Exercise in thermal analysis

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1 Background

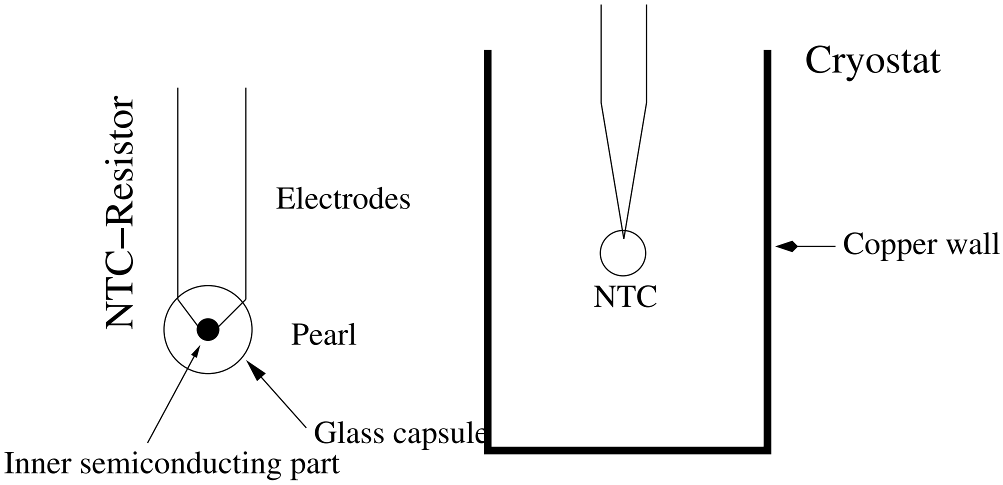
The exercise is designed to get you familiarized with a method used to determine the frequency dependent heat capacity of a material. The measurement method is part of a number of methods used in the “liquid-laboratory”. These methods, which were partly developed here on the site, are established with the aim of measuring a complete set of electrical and thermo-mechanical response functions.

In the preparation for this exercise, read this note thoroughly and try to work out the various tasks herein. After the exercise, a report should be written which contains a review of the theory behind the method (ie at least answering most “exercises” in this note), and a thorough analysis of the performed measurements. The measurements must be treated and the results compared with literature values and theory.

The heart of the method consists of a so-called NTC resistor (negative temperature coefficient) which has a resistance, R, that depends on temperature. The principle of the method is that the resistor simultaneously can deliver heat (Joule heat) and register the corresponding temperature response. By sending a known electrically current through the resistor and measuring the voltage across this, one can thus calculate both heat production and temperature response.

The NTC resistor has the form of a little pearl. It has a structure with a inner semiconductor portion and an outer glass capsule, and is connected through two legs (electrodes) (see figure 1). The NTC-resistor is placed in a thermostated chamber. We assume that the chamber walls, which consists of solid copper, can be thermostated so that the temperature here does not depend on the heat flow generated by the NTC resistor.

The thermal properties of a sample can be calculated if it is thermally coupled to the resistor. In the laboratory we work with two different “geometries” a thin



Figur 1: **Left** Sketch of the pearl with inner part and glass capsule. **Right** Pearl in cryostat.

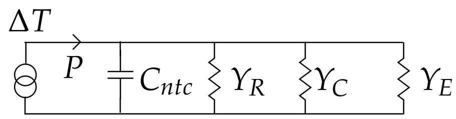
and a thick. In the thin geometry the liquid is applied onto the pearl and hangs like a drop around this (due to surface tension). The specific heat capacity of the liquid can then be determined from the drop's weight, the total heat capacity and the heat capacity of the pearl. In the thick geometry, the bead is placed in a container (which is much larger than the bead) filled with liquid. These two geometries each have their advantages and disadvantages.

Due to time constraints, we can in this exercise only determine the heat capacity of the pearl, total heat loss to the surroundings, and the heat conductivity of air.

2 Modeling the thermal system

To understand the thermal properties of the system consisting of the pearl and the cryostat, it is advantageous to provide a "network"model. In this model we let the voltage be the temperature difference between the pearl and the cryostat (ΔT) and the power is the heat flow (P), both assumed to be harmonic functions with a given frequency, and expressed as complex amplitudes. With these definitions we can talk about the complex frequency dependent thermal impedance ($Z = \Delta T/P$), thermal admitans ($Y = P/\Delta T$) and thermal capacity ($C = Q/\Delta T$, where Q is the total amount of heat, that is the integrated heat flow). [1]

[1] You have worked with this type of modeling on the " Physical modeling"course, if necessary see the notes from that course for a discussion of the generalized impedance.



