Measurement

There are many ways of measuring thermal conductivity, each of them suitable for limited range of materials, depending on the thermal properties and the medium temperature. In general, we can find two basic techniques of measurement.

- In the **steady state** technique the measurement is performed when the whole system is in equilibrium. Although it is a very simple technique because it is made with constant signals, it generally requires a long time to reach the equilibrium.

- In the **non-steady state** techniques all the measurement are performed while the system is heating up [8] .

Bellow are described some of the most commonly used techniques for measuring the thermal conductivity for bulk materials. One should note that there are also other methods for the characterization of thermal conductivity for thin film materials.

2.1 Steady-state techniques

The steady-state measurement of the thermal conductivity is done by measuring the temperature difference ΔT under a constant heat flow Q. One can distinguish four different ways of measuring the thermal conductivity using steady-state techniques.

2.1.1 Absolute technique

The Absolute technique for measuring the thermal conductivity is done by placing the sample between a heat source and a heat sink. Once a constant heat flow is supplied through the heat source and the steady state is reached, the resulting temperature drop across the sample is measured by temperature sensors (e.g. thermocouples or RTD sensors). The thermal conductivity of the sample can be extracted using Fourier's law of heat conduction (see Equation 1.2).

It is noted that heat convection and radiation must be minimized in order to obtain relevant results. In general, heat losses should be controlled to be less than 2% of the total heat flow through the sample [9]. To minimize those losses, it is preferable to conduct the measurements under vacuum with radiation shield. Another important factor to bear in mind is the heat conduction through thermocouple wires. For this reason it is preferable to use thermocouples with small wire diameter and low thermal conductivity. Other negative aspects of this technique are the long waiting time to reach steady state (it can take more than 2 hours) and the large size of the sample (in centimeter scale or even larger).

2.1.2 Comparative technique

This technique is based on the "comparison" of the parameters of an unknown material with those of one or more materials of known thermal properties. A temperature gradient is established along the test stack. Then, since the heat flow through the standard sample is the same as the one through the measurement target, the thermal conductivity of the unknown sample can be extracted from the following formula:

$$k_1 = k_2 \frac{A_2 \Delta T_2 L_1}{A_1 \Delta T_1 L_2} \tag{2.1}$$

where subscript 1 is associated with the measurement target and subscript 2 with the standard sample.

One can see that the comparative technique is very similar to the absolute one. However, the main benefit of the comparative method is that there is not need to determine the heat flow through sample, which is the biggest challenge in the absolute method [9].

2.1.3 Radial heat flow method

In the radial heat flow method, also referred as the cylinder method, the heat flows radially away from a central heater towards a heat sink. With this, a steady-state temperature gradient is established in the radial direction. The thermal conductivity is then extracted from the Fourier's law in cylindrical coordinate:

$$k = \frac{Q \ln(\frac{r_2}{r_1})}{2\pi H \Delta T} \tag{2.2}$$

where r_1 and r_2 are the radial positions of the temperature sensors, H is the sample height and ΔT is the temperature difference between temperature sensors.

Heat losses at the top and bottom of the cylinder will affect the uniform distribution of heat flux throughout the specimen. For this reason, it is important to make the cylinder long with respect to the cylinder radius. This allows to establish a uniform temperature distribution in the mid-section of the cylinder. Therefore, the main disadvantage of this method is that, in order to get accurate measurements, large specimen should be used. This implies a greater cost and more time to reach the steady state.

2.2 Transient techniques

Transient methods for measuring the thermal conductivity have become very popular as a result of the progress made in modern computing and data analysis.

The transient techniques measure the response when a signal is sent to create heat in the test sample. At the beginning, the specimen is in thermal equilibrium with the surrounding. Then, a heating pulse is supplied to the sample and the temperature change during time is recorded for determining the thermal conductivity of the sample.

The advantages of transient techniques is that they overcome many of the issues associated with the steady-state techniques described before (e.g. heat losses, contact resistance and long waiting time [9]).

2.2.1 Pulse-power method

This technique was presented by Maldonado at 1992 for measuring both heat conductivity and thermoelectric power [4]. Based on the absolute technique in the steady-state methods, in the pulse-power method the heater current that generates the thermal gradient is pulsed with a square wave while the bath temperature T_0 is slowly drifted. Since the thermal equilibrium is never reached, time between measurements is significantly reduced and a better resolution can be obtained.

The heat balance equation for the heater is written as the sum of the heat dissipated by the heater and the heat conducted through the sample:

$$\frac{dQ}{dt} = C\left(T_1\right)\frac{dT_1}{dt} = R\left(T_1\right)I^2\left(t\right) - K\left(T_1 - T_0\right) \tag{2.3}$$

where $\frac{dQ}{dt}$ is the time rate of change of heat in the heater, T_1 is the heater temperature, T_0 is the bath temperature, C is the heat capacity of the heater, R is the heat resistance, and K is the sample thermal conductance.

