Time-Resolved Temperature Measurement Device for Pulsed Laser Irradiation

by

Bilal Iqbal
Email: iqbalb@mcmaster.ca
Materials Science and Engineering
McMaster University

Date: Aug 12th, 2005

Supervisors
Dr. J. D. Embury
F. Lefevre-Schlick

ABSTRACT

This report presents the design of a time-resolved temperature measurement device that is used to measure the change in resistance of a Pt sample while it is being irradiated by laser pulse. The results are then interpreted to estimate the temperature reached by the sample. The device was initially designed to make nanosecond resolved resistance measurements but later improved to make picosecond and femtosecond resolved measurements.

Several resistors of Pt with variable length and thickness are deposited on a substrate and each resistor is connected to the circuit and irradiated one by one. During irradiation, changes in resistivity are recorded and changes in temperature are deducted from temperature. Details regarding the design of the circuit and shape of Pt sample to make accurate time resolved measurements are discussed in this paper.

This first part of experiment is essentially a reproduced version (with slight modifications) of a similar experiment conducted by J. A. Kittl, M. J. Aziz, D. P. Brunco and M. O. Thompson. [1]

ACKNOWLEDGEMENTS

I would like to thank my supervisors, Dr. J. D. Embury and F. Lefevre-Schlick, for giving me an exciting and equally challenging opportunity to work on this project. They continually encouraged and supported me throughout the life of this project.

I would also like to mention Dr. O. Marinov who took a special interest in this project and spent numerous hours discussing and suggesting possible designs for the device. I also thank Dr. J. Deen and Dr. Y. Haddara for their direction, guidance and assistance.

In addition, I would like to extend my deepest appreciation to the following:

- Photonics Group
  - Dr. Preston, Dr. Haugen, H. Tiedje
- Center for Electrophotonic Materials and Devices (CEMD) Group
  - Dr. Mascher, D. Stevanovic, D. Bruce

This project wouldn’t have been possible without the help of all these people.

TABLE OF CONTENTS

1 PROBLEM STATEMENT

To design a device that is able to measure fast changes in resistance of a platinum resistor during irradiation by ultrafast laser pulse.

2 OBJECTIVES

- To reproduce an existing nanosecond resolved temperature measurement device with slight modifications.
- To design a new device which allows measurement of changes caused by picosecond and femtosecond laser pulses.
- To design a sample resistor for laser irradiation which allows uniform distribution of heat, energy and avoids quantum effects.

3 INTRODUCTION

Since a decade, there had been an increasing interest in rapid heat treatments of metals, both from a scientific point of view (i.e., to study first stage of nucleation events such as phase transformation and recrystallization) and from a practical point of view to produce very fine scale materials or new phases showing new mechanical properties. When a material is exposed to laser irradiation, the surface of the material is rapidly heated and subsequently self-quenched by conduction (at rates up to 10^{12} K/s). Time-resolved temperature measurement then becomes crucial in order to gain information about the localized time/temperature profile during the laser heat treatment.

This experiment is conducted to accurately determine the temperature changes during a pulsed laser irradiation on a sample. The results obtained are then used in studying nucleation events for recrystallization in Cu and Fe and phase transitions in steels. The sample used in the experiment is prepared very carefully to obtain a uniform distribution of heat and energy throughout it’s mass.

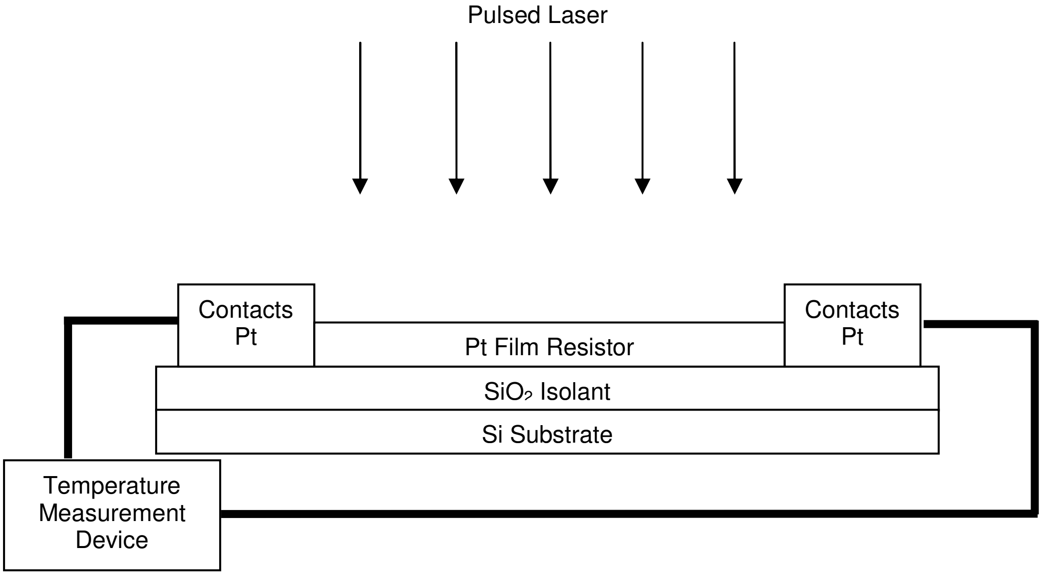


Figure 1: Device Structure

4 DEVICE DESIGN

The temperature measurement device has been developed in two stages;

- Reproduction of an existing circuit with slight design modifications.
- Preparation of the new circuit.