Figur 2: Electric equivalence network for the thermal system.

The thermal properties of the system can be described by the electric equivalence network shown on figure 2.

The current generator represent the inner part of the bead. It is seen that the current generator sends a current into a parallel connection of the heat capacity of the pearl and three thermal admittance's. The thermal properties of the bead and the surroundings is typically represented by the total thermal impedance $Z_{th} = \Delta T/P$.

In the following exercises you should consider the thermal admittance and how it can be found.

Exercises

Why are the 4 elements coupled in parallel? Which heat conduction abilities are represented by Y_R, Y_C og Y_E? How can we find C_{NTC} and the total conductivity from the measured thermal impedance Z_{th}?

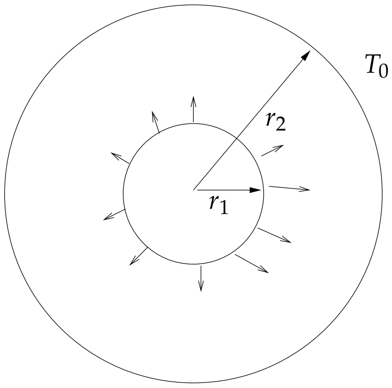
Think of a substance with the specific heat conductivity λ, which is placed between two concentric sphere shells of radius r_1 and r_2 (see figure 3). Calculate the temperature increment ΔT obtained in equilibrium at the inner shell, if we maintain the temperature T_0 on the outermost shell and sends a heat flow I_0 from the inner shell out into the medium. Now let the pearl be the inner shell and the cryostat the outer, and calculate an expression for the thermal admittance as seen at the inner shell assuming $r_2 \gg r_1$.

Calculate an expression for the thermal admittance when heat is transported by radiation. Finally calculate an expression for the thermal admittance due to heat loss through the electrodes.

The thermal conductivity of the air can be changed by changing the pressure of the cryostat chamber. How does the conductance λ depend on the pressure?

Sketches the theoretical thermal impedance as a function the frequency of the harmonic heat (power) input.

In the exercise we will be measuring the total thermal conductivity at various pressure, consider what we gain from this and how it enables us to find the thermal conductivity of air.



Figur 3: Sketch of two spheres for calculating the thermal admittance

3 A short section on complex notation

In what follows, we use complex notation to represent a periodic signal. In principle the same technique as in the " Physical modeling course", but in this context, we need to be a little more thorough. This is because the system is not linear and we therefore need to explicitly express the real time-dependent periodic signal on the basis of the complex quantities.

A periodic signal $A(t)$ can be generally written as a sum of harmonic terms:

$$A = A_0 + |A_1|\cos(\omega t + \phi_1) + \ldots + |A_n|\cos(n\omega t + \phi_n) \tag{1}$$

where A_0, $|A_k|$ are real amplitudes and ϕ_k phases. Such a sum of harmonic terms can be written as (if you can not see this immediately try to work it out)

$$A = \frac{1}{2}\left(A_0 + A_0 + |A_1|e^{i(\omega t+\phi_1)} + |A_1|e^{-i(\omega t+\phi_1)} + \ldots + |A_n|e^{i(n\omega t+\phi_n)} + |A_n|e^{-i(n\omega t+\phi_n)}\right). \tag{2}$$

By introducing the complex amplitude $A_k = |A_k|e^{i\phi_k}$ and a shorthand notation $E_k = e^{ik\omega t}$ the sum can be written as

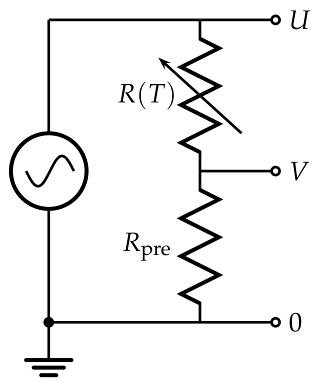
$$A = \frac{1}{2}(A_0 + A_1E_1 + \ldots + A_nE_n + c.c.), \tag{3}$$

where $+c.c.$ means that the complex conjugated of all terms in the parenthesis are to be added (including the conjugated of the real constant).