Since Equation 2.3 is nonlinear and difficult to solve analytically, Maldonado arrived at a solution by doing several simplification. The temperature difference T_1 and T_0 is very small and $C\left(T\right)$, $R\left(T\right)$, and $K\left(T\right)$ are smooth functions of T. Then, T_0 is used instead of T_1 as the argument of C, R, and K. Also, T_0 is assumed to be constant, since the temperature change is slow compared to the periodic current. The solution has a sawtooth form as shown in Figure 2.1. The difference between the smooth curves ΔT_{pp} yields a relations for the thermal conductance:

$$K = \frac{RI_0^2}{\Delta T_{pp}}\tanh\left(\frac{K\tau}{2C}\right) \tag{2.4}$$

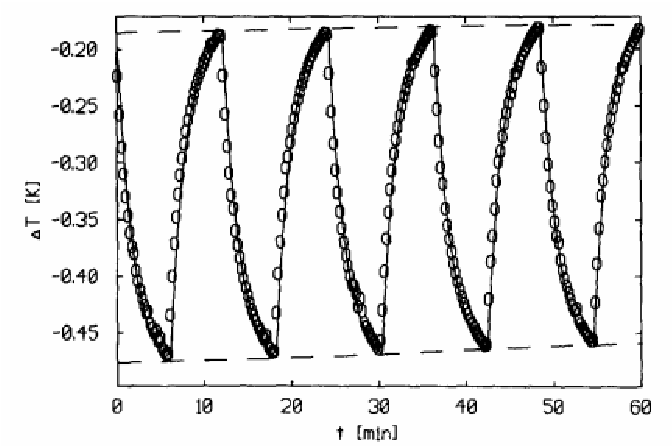

Figure 2.1: Temperature rise and fall for pulsed power using the "Maldonado" technique. From [4]. The graph shows the time dependence of the temperature difference across the sample ((-) represents simulation results and (o) represents experimental data).

It is noted that Equation 2.4 has K at both sides, so that it has to be solved by numerical iteration. The overall accuracy reported by Maldonado is less than 5% [4].

2.2.2 Transient plane source method

This technique uses a thin, plane, and electrically insulated resistive element, as shown in Figure 2.2, both as a temperature sensor and a heating source. Measurements are made by placing the heater between two test samples of the same material. The surface of those samples need to be as flat and smooth as possible in order to minimize the contact resistance with the heating element. The thermal properties of the test sample can be determined from one single transient recording by monitoring the increase of resistance in the heating element.

The transient plane source method has been proved to work for both fluids and solids with thermal conductivities ranging from 0.01 to 500 W/(mK) and a temperature range from cryogenic temperatures to 500 K [3].

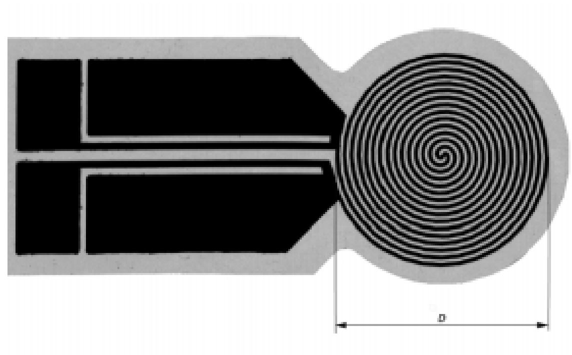

Figure 2.2: Thin film heater and sensor used for TSP. From [3].

2.2.3 Laser flash method

In the laser flash method a single pulse from a laser is used to heat instantaneously one face of the testing sample while the resulting temperature on the other face is monitored with an infrared detector (IR), making the measurement contactless (see Figure 2.3). In addition to this, there is not contact resistance between the sample and the heating and cooling plates, which means a significant advantage over other methods mentioned above.

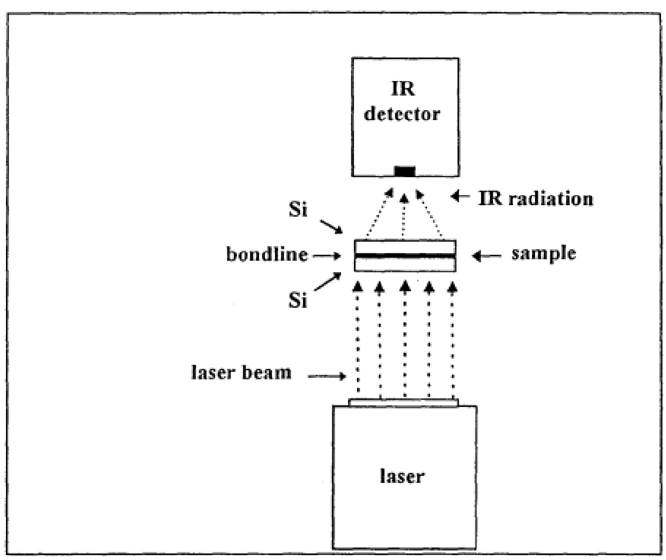

Figure 2.3: Schematic of laser flash diffusivity method. From [2].

