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**Thermal conductivity measurement of thin layers by the 3ω method**

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Concerns: Internship Report

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Abstract:
The market of LEDs increases exponentially over the years as well as their power. However, at the same time the mean time to failure should remain superior to 100,000 hours. The major difficulty comes from the mechanical stress in the LED structure, which is roughly proportional to the inverse of the thermal conductivity. Consequently, it is really important to determine thermal conductivity of the materials used in LEDs. In the same way, the thermal conductivity of films is lower than the thermal conductivity of the bulk material. Unfortunately, thermal conductivity measurement is difficult on two-dimensional structures. Then an appropriate method had to be developed. The $3\omega$ thermal conductivity measurement method has been used extensively to measure the thermal properties of bulk and thin film dielectric materials. Tests on different materials will allow to find materials with a higher thermal conductivity. The thermal conductivity measurement will be elaborated by two different methods defined the isotropic and anisotropic properties, the "slope method" and the 2D heat conduction.

Conclusions:
- The set-up is operational from 50Hz to 15kHz
- The experiment is fully automated and controlled by Labview software. It takes 12h30.
- The measurement accuracy of the thermal conductivity by the $3\omega$ method is:
  - 7% for bulk.
  - 10% and 15% for respectively isotropic and anisotropic film.
  - 0.2% repeatability.
- A thin dielectric layer shows a:
  - thermal conductivity equal to $1.28W.m^{-1}.K^{-1}$ and $1.23W.m^{-1}.K^{-1}$ for respectively SiO$_2$ and Si$_3$N$_4$ 500nm thin layer at 350°C.
  - thickness dependence.
  - temperature dependence, the thermal conductivity increase with temperature due to the phonon conductivity of semi-transparent glass.
- The interfacial resistance for SiO$_2$/Si$_3$N$_4$ is about $3.10^{-8}m^2.K.W^{-1}$. 
# Contents

1. **Introduction**  .................................................................................................................. 1
   1.1 History and Usefulness .................................................................................................. 2
   1.2 Thermal conductivity dependence .............................................................................. 4
   1.3 Internship goal and objective .................................................................................... 6

2. **The 3ω method** .................................................................................................................. 7
   2.1 Introduction .................................................................................................................... 7
   2.2 Theory of the 3ω method ............................................................................................. 7
      2.2.1 Principle ................................................................................................................... 7
      2.2.2 Measurements on bulk materials ........................................................................... 11
      2.2.3 General solution for the 2D heat conduction across a multilayer-film-on-substrate system [18] ................................................................. 12
      2.2.4 Analytical approach of the thermal amplitude $\Delta T_0$ ......................................... 13
      2.2.5 Thermal conductivity measurement for anisotropic materials ............................. 13
   2.3 Determination of the main parameters - "Slope Method" ............................................ 16
      2.3.1 Thermal conductivity of isotropic substrate $k_s$ ..................................................... 16
      2.3.2 Thermal conductivity of isotropic film $k_f$ ............................................................ 17
   2.4 Thermal conductivity contributions ............................................................................ 17
      2.4.1 Electronic contribution ........................................................................................... 18
      2.4.2 Phonon contribution .............................................................................................. 18
      2.4.3 Photon contribution .............................................................................................. 18
      2.4.4 Conclusion ............................................................................................................ 19

3. **Experimental details** ....................................................................................................... 20
   3.1 Sample .............................................................................................................................. 20
      3.1.1 Sample design ......................................................................................................... 20
      3.1.2 Sample preparation ............................................................................................... 21
   3.2 Experimental set-up ...................................................................................................... 23
   3.3 Software ........................................................................................................................ 25
      3.3.1 Labview, Thermal coefficient ............................................................................... 26
      3.3.2 Labview, 3ω method ............................................................................................. 27
   3.4 Statistics ........................................................................................................................ 30
      3.4.1 Repeatability analysis ............................................................................................ 30
      3.4.2 Reproducibility analysis ....................................................................................... 30
      3.4.3 Systematic error ................................................................................................... 31
   3.5 Measurement validation ............................................................................................... 32

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4 Results

4.1 General solutions ..................................................... 33
  4.1.1 Thermal conductivities ......................................... 33

4.2 SiO₂ ................................................................. 35
  4.2.1 Preliminary analysis .............................................. 35
  4.2.2 Bulk and film thermal conductivities ......................... 36
  4.2.3 Thickness and temperature dependence ...................... 38

4.3 Si₃N₄ ............................................................... 40
  4.3.1 Preliminary analysis ............................................. 40
  4.3.2 Thermal conductivities ......................................... 41

4.4 Thickness analytical prediction ................................... 43

4.5 Model precision .................................................... 44
  4.5.1 Film analysis .................................................. 44
  4.5.2 Substrate analysis .............................................. 44

References ............................................................... 48

A Temperature stability of the climate cabinet ..................... 51

B 3ω distortion .......................................................... 53

C Theoretical approach for the 3ω method .......................... 55

D Thermal wave .......................................................... 59

E Labview program ...................................................... 64

F Temperature calibration ............................................... 65

G Manual ................................................................. 66

H Symbols ............................................................... 68
Chapter 1

Introduction

A light-emitting diode (LED) is a semiconductor device that emits incoherent narrow-spectrum light when electrically biased in the forward direction (Figure 1.1). This effect is a form of electroluminescence. The colour of the emitted light depends on the chemical composition of the semiconductor material used, and can be near ultraviolet, visible or infrared.

Figure 1.1: LED - electrical model
1.1 History and Usefulness[6][7]

The phenomenon of electroluminescence was first observed in a piece of Silicon Carbide (SiC), in 1907 by Henry Joseph Round. The yellow light emitted by it was too dim to be of practical use and difficulties in working with Silicon Carbide meant that research was abandoned. Further experiments were carried out in Germany in the late 1920s by B. Gudden and R.W. Pohl, using phosphor materials made from Zinc Sulphide doped with Copper (ZnS:Cu), although once again, the low level of light produced meant that no in depth research was carried out. In 1936 George Destriau published a report on the emission of light by Zinc Sulphide (ZnS) powders, following the application of an electric current and is widely credited with having invented the term "electroluminescence". British experiments into electroluminescence, using the semiconductor Gallium Arsenide (GaAs) in the 1950s led to the first "modern" Light Emitting Diodes (LEDs), which appeared in the early 1960s.

The first commercial LEDs were only able to produce invisible, infra red light, but still quickly found their way into sensing and photo-electric applications. The first visible (red) light LEDs were produced in the late 1960s, using Gallium Arsenide Phoshphide (GaAsP) on a GaAs substrate. Changing to a Gallium Phosphide (GaP) substrate led to an increase in efficiency, making for brighter red LEDs and allowing the colour orange to be produced. By the mid 1970’s Gallium Phosphide (GaP) was itself being used as the light emitter and was soon producing a pale green light. LEDs using dual GaP chips (one in red and one in green) were able to emit yellow light. The use of Gallium Aluminium Arsenide Phosphide (GaAlAsP) LEDs in the early to mid 1980s brought the first generation of superbright LEDs, first in red, then yellow and finally green. By the early 1990’s ultrabright LEDs using Indium Gallium Aluminium Phosphide (InGaAlP) to produce orange-red, orange, yellow and green light had become available. The first significant blue LEDs also appeared at the start of the 1990’s, once again using Silicon Carbide. Ultrabright blue Gallium Nitride (GaN) LEDs arrived in the mid 1990s, with Indium Gallium Nitride (InGaN) LEDs producing high-intensity green and blue shortly thereafter. The ultrabright blue chips became the basis of white LEDs, in which the light emitting chip is coated with fluorescent phosphors. These phosphors absorb the blue light from the chip and then re-emit it as white light. This same technique has been used to produce virtually any colour of visible light and today there are LEDs on the market which can produce previously ‘exotic’ colours, such as aqua and pink.

In the end 1990s, cost-effective traffic light lamps using light emitting diodes (LEDs) were developed; prior to this date traffic lights were designed using incandescent or halogen light bulbs. Unlike the incandescent-based lamps, which use a single large bulb, the LED-based lamps consist of an array of LED elements, arranged in various patterns. When viewed from a distance, the array appears as a continuous light source (unless closely examined).

The operational expenses of LED-based signals are far lower than equivalent incandescent-based lights. As a result, most new traffic light deployments (for example in the United States) have been implemented using LED-based lamps; in addition many existing deployments of incandescent traffic lights are being replaced. (LEDs are also replacing incandescent lamps in many other applications, such as vehicle taillights). Many of the more exotic traffic signals would not be possible to construct without using LED technology.
LED-based lamps have nowadays numerous advantages over incandescent lamps; among them are:

- Much greater energy efficiency
- Much longer lifetime
- The ability to display multiple colors and patterns from the same lamp by multiple LEDs
- Much faster switching

Very soon, LEDs will be bright enough to light our homes, offices and perhaps our streets as well. The extreme energy efficiency of LEDs means that solar charged batteries can power LED units by night, bringing light to the Third World and other areas without mains electricity. Each decade, LED prices have fallen by a factor of 10 while performance has grown by a factor of 20. This behavior, known as Haitz’s law, is the LED equivalent of Moore’s Law in the chip industry. The Figure 1.2 show the Haitz’s Law in the case of LED flux. We can see that LED flux per package has doubled every 18-24 months for the last 30 years.

![Figure 1.2: Haitz’s law](image)

In spite of the exponential increasing of numerous parameters like power, flux, efficiency we are limited by the temperature which has its limit. Consequently it is necessary to find a solution to cool the device. In the same way the thermal and the conductive resistances must be decreased continuously.

LEDs are packaged with various materials, like polymers, dielectrics, ceramics and metals. Different design are elaborated and we can find a variety of power, colors and dimensions. An example is given in Figure 1.3.
All the materials used for the LEDs are interconnected and there are numerous interfaces. The III-V or II-VI semiconductors used to create various colors are grown on a sapphire substrate. After cutout, LEDs are connected to a support via flip-chip. An electrical insulator is used between the interconnection and the substrate to avoid leakage current. All these interfaces are shown in Figure 1.4. The heat needs to be extracted and the material’s thermal properties are most important.

### 1.2 Thermal conductivity dependence

Changes in temperatures cause thermal effects on materials. Some of these thermal effects include thermal stress, strain, and deformation. The first effect we will consider is thermal deformation. Thermal deformation simply means that as the "thermal" energy (and temperature)
of a material increases, so does the vibration of its atoms/molecules; and this increased vibration results in what can be considered a stretching of the molecular bonds - which causes the material to expand.

Hooke’s law can explain stress of elastic materials if we know the strain and the Young’s Modulus of the considered materials:

\[ \sigma = E \varepsilon \]  \hspace{1cm} (1.1)

Of course, if the thermal energy (and temperature) of a material decreases, the material will shrink or contract. Then, the thermal deformation is given by:

\[ \varepsilon = \frac{\Delta L}{L} = \alpha \Delta T \]  \hspace{1cm} (1.2)

The thermal stress which develops if a structure or member is completely constrained (not allowed to move at all) is the product of the coefficient of linear expansion and the temperature change and Young’s modulus for the material. This expression is obtained by combining equation (1.1) and (1.2):

\[ \sigma = \alpha \Delta T E \]  \hspace{1cm} (1.3)

The law of heat conduction also know as Fourier’s law states that the time rate of heat flow \( q \) is proportional to the gradient of temperature difference:

\[ q = kA \frac{\Delta T}{\Delta x} \]  \hspace{1cm} (1.4)

Where \( A \) is the transversal surface area, \( \Delta x \) is the thickness of the body of matter through which the heat is passing, \( k \) is the thermal conductivity dependent on the nature of the material at a given temperature, and \( \Delta T \) is the temperature difference through which the heat is being transferred.

However, if we are in a two-dimensional system where only two limits are involved, we may define a conduction shape factor \( S \) such that \[9\]:

\[ q = kS \Delta T \]  \hspace{1cm} (1.5)

So

\[ \Delta T = \frac{q}{kS} \]  \hspace{1cm} (1.6)

The value of \( S \) have been worked out for several geometries and are summarized by J.P. Holman\[9\]. A very comprehensive summary of shape factors for a large variety of geometries is given by Hahne and Grigull \[10\].

By combining the equation (1.3) and (1.6) we have:

\[ \sigma = \alpha E \frac{q}{kS} \]  \hspace{1cm} (1.7)
Thanks to this expression we can see that it is really important to take care of the thermal conductivity \( k \). As \( \sigma \) is proportional to \( 1/k \) we need to maximize the conductivity to decrease thermoelastic stress.

