Laboratory 1
Heat diffusion

Objective

The objective of this laboratory is to improve your understanding of solutions of the diffusion equation via measurements of the diffusion of the heat in a material. You will extend your knowledge about heat transport in solids. In this laboratory you also become more familiar with using lock-in amplifier for measurements of the amplitude and the phase of an electronic signal.

Preparation

Read:

- **Physical Properties of Materials, Second Edition** Published:June 28, 2011 by CRC Press Content:469 Pages | 273 B/W Illustrations Author(s):Mary Anne White
- Three-page document "What is a lock-in amplifier?"
- Journal of Applied Physics Volume 32, number 9 Flash Method for Determining Thermal Diffusivity, Heat Capacity, and Thermal Conductivity
- Review lecture slides

Look up Reference Values:
- Heat Capacities and thermal conductivities of all sample materials

Equipment and Samples

Flash Method
- Pyroelectric detector
- Lock-in amplifier with Signal Recovery software
- Optical chopper and controller
- Digital Oscilloscope and software
- Halogen lamp
- Optical density filters (2)
- Samples: disks made of graphite, steel shim, thicker steel plate, silicon, alumina
- Computer with plotting software such as MS Excel or Origin Pro

Angström Method
- Bread board,
- Validyne Data acquisition USB box with a circuit board using Easy Sense 2250 Software
- Samples: Cu, SiC Al_2O_3, Si_3N_4, and AlN rods with connected thermocouples and heaters.

Introduction

The heat capacity, c, of a material per unit volume is a thermodynamic property. It is a measure of how much the temperature changes when a small amount of energy is added or subtracted from the sample. Thermal conductivity (usually written as the Greek letter "kappa" κ) is a transport property. It is a linear transport coefficient that relates a temperature gradient with a heat flux. For isotropic materials or cubic crystals, κ is a scalar, for non-cubic crystals or materials with anisotropic microstructures κ is a tensor. In most cases of practical interest, the κ tensor is diagonal. The ratio of the thermal conductivity to the heat capacity per unit volume, is the thermal diffusivity, $D=\kappa/C$. In MKS unit system, the diffusivity is measured in m^2/s.

From Einstein – Smoluchowski equation, $\tau=L^2/D$, we can get a rough idea of the time τ that it takes for heat to diffuse distance L. For a Si wafer, L=500 microns, D=1 cm²/s, the time τ=2 msec. For polymers, where D=0.001 cm²/s, it takes several minutes for the heat to diffuse 1 cm. As one can see, in practical situations the time-scales of heat diffusion vary enormously. In scientific studies, the time scale of heat diffusion spans 27 orders of magnitude.
Thermal conductivity can be measured directly but usually it is determined from the heat diffusivity via the relation $\kappa=D\cdot C$. The most popular methods for measuring the diffusivity are the Flash Method and the Angström method.

Session 1:
Measurements of the frequency response of the pyroelectric detector for incident light pulses – calibration.

Set up or verify that the experimental station has been assembled as shown in the lecture slides.
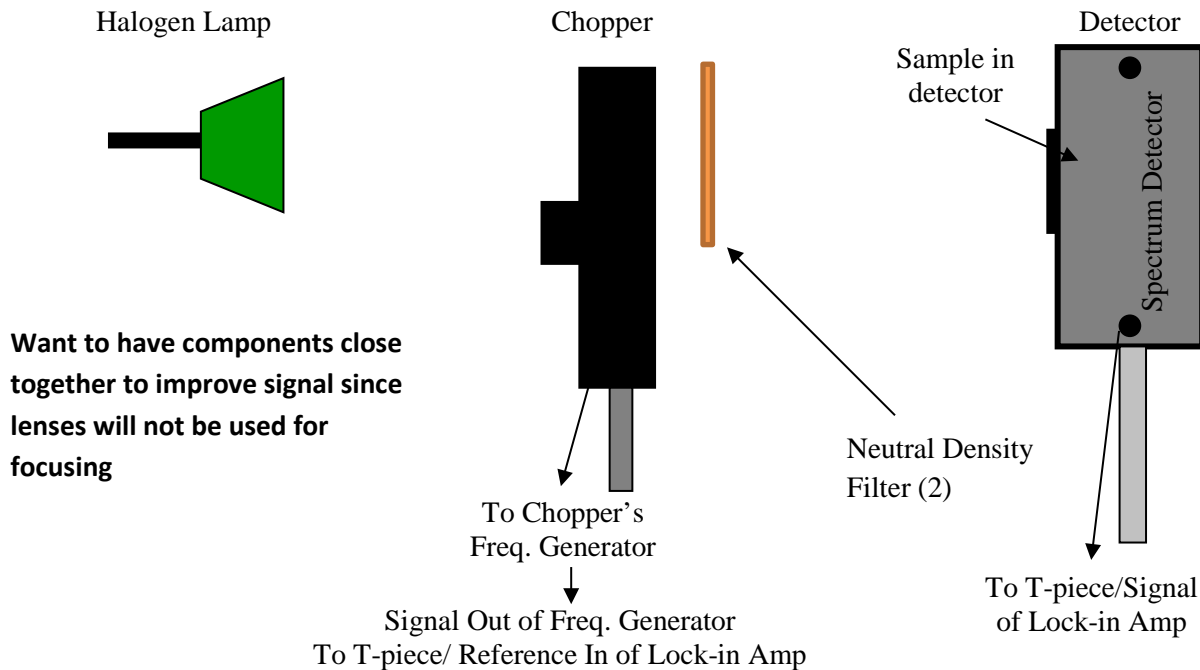
- Set up the filters in Signal Recovery Lock-in Amplifier Software for 1 – 500 Hz.