Both circuits follow the same basic principles but they differ in their functionality in terms of sensitivity. The reproduced circuit is able to measure changes caused by nanosecond laser pulses whereas the new circuit is designed keeping into consideration the use of picosecond and femtosecond laser pulses, which will induce changes in temperature in a time frame of the order of 10-12 seconds (phonon-phonon interaction in a solid).

Two main aspects are taken into account for the design of this device:

- **Sensitivity**

  Sensitivity is the ability of the circuit to sense a signal. The sensitivity should be very high for circuits dealing with measurements on a scale of nanoseconds and less.
- **Matching**

  Matching is the ability of load resistance (impedance) to totally eliminate the signal produced by the source once it completes one circulation. Matching is important because it eliminates rebounding signals. Hence an accurate result is obtained without repetition of the same signal.

There is usually a trade-off between sensitivity and matching so a balance needs to be maintained between the two.

4.1 General Device Architecture

The circuits for this device are based on a transmission line configuration because conventional resistance measurement circuits with constant voltage and current sources cannot be used when high speed measurements are involved. The maximum power transfer in a transmission line is obtained when the source and load impedances are the same. So the signals generated by the source and transferred through the source impedance are terminated by the load impedance completely. Hence, the same signal is not repeated over and over. The transmission line configuration is as follows:

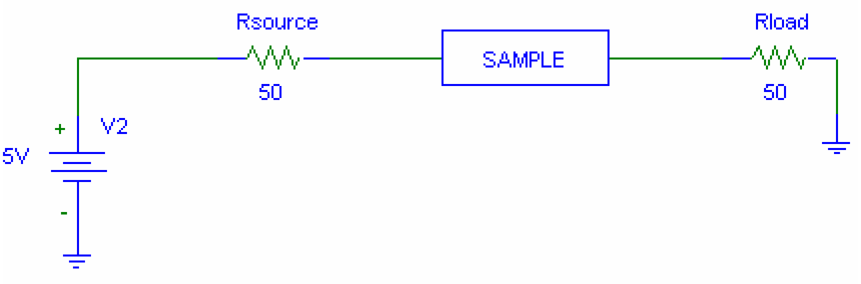


Figure 2: General Device Architecture

Therefore, maximum power can be achieved only when:

$$R_{source} = R_{load}$$

In this case, R_{source} and R_{load} have a resistance of 50 ohms each and hence this circuit will be able to prevent rebounding signals showing high matching. One of the standard impedances in transmission lines is 50 ohms and therefore, all the circuits for temperature measurement device are designed with 50 ohm source and load impedances in this project.

4.2 Reproduction of Existing Circuit

This circuit was initially designed by Aziz's Group at Harvard University. It has been slightly modified to provide more precise measurements. This circuit makes use of a band pass filter to transfer appropriate signals to the next phase. The circuit diagram is as follows:

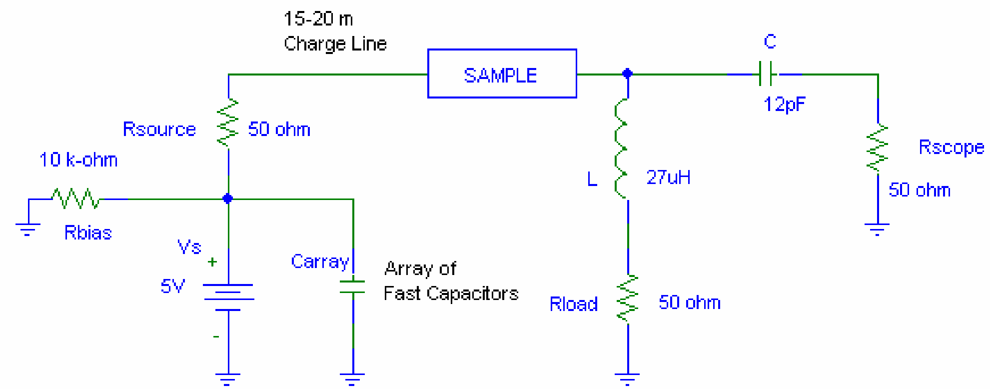


Figure 3: Reproduced Circuit

4.2.1 Circuit Principle

Circuit in Figure 3 is composed of three major blocks described below:

- Source
- Sample
- Load

Source

This block consists of a constant voltage source connected in parallel with an array of fast capacitors and a source impedance. Due to rapidly changing current at the sample resistor because of laser irradiation, the constant voltage source is unable to respond and hence fast capacitors are used to provide required current. In other words, capacitors are used to reduce the drop in voltage caused by rapid change in current when the sample resistor is irradiated with pulsed laser.