With the Table 1.1 we can find different results obtained at Lumileds Optoelectronics Division [11]. On the same way, they show that it’s really important to control temperature. If the junction temperature decrease about a factor 2 we observe that the estimated MTBF[1] expand about a factor 10. In the same time the failure rates decrease about a factor 9.5.

<table>
<thead>
<tr>
<th>Ambient Temp( °C)</th>
<th>Junction Temp( °C)</th>
<th>MTBF</th>
<th>Failure Rate (%/1K Hrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>85</td>
<td>124</td>
<td>1,333,000</td>
<td>0.075%</td>
</tr>
<tr>
<td>75</td>
<td>114</td>
<td>1,844,000</td>
<td>0.054%</td>
</tr>
<tr>
<td>65</td>
<td>104</td>
<td>2,596,000</td>
<td>0.039%</td>
</tr>
<tr>
<td>55</td>
<td>94</td>
<td>3,723,000</td>
<td>0.027%</td>
</tr>
<tr>
<td>45</td>
<td>84</td>
<td>5,449,000</td>
<td>0.018%</td>
</tr>
<tr>
<td>35</td>
<td>74</td>
<td>8,151,000</td>
<td>0.012%</td>
</tr>
<tr>
<td>25</td>
<td>64</td>
<td>12,489,000</td>
<td>0.008%</td>
</tr>
</tbody>
</table>

Table 1.1: Point typical performance in time which represents 60% confidence level. Values theoretically extrapolated from ageing at high temperature.

1.3 Internship goal and objective

The major goal of this work is to develop a recent method named 3.\( \omega \) method within the Photonic Materials and Devices department to be able to increase the thermal conductivity of the constituent materials inside LEDs in order to improve the power - reliability ratio. This method, well described and explained in Chapter 2 will enable to measure the thermal conductivity of thin layers and bulk. A lot of different parameters must be take in consideration to succeed in the measurement. However, to measure the thermal conductivity quickly enough we had to program and to automate the instruments. It was a real challenge to develop a new way of automation using mainly computer programming without human intervention. This part realized with the Labview[© software took an important place of the internship with all the optimization and the needed verification (Chapter 3). Thanks to the 3.\( \omega \) method theory and the experimental set-up we measured the thermal conductivity of different materials and look at the influence of the thickness (Chapter 4).

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1MTBF=Total device hours divided by the number of failure
Chapter 2

The \(3\omega\) method

2.1 Introduction

In all cases, the thermal conductivity of a film is lower than the thermal conductivity of the bulk material due to various defects becoming dominant at small dimensions. Unfortunately, thermal conductivity measurement is difficult especially on two-dimensional structures. Problems arise because of thermal radiation; heat loss in the temperature measuring probes and because of the power supply to the sample has to be known with high accuracy. Consequently a new method had to be found [12]. The three omega thermal conductivity measurement method has been used extensively to measure the thermal properties of bulk and thin film dielectric materials [12]-[17]. The method employs a metallic strip in contact with the surface (Chapter 3). When we applied an Alternating Current (Fig. 2.1), the strip emits a radial flow of heat from that is used both as heater and sensor. This method employs the thermal excitation of a sample with a plane-surface heater at frequency \(\omega\) and the observation of the \(3\omega\) thermal response of this metal element, whence the method’s name. The amplitude obtained for the response at \(3\omega\) depends on different parameters like frequency, temperature, thermal capacity and thermal conductivity of materials.

![Figure 2.1: Scheme of the metallic strip](image)

2.2 Theory of the \(3\omega\) method

2.2.1 Principle

As we applied an alternating current, we have the following equation

\[
I(t) = I_0 \cos(\omega t)
\]  

(2.1)

Where \(\omega\) is the angular modulation frequency of the electric current, heats the metal band and the sample at an angular frequency \(2\omega\) by Joule effect. On account of the increase of the temperature of the heater band there is a variation of the temperature according to the below-mentioned expression
\[ \Delta T = \Delta T_0 \cos(2\omega t + \Phi) \] (2.2)

Likewise, the resistance \( R \) oscillates at the angular frequency \( 2\omega \) and can be approximated as

\[ R = R_0(1 + \alpha R \Delta T) = R_0[1 + \alpha R \Delta T_0 \cos(2\omega t + \Phi)] \] (2.3)

The amplitude \( \Delta T_0 \) and the phase \( \Phi \) are directly linked to the thermal conductivity of the sample and to the angular frequency \( \omega \). If we consider the Ohm’s law, the voltage \( V \) across the heater is definite by

\[ V(t) = R(t)I(t) \] (2.4)

This expression is modulated in amplitude and is compound by an harmonic constant given such as

\[ V(t) = R_0I_0 \cos(\omega t) + \frac{1}{2} R_0I_0\alpha R \Delta T_0 \cos(\omega t + \Phi) + \frac{1}{2} R_0I_0\alpha R \Delta T_0 \cos(3\omega t + \Phi) \] (2.5)

The first term, which is just the normal dc voltage at the drive frequency, doesn’t contain any information on the amplitude \( \Delta T_0 \) and the phase \( \Phi \). However it is 1000 times more important than the other terms. The second term can’t be used to measure \( \Delta T_0 \) and \( \Phi \) for its frequency is the same as the first term, most important. The last term appears at the third harmonic of the leading frequency and it can be extracted to the signal thanks to Fast Fourier Transform (FFT) or lock-in amplifier. The amplitude of this term, \( V_{3\omega} \) is

\[ V_{3\omega} = \frac{1}{2} R_0I_0\alpha R \Delta T_0 \] (2.6)

Moreover the amplitude \( V_{3\omega} \) is function of the frequency \( f \). Thus we measure the amplitude \( V_{3\omega} \) at a constant temperature, for different frequencies \( f \), measuring the amplitude of the first harmonic \( V_\omega \). Considering a semi-infinite substrate we can apply the "slope method" (2.3). Borca-Tasciuc has compared two different methods and to conclude he show that the "slope method" is the better method if we keep some parameters in the good range of value [18]. Accordingly, we can fit the result with a linear equation:

\[ V_{3\omega} = \alpha_{3\omega} \ln(f) + b_{3\omega} \] (2.7)

\( \alpha_{3\omega} \) represent the slope of the line in semi-log scale whence the name "slope method".

Thanks to the equation (2.14) derived from a relation defined by Carslaw and Jaeger[19] and given by Cahill[14] we have:

\[ \delta T(r, t) = \frac{P}{\pi lk} K_0(qr) \] (2.8)
The reciprocal factor of the wave vector $q$ defines the wavelength of the thermal heat wave generated by electrical heating, such that

$$\frac{1}{q} = \sqrt{\frac{\alpha}{\omega}}$$  \hspace{1cm} (2.9)$$

The wavelength $\frac{1}{q}$ is often referred as the penetration depth of the heat wave, although damping distance might be more accurate. The magnitude of $\frac{1}{q}$ is an important parameter for designing the $3\omega$ method to probe materials of different thicknesses. The equation 2.9 shows the penetration depth of the thermal wave into a material decreases with increasing frequency.

In addition the penetration depth is associated with the thermal diffusivity $\alpha$ of a material. The thermal diffusivity $\alpha$ and the thermal conductivity $k$ are related to each other by the density $\rho$ and heat capacity $C$ of a material:

$$\alpha = \frac{k}{\rho C}$$  \hspace{1cm} (2.10)$$

Figure 2.2 shows the frequency dependence of the thermal penetration depth for 4 different materials: glass, nitride aluminium, silicon nitride and silicon. For example a penetration depth of 5$\mu$m is related to frequencies 1kHz, 50kHz and 200kHz for glass, silicon nitride and silicon, respectively. The huge difference in the required excitation frequency is due to the ability of the material to conduct heat. As higher the thermal conductivity of a material as higher a frequency is required.

For the $3\omega$ method, temperature oscillation is detected at the metal/sample interface by a metal line of line width $2b$.

**Figure 2.2:** Frequency dependence of the thermal penetration depth. I used the following parameters: $\alpha_{SiO_2}$=0.00834cm$^2$.s$^{-1}$; $\alpha_{AlN}$=0.112cm$^2$.s$^{-1}$; $\alpha_{Si_3N_4}$=0.133cm$^2$.s$^{-1}$; $\alpha_{Si}$=0.892cm$^2$.s$^{-1}$[20]
If we transform equation 2.8 to Cartesian coordinates, then the real space coordinate x is replaced by the Fourier space $\lambda$ to give:

$$\Delta T(x) = \frac{P}{\pi k}K_0(qx) = \frac{P}{\pi k} \int_0^\infty \frac{\cos(qxt)}{\sqrt{t^2+1}} dt = \frac{P}{\pi k} \int_0^\infty \frac{\cos(\lambda x)}{\sqrt{\lambda^2+q^2}} d\lambda$$

(2.11)

In general, $e^{i\lambda x} = \cos(\lambda x) + isin(\lambda x)$

(2.12)

The Fourier transform can be written as

$$\Delta T(x) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \Delta T(\lambda) e^{i\lambda x} d\lambda$$

(2.13)

$$\Delta T(x) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \Delta T(\lambda) \cos(\lambda x) d\lambda + \frac{i}{\sqrt{2\pi}} \int_{-\infty}^{\infty} \Delta T(\lambda) \sin(\lambda x) d\lambda$$

(2.14)

Combining equation 2.3 and equation 2.14 for $\Delta T(\lambda)$ yields [14]:

$$\Delta T(\lambda) = \frac{P}{\sqrt{2\pi l k}} \frac{1}{\sqrt{\lambda^2+q^2}}$$

(2.15)

Assuming heat enters the sample uniformly across the line width 2b the Fourier transformation [22] of the finite heat source of the line width 2b can be written as:

$$\frac{1}{2b} rect \left( \frac{x}{2b} \right) \rightarrow \frac{\sin(\lambda b)}{\lambda b}$$

(2.16)

The finite width of the metal line is included by taking the product of equation 2.14, after $\Delta T(\lambda)$ was substituted by equation 2.15 and equation 2.16 Then, the inverse transformation is used to get

$$\Delta T(x) = \frac{P}{\pi k} \int_0^\infty \frac{\sin(\lambda b) \cos(\lambda x)}{(\lambda b)\sqrt{\lambda^2+q^2}} d\lambda$$

(2.17)

Finally, equation 2.17 is integrated over the line width $-b < x < b$ and average by dividing by 2b [14]:

$$\Delta T = \frac{P}{\pi k} \int_0^\infty \frac{\sin^2(\lambda b)}{(\lambda b)^2 \sqrt{\lambda^2+q^2}} d\lambda$$

(2.18)

Equation 2.18 gives a general solution for the total temperature oscillation on a surface caused by a heat source of finite width 2b assuming one dimensional heat flow or an isotropic material, and neglecting the thermal boundary resistance between metal line and sample.
2.2.2 Measurements on bulk materials

Figure 2.3 and 2.4 schematically show the curvature of the temperature oscillation based on equation 2.18.

![Figure 2.3](image1)

**Figure 2.3:** Magnitude of the temperature in function of the frequency for a line width equal to $500\mu m$ in units of $P/\pi k$

![Figure 2.4](image2)

**Figure 2.4:** Magnitude of the temperature in function of the frequency for a line width equal to $50\mu m$ in units of $P/\pi k$

The frequency spectrum of the temperature oscillation for an infinite thick sample shown in Figure 2.3 can be divided into two regions [23]. At low frequencies, the thermal penetration depth is much larger than the heater width $2b$. Therefore, the temperature oscillation is nearly
linear versus logarithmic frequency. The linear curvature can be described by approximating equation 2.18. From the result of this approximation, the thermal conductivity can be determined by the "slope method" ([2.3]), which state that the thermal conductivity is inversely proportional to the slope of the linear curvature. These results can be extended to different materials (Figure 2.4) where the line width is really small if we compare it to the penetration depth.

At high frequencies, the penetration depth of the thermal wave is in the order of the line width 2b or smaller than 2b. In this case, the temperature oscillation shows an asymptotic curvature. Since an analytical solution for equation 2.18 in the asymptotic region is unknown [23], extracting the thermal conductivity from the temperature oscillation versus frequency plot is carried out by fitting the equation to measured data with the help of appropriate software. However, our experiment won’t use line widths in the same order of the penetration depth. Otherwise this region would be interesting if we work at high frequency because one could extract both specific heat and thermal conductivity.