- The distance between the Halogen Lamp and the Chopper should be approximately 50 mm, while the distance between the Chopper and the detector is approximately 60-65 mm.

- Use the optical chopper to modulate the light coming from a halogen lamp that reaches the pyroelectric detector from continuous into light pulses.
  - Record **the amplitude** and **the phase shift** of the measured signal.
  - Plot those values as a function of the frequency.

The range of frequencies should be 4.5 to 100 Hz. When you collect the data think about spacing the data points by a constant interval (e.g. 5, 10). However, try to obtain as accurate a plot as possible. Thus collect at least 20 data points. In the high frequency limit, the amplitude should decrease as $1/f$ and the phase should be $-\pi/2$ radians. Conduct your measurements until the phase shift saturates with the frequency. Determine the plateau regime.

Note: It may be preferable to start with the graphite sample (as described below), and then calibrate the detector without a sample. This allows easier determination of the necessary frequency range.

Session 2:
Measurement of the thermal diffusivity of materials using the Flash Method

- Measure the thickness of the sample with a caliper (remember to write the accuracy of the caliper as ±)
- Install the sample in the window of the pyroelectric detector

- Determine the thermal diffusivity of two samples (graphite and one other) by illuminating one side of the sample with modulated light and measuring a phase shift between temperature-response signal, measured by the pyrometer and the reference signal:

*Ideally, good results should be obtained for low frequencies (e.g **12 – 20 Hz** for carbon-coated 0.9±0.1 mm thick steel disk). However due to the external noise, sometimes it might not be possible to measure a stable phase shift at low frequencies. If that happens, try to find a region with a stable phase shift (±**10 Hz**) at higher frequencies (e.g. ~ **300 Hz**). **Keep in mind that at high frequencies you must switch the fan and the frequency controller to high frequency regime and adjust the filters in Signal Recovery software.** In your report, try to answer why there is only "a window" of stable frequencies.*

Theory

The heat equation can be derived using Fourier's Law of Conduction ($\vec{J} = -\kappa\vec{\nabla}T$) and the conservation of energy. In one-dimensional heat flow, this simplifies to:

$$D\frac{\partial^2 T}{\partial x^2} - \frac{\partial T}{\partial t} = 0 \tag{1}$$

Where D is the diffusivity, which can be defined as the ratio between thermal conductivity (κ) and heat capacity (C)

$$D = \frac{\kappa}{C} \qquad (2)$$

The partial differential equation (1) can be turned into an ordinary differential equation by looking for periodic in time solutions. For this thermal diffusion lab, heat is hitting the sample on one side at $x = 0$ and traveling through until it remits at $x = d$, where d is the sample thickness. The temperature, therefore, can be written as

$$\tilde{T} = \left[\frac{J_0 d}{\kappa}\right]\left(\frac{1}{qd}\right)\left(\frac{1}{\sinh(qd)}\right) \qquad (3)$$

Where

$$q^2 = \frac{i\omega}{D} \qquad (4)$$

and d is the sample thickness, κ is the thermal conductivity, ω is the angular frequency and D is the material's thermal diffusivity. Note that this equation has both real and imaginary solutions since it includes the term $i\omega$.

