Thermal conductivity measurements of nanometer-thick SiN membranes

THESIS

submitted in partial fulfillment of the
requirements for the degree of

BACHELOR OF SCIENCE
in
PHYSICS

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Leiden, The Netherlands, July 3, 2018

Thermal conductivity measurements of nanometer-thick SiN membranes

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Abstract

In this bachelor research project, the thermal conductivity of very thin (50 nm) suspended Silicon Nitride (SiN) membranes is studied. These membranes are used as mechanical resonators in optomechanical experiments where quantum decoherence properties are studied. When these optomechanical experiments are performed at low (mK) temperatures, heating of the membrane by laser absorption perturbs these measurements. Therefore, the thermal properties of these membranes are studied in this project at room temperature with a method which is in principle also applicable for cryogenic measurements. In this project different methods are studied and the 3ω method is expected to be the best suitable method and is therefore investigated in this project. Using this method, measurements have been done and a signal containing thermal properties of the membrane is obtained. However, due to the sample preparation, no thermal conductivty could be extracted from this signal.

Contents

Chapter 1

Introduction

In this bachelor research project the thermal properties of 50 nm thick SiN membranes are studied, with focus on thermal conductivity. These membranes are used in quantum optomechanics experiments. Quantum optomechanics focuses on the interaction between light and mechanical objects at low-energy scales. In experiments, of which a schematic is shown in figure 1.1, lasers are used to make interaction between light and matter possible using optical resonators, which are called cavities. Within such a cavity, a mechanical resonator can be placed, which will oscillate due to either thermal excitation or radiation pressure of the laser.

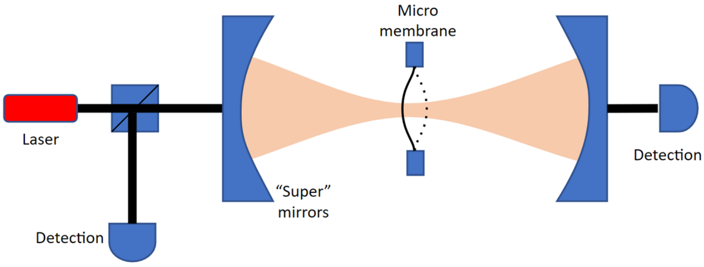

Figure 1.1: *Experimental setup for optomechanical measurements, where the optical cavity consists of the two mirrors and the micro membrane functions as mechanical resonator*

This oscillator can then show quantum mechanical behaviour: it can be in a superposition of being in two places at the same time. This kind of quantum state of motion allows us to investigate decoherence, which describes the transition between states that are described by quantum mechanics to states that have a classical description. Measuring these optomechanical oscillations provide us with a way to measure predictions of quantum mechanics and decoherence models and might give answers to fundamental questions in physics.

In the cavity optomechanics experiments at Leiden, silicon nitride (SiN) membranes are used as mechanical resonators. These membranes have a very high quality factor, which results in excellent coherence properties, which allows us to do precise measurements on the decoherence properties. Because we want to measure at low- energy scales, in order to measure the quantum ground state of the oscillations, measurements need to be done at very low (mK) temperatures. Since the membrane absorbs a small fraction of the laser light, the membrane heats up. By heating up the membrane, we add energy to the system and we partially lose the low energy scale behaviour, which makes it more difficult to study the decoherence properties. It is thus of big importance to gain knowledge about the thermal properties of these membranes at room temperature and, especially, at mK temperatures.

Another motivation for investigating the thermal properties of SiN membranes, apart from the optomechanics experiments, is the fact that these membranes are very thin (25-100 nm) and that the SiN membranes are not a crystalline solid, but an amorphous solid. Both these properties of the SiN membranes makes them interesting to investigate, because (1) in nanoscale materials, heat transfer differs from heat transfer in bulk materials, especially at low temperatures. And (2) because in amorphous solids, heat transfer is different than in crystalline solids. Therefore, a brief description of heat transfer in amorphous nanoscale materials can be found in chapter 2.

In this thesis, three different methods for measuring the thermal conductivity are studied: a steady state method using a heat camera, a steady state method using a line heater deposited on the membrane and a transient method (the 3ω method). However, the method involving a heat camera is not applicable to mK temperature measurements, due to the different wavelength of the black-body radiation at mK temperatures, which is not detectable by an infrared (IR) heat camera. For the steady state method involving a line heater deposited on the membrane, numerical calculations have been performed using COMSOL Multiphysics software. Studying these simulations we found that the steady state method might not be the best suitable method for thermal conductivity measurements, so only the 3ω method is actually performed in this thesis. Further in this thesis one can find the needed sample preparation, the experimental setup for 3ω measurements, the obtained results, a discussion of these results and conclusions from this research project, with a brief proposal for future thermal conductivity measurements on SiN membranes.

Chapter 2

Heat transfer in nanoscale amorphous materials

In this section, a brief description of heat transfer in nanoscale amorphous materials will be provided. Descriptions of heat transfer are different for the two types of measurement methods (steady state and transient) investigated in this thesis. For steady state methods, the temperature profile does not change in time and therefore heat transfer can be described by Fourier's law:

$$\mathbf{q} = -k\nabla T(x, y, z) \tag{2.1}$$

With $\mathbf{q}$ the heat flux in Watt per unit area, k the thermal conductivity of the material to be studied and $T(x, y, z)$ the steady temperature profile in three dimensions. For transient methods, the temperature profile does change in time, and therefore the 1D heat diffusion equation is used to describe heat transfer:

$$\frac{\partial^2 T(x,t)}{\partial x^2} = \frac{1}{D}\frac{\partial T(x,t)}{\partial t} \tag{2.2}$$

Where $T(x, t)$ is the temperature field and $D = \frac{k}{\rho c}$ the thermal diffusivity of the material to be studied, with k the thermal conductivity, ρ the mass density and c the specific heat.

In non-metallic materials, heat is transferred by vibrations of atoms. These vibrations can then be transmitted as waves through the material. These vibrations carry energy and are responsible for the thermal transport in the material. The quantization of energy of these lattice vibrations is called a phonon. The thermal conductivity of the material is directly related to the phonon mean free path length ($k \sim \Lambda$) [1], with Λ the phonon mean free path length. The phonon mean free path length is restricted (and so the thermal conductivity) by boundary scattering of phonons and by scattering of phonons due to defects in the lattice of the material. The fact that the SiN membranes in this study are amorphous and have nanoscale dimensions, cause the thermal conductivity of the membrane to be different from bulk crystalline materials. First, because the SiN membranes are amorphous, which causes the lattice of the material lack the periodicity that is present in crystalline materials. This lack in periodicity causes the phonon mean free path to decrease, because a lack in periodicity can be seen as a lattice defect, which decreases the thermal conductivity.