As discussed above, constant power supply is unable to respond to sub-microsecond time scales, a 15 – 20 meter charge line is used to isolate the source from the sample. This isolation causes a delay in propagation of the signal. The delay caused by the isolation is given by:

$$\nabla t = 2L / (2/3c) \qquad \textit{(where c = speed of light)}$$

The current in the sample resistor changes rapidly due to the change in resistance but the dispersion of current in the charge line reduces the dI/dt and relaxes the source bias requirements.

The 10 kilo-ohm resistor connected in parallel to the voltage source and the capacitors is used to create a potential difference so a voltmeter can be connected to measure the voltage.

Sample

This block contains the sample resistor which is irradiated by the laser pulse. This circuit is designed with a sample resistor of 100 ohms. The material for sample resistor must have good resistivity changes with changes in temperature such as the ones exhibited by platinum. Different properties of sample resistor are controlled by its geometry which are discussed in detail in the next section.

Load

This block is where the oscilloscope is connected for analysis of the given signal. As the measurements of interest are on the scale of nanoseconds, only high frequency signals needs to be processed by the scope. Therefore, a band pass filter is used which discards all frequencies between 295KHz and 265MHz.

Band Pass Filter

Ideally, the collective resistance of inductor (L) and resistance (R_{load}) should be 50 ohms but in practice, it is a little off. Assuming a negligible deviation, the low cut-off frequency is calculated as follows:

Inductor	L	27 μH
Resistor	R_{load}	50 ohm
Time Constant	$T = L/R$	5.40E-07
Cut-off Frequency	$1/2\pi T$	295 KHz

The high cut-off frequency is calculated as follows:

Capacitor	L	12pF
Resistor	R_{load}	50 ohm
Time Constant	$T = RC$	6.00E-10
Cut-off Frequency	$1/2\pi T$	265 MHz

For 30 ns laser pulse, the frequencies of interest will be around 300Mhz and above. Therefore, this circuit only processes the signals with frequencies higher than 265 MHz into the oscilloscope. All other signals are sent to the load impedance and terminated into the ground.

From the above calculations, it can be seen that the values are loosely calculated and will have no effect on the measurements as long as the frequencies of interest are passed into the oscilloscope.

4.2.2 Implementation

This circuit has been implemented on a piece of copper by restraining conductivity within small boxes and connecting them by means of components. Figure is shown below:

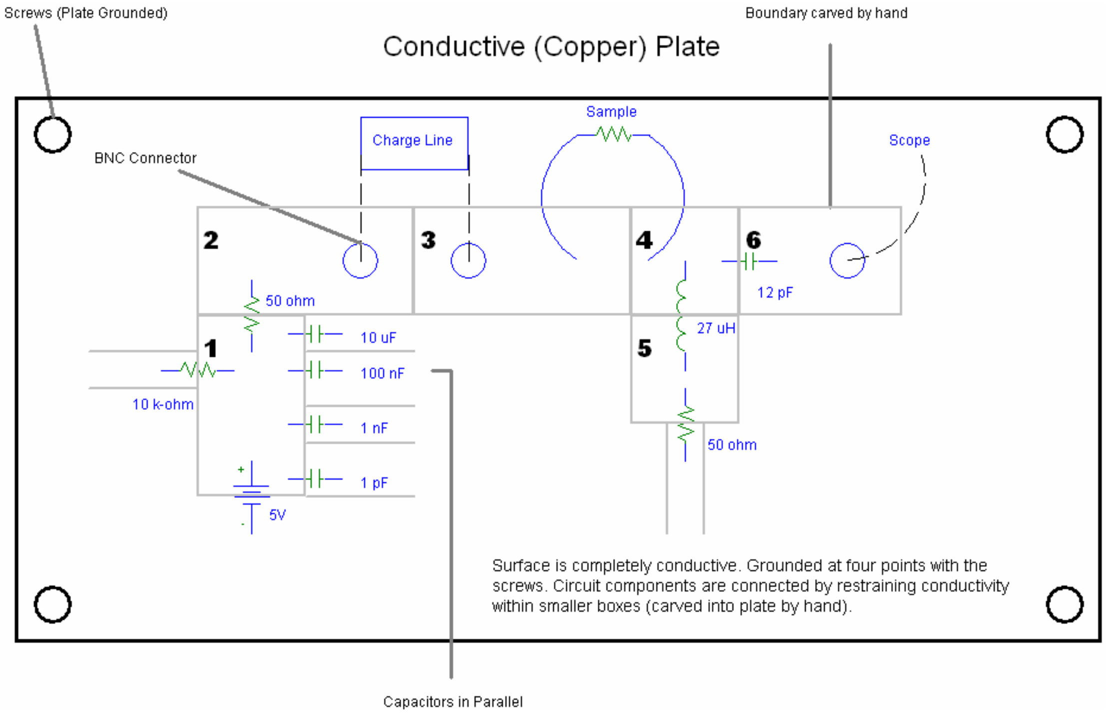


Figure 4: Circuit Implementation on Copper Plate

Conductivity is restrained within smaller boxes by scratching away the copper from their boundaries. Components are then soldered between the blocks.