For any given material's diffusivity, D, and thickness, d, there exists an angular frequency, ω, where the real and imaginary parts of the temperature equation are equal. This occurs when

$$|qd| = 2.22 \qquad (5)$$

Due to the pyroelectric detector's response, a calibration must be done where the detector's response to direct illumination is measured, followed by collection of data with the sample in front of the detector. Thus, the change in temperature is

$$\Delta T(\omega) \propto \frac{\Delta V(\omega)}{\Delta V_d(\omega)} \qquad (6)$$

where ΔV_d is the response from the direct illumination. As the voltage responses are complex, they can be written as

$$\frac{\Delta V(\omega)}{\Delta V_d(\omega)} = \frac{R * \exp(i\phi)}{R_d * \exp(i\phi_d)} \qquad (7)$$

$$\frac{\Delta V(\omega)}{\Delta V_d(\omega)} = \frac{R}{R_d}\exp\left(i(\phi - \phi_d)\right) \qquad (8)$$

where R is the voltage magnitude with the sample, R_d is the voltage magnitude under direct illumination (no sample), ϕ is the phase from the sample, and ϕ_d is the phase under direct illumination. The magnitude and the phase will be outputted by the lock-in amplifier for each frequency.

As the temperature equation is complex, the real and imaginary parts can be solved independently.

$$Re[\Delta T(\omega)] \propto \frac{R}{R_d}\cos(\phi - \phi_d) \qquad (9)$$

$$Im[\Delta T(\omega)] \propto \frac{R}{R_d}\sin(\phi - \phi_d) \qquad (10)$$

Plotting the absolute value of the real and imaginary parts of the temperature results in a crossover frequency where the values are equal. This crossover is the frequency where the response from the sample is 45° out of phase from the response under direct illumination. Once the crossover frequency has been determined experimentally, equations (4) and (5) can be used to determine the diffusivity.

Determining where the difference in phase is 45° is often more straightforward than trying to plot the real and imaginary components, and is an acceptable method for determining crossover frequency. In this case, plot the difference in phase angle as a function of chopper frequency, fit an equation to the line, and use this to determine the frequency with a 45° (or -45°) difference.

Measurement of the thermal diffusivity of a material via Angström method

Determine the thermal diffusivity of a sample (choose 2 of the 5 rods) by measuring the time of the heat transfer along the rod.

- **Make sure that the circuit is OFF**!
- Connect the thermocouple to the circuit board.
- Place the sample on a non-metal surface e.g. on the wooden table.
- Connect the heater attached to the sample to the power supply.

To avoid electrical shock, make sure that the sample or any other uninsulated wires connected to it does not touch the bread board, you or your colleague, or your precious TA.

- Start the Validyne Easy Sense software and set it according to the information sheet or instructions provided by TA
- Click the Start button in the Easy Sense program, type the name of your file and **WAIT**.
- Turn the heater ON and click SAVE in the Easy Sense program. The software will start measuring automatically. The data will be saved in a file that can be open via MS Excel.

DO NOT OPEN THE FILE BEFORE THE MEASUREMENT IS DONE! Otherwise the software will crash and you will lose you data.

- Conduct your measurement until temperature measured by the thermocouple increases ~10°C.

Fit two lines to your plot:

1) Before the heat reaches the thermocouple (flat line)
2) Afterwards, when the temperature at measured point increases (steep line)

The intersection of the lines will give you a time required for heat to be transferred from the heater to the thermocouple.

Diffusivity can be approximated from the following equation:

$$D = 0.15 \frac{L^2}{t}$$

Where L is the length of the rod, and t is the time required for heat transfer.

In your report

- In calibration part, show two plots: amplitude and phase shift as a function of the frequency.
- In the Flash Method part, also plot amplitude and phase shift as a function of frequency. Plot the real and imaginary parts of the temperature and use the crossover point to calculate diffusivity as described above. (Alternatively, plot the change in phase shift as a function of frequency, and determine where the phase difference is 45°. Use this frequency to calculate D.) Compare the obtained results with literature values and comment on the result.
- In the Angstrom Method, show plots of temperature as function of time, determine the time for heat transfer and the diffusivity. Comment on the results.

Remember to conduct error analysis!

Compare measured diffusivities and methods. Based on your results and collected knowledge from the literature, analyze which material for what thermal applications is better and why. Make sure to draw a final conclusion i.e. "a take home message".