Second, because the SiN membranes have nanoscale dimensions, the phonon mean free path can be of the same order as the thickness of the membrane, resulting in a relatively high percentage of phonons scattering at the surface of the membrane, which then results in a decrease of thermal conductivity. However, at room temperature, the phonon mean free path in SiN membranes is estimated to be less than 1 nm, which should not result in a big difference in thermal conductivity between membranes with a different thickness. Nevertheless, differences in thermal conductivity between 50 nm and 100 nm SiN membranes are observed at room temperature [2]. The phonon mean free path for low temperatures, however, is expected to be much longer than for room temperatures [3], and can exceed the thickness of the membrane, leading to a heavy decrease of thermal conductivity when the temperature is decreased.

Chapter 3

Methods

Measuring the thermal conductivity of membranes and thin films can be done by a variety of methods. The most commonly used methods can be divided in two different categories:

- Steady state methods;
- Transient methods

Both these methods will be described in this section and for steady state methods, supported with simulations using COMSOL Multiphysics software. Also, for the steady state methods, it will be explained why these methods are not the best suitable methods for measuring the thermal conductivity of the SiN membranes, and thus, why we have chosen to perform a transient (3ω) method.

3.1 Steady state methods

In steady state methods, a temperature gradient is created across the material to be studied. This temperature gradient does not change in time and therefore can be stated for every point in the membrane that:

$$q_{in} = q_{out} \tag{3.1}$$

Where q_{in} and q_{out} are the inward and respectively outward heat flux. By evaluating q and by measuring the temperature profile $T(x, y, z)$, equation 2.1 can be used to extract the thermal conductivity of the material. For steady state methods, two different measurement techniques are explored:

- Using infrared thermography (no contact with membrane);
- Using line heater/ temperature sensor on membrane (contact with membrane)

3.1.1 Steady state infrared thermography

An example of a non-contact steady state method is provided by Greppmair et al. (2017) [4]. Here an infrared (IR) camera is used to measure the steady temperature profile across a uniformly heated thin film. This thin film is uniformly heated by a LED, and the substrate of the thin film functions as a heat sink with a controllable temperature. Heat flows through the membrane to the heat sink, creating a transverse temperature profile, of which the black-body radiation in the IR spectrum is detected by an infrared camera. A schematic of this method is shown in figure 3.1. By measuring this temperature profile and by evaluating how much energy of the LED light is absorbed by the membrane, Fourier's law in polar coordinates can be applied:

$$q(r) = -k\frac{\partial T(r)}{\partial r} \tag{3.2}$$

With $q(r)$, the heat flow at a distance r from the middle of the membrane, k the thermal conductivity and $T(r)$ the temperature profile of the membrane. Using this equation, the thermal conductivity of the film or membrane can be calculated.

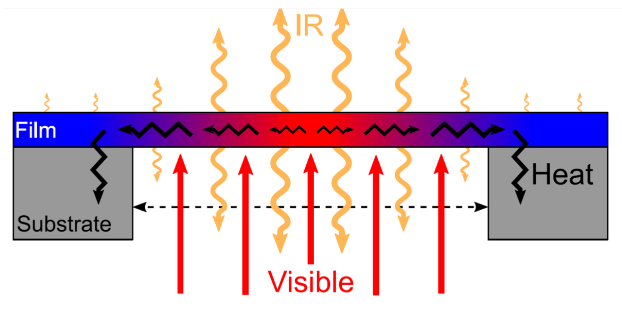

Figure 3.1: *Schetmatic of the steady state method using a heat camera. [4]*

A big problem arising from this method is that the total energy flow per unit time through the membrane is needed to calculate the thermal conductivity. To calculate this energy flow, a measurement of the absorption of the LED light by the membrane needs to be performed. This however, is relatively complicated and was not estimated as reachable within the scope of this bachelor project. Another general problem involving measurements using IR cameras arises from the fact that we ultimately want to measure at mK temperatures. The wavelength of the black-body radiation of the membrane increases at these low temperatures substantially, leading to the fact that this radiation cannot be detected by an IR camera anymore. This means that this method is not applicable to mK temperature measurements. About this method we can conclude that it is not compatible with the goal and possibilities of this research.

3.1.2 Steady state methods using a line heater/temperature sensor

One of the most straightforward methods involving a steady state method with direct contact is described by Zhao et al (2016) [5] (page 25-27). The experimental scheme is shown in figure 3.2:

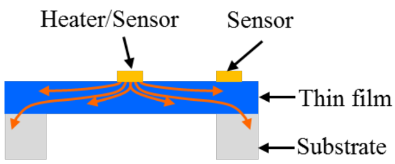

Figure 3.2: *Steady-state thermal conductivity measurement with a heater/sensor and an additional temperature sensor (Zhao et al 2016)[5]*

Here a line heater, which also functions at the same time as a thermometer, is placed at the middle of the membrane across the whole membrane, heating up the membrane with a total heating power per unit length Q, resulting in a steady state temperature profile across the membrane. Another thermometer is placed at distance $\frac{1}{2}L$ from this heater. By this measured temperature difference, $T_{heater} - T_{substrate}$, and the known total heating power per unit length, the thermal conductivity can be determined by applying Fourier's law in one dimension to this specific measurement setup [5]:

$$k = \frac{QL}{2d(T_{heater} - T_{substrate})} \tag{3.3}$$

With d the thickness of the membrane/thin film and $\frac{L}{2}$ the distance between the temperature sensors. Heat is created by Joule heating of the line heater by applying a current on the line heater. The temperature can be measured using the fact that the resistance of the metal heater/sensor is temperature depended, so by measuring the resistance of the metal line, the temperature can be determined. Although this method is theoretically relatively simple, there are a lot of difficulties to account for when performing this method:

- The temperature profile on the membrane is 2D instead of 1D;
- Not all the heat flows through the membrane: heat is also transferred by radiation, convection by the surrounding air and by conduction of the line heater to the contact pads and wires.