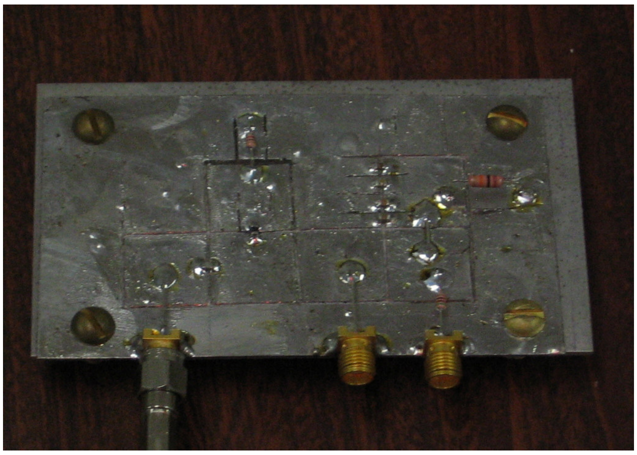


Figure 4: Nanosecond Resolved Temperature Measurement Device

4.3 Design of New Circuit

This circuit was designed with the goal to improve the existing circuit so that smaller changes in resistance (caused by picosecond and femtosecond laser pulses) can be measured. The circuit was proposed by Dr. O. Marinov and it was modified at several stages to accommodate additional features. The circuit is similar to a very basic transmission line configuration. Another feature of this circuit is that the sensitivity and matching can be varied by slightly modifying the circuit. This circuit has been designed in such a way that high, moderate and low levels of sensitivity and matching can be obtained and later, results can be compared to find a more accurate set of measurements. The circuit diagrams for each setting are as follows:

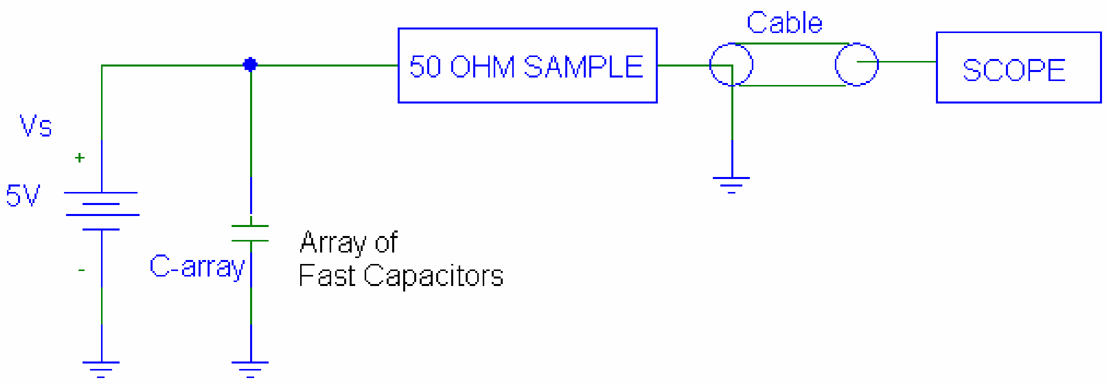


Figure 5(a): New Circuit – Setting 1

Figure 5(a) shows the first setting of the circuit which exhibits high sensitivity but low matching.

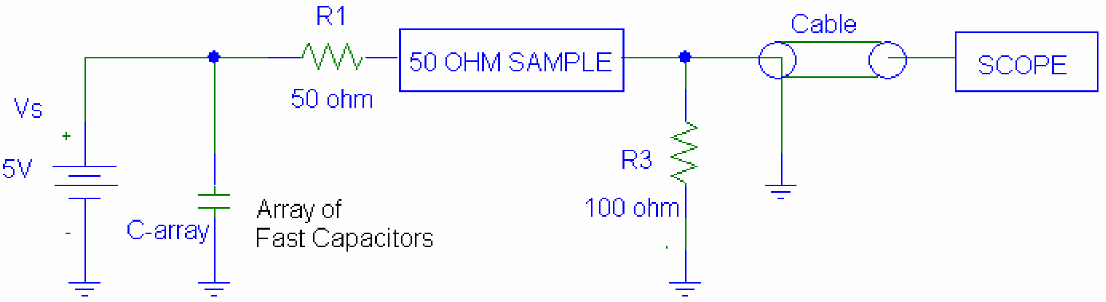


Figure 5(b): New Circuit – Setting 2

Setting 2 for new circuit in figure 5(b) has high matching but sensitivity is relatively low.