The samples are usually prepared for the measurement by spraying a layer of graphite on both sides. This layer acts as an absorber for the heating laser on one side and as an emitter for the IR detector on the other side [7].

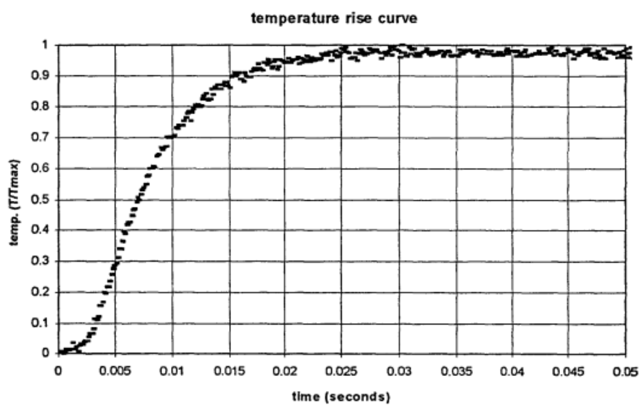

Figure 2.4: Back surface temperature rise. From [2].

Figure 2.4 shows the temperature rise in the normalized back surface of the measuring sample. This response curve is used to determine the thermal diffusivity:

$$\alpha = \frac{0.139d^2}{t_{1/2}} \tag{2.5}$$

where d is the sample thickness and $t_{1/2}$ is the time to reach on half of the maximum temperature on the back surface [6].

The thermal conductivity can then be obtained as follows:

$$k = \alpha\rho c_p \tag{2.6}$$

where ρ is the density of the material and c_p the specific heat. Both of them have to be measured from different experiments, which can lead to larger errors and uncertainties. The laser flash method is capable of measuring the thermal conductivity over a wide range of temperatures (-120 C to 2800 C) with a reported accuracy of less than 3% [9].

Chapter 3

Parallel Thermal Conductance Technique

The parallel thermal conductance technique (PTC) is a steady-state method for measuring the thermal conductivity for bulk materials. In contrast to other steady-state methods described above, in the PTC technique both the temperature senors and the heater are not attached to the sample. This implies a significant improvement over alternative methods, as attaching sensors and heaters directly to small samples (millimiter scale) can be very difficult and originate large heat losses and errors [10].

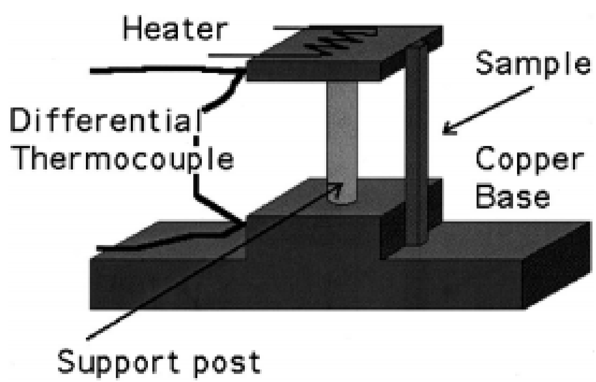

Figure 3.1: PTC sample holder. From [10].

The essence of the PTC technique is to develop a sample holder so that the temperature sensor and the heater can be attached to it. This configuration is illustrated in Figure 3.1.

Due to the presence of the sample holder, it is required a preliminary measurement of the sample holder thermal conductance (C_{base}) to quantify the thermal losses associated to it.

$$P = I^2 R = C_{base} \Delta T \tag{3.1}$$

The measurement target is then attached to the sample holder and the thermal conductance of the whole system (C_{total}) is measured.

$$P = I^2 R = C_{total} \Delta T \tag{3.2}$$

Once this is done, the parallel thermal conductance can be calculated by subtraction. This conductance is due to the sample, thermal contacts, and blackbody radiation from the sample. An estimation of the radiation losses due to the sample itself can be obtained by doing a third measurement. This is further explained in literature [10]. Furthermore, because of the small cross sectional area and low thermal conductivity of the sample, the thermal resistance of the thermal contacts can be ignored. The thermal conductivity of the sample is then obtained by multiplying the sample length and dividing by it the cross-sectional area.

$$K_{sample} = \frac{(C_{total} - C_{base})\, L}{A} \tag{3.3}$$

3.1 Sample holder

Proper design of the sample holder must be ensured in order to get relevant results when performing the PTC technique. The main issue that must be taken into consideration is to have a thermal conductance as low as possible. This will result in a better precision when subtracting the sample holder from the total thermal conductance. According to literature [10] the thermal conductance of the sample should be at least on the order of one-tenth that of the thermal conductance of the base line (sample holder). On the other hand, the sample holder must be able to support the sample, as well as the heater and the temperature sensors. Finally, in order to reduce the time to reach steady state, the heat capacity of the heater head should be minimized.

Part	Material	Thermal conductivity (K) @ 20 °C (W/mK)	Specific Heat (C_p) @ 20 °C (cal/gram °C)
Heater Plate	Copper 110	390	0.092
Support post	Polyester (PET)	0.15-0.4	0.30
Base plate	Copper 110	390	0.092

Table 3.1: Sample holder material selection

According to all these specifications, a selection of the different materials included in the sample holder (heater plate, support post and base plate) has been made (see Table 3.1).