- Because heat is transferred along the line heater, there is a temperature gradient along the line heater, leading to errors in measured temperature.

These problems have been studied using simulations with COMSOL Multiphysics software. In COMSOL, the geometry of the membrane is reproduced and a line heater of aluminum (width= 150 μm) is placed at the middle of the membrane. The geometry looks as follows:

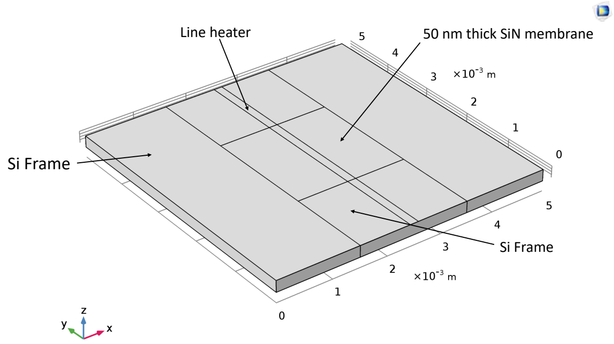

Figure 3.3: *Top view of the geometry in COMSOL*

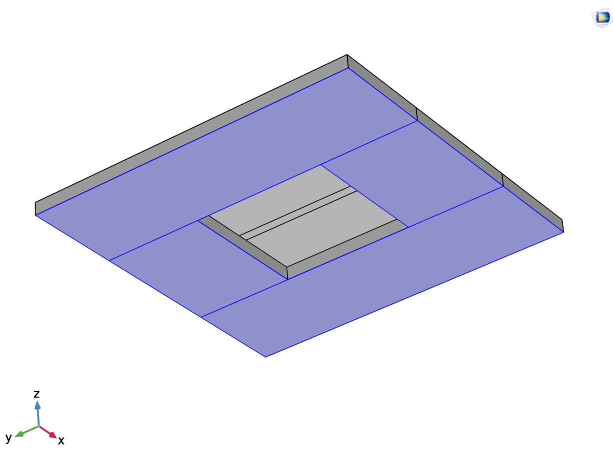

Figure 3.4: *View from below of the geometry in COMSOL*

For heat transfer simulations, the thermal conductivity of the SiN membrane is set to 5 W/(m K), and a heat flux of 4 $\times$ 10^5 W/m^2 is applied to the line heater, normal to the plane of the SiN membrane. The ambient temperature is set to 293.15 K and the parts in blue in figure 3.4 function as heat sink with a fixed temperature, also set to 293.15 K. These settings give the following temperature profile across the membrane:

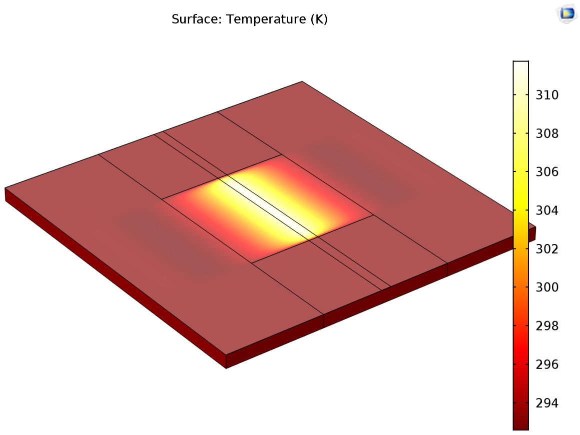

Figure 3.5: *Steady state temperature profile across the SiN membrane (COMSOL)*

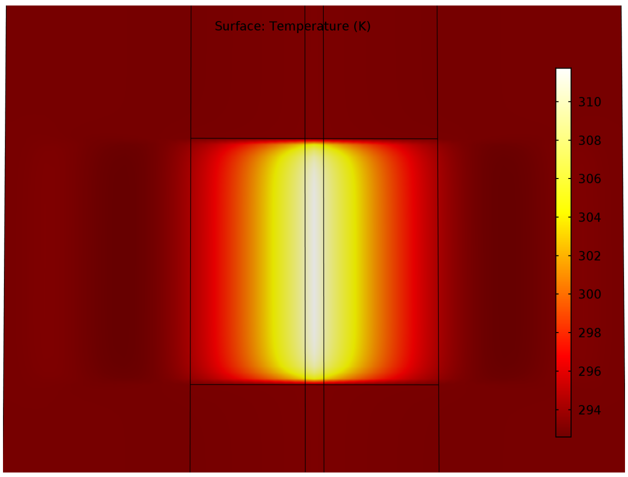

Figure 3.6: *Zoom of temperature profile across the SiN membrane (COMSOL)*

From figure 3.6 can be concluded that the temperature profile across the membrane is not 1D, but 2D. This is because near the frame, heat flows from the membrane directly in to the frame. To account for this, while still using equation 3.3, rough estimations have to be made, leading to substantial errors in the calculation of the thermal conductivity.

Another fact that can be observed from figure 3.6, is that there is a temperature gradient along the line heater. This is better shown in the following picture:

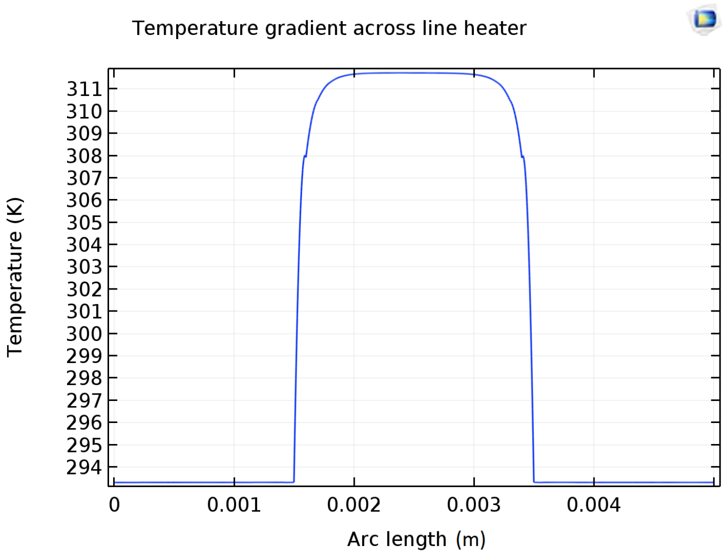

Figure 3.7: *Temperature gradient along the line heater (COMSOL)*

In this figure, it is shown that the temperature of the line heater on the frame (from 0 to 1.5 mm and 3.5 to 5.0 mm) is the same as the temperature of the heat sink, due to the high thermal conductivity of the crystalline Si. From 1.5 to 3.5 mm there is a strong increase in temperature over 0.5 mm, then is the temperature nearly constant over 1 mm, followed by a strong decrease of the temperature. When measuring the resistance, and thus the temperature of the heater, the average of the temperature over the line heater will be measured. This leads to an error in measuring the temperature difference, which then leads to an error in the thermal conductivity.