Some algebraic rules apply (convince yourself):
$E_0 = 1$, $E_kE_l = E_{k+l}$, and $E_kE_l^* = E_{k-l}$.

Exercise

Let $A = \frac{1}{2}(A_0 + A_1E_1 + c.c.)$ og $B = \frac{1}{2}(B_0 + B_1E_1 + c.c.)$, calculate $A \cdot B$ and express it in terms of the above introduced notation (that is give the complex amplitudes).



Figur 4: Circuit for measuring, U is the input voltage, V is the measures voltage over the pre-resistor R_{pre} and $R(T)$ is the temperature dependent resistance of the NTC-resistor.

4 The electric setup and the fundamental equations

The electric current/voltage relation is measured by the circuit shown on figure 4. The NTC resistor is placed in what is known as a voltage divider together with a known resistor, R_{pre}, which is temperature independent.

The voltage generator is custom build (by the workshop) and delivers a time dependent voltage $U(t) = A\cos(2\pi\nu t)$, where the amplitude A can be chosen between 0 and 10V in 256 steps, while the frequency ν can be changed between 1mHz and 100Hz.

The time-dependent output voltage $U(t)$ is measured using a 26-bit digital voltmeter. Measurements are presented as an array of 512 measurements taken at equal distances over a period of the input signal. In the design of the setup much care is taken to keep track of the phase between the input voltage and the measured voltage.

Exercises

Derive the voltage divider formula:

$$V(t) = \frac{R_{\text{pre}}}{R_{\text{pre}} + R(T(t))} U(t). \tag{4}$$

The temperature dependency of the bead can be described as: $R(T) = R_\infty e^{T_a/T}$, discuss why this is the case?

Assume that the temperature of the bead is close to the temperature of the cryostat, T_0, now show that one may express R as function of ΔT to first order as $R = R_0(1 + \alpha\Delta T)$ (where ΔT is the difference in temperature from the cryostat temperature. What is the physical interpretation of R_0?

Show that the temperature coefficient α can be expressed as $\alpha = \frac{d \ln R}{dT}$, and derive an expression for the dependence of T and T_a.

Now take advantage of this first order approximation to write $V(t)$ as function of $U(t)$ and $R(T)$ to first order in ΔT as:

$$V = \frac{1}{A+1}\left(1 - \frac{A\alpha}{1+A}\Delta T\right) U. \tag{5}$$

Finally derive the two following expression for the effect delivered to the bead:

$$P = \frac{1}{R_{\text{pre}}}\frac{A}{(1+A)^2}\left(1 + \frac{1-A}{1+A}\alpha_1\Delta T\right) U^2 \ \text{(to first order in } \Delta T) \tag{6}$$

$$P = \frac{(U-V)V}{R_{\text{pre}}}, \tag{7}$$

what is the advantage of each of these expressions?

5 From time to frequency domain

As described earlier we measure the time-dependent signal $V(t)$ when a periodic signal $U(t)$ is applied. We want to convert this signal to a sum of harmonic contributions. This is done in the computer by use of a so-called "Fast Fourier Transform" (FFT).

In matlab it looks like this:

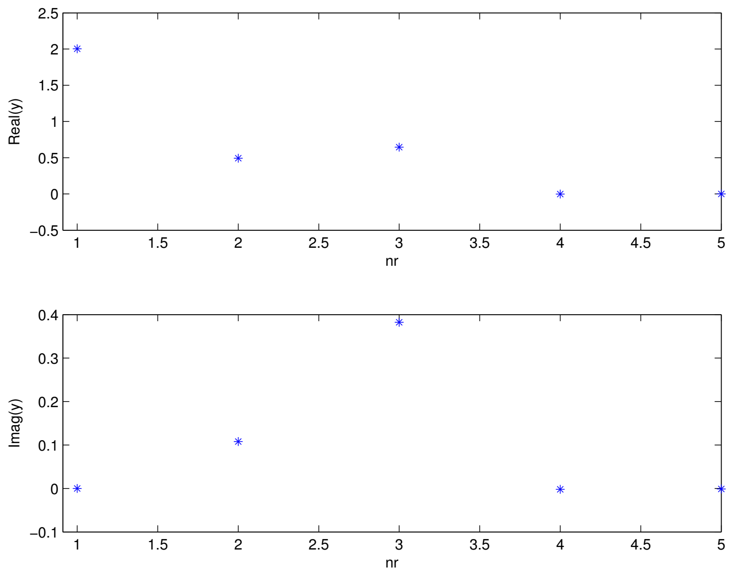
```
x=fread(multi,512,'int32')*iscale;
xfft=fft(x)/256;
```