2.2.3 General solution for the 2D heat conduction across a multilayer-film-on-substrate system [18]

The expression for the heater temperature rise is derived in the general case of a multi-layer film-on-finite/semi-infinite-substrate system with anisotropic thermophysical properties. The derivation is based on a two-dimensional heat-conduction model across the system and a uniform heat flux boundary condition between the heater and the top film. Neglecting the contributions from the thermal mass of the heater and the thermal boundary resistances, the complex temperature rise of a heater dissipating \( P \) peak electrical power per unit length is:

\[
\Delta T = \frac{P}{\pi k_y(1)} \int_0^\infty \frac{1}{A_1 B_1} \frac{\sin^2(\frac{b\lambda}{2})}{b^2 \lambda^2} d\lambda
\]

(2.19)

Where

\[
A_{i-1} = \frac{A_i \frac{k_y(i)B_i}{k_y(i-1)B_{i-1}} - \tanh(\Phi_{i-1})}{1 - A_i \frac{k_y(i)B_i}{k_y(i-1)B_{i-1}} \tanh(\Phi_{i-1})}
\]

(2.20)

\[
B_i = \left( k_{xy}(i) \lambda^2 + \frac{j2\omega}{\alpha_y(i)} \right)^{\frac{1}{2}}
\]

(2.21)

\[
\Phi_i = B_i d_i, \; k_{xy} = \frac{k_x}{k_y}
\]

(2.22)

In the above expressions, n is the total number of layers including the substrate, subscript i corresponds to the \( i \)th layer starting the top, subscript y corresponds to the direction perpendicular to the film/substrate interface (cross plane), b is the heater half width, k is the thermal conductivity of the layer, \( \omega \) is the angular modulation frequency of the electrical current, d is the layer thickness, and \( \alpha \) is the thermal diffusivity. The effect of the thermal conductivity anisotropy is introduced through the term \( k_{xy} \), which is the ratio of the in-plane to cross-plane thermal conductivity of the layer. For the substrate layer \( i=n \), and if the substrate is semi-infinite \( A_n=-1 \). When the substrate has a finite thickness, the value \( A_n \) depends on the boundary condition at the bottom surface of the substrate: \( A_n=-\tanh(B_n d_n) \) for an adiabatic boundary condition or \( A_n=-\frac{1}{\tanh(B_n d_n)} \) if the isothermal boundary condition is considered.
2.2.4 Analytical approach of the thermal amplitude $\Delta T_0$

In the case of a thin film deposed on the substrate, the temperature rise of the heater $\Delta T_0$ is composed of two parts: the first, $\Delta T_s$, is due to the substrate, and the second $\Delta T_f$ is due to the thin film:

$$\Delta T_0 = \Delta T_f + \Delta T_s \quad (2.23)$$

Thanks to equation 2.18 we obtain, if we consider that the heat flux is homogenous on all the width of the heater band, and the heater band is deposed on an electrical insulating material [14]:

$$\Delta T_s = \frac{P}{l\pi k_S} \int_0^\infty \frac{\sin^2(\lambda b)}{(\lambda b)^2 \sqrt{\lambda^2 + q^2}} d\lambda \quad (2.24)$$

But if we used the approximation where the thermal wavelength is much larger than the line width $\frac{\lambda}{q} \gg b$ and $|qr| \ll 1$, the complex temperature can be approximated as (Appendix C):

$$\Delta T_s = \frac{P}{l\pi k_S} \left[ \frac{1}{2} \ln \left( \frac{k_s}{C_p b^2 \rho} \right) - \frac{1}{2} \ln \left( 2\omega \right) + \eta - i\frac{\pi}{4} \right] = \frac{P}{l\pi k_S} F_{lin}(\ln(\omega)) \quad (2.25)$$

Where $\eta$ is a constant ($\eta=0.923$ [24, 25] or $\eta=1.05$ [25]) that is often fitted to the experimental data.

Since the interest of this report is to work with thin films, we have the following expression in the case of a thin film of thickness $d_f$. In that case the thin film behaves as a simple thermal resistance and adds a frequency-independent temperature oscillation to the result of equation 2.24:

$$\Delta T_f = \frac{P d_f}{2b k_f} \quad (2.26)$$

Thanks to the expressions 2.23, 2.25, 2.26 we know

$$\Delta T_0 = \frac{P}{l\pi k_S} \left[ \frac{1}{2} \ln \left( \frac{k_s}{C_p b^2 \rho} \right) - \frac{1}{2} \ln \left( 2\omega \right) + \eta - i\frac{\pi}{4} \right] + \frac{P d_f}{2b k_f} \quad (2.27)$$

This expression can be inserted in equation 2.6, which is function of $\Delta T_0$.

2.2.5 Thermal conductivity measurement for anisotropic materials

Equation 2.27 considers only the case of isotropic substrates although in some experimental situations the substrate thermal conductivity can be anisotropic. Thanks to the expression given by Borca Tasciuc we obtained an analytical expression of the "slope" using equation 2.19 for the case of a finite anisotropic substrate with adiabatic boundary condition on the bottom surface. If we divide the analytical slope by $-p/\pi l k_S \sqrt{k_{Sxy}}$ we obtained the following general expression for the normalized slope:
\[
\frac{d\Delta T/d\ln(\omega)}{-p/(\pi k_{Sy} \sqrt{k_{Sxy}})} = 
0.5 \int_{0}^{\infty} \frac{z^2}{B_S^3 \tanh(B_S \beta_S)} \ldots 
\left(1 + \frac{4B_S \beta_S}{(e^{B_S \beta_S} + e^{-B_S \beta_S})^2 \tanh(B_S \beta_S)} \right) \sin^2(\lambda) \frac{\lambda^2}{\lambda^2} d\lambda
\]  

(2.28)

where, \( z = q b / \sqrt{k_{Sxy}} \), \( \beta_S = \sqrt{k_{Sxy}}(d_S/b) \), and \( B_S = \sqrt{\lambda^2 + z^2} \).

The quantity \( z \) involves the thermal penetration depth, the heater half width and the effect of the substrate anisotropy. The quantity \( \beta_S \) as can be seen from the above is an expression for the ratio of the substrate thickness and heater half-width. Figure 2.5 below is a plot of the normalized slope versus \( z \) for different value of \( \beta_S \).

![Figure 2.5: Plot of normalized slope versus the dimensionless parameter z for different values of \( \beta_S \). The line at 0.5 value on the y-axis refers to the semi-infinite case.](image)

In Figure 2.5 the case of the semi-infinite substrate is given by \( \beta_S \to \infty \), i.e. large ratio of substrate thickness to heater half-width.
Figure 2.6: Plot of normalized slope versus the nondimensional parameter $q_dS$ for different values of $\beta_S$

The line source and semi-infinite approximations can be used with less than 1% error if $z<0.2$ and $q_dS > 5$. In this case, a simplified expression similar to the Equation 2.25 can be derived for an anisotropic substrate:

$$\Delta T = \frac{P}{\pi l k_{S_y} \sqrt{k_{Sxy}}} \left( \frac{1}{2} \ln \left( \frac{\alpha_{S_y} k_{Sxy}}{b^2} \right) - \frac{1}{2} \ln(\omega) + \eta \right) \quad (2.29)$$

If $z>0.2$, the line-source approximation is less applicable and the deviation between the approximation and exact solutions for the normalized slope increases rapidly as $z$ increases. If substrate thickness is finite, for small $z$ values the thermal wave is reflected by the surface of the substrate and the normalized slope can be very different from that predicted by the slope method.

If $q_dS > 5$, that is to say the penetration depth is at least 5 times smaller than the substrate thickness, indicates that the semi-infinite substrate thickness approximation is valid for the application of the slope method.

In the cross-plane thermal conductivity measurement, a simple one dimensional heat conduction model can be applied to obtain the thermal conductivity across a film, as long as all the necessary conditions are satisfied. Especially when the heater width is much larger than the film thickness. For anisotropic thermal conductivity measurement, a 4-wire $3\omega$ method is developed based on the two-dimensional heat conduction model. As shown in Figure 2.7 to be able to determine both the in-plane and the cross-plane thermal conductivity, the idea is to use a wide and a narrow heater.

The one with a narrow width comparable to the film thickness will measure the temperature drop due to both in-plane and cross-plane conduction. The one with a wide width is used to measure the temperature drop almost mainly due to cross-plane conduction.
In the case of a one-layer film with anisotropic thermal conductivities on a semi-infinite substrate, if we apply equation 2.19 to 2.22, the temperature rise is given by:

$$\Delta T = \frac{P}{\pi l k_f y} \int_0^\infty \frac{\sin^2(b\lambda)(b\lambda)^2\gamma_f}{(b\lambda)^2\gamma_f} \left( \frac{1 + (k_S\gamma_S)\tanh(\gamma_f d_f)/(k_f y\gamma_f)}{-\tanh(\gamma_f d_f) - (k_S\gamma_S)/(k_f y\gamma_f)} \right) d\lambda$$

(2.30)

where $\gamma_S$ and $\gamma_f$ are given by

$$\gamma_S = \left( \lambda^2 + q_s^2 \right)^{1/2}$$

(2.31)

$$\gamma_f = \left( k_{f,xy} \lambda^2 + q_{f,y}^2 \right)^{1/2}$$

(2.32)

The values of the in-plane and cross-plane thermal conductivity can be obtained by fitting simultaneously the temperature rises of both the narrow and wide heaters. We obtain in that case a unique solution.

2.3 Determination of the main parameters - "Slope Method"

2.3.1 Thermal conductivity of isotropic substrate $k_s$

There are different approaches that enable to find the thermal conductivity. For this work I have chose the method determined by Cahill in 1989 [14], which seems to be the more efficient with the different results obtained previously.

Thanks to the relation defined by Cahill, we can obtain the thermal conductivity as mentioned below:

$$k_s = \frac{V_{31}^2\ln(f_1^2)}{4\pi R^2(V_{3,1} - V_{3,2})} \frac{dR}{dT}$$

(2.33)

Where $V_{3,1}$ is the in-phase $3\omega$ voltage at frequency $f_1$ and $V_{3,2}$ is the in-phase $3\omega$ voltage at frequency $f_2$. In fact these parameters represent the slope of the curve $V_{3,\omega} = f(\ln(f))$, that is to say

$$\alpha_{3\omega} = \frac{V_{3,1} - V_{3,2}}{\ln(f^2)}$$

(2.34)
To determine $\frac{dR}{dT}$ we must find $\alpha_R$. Even so the temperature coefficient can be determined by the expression:

$$\alpha_R = \frac{1}{R} \frac{dR}{dT} \tag{2.35}$$

Thus, if we plot $R=f(T)$ we can determine the slope of the line if we fit the curve with a linear equation:

$$R = a_RT + b_R \tag{2.36}$$

Thanks to 2.35 2.36 we obtain:

$$\alpha_R = \frac{a_R}{a_RT + b_R} \tag{2.37}$$

These expressions enable us to determine a simplification of the equation 2.33 to this expression:

$$k_s = \frac{V_3^3 \omega a_R}{4\pi \alpha_3 \omega (a_RT + b_R)^2} \tag{2.38}$$

We can observe that $V_\omega$ is the most sensitive parameter and in that case all voltages are rms.$^1$

### 2.3.2 Thermal conductivity of isotropic film $k_f$

The thermal conductivity of the thin film [27] is determined using equation 2.39 which is derived from the second term in equation 2.27:

$$k_f = \frac{Pd_f}{2b(\Delta T_0 - \Delta T_s)} \tag{2.39}$$

Where $\Delta T_0$ is the temperature oscillation of the in-phase component for the substrate with the thin film and $\Delta T_s$ is the temperature oscillation of the in-phase component without the thin film. The temperature variation is obtained from the measurement of the third-harmonic voltage drop across the metal-line, $V_{3\omega}$, using the expression 2.6 by

$$\Delta T_0 = \frac{2V_{3\omega}}{\alpha_R I_0 R_0}, \quad V_\omega = I_0 R_0 \tag{2.40}$$

### 2.4 Thermal conductivity contributions

Heat transfer plays a fundamental role in the various phenomena that occur in our universe and can occur through three different mechanisms which are conduction, convection and radiation. In the case of the $3\omega$ method we consider only conduction. Indeed convection can be considered negligible because our device is positioned inside a copper box and the measurement begins after thermal stabilization of the whole equipment. Nevertheless we must consider contributions of phonons, photons and free carriers. We may say that the thermal conductivity measured by the $3\omega$ method is composed of three component consisting of the lattice component ($K_L$), the electronic component ($K_e$) and the radiative component ($K_R$).
2.4.1 Electronic contribution

The electronic thermal conductivity is given by the Wiedemann-Franz law which say that the thermal conductivity due to the electronic component \( K_e \) is proportional to the temperature \( T \):

\[
K_e = LT(qp\mu_y) \tag{2.41}
\]

with \( L \) = Lorenz number which is equal to \( 2.45 \cdot 10^{-8} \text{ W} \cdot \Omega \cdot K^{-2} \), \( q \) is the charge of the carrier, \( \mu_y \) is the carrier mobility in the cross plane direction, \( p \) is the carrier concentration.