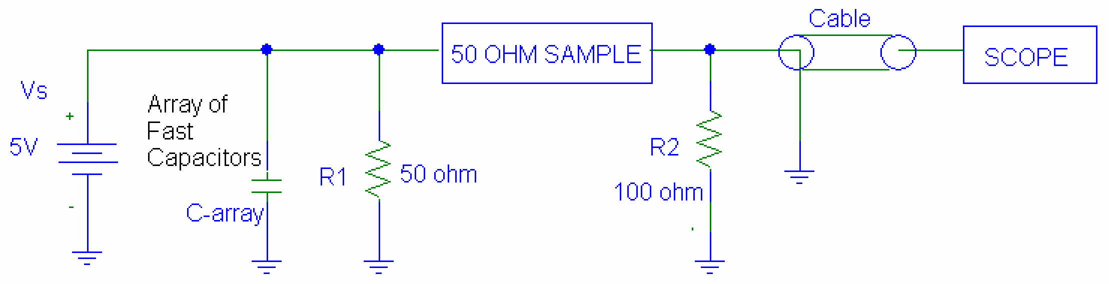


Figure 5(c): New Circuit – Setting 3

Circuit in figure 5(c) has moderate matching and sensitivity.

4.3.1 Circuit Principle

The circuit is based on transmission line configuration. It has a battery source connected in parallel with an array of fast capacitors (100pF, 1nF, 100nF and 10μF). These capacitors are used to deal with the fast changing current (dI/dt). With the laser irradiation, the resistance of the sample resistor changes rapidly causing a fast change in current and constant voltage source cannot be used to deal with this change without fast capacitors. In other words, these capacitors are used to reduce the drop in voltage making it possible to make fast measurements.

Once the sensitivity aspect of the circuit is taken care of, the circuit should be matched. The transmission line provides maximum matching when the source and load impedances are equal. In this circuit, there is a trade-off between matching and sensitivity. As the matching is increased, the sensitivity of the circuit starts to drop because the current through the capacitors has to go through the source impedance which causes a decrease in sensitivity.

To deal with the problem of sensitivity and matching trade-off, the three settings of the circuit works as follows:

4.3.2 Setting 1 – High Sensitivity, Low Matching

This setting has highest level of sensitivity because the current through the capacitors is passed into the sample resistor without passing through any source impedance. On the other hand, it has low matching because there is no source impedance or load impedance. There will be rebounding signals in this setting causing repetition in measurements. However in practice, the signal will eventually die down due to internal resistance / impedance of the circuit with passage of time.

4.3.3 Setting 2 – Moderate Sensitivity, High Matching

As discussed above, sensitivity is provided by the fast array of capacitors connected in parallel to the voltage source. These capacitors fulfils the requirement of fast changing current due to change in resistance of sample resistor which is caused by pulsed laser irradiation. Setting 2 has relatively low sensitivity because there is a 50 ohm source impedance and all the current passes through it. This impedance causes a reduction in sensitivity of the circuit. On the other hand, the circuit has high matching because equal source and load impedances are used. It can be seen in Figure 5(b) that the combined resistance of the circuit from the source point of view or the load point of view provides the same resistance i.e., 50 ohms.

4.3.4 Setting 3 – Moderate Sensitivity, Moderate Matching

This setting of the circuit is very similar to setting 2 except for the fact that the 50 ohm resistor is connected in parallel with the capacitors and the voltage source rather than in series. In this way, the sensitivity of the circuit has been increased because the impedance to the current is less than setting 2. On the other hand, matching of the circuit has also been somewhat preserved. Theoretically, for matching calculations, resistors in parallel can be considered to be in series but in practice, it has some impact on the matching. Hence, this setting provides low to moderate matching.

4.3.5 PCB Fabrication & Implementation

Instead of fabricating a separate schematic for each setting, a schematic was designed to accommodate all three settings. It was designed in a way that the circuit can easily be modified to switch between settings. For this purpose, jumpers/switches are used and hence, it is possible to switch between settings by simply connecting or removing the jumpers.

