$3w$ Method Lab Practice - Group 13

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1 Objectives and Experimental Setting

The main objective of this lab practice was to employ the variant of the well known $3w$ method [1], described in Ref. [2], in order to simultaneously measure the thermal conductivity coefficient of a dielectric thin-film (of silicon nitride SiN_x and silicon oxide SiO_2), κ_{film}, and that of a possibly conductive substrate (of doped silicon Si), κ_{subs}, without the need to employ a reference sample.

The experimental setup employed for this consisted on a 2 mm long aluminium Al metallic strip deposited over the thin-film (either a 450 nm SiN_x film or a 400 nm SiO_2), which when current is passed through, dissipates electric energy as heat, that is transversely transferred to the thin-film and substrate beneath it. The same Al strip not only acts as a heater, but also as the sensor for the temperature difference caused on the materials beneath it, as explained in the next section. In particular, two strips of different widths, 30 and 50 μm, were employed for each film, which is the trick to measure both thermal conductivities at once, as we will see.

The strip is connected in series with a load resistance $R_L = 50\Omega$ and a manually calibrated potentiometer. The current source for them allows manual control of the input alternate current (AC) frequency w. Finally, there is a voltmeter measuring the drop in the load (the amplitude of which will be denoted by V_L), a voltmeter measuring the drop in the strip (of amplitude V_S) and a voltmeter measuring the drop difference between the strip and the potentiometer (of amplitude V_{diff}), which is amplified 100 times. The three signals are converted to digital arrays for real time oscilloscope-like monitoring with a Python script, allowing the display of the Fourier transform of V_{diff} in real time.

2 Methodology

Following the derivations of Ref. [2], we know that for the j-th heater strip, the total in-phase (with the input AC current) temperature variation of the substrate and thin-film is given by:

$$\Delta T^{(j)}_{total}(w) = \Delta T^{(j)}_{subs}(P_j(w), ln(2w)) + \Delta T^{(j)}_{film}(P_j(w)) \tag{1}$$

with $\Delta T^{(j)}_{subs}$ the contribution of the substrate and $\Delta T^{(j)}_{fillm}$ the one of the thin-film, such that:

$$\Delta T^{(j)}_{subs} := \frac{P_j(w)}{\pi l \kappa_{subs}}\left[\frac{1}{2}ln\left(\frac{\kappa_{subs}}{C_{subs}}\right) - ln(b_j) + \eta - \frac{1}{2}ln(2w)\right] \tag{2}$$

$$\Delta T^{(j)}_{film} := \frac{P_j(w)h}{2b_j l \kappa_{film}} \tag{3}$$

where $P_j(w)$ is the power dissipated by the j-th strip when stimulated at a frequency w, l is the length of the strips (2 mm), $2b_j$ is the width of the j-th strip, h is the thickness of the thin-film and C_{subs}, η are fixed constants.

We can immediately see that the slope of the line $\Delta T^{(j)}_{total}/P_j(w)$ as a function of $ln(2w)$, that is, $S := \frac{-1}{2\pi l \kappa_{subs}}$, readily encodes a measurement for the thermal conductivity of the substrate κ_{subs}

as a function of known parameters.[1] Then, we can compute κ_{film} by acknowledging that the only modification induced by the strip change is b_j, which in the equation for the line $\Delta T^{(j)}_{total}/P_j(w)$ as a function of $ln(2w)$ only modifies the intercept and not the slope S. This means that the relative displacement of the line will encode κ_{film} as a function of known parameters (since the terms containing C_{subs}, η cancel out).

In particular, we have that this line displacement gives:

$$\frac{\Delta T^{(1)}_{total}}{P_1(w)} - \frac{\Delta T^{(2)}_{total}}{P_2(w)} = \frac{h}{2l\kappa_{film}}\left(\frac{1}{b_1} - \frac{1}{b_2}\right) + \frac{1}{\pi l \kappa_{subs}} ln\left(\frac{b_2}{b_1}\right) \tag{4}$$

theoretically independent of w. Therefore, in each w value we will have an estimate of κ_{film} as:

$$k_{film} = \frac{h(\frac{1}{b_1} - \frac{1}{b_2})}{2l\left[\frac{T^{(1)}_{total}}{P_1(w)} - \frac{T^{(2)}_{total}}{P_2(w)} - \frac{ln(b_2/b_1)}{2\pi l k_{subs}}\right]} \tag{5}$$

Now, the only question left would be how to compute $\Delta T^{(j)}_{total}(w)$ and $P_j(w)$ given our experimental setup.