This contribution takes an important place in the case of metals but stay quite negligible in the case of dielectric because there is no free carrier.

2.4.2 Phonon contribution

If we consider the simple expression for the thermal conductivity based on the Debye model:

\[
K_L = \frac{1}{3} C_p v \bar{\gamma} \tag{2.42}
\]

where \( k \) is the thermal conductivity, \( C_p \) specific heat, \( v \) molecular velocity and \( \bar{\gamma} \) is the mean free path between collisions. The molecular velocity can be determined by the following expression:

\[
v = \sqrt{\frac{3RT}{M}} \tag{2.43}
\]

with \( R \) the gas constant equal to \( 8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1} \).

In the case of a heater composed of Gold we have the following parameters \( M=196.96 \text{ g} \cdot \text{mol}^{-1}, k=116 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1} \), and \( C_p=0.914 \text{ J} \cdot \text{cm}^{-3} \cdot \text{K}^{-1} \). If we consider \( T=298.15 \text{K} \) we obtain for the velocity \( v=19,400 \text{ cm} \cdot \text{s}^{-1} \) and for the mean free path \( \bar{\gamma}=19.6 \mu \text{m} \). This value gives us an idea of the length scale involving phonon transport in the heater compared to its dimension. We see that the typical dimension of the heater thickness (0.4 \( \mu \text{m} \)) are inferior to \( \bar{\gamma} \) which show that we won’t probe the heater. In the case of a thin layer in silicon dioxide, if we take the parameters of SiO\(_2\), \( M=60.08 \text{ g} \cdot \text{mol}^{-1}, k=1.4 \text{ W} \cdot \text{cm}^{-1} \cdot \text{K}^{-1} \), and \( C_p=1.95 \text{ J} \cdot \text{cm}^{-3} \cdot \text{K}^{-1} \) we obtain \( \bar{\gamma}=0.02 \mu \text{m} \). However to be more accurate we should take in consideration the phonon wavelength, but it is not the goal of this report, we just want to have an idea of the intrinsic phenomenon.

2.4.3 Photon contribution

Thermal radiation corresponding to a mean temperature are present within every material and in thermal equilibrium this radiation is black body radiation. If the material is transparent for a particular photon it can pass through it without being disturbed. If not, photons will diffuse through the material in a manner similar to the flow of phonon. The photon thermal conductivity can be written as \([28]\):

\[
K_R = \frac{16}{3} \sigma_0 n^2 T^3\bar{\alpha}^{-1} \tag{2.44}
\]
Where $\sigma_0$ is the Stefan-Boltzmann constant, $n$ is the refractive index and $\tilde{\alpha}$ is the absorption coefficient.

The photon penetration depth is inversely proportional to $\tilde{\alpha}$:

$$\gamma = \frac{1}{\tilde{\alpha}}$$

(2.45)

Per example in the case of silicon dioxide different studies were done to measure the absorption coefficient\[29\]. We obtain a penetration depth about 0.1mm for a wavelength equal to 4$\mu$m showing that there is low interaction photon-matter (At 3$\mu$m we obtain 100cm). If we take the case at 7$\mu$m we can observe a penetration depth about 0.1$\mu$m which is at the limit of our considerations in comparison with the thickness of the studied layers. However we can also observe (Figure 2.8) that the peak at 7$\mu$m is really thin and we don’t really know what is the exact wavelength range generated by our device.

![Figure 2.8: Quartz absorption spectra\[30\]](image)

2.4.4 Conclusion

From these expressions we may conclude that the electronic thermal transport plays an important role and governs the thermal conductivity in the case of metals. Contrarily the thermal conductivity based on the phonon transport is dominant within dielectrics and in most cases (especially low temperature and thin film) photon thermal conductivity is negligible in comparison to the contribution by lattice vibrations.
Chapter 3

Experimental details

3.1 Sample

3.1.1 Sample design

The key component of the $3\omega$ measurement system is a thin metal line deposited on a dielectric material. As shown in Chapter 2, the metal line functions both as a heater by carrying electrical current and as thermometer by detecting the variation of the resistance (which in turn depends on the temperature and the thermal properties of the coating or substrate material). Four electrode pads are connected to the thin line: two pads function as current leads $I^+$ and $I^-$ and two pads are the voltage leads $V^+$ and $V^-$. The properties of the metal line and the electrode pads have to be optimized to match the $3\omega$ requirements (Equation C.11). Also, the dimensions of the metal line and electrode pads assembly should be feasible to be manufactured with reasonable effort.

The width of the thin metal band $2b$ must be between 5 and 100 $\mu$m and the length of the strip must be long compared to the width ($l \sim 4$ mm) (Figure 3.1) [24].

![Sample design](image)

Figure 3.1: Sample design consisting of the thin metal line of a width $2b$ and a length $l$ between two pads. The electrical connections to the pads are realized by wire bonding.
The metal of the wire and its contacts should have a high thermal conductivity and a low electrical resistance in order to achieve a high power input without increasing significantly the temperature of the metal line. In general, the materials which have an high electrical conductivity have also an high thermal conductivity. The behavior of these different conductivities is given by the Wiedemann-Franz law (Equation 2.41).

Moreover the metal line must be chemically inert to avoid metal line variation over time. To comply with this point we used gold. One drawback of noble metals is their poor adhesion to most dielectric materials requiring an intermediate adhesion layer, like titanium.

### 3.1.2 Sample preparation

The sample preparation including sample cleaning and supplying the electrodes was carried out in the cleanroom facility Philips MiPlaza by Eugene Timmering. The manufacturing steps are based on thin film technologies including mainly masking by lithographic processes and metallization by physical vapor deposition. A mask consisting of various device structure with varying heater widths was manufactured within Philips. Samples constituted by a thin dielectric layer use a silicon wafer (20-30 Ω.cm, p-type, <100>) as substrate. A schematic process is shown in Figure 3.2 and explained in the following paragraphs.

![Figure 3.2: Wafers process in the case of a thin dielectric layer](image)

**Thin layer**

The thin layers are realized in different ways. They must have a good contact with the substrate and properties close to the final materials used inside LEDs. We used two different dielectrics...
during this project, a silicon dioxide and a silicon nitride. It was impossible to deposit less than 20nm of Si$_3$N$_4$ due to our PECVD equipment contrarily to SiO$_2$ where we obtained 10nm. These thin layers are used to insulate the heater from the substrate or to be a study subject to see the influence of the thickness on the thermal conductivity. The process of each thin layer is explained in Chapter 4.

Heater

As gold is a noble metal, we need to use another layer to obtain a good adhesion with dielectric. In that way we used Ti which reacts with the dielectric interface to form in the case of silicon dioxide, TiO$_2$. Thanks to this property we obtain a good interface between dielectric and gold (Figure 3.3). 4000Å of gold and 200Å of titanium are deposited by physical vapor deposition.

![Figure 3.3: Localized oxidation of titanium at the interface Ti-SiO$_2$](image)

To enhance adhesion between photoresist and substrate Trimethylsilyldiethylamine (TMS-DEA) was applied via vapor techniques. This very thin TMSDEA layer could react to form reactive trimethylsilyl groups bound to the surface. The wafer was placed on the chuck of a Karl-Suess spinner and fixed by vacuum. Subsequently a positive photo-sensitive polymeric material HPR504 was applied during 30 sec with a rotation speed of 4,000rpm. The resist is soft baked 60 sec at 90°C. Then the wafer is placed beneath the 3omega mask and exposed to UV at 7.2mW.cm$^2$ during 3sec. The insulated resist is removed by HPR-developer following by both a Descum and a hard bake at 125°C. This hard bake enables to stabilize the remaining resist and the Descum enables to obtain a hydrophilic surface. Following that, the gold was etched in a Transene gold-etch, 30 sec over etch and TiN was etched in a Titanium-etch, 5 sec over etch.

Final preparation

To protect the sample during the dicing phase, wafers are covered by a resist, baked during 30 min at 125°C, which is removed with fuming nitric acid before doing wire-bonding. On each wafer we have 8 patterns reproduced numerous times. Cor Rijpert dices it and we chose the better samples, without any visible default, of each pattern. Ruud Smulders glues them on a DIL Lead side brazed package with an epoxy glue and bonds the contact pads of the sample to the pins of the DIL with thin gold wires (Figure 3.4).
3.2 Experimental set-up

The experimental set-up (Figure 3.5) is composed of an ultra low distortion function generator model Stanford Research Systems DS360, a resistor used as a reference and an Analogical Digital Converter card model NI PCI 4474. We use a climate cabinet Vötsch Heraus EK220 which is controlled by a Eurotherm regulator model 988D and connected to a computer thanks to a RS 232 connection. The sample is positioned in a copper box which enables both to stabilize the temperature and to decrease the variation due to convection (Figure 3.6).

The instruments are controlled under Labview©, programmed with the cooperation of Albert Geven and Henk Hessel. The length of the BNC cables has been minimized to 50cm. This is really important at high frequency. The generated signal is a low distortion sine-wave signal (less than 105dB, Appendix B). We extract the sine-wave input-output both to the resistance and to the sample (Figure 3.1). Thanks to an appropriate function we can define the rms voltage on
the resistors. The subtraction of the reference signal to the sample signal with a factor given by \((\text{AI}_0-\text{AI}_1)/(\text{AI}_2-\text{AI}_3)\) (Figure 3.7) enables to suppress the first harmonic to obtain mainly the third harmonic signal. This circuit was defined in collaboration with Jim Oostven and Henk Hessel. If we use a reference resistor \((R_{\text{ref}})\) in the same order of magnitude of the sample we can extract thanks to an FFT analysis the amplitude of the third harmonic signal for each frequency with an high S/N ratio. In contrast to a lock-in amplifier, the FFT enable us to obtain quickly, thanks to a software computation and without resistor adjustment, the \(V_{3\omega}\) amplitude.

Figure 3.6: Sample positioned in a copper box inside the climate cabinet

![Copper box inside climate cabinet](image)

Figure 3.7: Scheme of the circuit of measurement

![Circuit diagram](image)

It is also possible to use the Stanford DS360 output (+ and -) to obtain 2 opposite signals
We measure, in the same way the sine-wave input-output both to the resistance and to the sample. This method allows to apply the same power on each resistor but there is no real advantage compared to the circuit with only one sine-wave (Figure 3.7).

The automation allows us to perform measurements in an obscure laboratory without intervention (excepted to change the sample). Moreover it saves a lot of time for each experiment. The duration of each experiment which is equal to 12h30 can be launched each evening and the results can be extracted the next day.

3.3 Software

To be able to control the different instruments and to automate it, we need to use software like Labview\textcopyright. This is a revolutionary graphical programming development environment based on the G programming language for data acquisition, control, data analysis, and data presentation. Thanks to its intuitive interface, this make possible to control easily and quickly the different instruments and to observe our experiment evolution.

We realized 2 programs, the first one (§3.3.1) enables to obtain once and for all the temperature calibration of the sample. These values are used to measured the temperature with the heater during the experiment defined in the paragraph 3.3.2 and to calculate the thermal conductivity with the appropriate software.
3.3.1 Labview, Thermal coefficient

All samples must be calibrated to be able to obtain the temperature from the resistance variation. To that goal we realized a program which allows to control up to 10 samples simultaneously (Figure 3.9). Each sample, positioned in the oven, is connected to a Keithley 2000 Multimeter by the 4-wire method to measure its resistance. At the same time, a Keithley 2700 connected to a type K thermocouple measures the variation of temperature inside the climate cabinet.

Figure 3.9: Picture of the samples connections

The software allows to set easily the instrument parameters like GPIB, thermocouple(s) channel(s), calibration parameters for the thermocouple (Appendix F), parameters of each samples, temperature range . . . The interface is given in Figure 3.10.

Figure 3.10: Labview© interface - software TempCoeff enabling to define the sample thermal resistance

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Thanks to Figure 3.11 and equations 2.36-2.37 we can obtain the parameters $a_R$, $b_R$ and $\alpha_R$ necessary for each sample by a linear fitting.