The first line retrieves the 512 measurements of voltage (this is multiplied by a factor from the voltmeter). In the second line the discrete Fouier transform is taken of this signal (to get the absolute values to fit you must divide by half the number of points (see also matlab help page about fft)). The result is an array where the first place contains 2 times the amplitude of the 0 harmonic, the 2nd place the complex amplitude of the first harmonic, 3rd place the complex amplitude to the second harmonics and so on (why we get 2 times the amplitude to the 0'te harmonics lie in the definition of the Fouier transformation, which we do not get into here). A simple example is illustrated in Figure 5.

```
%number of points
n=512

% array with evenly spaced points between 0 and 2pi
x=linspace(0,2*pi,n)

%A function
y=1+0.5*cos(x+12/180*pi)+0.75*cos(2*x+30/180*pi)
% the FFT transform scaled by n/2
yfft=fft(y)/n*2
```



Figur 5: Example of the resulting values after transforming a signal sampled over one periode. Only the first 5 lines of the array is shown.

In the following we work in the frequency domain. In general we define (only including up to 4th order terms)

$$\begin{aligned}
V(t) &= \frac{1}{2}\left(V_0 + V_1E_1 + V_2E_2 + V_3E_3 + V_4E_4 + c.c.\right) \\
U(t) &= \frac{1}{2}\left(U_0 + U_1E_1 + U_2E_2 + U_3E_3 + U_4E_4 + c.c.\right) \\
P(t) &= \frac{1}{2}\left(P_0 + P_1E_1 + P_2E_2 + P_3E_3 + P_4E_4 + c.c.\right) \\
\Delta T(t) &= \frac{1}{2}\left(T_0 + T_1E_1 + T_2E_2 + T_3E_3 + T_4E_4 + c.c.\right).
\end{aligned}$$

We likewise define the thermal impedance for all the harmonic, that is:

$$\begin{aligned}
Z_{th,1} &= T_1/P_1 \\
Z_{th,2} &= T_2/P_2 \\
\vdots & \quad \vdots
\end{aligned}$$

.

Exercises

Generally, we measure the frequency dependent thermal impedance $Z_{th,i}(\omega)$. What is the relationship between $Z_{th,1}(\omega)$ and $Z_{th,2}(\omega)$? (This is a very important but difficult point, use some time thinking about it).

Convince yourself that the values seen in figure 5 fit with the function which has been transformed.

6 Calibrating the temperature dependent resistance of the bead

When the bead sits in the cryostat its temperature-dependent resistance can be determined by measuring V at different temperatures and by pre-measuring U and R_{pre} which does not depend on temperature. When R is measured this way by sending a current through the bead, the bead will not have the same temperature as the cryostat.

Assume hereafter that the generator is "perfect", that is it delivers a pure harmonic signal, $U = \frac{1}{2}(U_1E_1 + cc)$ and that the measured voltage can be written as $V = \frac{1}{2}(V_0 + V_1E_1 + cc)$ (that is we ignore any higher-order harmonic parts).

Exercises

Express the heat-effect of the pearl as a harmonic series using equation 7, what is the dominant part? How does temperature vary on the bead (which terms must be included if the temperature is written as a sum of harmonic terms).

Show that the DC amplitude of the heating effect can write as:

$$P_0 = \frac{1}{4R_{\text{pre}}}((U_1 - V_1)V_1^* + (U_1^* - V_1^*)V_1) \tag{8}$$

Show that the first harmonic in the measured signal (V_1) can be expressed as:

$$V_1 = \frac{1}{A+1}\left(U_1 - \frac{A\alpha}{1+A}T_0 U_1 - \frac{1}{2}\frac{A\alpha}{1+A}T_2 U_1^*\right) \tag{9}$$

(where $A = R_0/R_{pre}$).

By measuring at different amplitudes of U one can by extrapolation determine R at the current cryostat temperature, how is this done? (Note that the term $\frac{1}{2}\frac{A\alpha}{1+A}T_2U_1^*$ can be assumed to be small).

Based on this expression it is also possible to find the DC level of the thermal impedance (Z_0), how?