On the one hand, we placed the load resistance R_L in order to know the amplitude of the AC input current, I, which by Ohm's law is $I = R_L V_L$. This number will depend on w due to the manual variation of the peak-to-peak voltage on the source. With this and the voltage drop in the strip, V_S, we can compute the (average) power dissipated by the strip as $P_j(w) = IV_S/2$ (where the factor two accounts for the temporal average for a resistor).

On the other hand, as shown in Ref. [2], given the AC input current of magnitude I, the voltage drop in the heater strip has primarily three contributions in Fourier space: a constant part, a part of frequency w and a part in $3w$, due to the heating. The $3w$ component has a theoretical magnitude:

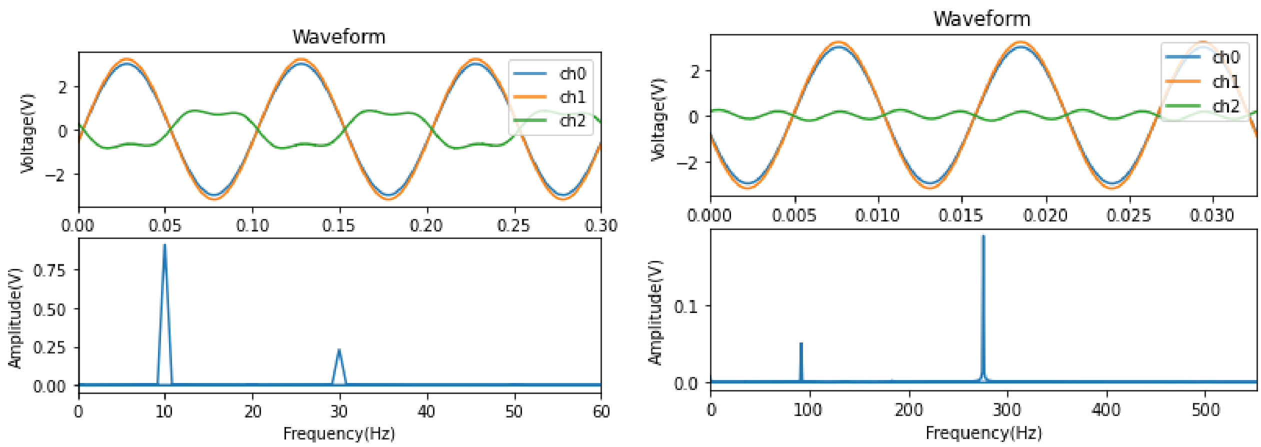
$$V_{3w} = \frac{1}{2} I R_S \Delta T^{(j)}_{total} C_{RT} \tag{6}$$

where R_S is the strip resistance, such that $V_S = IR_S$, and $C_{RT} := \frac{1}{R}\frac{\mathrm{d}R}{\mathrm{d}T}$ is the resistance-temperature coefficient of the strip, known to be for Al of $0.0021\ 1/K$ [3]. Then, if we knew V_{3w}, we could compute:

$$\Delta T^{(j)}_{total} = 2\frac{V_{3w}}{V_S C_{RT}} \tag{7}$$

Finally, because in the potentiometer, there is no noticeable heating effect, there will be no $3w$ component drop there. As such, since we can monitor the Fourier space decomposition of the voltage difference between the potentiometer and the strip, of amplitude V_{diff}, the potentiometer can be adjusted until the drop in the potentiometer is exactly the same as the constant and w frequency components of the drop in the strip. At that point, the Fourier decomposition of the difference should show no peak at 0 frequency nor w and instead show a main peak at $3w$. Then, and only then, V_{diff} will be equal to V_{3w} (with the amplification). In Figure 1, the interface employed to monitor the voltage drops and the Fourier decomposition of the signal difference between the strip and the potentiometer is shown. In Figure 1.(a), the potentiometer is still not adjusted in order to compensate the constant and w components of the strip's drop. Note how weak the $3w$ amplitude is relative to the dominant w. In Figure 1.(b), almost only the $3w$ component is left, time at which $V_{diff} \simeq V_{3w}$ (up to amplificaiton).

[1] In fact, we will have two estimates for the parameter as given by each strip, since the slope S is the same irrespective of j and $j \in \{1, 2\}$.