![Figure 3.11: Heater resistance versus Temperature for several samples](image)

**3.3.2 Labview, 3ω method**

The goal of this software realized during my internship is to be able to extract automatically a $V_{3\omega}$ amplitude frequency spectrum over a temperature range. A simplify flow chart can be found in Appendix E.

This software is divided in four pages named: "Setting", "Meas", "Spectrum" and "Amplitude" (Figure 3.12). Thanks to its interface it is possible to define all the parameters necessary for the experiment. The page "Settings" enables to define the materials, the thickness and to add some comments about the goal of each experiment. On this page we can also set all the temperatures of the climate cabinet during the experiment with a stabilization time. We can control in the same manner the Stanford DS360 with its parameters, output function, DC offset, voltage, impedance and GPIB address. Another box named Meas params enables to choose the reference resistance and the way of the frequency, that is to say decreasing or increasing. Likewise we can define the place of each temperature measurement knowing that we have 49 frequencies.
On the page "Meas" we observe and we control voltages on the four input (Figure 3.13).

After numerical analysis we can obtain the $3\omega$ sine-wave and the FFT spectrum of this signal (Figure 3.14). We can check the amplitude and the proportion of first and third harmonic in the resulting signal.

The last page "Amplitude" gives the most interesting results (Figure 3.15). On this page...
we can observe the $3\omega$ signal frequency spectrum. This curve enables to define the thermal conductivity of the analyzed materials (See Equation 2.38 and 2.39).

For security, all the measurement are saved before changing temperature. This also allows to control the measurement during the experiment and, if necessary, to begin to extract them.
3.4 Statistics

Measurement errors are composed of repeatability, reproducibility and systematic errors.

3.4.1 Repeatability analysis

The repeatability is the ability of an instrument or a group of instruments to give the same measurement under repeated identical conditions.

We launched 16 experiments in the same conditions. For this analysis we used a sample composed of a 10nm thin layer of SiO$_2$ with a heater width equal to 10µm. We put down the sample into the climate cabinet regulate at 323K with 5% relative humidity. We took a reference about 22Ω and we started the software, realized under Labview©, Spectrun3w_QMH.vi. The results are mentioned in the Figure 3.16.

![Repeatability Analysis](image)

Figure 3.16: Repeatability analysis for a sample constituted with a 10nm thin layer SiO$_2$ with a heater width equal to 10µm at 329K. The repeatability is 0.34W.m$^{-1}$.K$^{-1}$≈0.2%

Thanks to this experiment we can see that there is a low thermal conductivity variation after numerous experiments. We obtained a standard deviation equal to 0.34. The thermal conductivity repeatability can be given, in that case, with a relative error equal to 0.2%.

3.4.2 Reproducibility analysis

The reproducibility is the ability of an experiment to be accurately reproduced by other operators using other samples.

As this experiment is automatic all the parameters are included into the software, the unique interventions are to change the sample and to launch the software. Thus we can think that there is only sporadically variation between each person which are negligible.

The sample doesn’t really influence the results if the quality of the thin layer is practically the same. The heater won’t change the results if it has a good contact with the thin layer, however its dimensions must be checked with accuracy.
3.4.3 Systematic error

There exist different systematic errors coming from the equipment, the sample but also from the operator. It is difficult to see these errors because the only mean to observe the error is to remove it. Repeating the measurement won’t change anything but averaging a large numbers of results. The only way is to calibrate the different instruments with other instrument more accurate.

- Some possible sources of systematic errors have been checked and illustrated largely:
  
  - Appendix A: climate cabinet behavior, if we wait 2h there is only a temperature variation of 0.2%
  
  - Appendix B: Stanford DS360 with the NI PCI 4474 card which showed a precision about at least 1\(\mu\)V and a 3\(\omega\) distortion inferior to -103.5dB.
  
  - Appendix F: calibration of the Keithley 2700 connected to a type K thermocouple

However all these verifications don’t remove the systematic error and confirm the idea that it is impossible to obtain something really perfect however we can approach this perfection by checking all our instruments.

- Each sample characteristic dimensions are measured by microscope and ellipsometry and these instruments must be well calibrated. We can observe (Figure 4.4) some irregularity on the edge of the heater which involved another error. We must take in consideration the heater width and length with the better accuracy. Per example the length of the heater is taken at the middle of each perpendicular connection and the heater width at the minimum dimension (Figure 3.17).

- Measurement errors of parameters in our models.

- Model simplifications leading to bias in parameter estimations.

- The operator can make error that he doesn’t know the existence.

![Figure 3.17: Sample characteristic dimensions measurement](image-url)
Systematic errors are difficult to detect and cannot be analyzed statistically, because all of the data is off in the same direction (either to high or too low). Spotting and correcting for systematic error takes a lot of care. Moreover there is another systematic error coming from the fixed parameters used during the extraction of our results like, for example, substrate thermal diffusivity.

### 3.5 Measurement validation

To be sure about our results we measured the thermal conductivity of silicon which is well known in literature [20]. Using different temperatures between 328 and 428K, we measured the thermal conductivity considering the isotropic case (§2.3). The results are mentioned in the Figure 3.18 which are within the thermal conductivity measurement accuracy range.

![Figure 3.18: Measurement of the thermal conductivity of the silicon substrate by the 3ω method compared to literature values [20]](image)

The variation that we can observe between our results and the reference may be explained by the quality of the silicon. We can say that the isotropic case model is well validated with our experiment.
Chapter 4

Results

Due to different parameters like impurities, lattice parameter, stress, dangling bonds and lacunases, the thermal conductivity of the material is modified. The thermal conductivity in the bulk case considers the whole of the material and defects become negligible, but if we decrease the thickness of such materials we increase the defects proportion. At the interface between the thin layer and the substrate there is compulsorily some dangling bond because of the lattice parameter. The thermal conductivity which is mainly engendered by the phonon conductivity, in the case of dielectric, would ergo be decreased. Then it is interesting to define the variation of the thermal conductivity versus the thin layer thickness. We observed in different articles\[25\] that the thermal conductivity of a dielectric thin layer is the same as a bulk over 500nm. That is why we choose, in the case of the dielectric SiO$_2$ and Si$_3$N$_4$ the following thicknesses : 50-100-200-500nm. It is interesting to begin our measurement with those materials because we have some information in the literature which can help us to validate our experiment.

4.1 General solutions

4.1.1 Thermal conductivities

We must consider two different cases, firstly we must define the thermal conductivity of the substrate to be able to extract the thermal conductivity of the film, in that case we consider the substrate isotropic. Secondly we can define the thermal conductivity of the film in the case of the "slope method" (§2.3), thus we obtain the isotropic thermal conductivity or to be more accurate an apparent thermal conductivity. Another way is to use the general solution for the 2D heat conduction (§2.2.3) and fitting two curves coming from the same sample thickness with a wide and a narrow heater width. The better software enabling to comply with equation 2.19 is Matlab©. We will compare both cases in this paragraph.
Isotropic case

Typical results obtained with Labview are showed in Figure 4.1.

![Variation of the amplitude $V_{3\omega}$ versus frequency](image1)

The isotropic case enables to define quickly the thermal conductivity of a considered material. Using the slope of the curve $V_{3\omega}$ versus frequency we can extract the thermal conductivity of the substrate (Equation 2.38). To define the thermal conductivity of a thin film we must simulate the substrate contribution to the temperature rise (Equation 2.27). After substraction of this thermal contribution (Equation 2.23) we can define the thermal conductivity of the film using Equation 2.39 and 2.40. Figure 4.2 show the employed method, we can observe that the slope of the two curves is similar and the resulting $\Delta T_f$ is constant over the frequency.

![Measured temperature oscillation (rms) versus frequency](image2)
Anisotropic case

This case, more elaborated than the first one, uses equations 2.19 to 2.22 which are translated for a sample, with a thin layer having anisotropic thermal conductivities on a semi-infinite substrate by the equations 2.30 to 2.32. We used a software realized under Matlab® program which enables to estimate the fitting parameters with different methods (Simplex, Gauss-Newton, Levenberg-Marquardt . . .). The study will be done using two parameters $k_{fy}$ and $k_{fxy}$ ($k_{fy}$ $k_{fx}$ are collinear) which are respectively the cross plane thermal conductivity and the ratio of the in-plane and cross-plane thermal conductivities. We must fit with the same parameters $k_{fy}$ and $k_{fxy}$ to the temperature rises obtained by a wide and a narrow heater. For example in the case of silicon dioxide thin layer we obtain the following fit (Figure 4.3).

![Figure 4.3: Temperature rise for a wide and a narrow heater on a 500nm SiO2 thin layer. Average power input is 197mW.](image)

The result seems to show a good fit of our experimental points. Values are coherent with those expected. However to be sure about the accuracy of our fit we must consider different parameters like the impact of :

- the integration parameters
- the noise
- the distance between the experimental points and the fitting curves

This part will be developed in paragraph 4.5.

4.2 SiO$_2$

4.2.1 Preliminary analysis

Silicon dioxide is used as an insulator between the interconnection and the substrate of LEDs (Figure 1.4). A wet oxidation at 1000 °C and a dry oxidation at 900 °C enabled to realize different kinds of silicon dioxide. After processing some studies were done on these samples like
Ellipsometry by Eugene Timmering. This technique gave us the thickness of the silicon dioxide in 9 points (4 on the edge of the wafer, 4 in each quarter and 1 in the center). The results are given in the Table 4.2.3.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>Min.(Å)</th>
<th>Max.(Å)</th>
<th>Mean(Å)</th>
<th>Standard deviation(Å)</th>
<th>Uniformity(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>D10</td>
<td>99.5</td>
<td>104.7</td>
<td>102.056</td>
<td>1.642</td>
<td>2.55</td>
</tr>
<tr>
<td>D50</td>
<td>486.0</td>
<td>501.7</td>
<td>490.633</td>
<td>5.038</td>
<td>1.60</td>
</tr>
<tr>
<td>W100</td>
<td>995.5</td>
<td>1058.9</td>
<td>1021.522</td>
<td>24.029</td>
<td>3.10</td>
</tr>
<tr>
<td>W200</td>
<td>1948.3</td>
<td>2016.7</td>
<td>1963.089</td>
<td>21.469</td>
<td>1.74</td>
</tr>
<tr>
<td>W500</td>
<td>4956.4</td>
<td>5015.6</td>
<td>4982.489</td>
<td>18.710</td>
<td>0.59</td>
</tr>
</tbody>
</table>

Table 4.1: Thickness of silicon dioxide measured by Ellipsometry

Due to the limited time we had to make some choice between the samples and define which are the more interesting. It’s really interesting to be sure about our substrate, that is why we used 2 samples from Wafer D10 which is covered by a thin insulator layer to define with accuracy the thermal conductivity of the silicon. In the same way we will be able to detect any anisotropy. The Wafer W500 has a thick enough layer to consider that its thermal conductivity is close to the bulk, thus we can check our experiment with data from the literature. For Wafer D50, W100 and W200 we used only one sample.

For each sample we checked the heater width with a microscope (magnifying x50-x100), we observed that over-etching has caused a lot of damage for the thin heater and variation of the width about ±18% (Figure 4.4). Heaters with a band width inferior to 10µm don’t work in the majority of the cases. This last observation is the same for silicon nitride.

![Figure 4.4: Thin layer of SiO₂ about 500nm with a theoretical band width about 15µm](image)

We can observe on the Figure 4.4 that the etched band is not linear but chiselled. The measure of the effective width is then defined by taking the minimum width, avoiding etch variations.

To resume we have the following sample specifications (Table 4.2).

### 4.2.2 Bulk and film thermal conductivities

An important point, enabling to determine the thermal conductivity of the thin layer, is the extraction of the substrate thermal conductivity. This value must be measured for each sample.
### Table 4.2: SiO₂ sample specifications

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mean Film thickness(Å)</th>
<th>Heater width(µm)</th>
<th>$a_R(ΩK^{-1})$</th>
<th>$b_R(Ω)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>D10-15</td>
<td>102.056</td>
<td>14.9</td>
<td>0.0585</td>
<td>9.6167</td>
</tr>
<tr>
<td>D10-35</td>
<td>102.056</td>
<td>36.2</td>
<td>0.0238</td>
<td>4.0233</td>
</tr>
<tr>
<td>D50-15</td>
<td>490.633</td>
<td>8.42</td>
<td>0.104</td>
<td>17.538</td>
</tr>
<tr>
<td>W100-15</td>
<td>1021.522</td>
<td>12.8</td>
<td>0.07</td>
<td>12.182</td>
</tr>
<tr>
<td>W200-15</td>
<td>1963.089</td>
<td>10.8</td>
<td>0.0774</td>
<td>13.204</td>
</tr>
<tr>
<td>W500-15</td>
<td>4982.489</td>
<td>14</td>
<td>0.0642</td>
<td>10.649</td>
</tr>
<tr>
<td>W500-35</td>
<td>4982.489</td>
<td>35.4</td>
<td>0.0244</td>
<td>4.1873</td>
</tr>
</tbody>
</table>

In order to calculate the temperature rise of the substrate (Equation 2.27). These results are shown for different thickness of silicon dioxide in Figure 4.5.