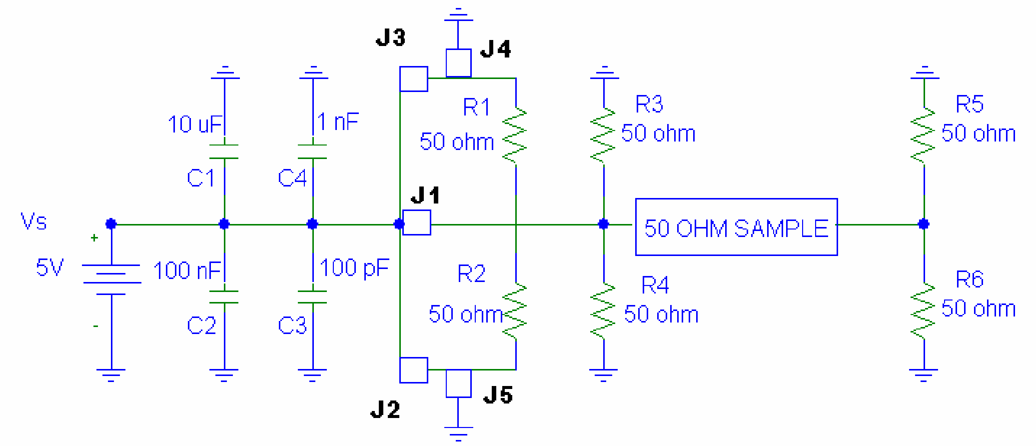


Figure 6: New Circuit – Schematic

It is possible to switch between different settings using the following approach:

Setting 1: This setting only requires the use of voltage source, capacitors and the sample resistor. So R1, R2, R3 and R4 should not be soldered at all and J1 (Jumper/Switch) should be closed with all other switches (J2, J3, J4 and J5) open. This will cause the current to flow directly to the sample resistor.

Setting 2: This setting requires the use of voltage source, capacitors, the sample resistor and all other resistors. Therefore, only J3 and J2 needs to be closed to use setting 2.

Setting 3: This setting requires the use of voltage source, capacitors, the sample resistor and other resistors but 50 ohms needs to be in parallel. Therefore, only J1, J4 and J5 needs to be closed.

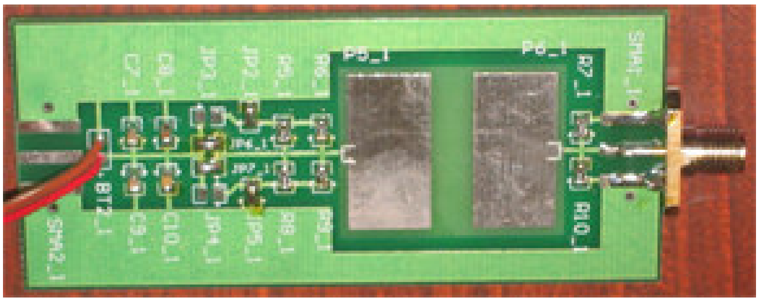


Figure 7: New Circuit (with Setting 3)

5 SAMPLE RESISTOR

The time-resolved temperature measurement device has been developed to measure the changes in resistance of the metals of interest so their properties can be further explored. The metals which are of special interest to this group are iron, copper and steels. The properties exhibited by platinum are very similar to these metals and it also shows good resistivity changes with the change in temperature which is a required property for this device. It makes platinum a very good candidate for experimentation and hence, it is used as the material for the sample resistor.

Apart from having good resistivity changes, there should also be uniform distribution of heat and energy so that the measurements are homogeneous across the resistor. This can be controlled by designing the resistor with a specific geometry. Moreover, experiments must be conducted on several thicknesses of platinum film to determine a specific trend of the results obtained.

The sample then needs to be connected to the circuit by means of some connection pads. To have accurate measurements, these pads should have ideally no resistance which in practice can be made negligible if a proper geometry for the connection pads is selected.

5.1 Constraints

Following are some of the constraints on the design of the sample resistor:

Uniform Heat and Energy Distribution:

The heat and energy should be transferred uniformly on the whole surface and volume of the platinum film. To ensure this, the sample resistor has been designed while keeping into account the following limitations:

- *Length of Pt film < 100 um* – Ensures uniform distribution of energy from the laser spot.

- *Thickness of Pt film < 150 nm* – Ensures homogenous depth of penetration of heat.

Quantum Effects:

It has been observed that for a film of below 10 nm, the resistivity of metallic films increases over that given by the bulk resistivity of the material.[3] This effect is called quantum effect. To avoid it, the thickness of the film has to be greater than 10 nm. To leave some cushion space, the minimum thickness of Pt film for the sample resistor in this experiment is chosen to be 20 nm.

Circuit-specific Constraints:

When the circuits are designed, they are made to work with a specific resistance. For this purpose, the sample resistor must be designed with that specific resistance for each circuit. The resistance used in the reproduced circuit is 100 ohms whereas the one used in the new circuit is 50 ohms.

Practical Limitations:

There are some practical limitations in the design of such a sample resistor. The resistor's dimensions are critical and extremely small which makes the mask design and fabrication hard. Here are some of the practical constraints:

- *Dimensions > 1 μm* – Maximum resolution of e-Beam used for mask design and fabrication process.
- Contact pads / Connection Pads always have some resistance, though, it can be minimized by proper selection of geometry.