The biggest problem, however, in steady state methods is the energy loss by radiation [6]. The impact of energy losses by radiation can roughly be quantified by the ratio of the amount of heat loss by radiation and the amount of heat transport by conduction of the membrane: $\frac{Q_{rad}}{Q_{cond}}$. An estimation of this ratio is given by [6]:

$$\frac{Q_{rad}}{Q_{cond}} \approx \frac{h_{rad} w^2}{kd} \tag{3.4}$$

With w the half length of the membrane, d the thickness of the membrane, k the thermal conductivity of the membrane and h_{rad} the heat transfer coefficient by radiation in W/(m^2K), which is equal to [6]:

$$h_{rad} = 4\epsilon\sigma T_{avg}^3 \tag{3.5}$$

With ϵ the emissivity of the membrane, σ the Stefan-Boltzmann radiation constant and T_{avg} the average temperature of the membrane and its surroundings. Because the membranes used in this thesis are very thin (50 nm) and the thermal conductivity is predicted to be roughly around 3-15 W/(m K), the ratio $\frac{Q_{rad}}{Qcond}$ is expected to be relatively high. In addition to that, the emissivity, ϵ, is unknown for the membrane and needs additional measurements. This uncertainty leads directly to an uncertainty in h_{rad} and consequently to an uncertainty in the ratio $\frac{Q_{rad}}{Qcond}$. Together with the fact that this ratio is very high leads this to substantial errors by radiation in thermal conductivity measurements.

So by errors due to the 2D in stead of 1D temperature profile, the temperature gradient along the line heater/sensor and the substantial radiation losses, this steady state method is expected not to be suitable for thermal conductivity measurements. Therefore, we have chosen to perform a transient (3ω) method, of which the errors due to radiation are estimated as relatively small (around 1%) [5]

3.2 The 3ω method

In the 3ω method, a metal line heater is deposited on the membrane, which also functions at the same time as thermometer, just as in the steady state method described in section 3.1.2. Here an AC current is applied to the line heater:

$$I_h(t) = I_0 \cos(\omega t) \tag{3.6}$$

Here, I_0 is the amplitude of the current at frequency ω. Due to Joule heating, heat is dissipated by the line heater:

$$P_h(t) = I_h(t)^2 R_{s,0} \tag{3.7}$$

Where $R_{s,0}$ is the resistance of the line heater. Using trigonometric identities, the dissipated power can be written as follows:

$$P_h(t) = \frac{1}{2} I_0^2 R_{s,0}(1 + \cos(2\omega t)) \tag{3.8}$$

From equation 3.8, the power dissipated by the line heater, due to Joule heating can be separated in two parts:

$$P_{DC} = \frac{1}{2} I_0^2 R_{s,0} \tag{3.9}$$

$$P_{AC}(t) = \frac{1}{2} I_0^2 R_{s,0} \cos(2\omega t) \tag{3.10}$$

Here, P_{DC} is a constant component and P_{AC} corresponds to the power that is depended on the second harmonic oscillation of 2ω. The dissipated power by the line heater causes the temperature of the line heater and the substrate to change. The temperature changes of the substrate can be expressed as follows [7]:

$$\Delta T(t) = \Delta T_{DC} + |\Delta T_{2\omega}| \cos(2\omega t + \phi_{2\omega}) \tag{3.11}$$

With ΔT_{DC} the temperature increase corresponding to P_{DC}, $|\Delta T_{2\omega}|$ the absolute value of the temperature change corresponding to $P_{AC}(t)$ and $\phi_{2\omega}$ the phase due to the lag between temperature changes and heat flux. Temperature changes induce a change in resistance in the following way:

$$R_s(t) = R_{s,0}(1 + \alpha \Delta T(t)) \tag{3.12}$$

Where α is the temperature coefficient of resistance (TCR), which is defined as follows:

$$\alpha = \frac{dR}{RdT} \tag{3.13}$$

When equation 3.12 and 3.11 are combined, the following formula for the resistance of the line heater is obtained:

$$R_s(t) = R_{s,0}(1 + \alpha \Delta T_{DC} + \alpha |\Delta T_{2\omega}| \cos(2\omega t + \phi_{2\omega})) \tag{3.14}$$

When applying Ohm's law, the voltage across the heater results from the product of the input current (equation 3.6) and the resistance of the heater (equation 3.14) [7] :

$$\begin{aligned} V(t) = R_{s,0} I_0 (1 + \alpha \Delta T_{DC}) \cos(\omega t) + \frac{1}{2} \alpha |\Delta T_{2\omega}| R_{s,0} I_0 \cos(\omega t + \phi_{2\omega}) \\ + \frac{1}{2} \alpha |\Delta T_{2\omega}| R_{s,0} I_0 \cos(3\omega t + \phi_{2\omega}) \end{aligned} \tag{3.15}$$

Because the signal containing the thermal properties of the membrane, $\Delta T_{2\omega}$, at 1ω is small compared to the signal at 1ω due to DC heating and the input voltage, the voltage at 3ω needs to be measured:

$$V_{3\omega}(t) = \frac{1}{2} \alpha |\Delta T_{2\omega}| R_{s,0} I_0 \cos(3\omega t + \phi_{2\omega}) \tag{3.16}$$

The absolute value of the 3ω signal can then be written as follows:

$$|V_{3\omega}| = \frac{1}{2} \alpha |\Delta T_{2\omega}| R_{s,0} I_0 \tag{3.17}$$