**Figure 4.5: Measurement of the thermal conductivity of the silicon substrate for different sample**

They agree reasonably well with the literature [20] with an estimated error $\sigma$ equal to 7%. However we can see that if we increase the thickness of the intermediate layer, the silicon thermal conductivity decreases slightly. This influence can be explained by the fact that we probe more the film than the substrate when the thickness increase.

Now we will compare the results obtained with the offset and the two-layer model. We could see in the Table 4.3 how the good agreement of the two models occurs because the substrate is thicker than the longest thermal diffusion length, which occurs at the lowest modulation frequency.

Moreover the heater used in the case of the offset method being narrow, we probe especially the cross plane thermal conductivity ($k_{xy}$). We observed that $k_{xy}$ is unstable when we change some parameters, the thermal diffusivity of both film and substrate influence this result. The in-
Table 4.3: Comparison of the fitting results from the offset method, case (a) and two-layer model, case (b) for the silicon dioxide thermal conductivity. Values are given for a temperature of 350 °K.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Offset</th>
<th>Two-layer model</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( k )</td>
<td>( k_y )</td>
</tr>
<tr>
<td>W.m(^{-1}).K(^{-1})</td>
<td>W.m(^{-1}).K(^{-1})</td>
<td>W.m(^{-1}).K(^{-1})</td>
</tr>
<tr>
<td>D10</td>
<td>0.26</td>
<td>-</td>
</tr>
<tr>
<td>D50</td>
<td>0.76</td>
<td>-</td>
</tr>
<tr>
<td>W100</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>W200</td>
<td>1.13</td>
<td>-</td>
</tr>
<tr>
<td>W500</td>
<td>1.28</td>
<td>1.25</td>
</tr>
</tbody>
</table>

The cross-plane value is not accurate enough to be measured whereas the cross-plane thermal conductivity for the anisotropic case show an uncertainty about 15%.

4.2.3 Thickness and temperature dependence

The thermal conductivity is slightly lower in comparison with the values given by the literature. However this can be explained by the quality of the thin layer which can differ between different processes. The results are shown in Figure 4.6 and show an increase of the thermal conductivity whilst the temperature increases. However if we consider that we should be in the same condition we obtain an error equal to 10%.

Figure 4.6: Measurement of the thermal conductivity of the silicon dioxide versus temperature

The different measurement done on our sample showed a thickness dependence. The thermal conductivity is seen to decrease with decreasing thickness (see Figure 4.7). Figure 4.8 shows the thermal resistance plot, for the results given in Figure 4.7, \( R = \frac{d}{k_{SiO2}} \).
Figure 4.7: Measurement of the thermal conductivity of the silicon dioxide versus its thickness at 3 temperatures as a function of film thickness \( d_f \).

Figure 4.8: Plot of the thin film resistance of the silicon dioxide films as a function of thickness. The nonzero intercept, \( R_i \), represent a fixed thermal resistance present in all the samples.

The apparent decrease of \( k_{SiO_2} \) could be defined as a thermal resistance layer within the structure. If we fit with a linear curve through the experimental points, we obtain by the intercept an interfacial resistance \( R_i \) equal to \( 2.96 \times 10^{-8} \text{m}^2 \cdot \text{K} \cdot \text{W}^{-1} \) at 340 °K. Different explanation may
be formulate but we can’t say if this resistance is between the film and the substrate or between
the film and the heater. This resistance could be due to defects like dangling bonds, lacunas or
interstitials. Our values for $R^i$ are comparable to those estimated by Käding and co-workers for
Au/Cr/SiO$_2$/Si prepared in a manner similar to our[31].

### 4.3 Si$_3$N$_4$

#### 4.3.1 Preliminary analysis

Like silicon dioxide, silicon nitride is used as an insulator between the interconnection and the
substrate of LEDs. The thickness parameters are obtained by ellipsometry and are given in the
Table [4.4]. This dielectric was grown by PECVD at 400 °C and it was impossible to obtain a
thickness inferior to 20nm.

<table>
<thead>
<tr>
<th>Wafer</th>
<th>Min.(Å)</th>
<th>Max.(Å)</th>
<th>Mean(Å)</th>
<th>Standard deviation(Å)</th>
<th>Uniformity(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NS20a</td>
<td>277.0</td>
<td>282.1</td>
<td>278.556</td>
<td>1.819</td>
<td>0.92</td>
</tr>
<tr>
<td>NS20b</td>
<td>226.0</td>
<td>229.2</td>
<td>227.189</td>
<td>0.980</td>
<td>0.70</td>
</tr>
<tr>
<td>NS20c</td>
<td>200.7</td>
<td>204.6</td>
<td>202.244</td>
<td>1.235</td>
<td>0.96</td>
</tr>
<tr>
<td>NS50</td>
<td>578.5</td>
<td>586.4</td>
<td>581.044</td>
<td>2.263</td>
<td>0.68</td>
</tr>
<tr>
<td>NS100</td>
<td>1014.0</td>
<td>1023.3</td>
<td>1018.244</td>
<td>3.073</td>
<td>0.46</td>
</tr>
<tr>
<td>NS200</td>
<td>2006.8</td>
<td>2041.2</td>
<td>2021.433</td>
<td>10.363</td>
<td>0.85</td>
</tr>
<tr>
<td>NS500</td>
<td>4993.6</td>
<td>5081.1</td>
<td>5033.244</td>
<td>28.179</td>
<td>0.87</td>
</tr>
</tbody>
</table>

Table 4.4: Thickness of silicon nitride measured by Ellipsometry

For this material we didn’t use the Wafer NS20c because there was a problem with gold and
we observe different contrasts which could come from roughness, unflatness or grain (Figure
4.9).

![Figure 4.9: Thin layer of Si$_3$N$_4$ about 20nm with a theoretical band width of about 25µm](image)

The general observations are the same as SiO$_2$, we can resume the different samples used
during our experiment in Table 4.5.
### Table 4.5: Si$_3$N$_4$ sample specifications

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mean Film thickness(Å)</th>
<th>Heater width(µm)</th>
<th>$a_H(\Omega K^{-1})$</th>
<th>$b_H(\Omega)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NS20a-15</td>
<td>278.556</td>
<td>14</td>
<td>0.0652</td>
<td>11.321</td>
</tr>
<tr>
<td>NS20b-15</td>
<td>227.189</td>
<td>14.05</td>
<td>0.0631</td>
<td>10.612</td>
</tr>
<tr>
<td>NS50-15</td>
<td>581.044</td>
<td>13.85</td>
<td>0.0631</td>
<td>10.612</td>
</tr>
<tr>
<td>NS100-15</td>
<td>1018.244</td>
<td>12.8</td>
<td>0.0674</td>
<td>11.525</td>
</tr>
<tr>
<td>NS200-15</td>
<td>2021.433</td>
<td>13.2</td>
<td>0.0666</td>
<td>11.298</td>
</tr>
<tr>
<td>NS500-10</td>
<td>5033.244</td>
<td>8.25</td>
<td>0.1129</td>
<td>18.96</td>
</tr>
<tr>
<td>NS500-15</td>
<td>5033.244</td>
<td>13.9</td>
<td>0.0642</td>
<td>11.003</td>
</tr>
<tr>
<td>NS500-35</td>
<td>5033.244</td>
<td>35.6</td>
<td>0.0247</td>
<td>4.2864</td>
</tr>
</tbody>
</table>

#### 4.3.2 Thermal conductivities

As for silicon dioxide samples we must determine first the substrate thermal conductivity (Figure 4.10). The results obtained in that case are close to the literature values.

![Figure 4.10: Measurement of the thermal conductivity of the silicon substrate for different sample](image)

After extraction of the thermal conductivity by the previous method ("slope method") we obtained the following results (Figure 4.11).
The obtained results are slightly lower than the thermal conductivity of silicon dioxide. The theory given by L. van der Tempel on the thermal conductivity (phonon) of glass agree well with our experiment showing that we have a phonon contribution. Like silicon dioxide, the different measurement done on our samples showed a thickness dependence. The thermal conductivity is seen to decrease with decreasing thickness (see Figure 4.12).

The SiO$_2$ and Si$_3$N$_4$ thermal conductivity behavior in function of the film thickness is rel-
atively the same. We obtain in that case a thermal resistance $R_i$ equal to $4.05 \times 10^{-8} \text{m}^2 \cdot \text{K} \cdot \text{W}^{-1}$ at $340 \, ^\circ \text{K}$ suggesting that we have more interfacial default than silicon dioxide. Indeed the thin layer are obtained by different manner, silicon dioxide is obtained by oxidation whereas silicon nitride is obtained by PECVD. We can understand that it is more difficult to deposit a thin layer than to grow it with good interface. However these values are really low and we can not really conclude.

### 4.4 Thickness analytical prediction

The striking thickness dependence of the thermal conductivity observed in the present report was interpreted using the approach proposed by Lambropoulos et al. [33]. They considered that a thermal resistance may develop at the film/substrate interface. This interfacial resistance $R_i$ would affect the thermal resistance of the film. In developing an equation for the apparent thermal conductivity, they argued the presence of such a large interfacial resistance implied that the temperature was virtually discontinuous across the interface. The following temperature discontinuity was taken to be a linear function of the heat flux across the interface. When the thermal properties of the interface were combined, they established the equation 4.1:

$$k(d) = k_{film}^{intrinsic} \frac{d}{R_i k_{film}^{intrinsic} + d}$$  \hspace{1cm} (4.1)

In equation 4.1, $k(d)$ is the experimentally determined, thickness-dependent apparent thermal conductivity of thin SiO2 films, $k_{film}^{intrinsic}$ is the intrinsic thermal conductivity of the film, $d$ is the thickness of the layer and $R_i$ is the interfacial resistance. Figure 4.13 shows that this equation fits the experimental results well and establishes values for the intrinsic thermal conductivity of silicon dioxide films at 1.36 W.m$^{-1}$K$^{-1}$ and the interfacial resistance at $2.8 \times 10^{-8} \text{m}^2 \cdot \text{K} \cdot \text{W}^{-1}$ at $340 \, ^\circ \text{K}$. This value is close to the value obtain in paragraph 4.2.3.

![Figure 4.13: Silicon dioxide thermal conductivity versus thickness at 340 °K. The fitting curve is the least squares curve fit conforming to equation 4.1.](image)

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If we plot the thermal conductivity of silicon dioxide using the parameters defined above we can estimate what are the interesting thickness enabling to know the thermal conductivity behavior versus thickness (interesting choice: 10, 100, 1000nm).

![Thermal conductivity estimation for silicon dioxide](image)

**Figure 4.14**: Thermal conductivity estimation for silicon dioxide correspondingly to equation 4.1

### 4.5 Model precision

This part will consider only the anisotropic case after fitting by Matlab.

#### 4.5.1 Film analysis

All the analyses using equation 2.30 show a good accuracy for the parameter $k_y$. However we observed important variations of the parameter $k_{xy}$ when we modify the thermal diffusivity of both film and substrate. The fitting is always correct with a residue repartition similar to a gaussian centered on 0. The influence of the integration parameters is less important when we choose limit higher than $1.10^{-9}$ to $1.10^9$, the variation being about less than 0.1%. The result seems to be convergent with increasing the integration parameters.

#### 4.5.2 Substrate analysis

This analysis use only the substrate as an anisotropic material. Equation 2.29 enable to extract the cross-plane and the in-plane thermal conductivity by a fitting similar to the equation 2.30. The results show a good accuracy between the model and the experiment. The value of $k_y$ and $k_{xy}$ show an isotropic behavior, like we could expect (Figure 4.15).
The value of the cross-plane thermal conductivity is close to the value given by the literature (σ=7%) and show a difference about 5% which seems to be more accurate than the "slope method" but we have not enough results to conclude. The final residue show the following repartition (Figure 4.16):

Figure 4.16: Residual between the model and our experimental points

The residual scattering seems to be homogeneous, only two points show an absurd behavior. If we distribute the residue versus their value we obtain the figure 4.17:

Figure 4.17: Distribution of the residue versus their value
Figure 4.17: Repartition of the residue versus their value

The repartition is centered on 0 and follows a Gaussian law.
Conclusion

Due to the limited time we had not measure materials with high thermal conductivity.