Due to its low heat capacity, copper 110 has been chosen for the heater plate. This will minimize the required time to reach steady state. Additionally, polyethylene terephthalate (PET), commonly known as polyester, has been selected for the support post as it has a very low thermal conductivity. Finally, since there is no requirement for the base plate, copper 110 has been used for it.

The design and dimensions (in mm) of the sample holder are shown in Figure 3.2.

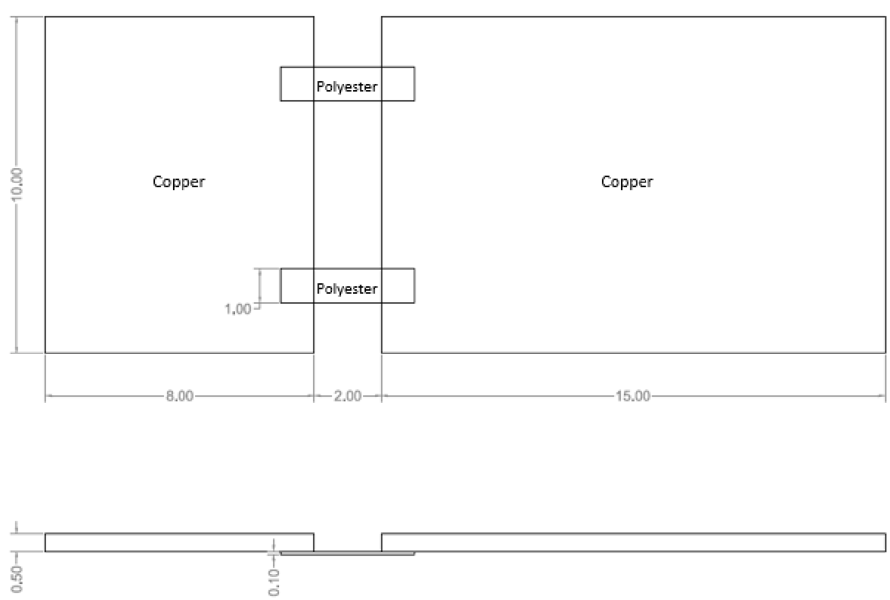

Figure 3.2: Sample holder drawings

An aluminum support for the heat sink has also been designed so that heater plate is isolated from the ground. Both the heat sink and the support are in thermal equilibrium with the ground. Two screws of stainless steel have been used to fix the support to the heat sink. The whole assembly is shown in Figure 3.3.

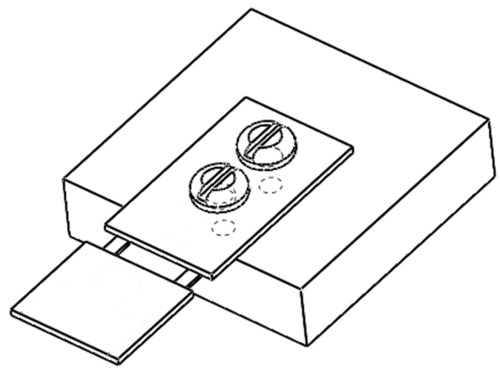

Figure 3.3: Sample holder assembly

3.2 Heating element

In order to provide thermal energy to the system, it is required to use a heater that must be attached to the sample holder. However, most of the heaters that are commercially available exceed the dimensions of the heater base. This problem led to the study of alternative ways of supplying energy to our system.

Literature [1] and [5] show that a strain gauge bonded to a copper disk or block can be used quite successfully in these applications. With this purpose, a 3x2 mm strain gauge (see Figure 3.4) with a nominal resistance of 350 Ω has been used as a heating element.

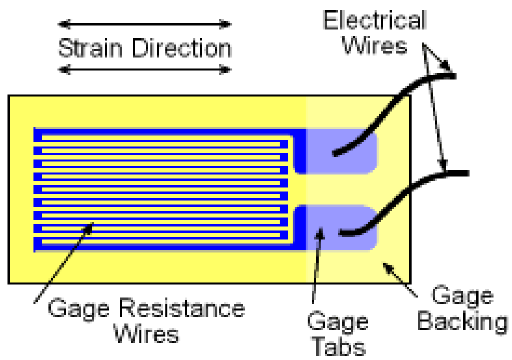

Figure 3.4: Strain gauge. From *www.omega.com*.

The gauge has been bonded to the surface of the heater plate using ethyl-based cyanoacrylate. The thermal energy dissipated by the strain gauge can be calculated as follows:

$$P = I^2 R \tag{3.4}$$

However, due to a resistance change of approximately 1% a small voltage correction must be made in order to maintain a constant power input [1].

3.3 Temperature sensors

In order to measure the temperature difference ΔT between the heater and the base plate, it is required to use a temperature sensor in both positions.