5.2 Calculations

There are a number of calculations to determine different dimensions for the shape of the resistor. All these calculations can be done using the following relation once the constraints have been identified:

$$\mathbf{R = \rho\, L/A = \rho\, L/\,(t * w)}$$

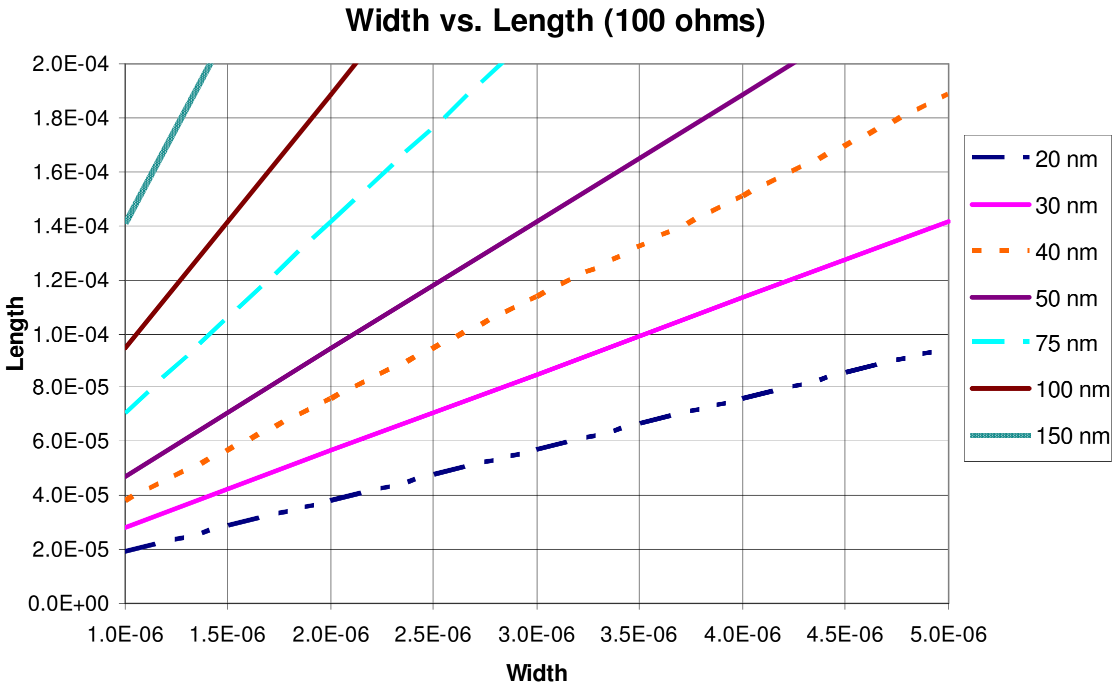
where R is the resistance, ρ is the resistivity, L is the length, t is the thickness and w is the width

5.2.1 Dimensions of the Platinum Resistor

Following is the graph that was used to determine different possibilities for length and width of the platinum sample.

Data that has been used to calculate these values is as follows:

Resistance = R = 100 ohms
Resistivity = ρ = 1.06E-07 ohm-meter



Graph 1: Platinum film - Width vs. Length

As it is costly to develop separate design masks for each resistance, a method was devised to use the same mask for different resistances. It was derived from the following relation:

$$\mathbf{R = \rho L / (t * w) = K/t}$$

$$\text{where } K = \rho L / w$$

It can be seen that the resistance is inversely related to the thickness. So if the thickness is doubled, the resistance reduces to half. The same principle has been used to produce resistors with 50 ohm resistance.

So the dimensions for the platinum film have been determined by fixing the thickness, setting the width in a specific range (from 1.0 μm to 2.5 μm) and finding the length to obtain a particular resistance.