- The set-up is operational from 50Hz to 15kHz

- The experiment is fully automated and controlled by Labview software. It takes 12h30.

- The measurement accuracy of the thermal conductivity by the $3\omega$ method is:

  - 7% for bulk.

  - 10% and 15% for respectively isotropic and anisotropic film.

  - 0.2% repeatability.

- A thin dielectric layer shows a:

  - thermal conductivity equal to 1.28W.m$^{-1}$.K$^{-1}$ and 1.23W.m$^{-1}$.K$^{-1}$ for respectively SiO$_2$ and Si$_3$N$_4$ 500nm thin layer at 350°K.

  - thickness dependence.

  - temperature dependence, the thermal conductivity increase with temperature due to the phonon conductivity of semi-transparent glass.

- The interfacial resistance for SiO$_2$/Si$_3$N$_4$ is about 3.10$^{-8}$m$^2$.K.W$^{-1}$.
Bibliography


[29] E. Loenen, L. van der Tempel, Determination of absorption coefficient of glasses at high temperatures, by measuring the thermal emission, Philips Unclassified Report 020/96


Appendix A

Temperature stability of the climate cabinet

The thermal conductivity is temperature dependent. If we want to obtain a satisfactory accuracy we need to check the stability of the climate cabinet Vötsch Heraeus EK220. The oven is equipped with by a temperature and humidity controller which can be set to obtain a good regulation of both temperature and relative humidity. That is why we measured during five hours, after stabilization of the instrument, both rise and variation of temperature with a thermocouple type K connected to a Keithley Multimeter. The experiment set point was $T=373 \, ^\circ\text{K}$ and we put the thermocouple in the same copper box as used around the $3\omega$ sample which enabled to stabilize the thermal exchange. This box should increase the temperature stability. (Fig. A.1).

![Temperature stability of the climate cabinet](image.png)

Figure A.1: Temperature stability of the climate cabinet

We observe that it is necessary to wait at least 1h30 to obtain 0.7% of temperature variation in
the course of the measurement of our sample characteristics (if we assume a measurement time
about 30min). If we wait 2h30 before starting the experiment we reach 0.05\% variation (Fig.
[A.2]). During the experiment we observed an offset between the oven and Keithley thermocouple
of 5\%. Consequently it is important to put the sample in the appropriate place and to control its
temperature accurately to its surface. The temperature plays a part in the determination of the
thermal conductivity as we saw in Chapter 2.

Figure A.2: Zoom around 105°C to see the variation of temperature

We see that if we wait 2h there is only a temperature variation of 0.2\%. This will engender
a thermal conductivity error <0.4\% according to the equation for k in Chapter 2.
Appendix B

3\omega distortion

It is important to know the 3\omega distortion of each instrument used. These distortions can entail V_{3\omega} and biased the thermal conductivity measurement (2.38). Correspondingly we measure both, the 3\omega distortion of a ultra low distortion function generator model Stanford Research Systems DS360 with a NI PCI 4474 (24bits) and a NI PCI 6259 (16 bits) Analogical-Digital conversion card. Accordingly the detection of the distortion is determined by FFT\(^1\) which enabled to find the distortion in 2\omega and 3\omega of the generator. The different measures show important differences between the two NI PCI cards yielding better results with the PCI 4474. In this way we obtain for different frequencies and a V_{rms}\(^2\) of 5V the following results:

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>Amplitude V3\omega(\mu V)</th>
<th>dB</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>26.7</td>
<td>-105dB</td>
</tr>
<tr>
<td>1000</td>
<td>26.4</td>
<td>-105dB</td>
</tr>
<tr>
<td>2000</td>
<td>26.5</td>
<td>-105dB</td>
</tr>
<tr>
<td>3000</td>
<td>25.6</td>
<td>-105dB</td>
</tr>
<tr>
<td>5000</td>
<td>25.7</td>
<td>-105dB</td>
</tr>
<tr>
<td>7000</td>
<td>28.0</td>
<td>-105dB</td>
</tr>
<tr>
<td>10000</td>
<td>27.7</td>
<td>-104.5dB</td>
</tr>
<tr>
<td>15000</td>
<td>33.8</td>
<td>-103.5dB</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>Amplitude V3\omega(\mu V)</th>
<th>dB</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>12.6</td>
<td>-117dB</td>
</tr>
<tr>
<td>1000</td>
<td>14.4</td>
<td>-113dB</td>
</tr>
<tr>
<td>2000</td>
<td>19.3</td>
<td>-107dB</td>
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<tr>
<td>3000</td>
<td>24.5</td>
<td>-103dB</td>
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<tr>
<td>5000</td>
<td>39.3</td>
<td>-100dB</td>
</tr>
<tr>
<td>7000</td>
<td>61.4</td>
<td>-97dB</td>
</tr>
<tr>
<td>10000</td>
<td>86.0</td>
<td>-95dB</td>
</tr>
<tr>
<td>15000</td>
<td>120.0</td>
<td>-92dB</td>
</tr>
</tbody>
</table>

Table B.1: Distortion analysis of the Stanford DS360 with a NI PCI 4474 and a NI PCI 6259

The first measurement was done with an amplifier to obtain the same power between the Stanford output and the circuit resistors. The value obtained showed that the amplifier generates a lot of third harmonic which engendered bad results, consequently I left this one and I measured the power on the sample for a given voltage.

\(^1\)Fast Fourier Transform
\(^2\)root mean square

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Another important parameter is to control the accuracy for the extraction of the third harmonic amplitude. For this experiment I used the Stanford DS360 both, as generator of first and third harmonics. In that way we chose to use a Krohn Hite model 3103A filter which is a variable band pass filter with an adjustable low and a high cutoff frequency. Thanks to this instrument we can extract from a synchronous square wave 3 times the fundamental frequency. An attenuator is used to be able to choose different amplitudes for the $3\omega$ generated signal. Thus, defining different amplitude for the $3\omega$ generated signal we are able to see if the FFT software combined with a NI PCI 4474 extract with good accuracy the $3\omega$ signal. The different results obtained are mentioned below at 2 different frequencies:

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>3rd harmonic ref (mV$_{rms}$)</th>
<th>3rd harmonic measure (mV$_{rms}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15 000</td>
<td>112.5</td>
<td>106</td>
</tr>
<tr>
<td></td>
<td>11.69</td>
<td>11.69</td>
</tr>
<tr>
<td></td>
<td>1.171</td>
<td>1.171</td>
</tr>
<tr>
<td></td>
<td>0.122</td>
<td>0.122</td>
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<tr>
<td></td>
<td>0.070</td>
<td>0.070</td>
</tr>
<tr>
<td></td>
<td>0.063</td>
<td>0.063</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
<td>0.067</td>
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Table B.2: Accuracy for the amplitude extraction. In that case I used the Stanford DS360 under 1Vrms with a third harmonic synchronous generation. Different filters enable to realise this control.

The results mentioned in the Table B.2 show for 2 frequencies a good accuracy for small signals. Values extracted with our sample are in the order of 1000µV$_{rms}$, that is to say the variation observed for values superior to 12mV$_{rms}$ won’t change the results obtained for our experiment. The range of accuracy is sufficient and we can observed a precision about at least 1µV$_{rms}$. Consequently we will use the NI PCI4474 for all our experiments with the Stanford DS360.
Appendix C

Theoretical approach for the $3\omega$ method

The basic conduction heat transfer equation in three dimensions is given by equation [9]:

$$\frac{k}{\rho C_p} \nabla^2 T + \frac{Q}{\rho C_p} = \frac{\partial T}{\partial t} \quad (C.1)$$

Equation (C.1) as transformed into cylindrical coordinates gives equation (C.2):

$$\frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial T}{\partial r} \right) + \frac{1}{r^2} \left( \frac{\partial^2 T}{\partial \theta^2} \right) + \frac{\partial^2 T}{\partial z^2} = \frac{1}{\alpha} \frac{\partial T}{\partial t} \quad (C.2)$$

with $r, \theta, z$ the cylindrical coordinates and $\alpha$ the thermal diffusivity.

In the case where heat transfer is independent of $\theta$ and $z$ we obtain the following expression:

$$\frac{1}{r} \frac{\partial}{\partial r} \left( r \frac{\partial T}{\partial r} \right) = \frac{1}{\alpha} \frac{\partial T}{\partial t} \quad (C.3)$$

i.e.

$$\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} = \frac{1}{\alpha} \frac{\partial T}{\partial t} \quad (C.4)$$

Equation (C.4) is checked when heat flows through a cylinder (with initial and boundary conditions are independent of $\theta$ and $z$), and the axis of the cylinder coincides to the $z$ axis (see Figure C.1).

The heat flow can be defined by a solution of the form:

$$T(r, t) = u(r) \exp(i\omega t) \quad (C.5)$$

Substituting equation (C.5) into equation (C.4) we obtain the modified Bessel’s equation of order 0 [35, 36, 37]:

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Figure C.1: Boundary condition for a heat flows radially perpendicular to the z axis

\[ r^2 \frac{\partial^2 u}{\partial r^2} + r \frac{\partial u}{\partial r} - r^2 \frac{i \omega u}{\alpha} = 0 \]  

(C.6)

The general solution of equation C.6 is given by the equation C.7 consisting of the modified Bessel function of the first kind of order 0 (Equation C.8) and the modified Bessel function of the second kind of order 0 (Equation C.9).

\[ u(r) = A I_0 \left( \sqrt{\frac{i \omega \alpha}{r}} \right) + B K_0 \left( \sqrt{\frac{i \omega \alpha}{r}} \right) \]  

(C.7)

\[ I_0(x) = \sum_{m=0}^{\infty} \frac{x^{2m}}{2^{2m}(m!)^2} \]  

(C.8)

\[ K_0(x) = - \left( \ln \frac{x}{2} + \gamma \right) I_0(x) + \sum_{m=1}^{\infty} \frac{h_m x^{2m}}{2^{2m}(m!)^2} \]  

where \( h_m = \sum_{m=1}^{\infty} \frac{1}{m} \)  

(C.9)

and \( \gamma \) is Euler’s Constant (0.577215...)

Therefore the temperature distribution (Equation C.5) for an infinite cylinder whose axis corresponds with the z coordinate is given by the following expression:

\[ T(r, t) = \left[ A I_0 \left( \sqrt{\frac{i \omega \alpha}{r}} \right) + B K_0 \left( \sqrt{\frac{i \omega \alpha}{r}} \right) \right] \exp(i \omega t) \]  

(C.10)

Equation C.10 shows that the temperature distribution oscillates in time. If we consider only the amplitude term we could see that there is two unknown parameters (A_0 and B_0). Thanks
Figure C.2: Bessel function of the first kind and 0 order as a function of x

Figure C.3: Bessel function of the second kind and 0 order as a function of x

to a straight-forward calculation under Matlab we obtained that the first parameter diverges (which depends on \(I_0(x)\)) for large values of x while the second parameters (which depends on \(K_0(x)\)) converges for large values of x (Fig. C.2 and C.3).

Now, we consider a long infinitely thin conductor covering a semi infinite medium supposed to be non-conducting.

\[
\begin{align*}
l & \gg b \gg d_f \text{ and } \frac{1}{q} \ll d_S
\end{align*}
\]  
(C.11)

The conductor is defined as a heater and in that case we can apply equation C.10 and treat the heater as a boundary condition. As we saw, the temperature distribution diverges consequently for very large r in order to keep finite solution we set coefficient \(A_0\) equal to 0. This allows us to define expression C.12 which only depends on the constant \(B_0\).

\[
T(r, t) = \left[ BK_0 \left( \sqrt{\frac{\alpha}{\nu_0}} r \right) \right] exp(\omega t)
\]  
(C.12)

As we saw in section 2.2.1 the resulting temperature distribution due to the heat flux will
oscillate twice the frequency of the applied current. Therefore if we consider a current carrying conductor on top of a semi-infinite medium, the temperature rise would be given by equation:

$$\delta T(r, t) = BK_0 \left( \sqrt{\frac{i2\omega}{\alpha}} \right)^k e^{x2\omega} (C.13)$$

Thanks to the equation defined by Carslaw and Jaeger [19] and given by Cahill [14] we can evaluate the unknown constant $B$ as:

$$\delta T(r, t) = \left[ \frac{P}{\pi lk} K_0 \left( \sqrt{\frac{i2\omega}{\alpha}} \right)^k e^{x2\omega} \right] (C.14)$$

Where $P$, $l$ and $k$ are respectively, applied power, conductor length and thermal conductivity.