In literature [10] the measurement of the temperature was made by thermocouples. However, we have considered the option of using RTD sensors as they provide very accurate readings. Below is explained the principle of measurement of this devices and the different configurations that are available.

3.3.1 RTD sensors

RTDs (resistance temperature detector) are temperature sensors which measurement principle is that the electrical resistance of a material changes with temperature. Platinum is the most widely used RTD element type, although nickel and copper alloys are also very common. The reason why Platinum is the most popular RTD element is that it has a wide temperature range and a good accuracy.

RTDs are generally characterized by their temperature coefficient of resistance. The temperature coefficient of resistance (α) symbolizes the resistance change factor per degree of temperature change and it is expressed in ohms/ohms/°C or more typically %/°C. The equation below defines α:

$$\alpha = \frac{R_{100} - R_0}{100R_0} \tag{3.5}$$

where R_{100} represents the resistance of the sensor at 100 °C and R_0 the resistance of the sensor at 0 °C.

RTDs can be found in two different constructions:

- **Wire-wound** devices are manufactured by winding a small diameter of wire into a coil on a suitable winding bobbin.

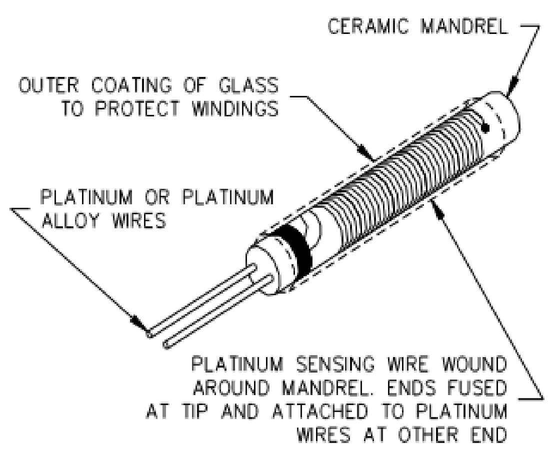

Figure 3.5: Wire Wound Element. From *www.pyromation.com.*

- In **Thin-film** elements a thin layer of the base metal is deposited onto a ceramic substrate. Because this configurations can reach higher resistances with less metal, they tend to be less costly than the wire-wound devices.

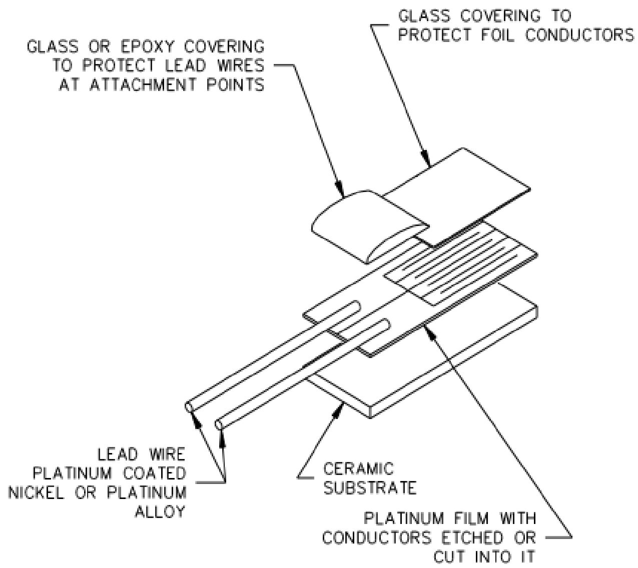

Figure 3.6: Thin Film Element. From *www.pyromation.com*.

Since RTD is a resistive element, errors in the measurement may arise due to the presence of any resistance elsewhere in the circuit. Usually, this additional resistance is in the lead wires attached to the senor, especially when they have a long extension. This issue is known as the lead wire error.

In order to minimize this factor the wiring configuration can be modified. Although RTD is inherently a 2-wire device, a third wire can be added to the circuit to help compensate the lead wire resistance, and thus provide a more accurate reading of the temperature. Also, a fourth wire can be added to the configuration in order to determine the actual resistance of the lead wires and remove it from the sensor measurement. The 4-wire constructions is therefore the most accurate one as it cancels the lead wire error (see Figure 3.7).

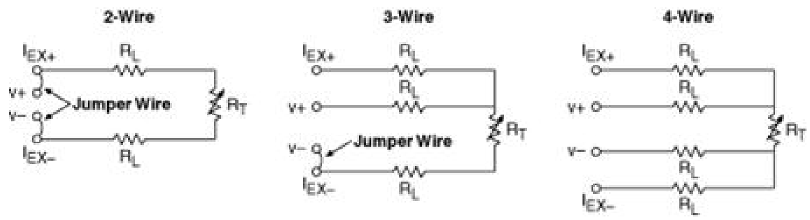

Figure 3.7: Two-, Three-, and Four-Wire Connection Diagrams. From *www.ni.com.*

In general, thermocouples are better than RTDs in terms of cost, reaction and ruggedness. However, RTDs provide more accurate and repeatable readings, which means that their measurements are more stable. Table 3.2 reflects and compares the attributes of thermocouples and RTD sensors.