The measurement of $V_{3\omega}$ appearing across the membrane is most commonly done with a lock-in amplifier. Measuring this voltage directly on the membrane is very difficult, because the voltage at 1ω is expected to be a factor 10^3 higher than the $V_{3\omega}$ signal. In order to suppress the voltage at 1ω, and be able to measure $V_{3\omega}$ with high precision, a Wheatstone bridge is needed. With a Wheatstone bridge the first harmonic is heavily suppressed, while the third harmonic can be measured. A schematic of a Wheatstone bridge can be found in figure 3.8. In our experimental setup, a slightly different Wheatstone bridge is used, which is shown in figure 5.3. The Wheatstone bridge output, $W_{3\omega}$, and $V_{3\omega}$ are related in the following way [9]:

$$V_{3\omega} = \frac{R_s + R_1}{R_1} W_{3\omega} \tag{3.18}$$

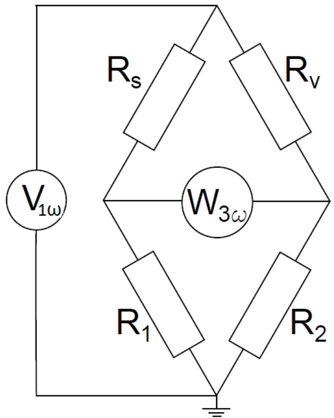

Figure 3.8: *Schematic of a Wheatstone bridge from thesis by Hanninen (2013) [8] Here, R_s corresponds to the line heater, R_1 and R_2 are other resistors and R_v is a variable resistor to balance the bridge. $W_{3\omega}$ is the Wheatstone bridge output and $V_{1\omega}$ is the Wheatstone bridge input.*

In order to extract thermal properties of the membrane from equation 3.17, the expected temperature oscillations, $\Delta T_{2\omega}$, need to be calculated. These oscillations are solutions from the 1D heat diffusion equation (equation 2.2). Solutions of this equation are provided by Sikora et al (2013) [10] and the erratum of this paper [11]. These solutions of the temperature field, leading to an expression for $\Delta T_{2\omega}$, together with equation 3.17 result in the following output of the Wheatstone bridge [11]:

$$|W_{3\omega}^{rms}| = \frac{\alpha (V_{1\omega}^{rms})^3 R_1 R_{s,0}^2}{4K(R_{s,0}+R_1)^4 \left[1+\omega^2\left(4\tau^2+\frac{2l^4}{3D^2}+\frac{4\tau l^2}{3D}\right)\right]^{\frac{1}{2}}} \tag{3.19}$$

With $|W_{3\omega}^{rms}|$ the absolute root mean square (rms) output of the Wheatstone bridge, $V_{1\omega}^{rms}$ the rms voltage at 1ω applied to the Wheatstone bridge ($V_{1\omega}$ in figure 3.8), R_1 the in-series resistor of the Wheatstone bridge (also see figure 3.8), $K = k\frac{S}{l}$ the thermal conductance of the membrane, with S the section of the membrane perpendicular to the heat flow, l the half-width of the membrane and τ the sum of the heat capacity of the membrane and the heat capacity of the line heater, divided by the thermal conductance of the membrane K.

For low frequencies, the ω term in equation 3.19 can be neglected and the following ω independed formula for $|W_{3\omega}^{rms}|$ is obtained [11]:

$$|W_{3\omega}^{rms}| = \frac{\alpha (V_{1\omega}^{rms})^3 R_1 R_{s,0}^2}{4K(R_{s,0} + R_1)^4} \tag{3.20}$$

This equation predicts a constant $|W_{3\omega}^{rms}|$ for low ω and will be used to measure the thermal conductivity of the membrane. To use this equation, $|W_{3\omega}^{rms}|$ will be measured for a range of frequencies, and plotted as a function of ω to check whether its behaviour is similar as predicted by equation 3.19.

In addition to the 3ω measurements using a lock-in and Wheatstone bridge, a TCR measurement of the line heater (α in equation 3.20) is needed in order to calculate the thermal conductivity.

Chapter 4

Sample preparation

Before going in detail about the sample preparation of the SiN membranes for thermal measurements, some technical information of these membranes will be provided here. The suspended membranes used in the experiments are very thin: 50 nm. The membrane samples consist of a SiN membrane placed on a relatively thick (500 μm) Si frame. These frames have the size 5.0×5.0 mm^2 while the membranes are 1.0×1.0 mm^2 in size. Schematics including a top view and a cross section are given in figures 4.1 and 4.2.

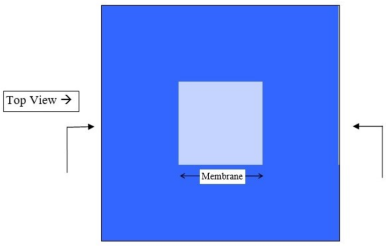

Figure 4.1: *Top view of the suspended membrane (Norcada 2015) [12]*

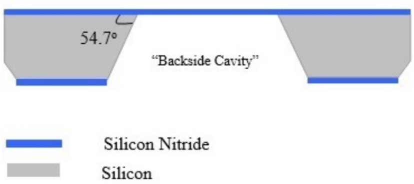

Figure 4.2: *Cross section of the membrane (Norcada 2015) [12]*

For thermal conductivity measurements, a metal line is needed to be deposited on the membrane, which functions as line heater and thermometer. In this research this is achieved by sputtering with an Ag target and the use of a 50 μm optical slit as a shadow mask. The membrane is then placed beneath the optical slit in the following way:

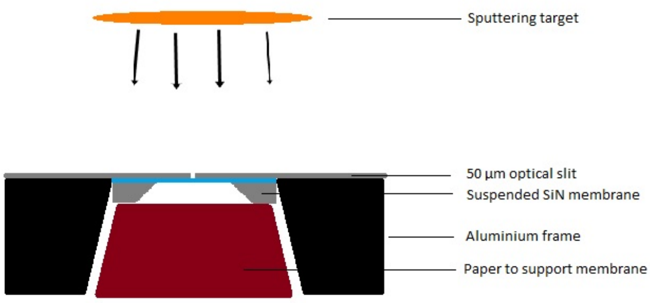

Figure 4.3: *Setup for sputtering an Ag line heater on the membrane*

Sputtering was done using the Leybold LH Z400 rf sputtering system, with a deposition rate for Ag of 19 nm/s. Sputtering has been done for 12 minutes. After sputtering, contacts with the line heater and 4 wires are made using silver paint, which is shown in figure 4.4. Microsocopy photos of the line heater are shown in figures 4.5 and 4.6