The modified Bessel function of the first kind of order 0 and the modified Bessel function of the second kind of order 0 may be approximated by equation [C.15] and [C.16].

$$K_0(x) = -\left( \ln \frac{x}{2} + \gamma \right) I_0(x) + \frac{1x^2}{4(1)^2} + \ldots (C.15)$$

$$I_0(x) = 1 + \frac{1x^2}{4(1)^2} + \ldots (C.16)$$

In the limit $|qr| \ll 1$, using equation [C.16] and [C.15] into equation [C.14] we get the expression for the temperature rise depending on the thermal properties of the thermal material, the frequency, distance and time:

$$\delta T(r, t) = \frac{P}{\pi lk} \left( \frac{1}{2} ln \frac{\alpha}{r^2} + ln(2) - \frac{1}{2} ln(2\omega) - \frac{i\pi}{4} - \gamma \right) e^{x2\omega} (C.17)$$
Appendix D

Thermal wave

The $3\omega$ method which is used to measure the thermal conductivity is essentially due to a thermal wave[19]. Then we can begin with solution for the one-dimensional heat equation for the special case when the temperature distribution is assumed to be oscillatory:

$$\alpha \frac{\partial^2 T}{\partial x^2} = \frac{\partial T}{\partial t}$$  \hspace{1cm} (D.1)

if at $x=0$ also an oscillatory temperature is prescribed, we have:

$$T(0, t) = A\cos(\omega t)$$  \hspace{1cm} (D.2)

which yields the solution:

$$T(x, t) = A\exp(-\zeta x)\cos(\omega t - \zeta x)$$  \hspace{1cm} (D.3)

where $\zeta = \sqrt{\frac{\omega}{2\alpha}}$

Equation [D.3] represents a temperature wave of wave number $\zeta$ and frequency $\omega$. These waves have several properties which are interesting for the $3\omega$ method. These are illustrated in Figure [D.1] if we assume copper as the heat conducting medium with $\alpha=1.17\text{cm}^2\cdot\text{s}^{-1}$ and plot just the steady state solution vs. $x$.

This figure shows that the effect of the exponential term in equation [D.3] would be to attenuate the temperature drastically as a function of both the frequency and distance. This has significant implications in that if there are any higher harmonics present, they will be damped out much more rapidly than the fundamental frequency. Therefore, the damping term plays a fundamental role in determining the effective thickness that the thermal waves may propagate into a given material. Now we turn our attention to the oscillatory term in equation [D.3] which enabled us to obtain the Figure [D.2]
Figure D.1: Effect of the frequency on the damping term for a copper sample

Figure D.2: Effect of the frequency on the temperature oscillation for a copper sample

Figure D.2 shows that there is a phase shift when there is an increasing frequency, which has significant implications in the measurement of thermal properties. At present we consider the temperature distribution within the medium as a function of position and time separately. We can plot the temperature fluctuations within the medium at a specific position as a function of the dimensionless parameter $\omega t$ and the temperature distribution at a particular frequency as the time changes. In these cases we used a normalized temperature.
Figure D.3: Normalized temperature for a copper sample at the position $x=70\mu m$

Figure D.4: Normalized temperature versus depth for $\omega=1000s^{-1}$, at different values of the dimensionless parameter $\omega t$

If we consider a periodic flux of heat $\Phi$ at the surface:

$$\Phi = \Phi_0 \cos(\omega t) \quad (D.4)$$

and the Fourier’s first law of heat conduction,

$$\Phi = k \frac{\partial T}{\partial x} \quad (D.5)$$
the temperature distribution in the medium which is heated at its face $x=0$ by a periodic flux (Equation D.4) is given by:

$$T(x, t) = \frac{\Phi_0}{\zeta k \sqrt{2}} e^{\exp(-\zeta x)} \cos(\omega t - \zeta x - \frac{\pi}{4})$$  \hspace{1cm} (D.6)

The expression D.6 enabled us to determine for multilayer, what is the thermal wave repartition in the whole structure. I used Matlab© to define this repartition with continuous boundary condition. The following example, Figure D.5 shows a multilayer SiO$_2$-Si with typical dimension 0.5/2000µm.

Figure D.5: Normalized temperature versus distance from the sample surface for $\omega=1000\text{s}^{-1}$ in two different cases, with and without periodic heat flux
In Figures D.5-D.6 we see that a periodic heat flux decreases the penetration depth in the case of a film with low thermal conductivity. One interesting point comes from the fact that the maximum temperature is observed inside the film, this shows that we really test the film. However we must take the substrate into consideration because its internal temperature variation is not negligible. These observations agreed well with the theory shown in Chapter 2.
Appendix E

Labview program

Figure E.1: Flow chart used under Labview to extract the $3\omega$ amplitude
Appendix F

Temperature calibration

A lot of different parameters must be checked to enhance our measurement accuracy. In this appendix we will see the correction that we must apply to the temperature measured with a Keithley 2700 connected with a thermocouple type K. To that end we used a calibration set ISOTECH Isocal-6 Venus 2150B and an high precision thermometer LABFACILITY Labcal plus to calibrate our instrument. The correction that we must apply is plotted in Figure F.1.

Figure F.1: Calibration of the Keithley 2700 connected to a type K thermocouple
Appendix G

Manual

The goal of this appendix is to give the main parameters necessary to launch the experimental set-up realized for the \(3\omega\) method.

1. Contact Eugene Timmering to process your sample with the mask "3omega".

2. To separate individual samples, contact Cor Rijpert who dices it. Ruud Smulders can glue the samples on a DIL Lead side brazed package with an epoxy glue and the contact pads must be realized by wire bonding.

3. First, to calibrate your samples, you must connect them in a specific box (Figure 3.9) inside the climate cabinet (room WZk.1.16). Each sample must be connected to a Keithley 2000 with 4 wires. The thermocouple connected to a Keithley 2700 must be positioned near to the samples. Now you can switch on all the Keithley instruments, the climate cabinet Heraeus EK220 and the computer. Launch the software realized under Labview TempCoeff.vi (shortcut on the desk). You can define all the parameters necessary for your experiment (§3.3.1).

4. Next you can begin to measure the thermal conductivity of your sample(s). Use only one sample and deposit it inside the climate cabinet Heraeus EK220 on the specific support inside the copper box (Figure 3.6).

5. Choose your resistor (R=22\(\Omega\) or 3.8\(\Omega\)) and connect it like the scheme defined in Figure 3.7. Now you can switch on the ultra low distortion function generator model Stanford Research Systems DS360 and the computer.

6. Launch the software realized under Labview named Spectrum3w_QMH.vi (Shortcut on the desk) and start it, then choose all the different parameters that you want on the first page named "Settings". To begin your experiment click on the button "Meas". All the information obtained during your experiment will be mentioned in the different pages (Figure 3.12 to 3.15). The results which will enable to obtain the thermal conductivity will be in the last page named "Amplitude".

7. The results are saved in the folder C:\results in a textfile format, this directory may be changed as well. You can exploit your results with Excel or Matlab© using the different equations defined in Chapter 2. You can use both, the isotropic and anisotropic case dependent of the material that you will use. The Excel file named Spectrum3w_results.xls could help you to exploit your results. To fit your results with Matlab you must use the
folder Fitting.zip realized by Dr F. Baillet and launched the files F2_LSQ.m, F2_NM.m and F2_UNC.m. These files using different Matlab function enables to find out what is the better fitting. Using % you can define in each Script the method that you want with the initial parameters. Before that you must create a file .txt which contain 3 columns having the frequency, the amplitude variation of the first sample and the second sample. Think to change the value of b (heater 1/2 width) and P (power applied on your sample).
Appendix H

Symbols

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Related to</th>
</tr>
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<td>F</td>
<td>Film</td>
</tr>
<tr>
<td>S</td>
<td>Substrate</td>
</tr>
<tr>
<td>H</td>
<td>Heater/Sensor</td>
</tr>
<tr>
<td>( \omega )</td>
<td>First harmonic</td>
</tr>
<tr>
<td>( 3\omega )</td>
<td>Third harmonic</td>
</tr>
<tr>
<td>x</td>
<td>In plane direction</td>
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<tr>
<td>y</td>
<td>Cross plane direction</td>
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<td>R</td>
<td>Radiation (photon)</td>
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<tr>
<td>L</td>
<td>Lattice (phonon)</td>
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<td>e</td>
<td>Electronics</td>
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Table H.1: Symbols - Subscripts

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<tr>
<td>( \eta )</td>
<td>Constant enabling to adjust experimental data</td>
</tr>
<tr>
<td>( \gamma )</td>
<td>Euler's Constant</td>
</tr>
<tr>
<td>L</td>
<td>Lorenz number</td>
</tr>
<tr>
<td>( \sigma )</td>
<td>Standard deviation</td>
</tr>
<tr>
<td>( \sigma_0 )</td>
<td>Stefan Boltzmann constant</td>
</tr>
<tr>
<td>q</td>
<td>Charge of the carrier</td>
</tr>
<tr>
<td>R</td>
<td>Gas constant</td>
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<tr>
<td>n</td>
<td>Refractive index</td>
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Table H.2: Symbols - Dimensionless parameters and constant

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<tr>
<td>b</td>
<td>[m]</td>
<td>Heater/Sensor width</td>
</tr>
<tr>
<td>l</td>
<td>[m]</td>
<td>Heater/Sensor length</td>
</tr>
<tr>
<td>d</td>
<td>[m]</td>
<td>Thickness</td>
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Table H.3: Symbols - Sample parameters
### Symbols - Thermal properties

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<th>Definition</th>
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<td>$k$</td>
<td>[W.m(^{-1}).K(^{-1})]</td>
<td>Thermal conductivity</td>
</tr>
<tr>
<td>$C_p$</td>
<td>[W.s.kg(^{-1}).K(^{-1})]</td>
<td>Specific heat capacity</td>
</tr>
<tr>
<td>$\rho$</td>
<td>[kg.m(^{-3})]</td>
<td>Density</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>[m(^2).s(^{-1})]</td>
<td>Thermal diffusivity</td>
</tr>
<tr>
<td>$T$</td>
<td>[K]</td>
<td>Temperature</td>
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<tr>
<td>$\alpha_R$</td>
<td>[K(^{-1})]</td>
<td>Temperature coefficient of resistivity</td>
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<tr>
<td>$\Delta T$</td>
<td>[K]</td>
<td>Temperature rise</td>
</tr>
<tr>
<td>$1/\bar{q}$</td>
<td>[m]</td>
<td>Thermal penetration depth</td>
</tr>
<tr>
<td>$\bar{\alpha}$</td>
<td>[m(^{-1})]</td>
<td>Absorption coefficient</td>
</tr>
<tr>
<td>$\bar{\gamma}$</td>
<td>[m]</td>
<td>Penetration depth</td>
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<tr>
<td>$\mu$</td>
<td>[cm(^2).V(^{-1}).s(^{-1})]</td>
<td>Carrier mobility</td>
</tr>
<tr>
<td>p</td>
<td>[cm(^{-3})]</td>
<td>Carrier concentration</td>
</tr>
<tr>
<td>$v$</td>
<td>[cm.s(^{-1})]</td>
<td>Molecular velocity</td>
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Table H.4: Symbols - Thermal properties

### Symbols - Electrical properties

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<td>[A]</td>
<td>Intensity</td>
</tr>
<tr>
<td>$R$</td>
<td>[\Omega]</td>
<td>Resistance</td>
</tr>
<tr>
<td>$P$</td>
<td>[W]</td>
<td>RMS power</td>
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<tr>
<td>$f$</td>
<td>[Hz]</td>
<td>Frequency</td>
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<td>Angular frequency</td>
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<tr>
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<td>[\Omega]</td>
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<tr>
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<td>[V]</td>
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</tr>
<tr>
<td>$b_{3\omega}$</td>
<td>[V]</td>
<td>Ordinate at the origin of the linear function $f_{3\omega}(\ln(\omega))=V_{3\omega}$</td>
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<tr>
<td>$\phi$</td>
<td>[W]</td>
<td>Flux of heat</td>
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Table H.5: Symbols - Electrical properties