Parameter	**Thermocouple**	**RTD**
Typical Measurement Range	-267 °C to 2316 °C	-240 °C to 649 °C
Interchangeability	Good	Excellent
Long-term Stability	Poor to Fair	Excellent
Accuracy	Medium	High
Repeatability	Poor to Fair	Excellent
Sensitivity	Low	Good
Response	Medium to Fast	Good
Linearity	Fair	Good
Self Heating	No	Low
Tip Sensitivity	Excellent	Fair

Table 3.2: RTD vs Thermocouple comparison chart

In order to evaluate both sensors and decide which one is better for the PTC technique, we have performed several measurements using RTDs and thermocouples. Table 3.3 shows the specifications reported by the manufacturer for each of the sensors that have been used.

Sensor	Temp. Range	Limits of Error (whichever is greater)
Thermocouple T-type	-270 °C to 370 °C	1.0 °C or 0.75 %
RTD Sensor Thin film 2-wire	-70 °C to 600 °C	Class A (IEC 60751) 0.19 °C @ 20 °C

Table 3.3: Thermocouple and RTD specifications reported by the manufacturer

One may realize that the RTD sensor that has been selected has a 2-wire configuration, and thus it is not the most accurate one as it is highly exposed to the lead wire error. However, this was the only thin film RTD with flat surface that was available in such small dimensions. Other options with 3 and 4-wire were excluded because of the large dimensions and cylindrical shape.

Both the RTD and thermocouple have been attached to a copper sheet using a high thermally conductive epoxy. A flexible kapton heater has also been attached in order to supply thermal energy to the system. The idea behind this is to check the response of both sensors and see if they agree in their readings.

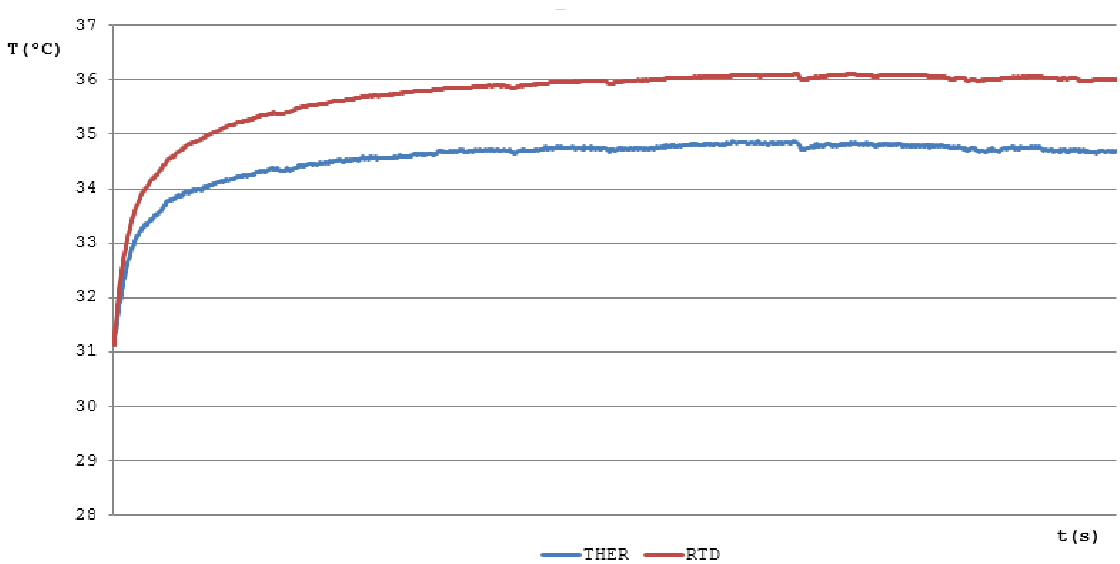

Figure 3.8: RTD vs Thermocouple response curves

Figure 3.8 shows the response curve of the temperature sensors when a constant voltage of 5 V is supplied. It is deducted from the graph that the time to reach steady state is greater in the RTD (τ_{RTD} = 12 min) than in the thermocouple (τ_{Ther} = 11 min). This agrees with Table 3.2. When steady state is reached, both temperatures differ in 1 °C. Although this difference is inside the thermocouple and RTD tolerance, it is excessively large. Some of the reasons for such a great difference are listed below:

- The distribution of the temperature is not 100 % symmetrical throughout the copper sheet, and thus the temperature sensors are not measuring the same point.
- Since the thermocouple does not have a flat surface, it is not in good contact with the surface of the copper sheet and so it is reading a lower temperature.
- The temperature measured by the RTD is higher because of the lead wire error.

If we have a closer look to the measurements done by both sensors (see Figure 3.9), one can see that the readings of the RTD are more stable and repeatable than the ones made by the thermocouple. This further supports Table 3.2.