7 The 3ω method

We now have (hopefully) seen that a purely harmonic input, give rise to a heat flow which varies as 2ω, and thus a temperature which varies with 2ω. As the resistance of the NTC-bead is dependent on temperature, its resistance also vary with 2ω. The voltage drop across the NTC-resistor is give by Ohm's law (the product of resistance and current) and as the current has a dominant first harmonic part, one gets a 3 harmonic term in the voltage across the resistor.

That is you can find the temperature amplitude by studying the third harmonics in the measured signal.

Exercises

Show that the amplitude of the third harmonic in the measured signal (V_3) can be written as:

$$V_3 = -\frac{1}{2}\frac{A\alpha}{(A+1)^2}T_2U_1. \tag{10}$$

Furthermore show that the amplitude of the second harmonic of the power can be written as:

$$P_2 = \frac{1}{2R_{\text{pre}}}(U_1 - V_1)V_1. \tag{11}$$

Use equation 8, 9, 10 and 11, to finde the frequency dependent thermal impedance and the DC impedance.

At which thermal frequency is the frequency dependent thermal impedance evaluated?

8 The two measurements

First exercise

The NTC-resistor is mounted in a specimen holder with a pre-resistor R_{pre}, whose value is determined with a multi-meter. The specimen holder is placed in a cryostat and connected to the measuring setup. Test how everything works.

The temperature dependence of the NTC resistor is found by measuring V and U at (least) 3 different amplitudes (50, 75 and 100) and at (least) three different temperatures (300K, 275k and 250K). The measurement is done at 10Hz. Based on these measurements find T_a and R_∞ and an initial guess of the DC thermal impedance (called hereafter *DC* method), and DC temperature amplitude (consider how this is related to the amplitude of the second harmonic heat flow).

Consider how we "online" may follow if the temperature has reached its equilibrium.

Second exercise

At a suitable temperature eg. 200K measure V and U as a function of frequency in the range 0.1 to 100Hz, and analyze the result.

Evacuated the sample chamber and repeat the measurement.

If you want we can now create a somewhat longer measurement over a few days, so we get better low-frequency data.

Calculated the thermal impedance in two ways, respectively by the "DC"-method and the "3ω- method".

Determine the thermal conductivity of air and heat capacity of the pearl from the two measurements, and compared with literature values.

9 Background literature

The following articles contains the background of the discussed method. It is not necessary to read these to do the exercise or write the report, but the references are include for completeness.

Measuring the frequency dependent specific heat by using the pearl and a thick layer of liquid. The articles contains a detailed review of the 3ω technique:

- Bo Jakobsen, Niels Boye Olsen & Tage Christensen. Frequency dependent specific heat from thermal effusion in spherical geometry. *arXiv:0809.4617v1 [cond-mat.soft]*, 2008. (Note that it is version 1 of this paper that is most relevant for this exercise).
- Bo Jakobsen, Niels Boye Olsen & Tage Christensen. Frequency-dependent specific heat from thermal effusion in spherical geometry. *Phys. Rev. E*, 2010, **81**, 061505.

Measuring the frequency dependent specific heat by using the peal and a thin layer of liquid:

- Tage Christensen & Niels Boye Olsen. Thermoviscoelasticity of glass-forming liquids. *J. Non-Cryst. Solids*, 1998, **235–237**, 296–301.

Detailed descriptions of the cryostat and the electric setup can be found in the following two articles:

- B. Igarashi, T. Christensen, E. H. Larsen, N. B. Olsen, I. H. Pedersen, T. Rasmussen & J. C. Dyre. A cryostat and temperature control system optimized for measuring relaxations of glass-forming liquids. *Rev. Sci. Instrum.*, 2008, **79**, 045105
- B. Igarashi, T. Christensen, E. H. Larsen, N. B. Olsen, I. H. Pedersen, T. Rasmussen & J. C. Dyre. An impedance-measurement setup optimized for measuring relaxations of glass-forming liquids. *Rev. Sci. Instrum.*, 2008, **79**, 045106.

An introduction to modeling by the use of electric network models can be found in:

- Niels Boye Olsen, "Modellering med elektriske netværk — Noter fra Fysik B kurset", IMFUFA tekst nr. 466 (2009) (In danish).
- Tage Christensen, "Notes for the Physical modeling Course" (found online at `http://dirac.ruc.dk/~tec/FysMod`).