(a) (b)

Figure 1: In each sub-figure, above, the time evolution of the detected voltage drops with (ch0,ch1,ch2) respectively indicating the voltgae drops in the load resistence, in the strip and the amplified difference between the strip and the potentiometer. Below, the Fourier decomposition magnitudes for the difference drop (ch2). In (a) the drop in the potentiometer does not fit the one in the strip. In (b) a better fit is found, which allows a more reliable extraction of V_{3w}, following the text. Note that in the upper plot of (b) one can readily see an almost pure sinusoid of frequency $3w$, while in (a) it has clearly both w and $3w$ contributions.

2.a Statistical Analysis

As we saw, theoretically, $\Delta T^{(j)}/P_j = \beta_j + S\ ln(2w)$, for the two strips $j \in \{1,2\}$ with

$$\beta_j := \frac{1}{\pi l \kappa_{subs}}[ln(\frac{\kappa_{subs}}{C_{subs}}) - ln(b_j) + \eta] \text{ and } S := -\frac{1}{2\pi l \kappa_{subs}},$$

such that $k_{subs} = -\frac{1}{2\pi l S}$.

Let us denote $y_j := \Delta T^{(j)}/P_j$ and $x := ln(2w)$. Then, if we denote by $\{(x^k, \hat{y}_j^k)\}_{k=1}^N$, the measured pairs of values, and $\bar{x}, \bar{y}_j$ their averages, assuming that the measurements had an independent and identically distributed additive Gaussian noise $\varepsilon_j \sim N(0,\ \sigma_{\varepsilon_j})$ of standard deviation σ_{ε_j}, such that $\hat{y}_j^k = y_j(x^k) + \varepsilon_j$, we can estimate the slope S for each j using classical mean squares regression analysis to get[2]

$$\hat{S}_j \sim N(S,\ \sigma_{S_j}) \quad \text{with} \quad \sigma_{S_j}^2 = \sigma_{\varepsilon_j}^2 / \sum_k (x^k - \bar{x})^2.$$

With them, we can straightforwardly get the estimators of the substrate thermal conductivity as

$$\hat{k}_{subs_j} \sim N(k_{subs},\ \sigma_{k_{subs}j}) \quad \text{with} \quad \sigma_{k_{subs}j} \simeq \frac{1}{4\pi l}\frac{\sigma_{S_j}}{S_j^2}.$$

Finally, we can get a global estimate of k_{subs} with a reduced variance through the mean random variable $\hat{k}_{subs} = (\hat{k}_{subs_1} + \hat{k}_{subs_2})/2$, which follows a normal distribution as well

$$\hat{k}_{subs} \sim N(k_{subs},\ \sigma_{k_{subs}}) \quad \text{with} \quad \sigma_{k_{subs}} = \frac{1}{2}\sqrt{\sigma_{k_{subs}1}^2 + \sigma_{k_{subs}2}^2}. \tag{8}$$

This allows a trivial confidence interval computation for the coefficient.

On the other hand, because in theory $y_1 - y_2 = \beta_1 - \beta_2 =: \gamma$, such that

$$k_{film} = \frac{h(1/b_1 - 1/b_2)}{2l(\gamma - ln(b_2/b_1)/2\pi l k_{subs})},$$

[2]By $\hat{X} \sim N(\mu, \sigma)$ we mean that the random variable $\hat{X}$ follows a normal distribution of expectation μ and standard deviation σ. We denote by the same letter with a "hat" $\hat{X}$, the statistical estimator of a deterministic but unknown parameter X.

we can first estimate β_j using classical mean squares regression as:

$$\hat{\beta}_j \sim N(\beta_j, \sigma_{\beta_j}) \quad \text{with} \quad \sigma^2_{\beta_j} = \sigma^2_{\varepsilon_j}\left[\frac{1}{N} + \frac{\bar{x}^2}{\sum_k (x^k - \bar{x}^2)}\right]$$

Then, it is easy to see that because $\hat{\gamma} = \hat{\beta}_1 - \hat{\beta}_2$,

$$\hat{\gamma} \sim N(\beta_1 - \beta_2,\ \sigma_\gamma) \quad \text{wtih} \quad \sigma_\gamma = \frac{1}{2}\sqrt{\sigma^2_{\beta_1} + \sigma^2_{\beta_2}}.$$

Finally, some manipulations lead us to:

$$\hat{k}_{film} \sim N(k_{film},\ \sigma_{k_{film}}) \quad \text{where} \tag{9}$$

$$\sigma^2_{k_{film}} = \left(\frac{c}{(\gamma - a/k_{subs})^2}\right)^2 \sigma^2_\gamma + \left(\frac{ac}{(\gamma k_{subs} - a)^2}\right)^2 \sigma^2_{k_{subs}}$$

with the known constants $a := \frac{ln(b_2/b_1)}{\pi l}$ and $c := \frac{h}{2l}(\frac{1}{b_1} - \frac{1}{b_2})$. With this, we can also get a trivial confidence interval for κ_{film}.