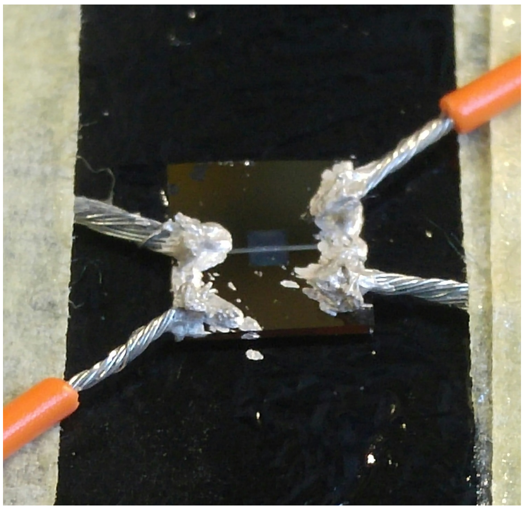

Figure 4.4: *Sample with four wires attached to the line heater*

From figures 4.5 and 4.6 the following properties of the line heater can be extracted: The width of the line heater is around 140 μm ($\pm$ 10 μm) and the length of the line heater across membrane and frame is 225 μm ($\pm$ 20 μm).

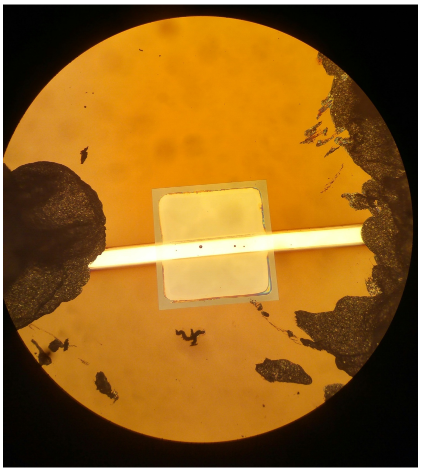

Figure 4.5: *Ag line heater on membrane microscopy image*

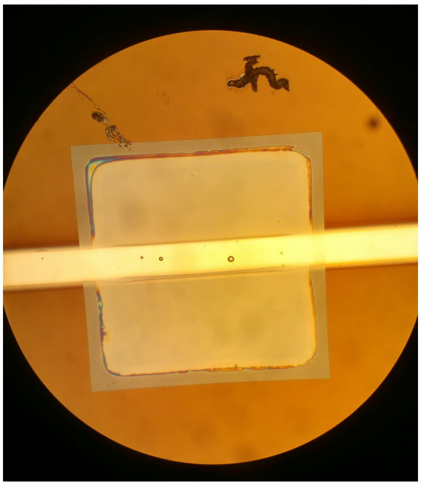

Figure 4.6: *Ag line heater on membrane zoomed microscopy image*

The resistance of the line heater and the contact between the wires and silver paint is measured using a 4-point measurement using the Keithley 2100 digital multimeter, obtaining a resistance of 13.2 Ω. Because the resistance of the contacts is estimated around 0.5 Ω, the resistance of the line heater itself is estimated at 12.2 Ω ($\pm$ 1 Ω). The resistance of the heater on the actual 1.0 $\times$ 1.0 mm^2 membrane is then estimated at 5.4 Ω ($\pm$ 0.8 Ω). This error in resistance is relatively large, due to the very low resistance of the line heater itself.

Chapter 5

Experimental setup

5.1 Setup for TCR measurement

In order to measure the temperature coefficient of resistance (TCR), the membrane with line heater is heated by a heating plate. A pt-100 is placed near the membrane, which is assumed to have nearly the same temperature as the line heater on the membrane. Photos of this setup are shown in figures 5.1 and 5.2. When heating the membrane using the heating plate, the resistance of both the pt-100 and the line heater is measured. The resistance of this pt-100 gives then relatively precise the temperature of the membrane and line heater. Together with the resistance of the line heater for different temperatures the TCR of the line heater can be determined using equation 3.13.

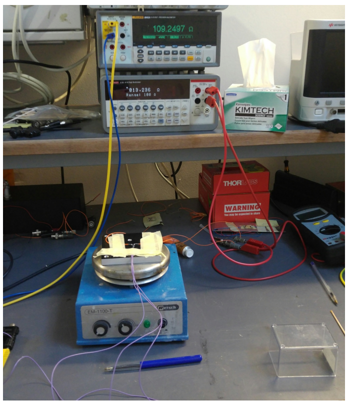

Figure 5.1: *Experimental setup TCR measurement using pt-100*

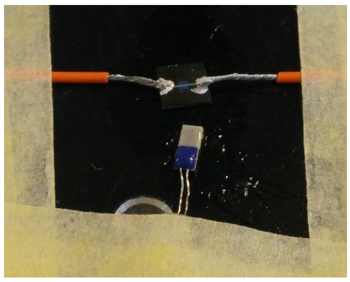

Figure 5.2: *CLose up of the TCR setup, where the membrane and the pt-100 are shown*

5.2 Setup for 3ω measurements

To create an AC current through the line heater on the membrane with a frequency of 1ω and to measure the 3ω voltage appearing across the line heater, a Zurich HF2LI lock-in amplifier is used. In order to measure the 3ω voltage, which is 10^5 weaker than the voltage at 1ω, the voltage at 1ω needs to be suppressed. This is accomplished using a Wheatstone bridge, of which a schematic is shown in figure 5.3

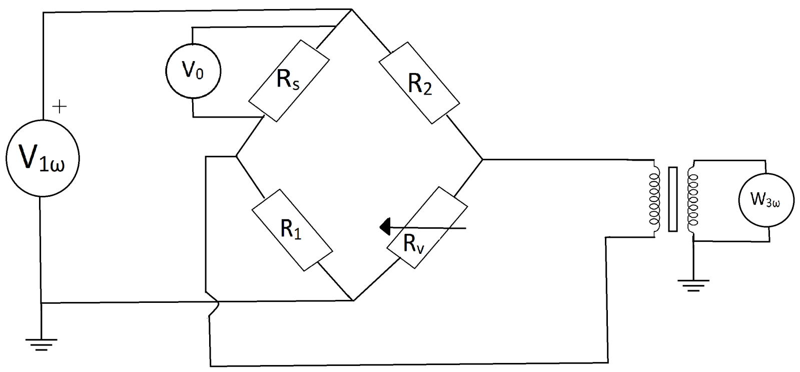

Figure 5.3: *Schematic of the Wheatstone bridge to suppress the 1ω voltage. R_s is the line heater, R_v is a variable (0-111 kΩ) resistor to balance the Wheatstone bridge, R_1 is a resistor of 13 Ω and R_2 is a resistor of 89 kΩ. A 1:1 transformer is also included in the setup and $V_{1\omega}$ corresponds to the output of the lock-in, where $W_{3\omega}$ and $V_{1\omega}$ correspond to the input of the lock-in.*