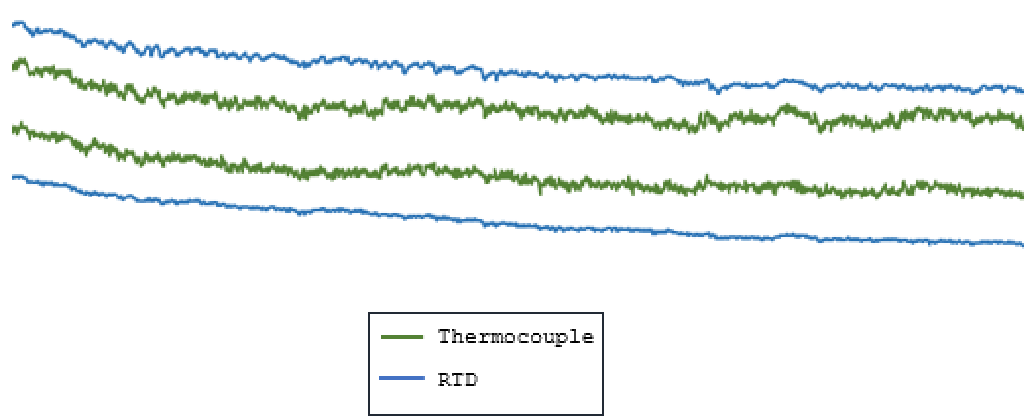

Figure 3.9: RTD vs Thermocouple accuracy

Finally, it is important to mention that the thermocouples are way more rugged than the RTDs. It has been proven that RTDs need a very careful handling, and yet they tend to break very easily. Due to this fact, thermocouples are a better option for measuring the temperature in the PTC method.

3.4 Data acquisition

Literature [10] presents a very complex and automated procedure for measuring the thermal conductivity of the sample at different temperatures. In our case, the data acquisition process is more straightforward as the thermal conductivity is only measured at room temperature.

Since all the measurements need to be done in a vacuum environment, it is required to use a vacuum chamber. Table 3.4 shows the desired specifications of vacuum for conducting the experiment.

Specifications	
Vacuum	10^{-6} torr (ideally)
Temperature range	room temperature
Feedthroughs	6 (minimum)

Table 3.4: Vacuum chamber specifications

Several vacuum chambers with this specifications are commercially available. However, because of their high economic cost, it has been decided to do the first measurements with a handmade vacuum chamber (see Figure 3.10), the specifications of which are shown in Table 3.5.

Specifications	
Vacuum	20 torr
Temperature range	room temperature
Feedthroughs	16

Table 3.5: Handmade vacuum chamber specifications

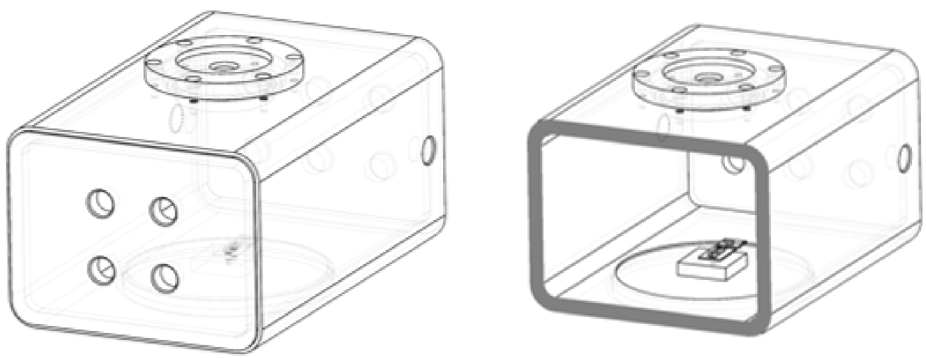

Figure 3.10: Handmade vacuum chamber

At the same time, it has been used a data acquisition system (*Agilent 34970A*) for processing all the readings of the thermocouples and a current source meter to supply a constant power across the strain gauge terminals. Figure 3.11 shows the complete data acquisition system.