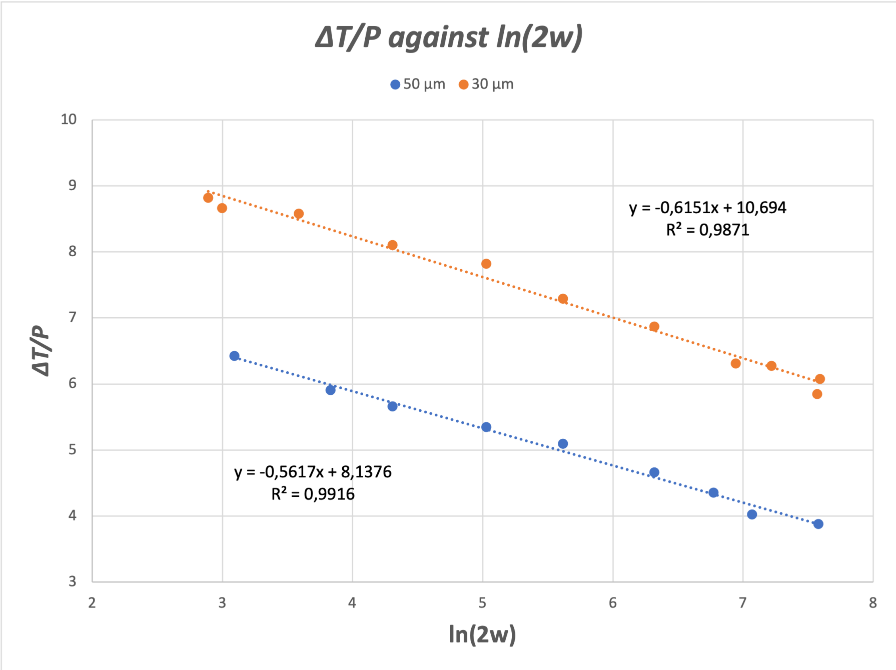
3 Results and Discussion

After plotting $\Delta T^{(j)}/P_j$ against $ln(2\omega)$ (see Figure 2), we have calculated the slopes, the intercepts and their standard deviations as explained in Section 2, using the data provided by the instructors. With them we could calculate the thermal conductivities following the computation of all the intermediate estimators explained in the same section. We can see the result of these calculations in Table 1.

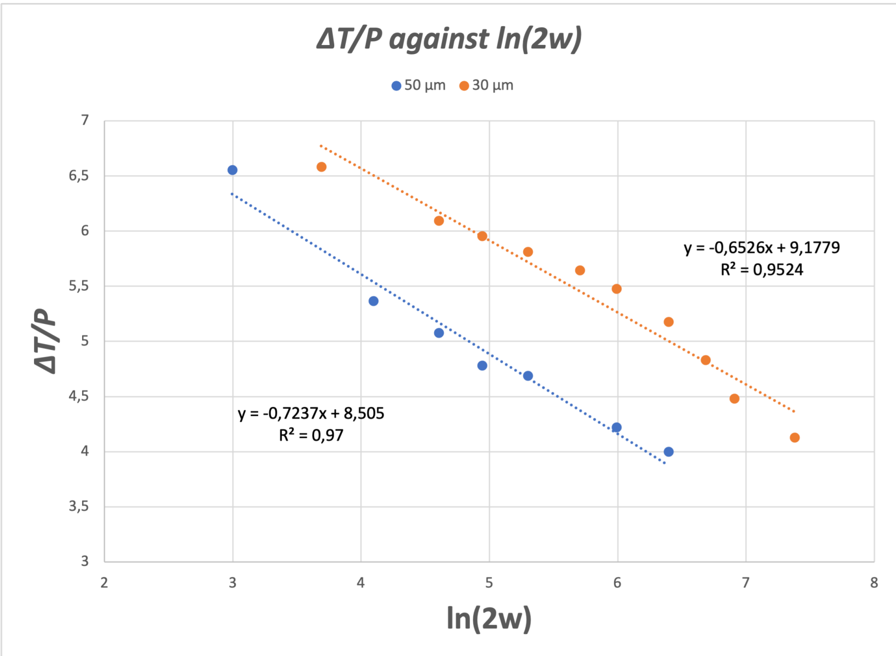
Table 1: Calculated parameters and their respective standard deviations, following the notation in Section 2.