For the 3ω method, the voltage over the line heater, V_0, needs to be measured simultaneously with the 3ω voltage. This is done by connecting 2 of the 4 wires on the membrane to input of the lock-in, while the other two wires are connected to the Wheatstone bridge. The variable resistance and R_2 are part of a Kelvin Varley voltage divider, placed in a metal box together with the transformer. The membrane with line heater and resistance R_2 are placed in a different metal box. These boxes are connected by a LEMO cable. All the components of the Wheatstone bridge are placed in a metal box to prevent noise from the surroundings.

Using this experimental scheme, the signal at 1ω is heavily suppressed by a factor of 10^5 and is of the same order of magnitude as the signal at 3ω, enabling us to measure the 3ω signal with high precision.

Chapter 6

Results and discussion

6.1 TCR measurements

The TCR measurements result in the following graph:

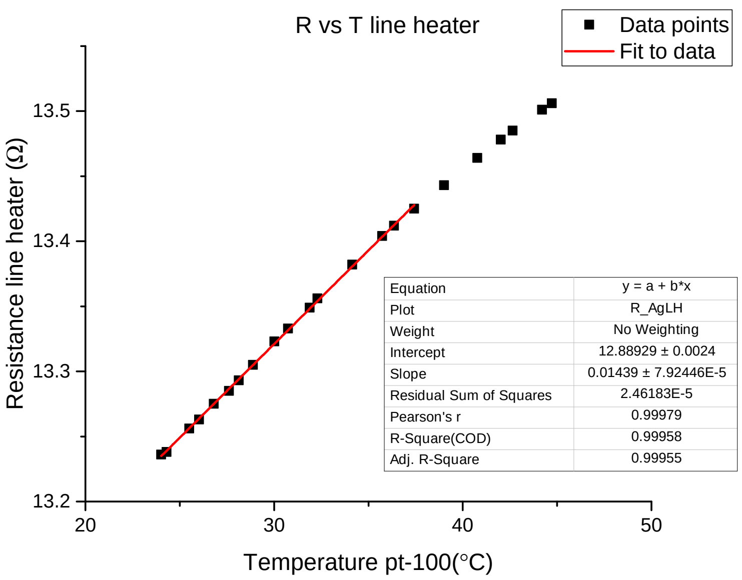

Figure 6.1: *Graph of the resistance of the line heater as a function of temperature measured by the pt-100, with a linear fit to the data points with linear behaviour.*

In this graph a linear relationship is shown for temperatures up to roughly 36 °Celcius. Above this temperature, the data points show less linear behaviour and are therefore not included in the fit from which the TCR of the line heater is determined. For this fit, the slope and the intercept (value of R where T=0 °Celcius) are shown in the box in figure 6.1. From this slope and the intercept, the TCR of the line heater, α is determined using the following formula strongly related to equation 3.13:

$$\alpha = \frac{1}{R_0}\left[\frac{\partial R}{\partial T}\right]_{T=23°C} \tag{6.1}$$

With R_0 the resistance of the line heater at T=23 °C and $\frac{\partial R}{\partial T}$ the slope of the fit to the data in figure 6.1. Using the values R_0=12.22 Ω and $\frac{\partial R}{\partial T}$ = 0.0144 Ω K^{-1}, a value of α= 0.0012 K^{-1} ± 0.0001 is obtained. This error is due to the large error in resistance (R_0) of the line heater, the small statistical errors are therefore not taken into account.

The obtained value of α differs greatly from the TCR of bulk silver which is around 0.0038 K^{-1}. This difference arises from the fact that the line heater in this study is produced by sputtering, which changes the electrical properties of the material. In this study, also sputtering has been done with Platinum/ Palladium (Pt/Pd) alloys, which resulted in the similar differences in TCR values between bulk- and sputtered values.

6.2 3ω measurements

3ω measurements have been performed with the ZiControl software, where the Wheatstone bridge output, $W_{3\omega}^{rms}$ at 3ω, is measured for 80 different values of the frequency of the input voltage, ω, between 1 Hz and 1 KHz on a logarithmic scale. For every value of ω, $|W_{3\omega}^{rms}|$ has been measured 64 times and is averaged over these 64 values. This results in the following graph:

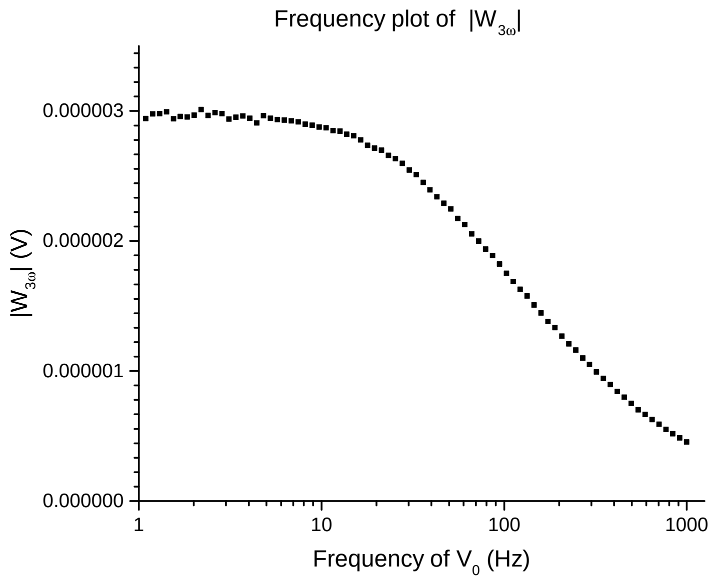

Figure 6.2: *Graph of the absolute rms Wheatstone bridge output versus the frequency of the input signal, applied to the Wheatstone bridge. The input voltage $V_{1\omega}$ (rms) has been set to 0.897 V*