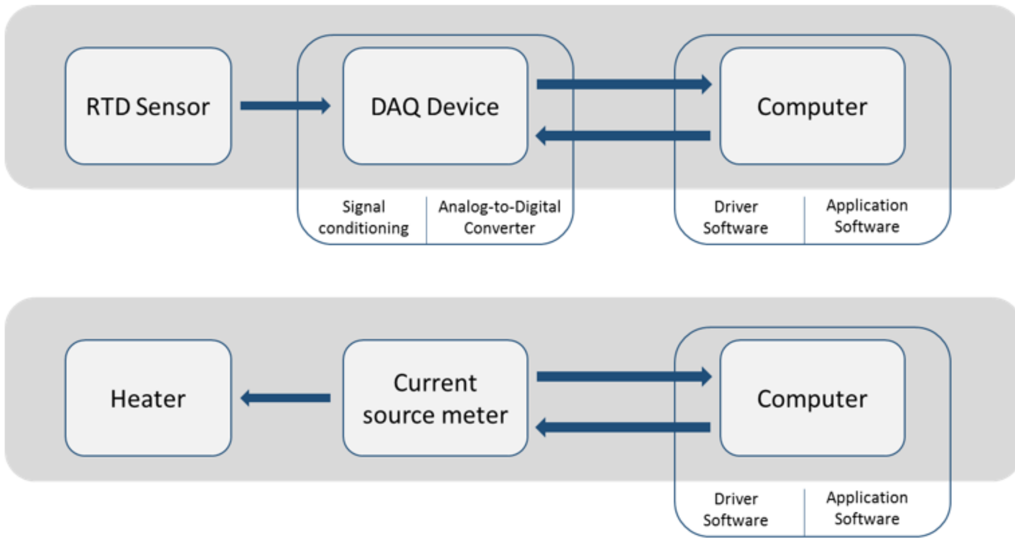

Figure 3.11: Data acquisition system

All the measurements have been conducted supplying 4 different voltages (4V, 4.5V, 5V and 5.5V). By fitting the linear relation, the thermal conductance of the sample holder and the whole system have been extracted. By subtraction, the thermal conductance of the sample and the thermal contacts have been obtained. The thermal conductivity, k, is derived from the thermal conductance and the dimensions of the sample.

3.5 Initial measurements and results

Since the handmade vacuum chamber had to undergo some modifications, first measurements have been done at ambient conditions. A standard 4x2x0.5 mm copper 110 sample has been selected for testing the measurement device.

After conducting the experiment at four different voltages (4V, 4.5V, 5V and 5.5V) and fitting the linear relation between the heating power and the temperature difference, the following figure (see 3.12) is obtained:

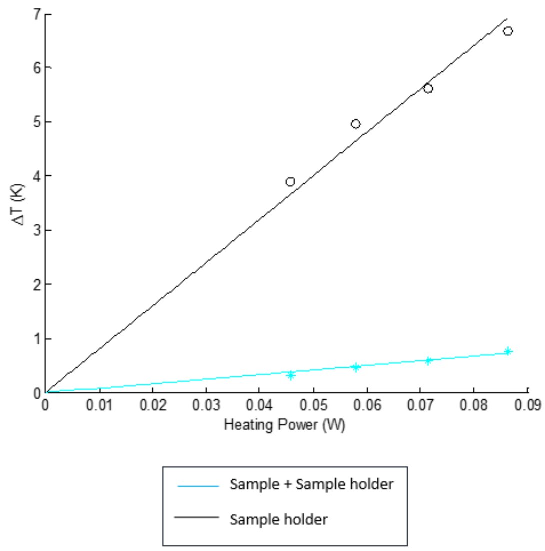

Figure 3.12: Copper 110 heating power vs. temperature difference

The thermal conductivity (k) can be extracted by subtraction with the dimensions of the sample. The value that has been obtained is:

$$k = 430\ W/mK \tag{3.6}$$

which is significantly higher than the one provided by the manufacturer ($k_{manu} = 390\ W/mK$). This can be due to the following uncontrolled factors:

- **Air convection**: Because the measurement is done at ambient conditions, part of the heating power is lost by convection. According to this, the measured thermal conductivity (k) should be greater than the one provided by the manufacturer (k_{manu}). This agrees with the value of thermal conductivity that has been obtained.

- **Non-symmetry**: the support post may be slightly offset to the side, then the temperature distribution is non-symmetric.

- **Thermal resistance of the contacts**: Since the copper has a high thermal conductivity, the thermal resistance of the contacts should be considered.

- **Dimensions of the sample**: The length and cross-sectional area of the sample must be determined with accuracy since the precision to which this dimensions can be measured can result in large uncertainties in the measurement of the thermal conductivity.

The problem with the air convection could be solved easily by performing the measurements in a vacuum environment. The effect of the non-symmetry is difficult both to quantify and amend, since the sample holder is built by hand. The thermal resistance of the contacts could be neglected if lower values of thermal conductivity were measured. Finally, the dimensions of the sample could be determined with better accuracy and precision if they were measured by Computer Numerical Control (CNC).

Chapter 4

Conclusions

In this report it has been described a conceptually simple and fast steady-state technique for measuring the thermal conductivity for bulk materials. The main advantage of the PTC method over other steady-state techniques is that both the heater and the temperature sensors are not attached to the sample, and thus the heat losses across this elements are minimized.

Although the PTC technique has already been implemented by other authors [10], some modifications have been introduced in order to improve its performance. First, the sample holder has been redesigned in order to secure the sample stability. Moreover, due to the small scale of the measurement device, it has been used a strain gauge as a low power heater on the basis of literature [1] and [5]. Finally, it has been assessed the possibility of using RTDs over thermocouples as temperature sensors, and although RTDs may have a better accuracy, they have not been considered a good option taking their fragility into account.

Once the measurement device has been tested, it has been proven that there are some factors such as the position of the temperature sensors or the cross-sectional area of the sample that must be precisely determined in order to avoid a large degree of uncertainty in the calculated thermal conductivity.

Future work should focus on performing the measurements in a vacuum environment as well as extending the temperature range within which the thermal conductivity is obtained.

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