	SiO_2		SiN_x	
	Estimator	Standard deviation	Estimator	Standard deviation
S_1 (K/W)	-0,615	0,024	-0,653	0,052
S_2 (K/W)	-0,562	0,020	-0,723	0,057
$k_{subs,1}$ (W/mK)	129,4	2,5	121,9	4,8
$k_{subs,2}$ (W/mK)	141,7	2,5	109,9	4,3
k_{subs} (W/mK)	135,5	1,7	115,9	3,2
β_1 (K/W)	10,69	0,13	9,17	0,30
β_2 (K/W)	8,14	0,11	8,51	0,29
γ (K/W)	2,56	0,087	0,67	0,21
k_{film} (W/mK)	1,181	0,046	9,3	6,0

After all the calculations, we obtained the objective parameters summarised in Table 2, with 90% confidence intervals. As it can be seen in the table, we got a thermal conductivity for SiO_2 of $(1,18 \pm 0,08) W/mK$ (with an $\alpha = 0.1$ significance) which is very close to the one obtained in Ref. [2], of $1,35 W/mK$. Likewise, our computed thermal conductivity for SiN_x, is not either far from the one obtained by Ref. [2]. We got a value of $(9,3 \pm 7,7) W/mK$ while Ref. [2] mentions a value of $2,23 W/mK$, which gets inside the confidence interval of our estimate. The differences in any case, could be due to the fact that the values of Ref. [2] were obtained for thin-films of 180 nm thickness, while our thin-films have thicknesses of 400 nm and 450 nm, for the SiO_2 and SiN_x samples respectively. Although the bulk thermal conductivity coefficients should not depend on the volume of the material, when talking about thin-films, surface effects may play a significant role, making the coefficients vary with the thickness of the material. If this is the case, our measurements indicate that the thermal coefficient gets bigger for a thicker thin-film (ours, compared to the cited reference).



(a) (SiO_2) sample



(b) (SiN_x) sample

Figure 2: $\Delta T^{(j)}/P_j$ (K/W) against $ln(2\omega)$ with w in Hz plotted for the analysed samples. See the inset for the colour-code. The the scattered dots represent the experimentally measured data, while the lines represent their linear regression, which is indicated by the fitted formulas (note the R^2 fitting coefficient is close to 1 in all cases, indicating the underlying linear nature of the data).

When it comes to the thermal conductivity of the doped Si substrate, we have obtained a different value for each sample, $(135, 52 \pm 2, 87) W/mK$ for the SiO_2 sample and $(115, 95 \pm 5, 3) W/mK$ for the SiN_x sample, while the literature mentions a value of $92 W/mK$. It is a good signal that they are all in the same order of magnitude, indicating that the substrate material is indeed similar. However, as explained by Ref. [2], the ammount of doping causes an important variation of the thermal conductivity. Therefore, with our measurement we can assert that the two samples do not have the exact same doping profile, at least in the neighbourhood of the deposited heater.

Table 2: Obtained thermal conductivity of the substrate and the thin-film from both samples, following the notation in the text. The uncertainty reflects 90% confidence intervals computed with the obtained standard deviation, as explained in Section 2.

	SiO_2	SiN_x
$k_{subs}(W/mK)$	$135,5 \pm 2,9$	$115,9 \pm 5,3$
$k_{film}(W/mK)$	$1,180 \pm 0,080$	$9,3 \pm 7,8$

4 Conclusions

In conclusion, we have successfully employed the version of the $3w$ method by Ref. [2], to predict the thermal conductivity coeffícents for SiO_2 and SiN_x thin-films, together with their doped Si substrates, matching the results previously found on the literature. This proves the validity of the employed experimental and analytic protocols.

References

[1] D. G. Cahill, "Thermal conductivity measurement from 30 to 750 k: the 3ω method," *Review of scientific instruments*, vol. 61, no. 2, pp. 802–808, 1990.

[2] J. Alvarez-Quintana and J. Rodriguez-Viejo, "Extension of the 3ω method to measure the thermal conductivity of thin films without a reference sample," *Sensors and Actuators A: Physical*, vol. 142, no. 1, pp. 232–236, 2008.

[3] "Medida de la conductividad térmica mediante el método $3w$," *Practice Guide - Physics at the Nanoscale (Universitat Autònoma de Barcelona - UAB)*, 2022-23.