For low frequencies, up to around 7 Hz, $|W_{3\omega}^{rms}|$ is constant, as predicted in section 3.2. Using equation 3.20, the thermal conductivity can be extracted from this constant signal. However, due to differences in sample preparation and the model on which equation 3.20 is based, the formula needs to adjusted. This arises from the fact that the line heater in this thesis is not entirely placed on the membrane, but also partly on the frame. Because the 3ω signal of the frame is expected to be much lower than of the membrane, because the conductance, K, of the frame is much higher than that of the membrane, it generates therefore a negligible 3ω signal. The adjustment in formula corresponds to a difference in resistance used for heating, generating the 3ω on the membrane ($R_{m,0}$) and the resistance of the full line heater ($R_{s,0}$), which is needed to convert the $V_{3\omega}$ on the line heater to the Wheatstone bridge signal using equation 3.18. When taking this differences in to account, the following equation for $|W^{rms}_{3\omega}|$ is obtained:

$$|W^{rms}_{3\omega}| = \frac{\alpha (V^{rms}_{1\omega})^3 R_1 R^2_{m,0}}{4K(R_{s,0} + R_1)^4} \tag{6.2}$$

Using this equation, an attempt has been made to predict the measured $|W^{rms}_{3\omega}|$ for low frequencies. Using α = 0.0012 K^{-1}, R_1 =13 Ω, $R_{m,0}$ =5.4 Ω, $R_{s,0}$ =14.4 Ω, $V^{rms}_{1\omega}$ = 0.897 V and $K = k\frac{S}{l}$, with S = 50 x 10^{-12} m^2 and l = 0.5 mm. Because the thermal conductivity is expected to have a value roughly around 5 W/(m K), this value for k has been used in the prediction of $|W^{rms}_{3\omega}|$. The predicted value of $|W^{rms}_{3\omega}|$ is 0.29 V, which is of order 10^5 higher than the measured signal. In the following subsection a discussion of the substantial difference between the expected and measured value of $|W^{rms}_{3\omega}|$ will be provided.

Discussion of differences between measured and predicted $|W^{rms}_{3\omega}|$

The fact that measured signal is significantly weaker than the expected signal, is partly due to the fact that heat generated in the line heater does not flow enirely through the membrane, creating a 3ω signal, but flows mostly through the line heater to the frame, creating no signal at 3ω. This arises from the fact that the thermal conductance of the membrane ($K \approx 5 \times 10^{-7}$ W/K) is lower than the roughly estimated thermal conductance of the line heater ($K_{lh} \approx 10^{-5}$ W/K). The thermal conductance of the line heater is estimated using $k_{lh} \approx 250$ W/(m K) [13], $S_{lh} \approx 10^{-11}$ m^2 and average distance to frame of $\frac{1}{4}$ mm. The ratio of these conductances is then: $\frac{K}{K_{lh}} = \frac{1}{20}$. This ratio of conductances is equal to the ratio of heat flow through the membrane and the heat flow through the line heater, $\frac{q_m}{q_{lh}}$. Because the temperature oscillations are expected to be linear related to the heat flow through the materials [11] and the because the 3ω voltage is linear related to the temperature oscillations, $|W^{rms}_{3\omega}|$ is expected to be $\frac{1}{20}$ times the expected value, resulting in a voltage of 14.5 mV. This however is not nearly the measured value and this problem can therefor not fully explain the big discrepancy between measured - and predicted signal.

Another cause of this big difference is the fact that the theoretical model predicting the 3ω signal is based on a infinitely small line heater width, while the line heater width in this bachelor project is around 140 μm.

Therefore for the 3ω measurements on thin films or membranes, line heaters with much smaller widths (5-10 μm) are usually used [10]. The effect of finite line heater width is studied by Ftouni et al (2013) [14], who stated that the line heater width should maximally be a fraction $\frac{1}{10}$ of the width of the membrane. This recommendation is not followed in this bachelor project, leading therefore in errors in thermal conductivity measurements. The quantitative impact of the too large line heater width is hard to estimate and is therefore not studied in this project.

These two causes for the difference between measured and predicted signal cannot completely explain this difference, and this discrepancy between measured and predicted signal is therefore subject for further studies.

Chapter 7

Conclusions and proposal for future studies

From this bachelor research project we can conclude that the best method for measuring the thermal conductivity of nm thick SiN membranes is the transient 3ω method. To implement this method, a line heater is deposited on the membrane using relatively simple sample preparation. Using this sample preparation, a 3ω signal of the membrane and line heater is detected. However, due sample preparation, no thermal conductivity of the SiN membrane could be extracted from this 3ω measurements.

For future studies the following improvements are proposed:

1. The line heater width should be reduced from 140 μm to around 5-10 μm;
2. The thermal conductance of the line heater should be reduced;
3. The resistance of the line heater should be increased;
4. The line heater should be entirely placed on the membrane

1: As stated in the discussion of the 3ω results, a ratio of $\frac{1}{10}$ of line heater width and the width of the membrane should be respected. To accomplish this, the line heater width should be heavily reduced.
2: As explained in the discussion of the 3ω results, the relatively low thermal conductance, K_{lh} of the line heater causes the heat to flow mostly through the heater to the frame, creating no 3ω signal. Therefore K_{lh} should be decreased, this can be accomplished using a material with lower thermal conductivity and using a smaller line heater width and height.

3: Because the resistance of the line heater used in this research project is relatively small (13.2 Ω), the error in this resistance due to the resistance of the contact pads is relatively large, leading to large errors in thermal conductivity measurements. By using a higher resistance of the line heater, these errors are heavily reduced.
4: Because the line heater in this project is placed on the frame and the membrane, estimations have to be made for the amount of resistance of the line heater on the membrane itself, creating a signal at 3ω. These estimations are quite rough and lead to large errors in resitance of the line heater on the membrane, which then leads to high errors in thermal conductivity measurements.

Chapter 8

Acknowledgements

First of all, I would like to thank my supervisor, Wolfgang Löffler, for helping me on daily basis with everything concerning my bachelor project. This includes all scientific discussions, all tips he gave to make progress in this project and learning me a lot on my path to become a researcher.

Second, I would like to thank Kier Heeck, who helped me with everything that had to with electronic circuits, lock-in amplifiers, soldering and for his attempt to measure the temperature of the membrane by using the change in inductance of a coil, due to temperature change of small metal disk deposited on the membrane.

Last, I would like to thank Douwe Scholma and Thomas Mechielsen, who helped me to use different sputtering machines and to use the profilometer.

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