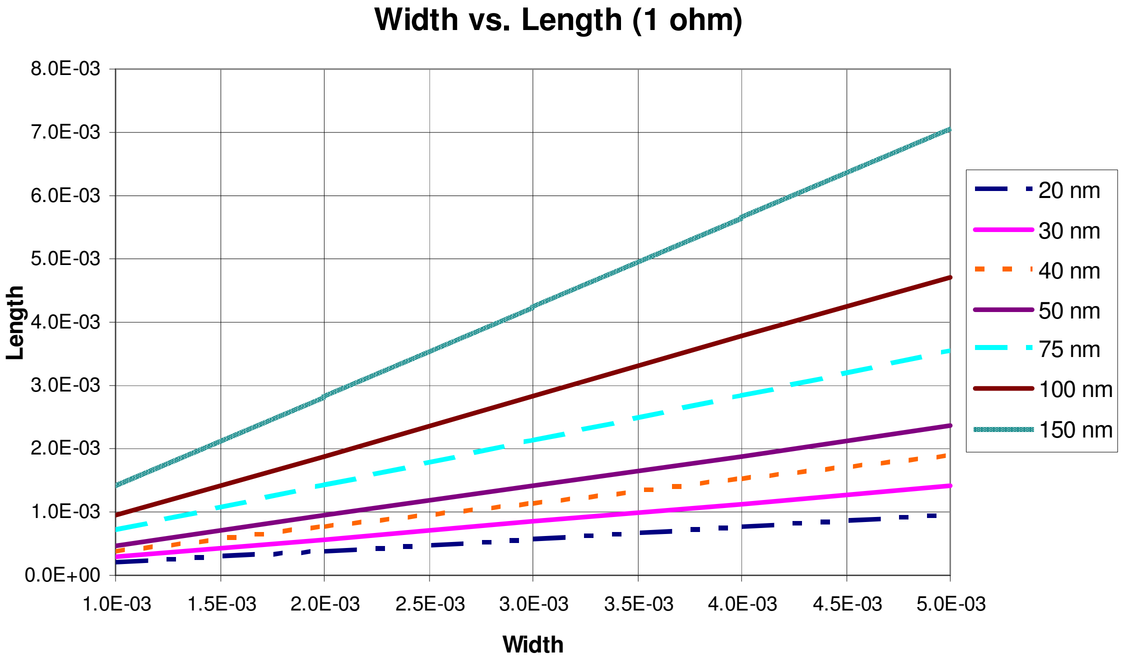
5.2.2 Dimensions of Connection Pads

Connection pads are used to connect the resistor to the circuit but it is required that these pads do not interfere with the measurements. Ideally, they should have no resistance which is impossible in practice. Hence, the dimensions have to be chosen in a way that their resistance is minimized and can be considered as negligible.

The following graph shows a number of different possibilities for length and width of the connection pads which makes the resistance of the sample negligible i.e., less than 5 ohms (less than 5% for 100 ohms resistor and less than 10% for 50 ohms resistor).

The data used to calculate the values is as follows:

Resistance = R = 1 ohms
Resistivity = ρ = 1.06E-07 ohm-meter



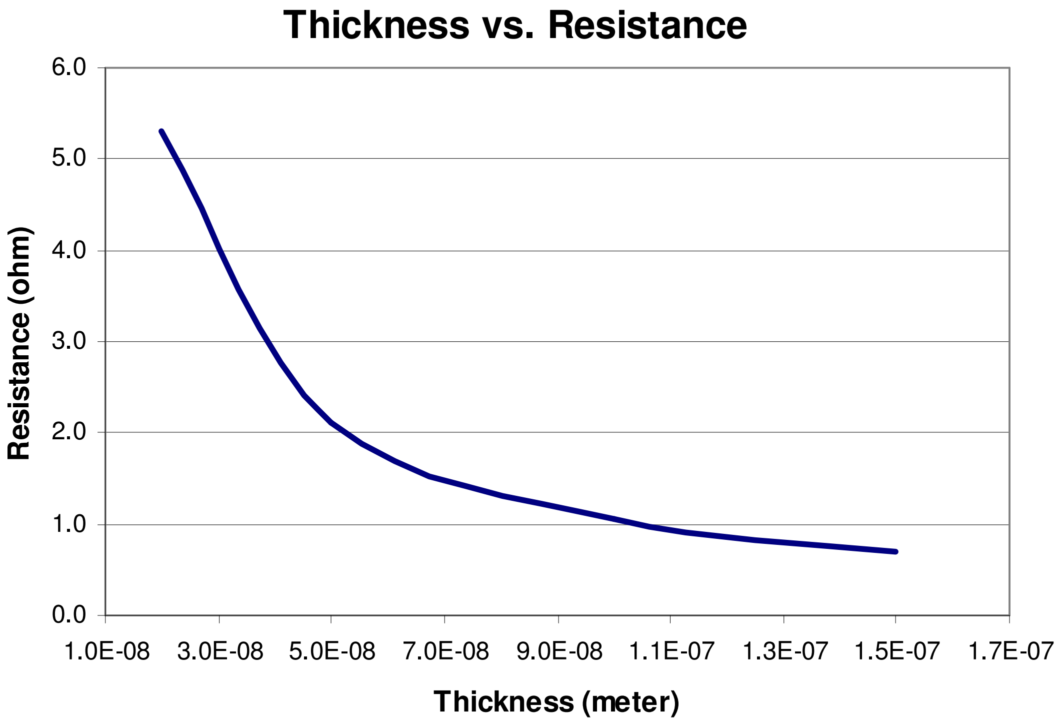
Graph 2: Connection Pads - Width vs. Length

The following table provides different possible resistances for the connection pads when the dimensions are fixed to be 2 x 2 mm and the thickness is varied from 20 nm to 150 nm.

Resistivity (ohm-meter)	1.06E-07
Length (meter)	2.00E-03
Width (meter)	2.00E-03

Thickness (meter)	**Resistance (ohm)**
2.00E-08	5.30
5.00E-08	2.12
1.00E-07	1.06
1.50E-07	0.71

The following graph is obtained when the values in the given table are plotted:



Graph 3: Connection Pads - Thickness vs. Resistance

5.3 Shape of the Resistor

The resistor has been given a shape such that a platinum film with required resistance (50 ohm or 100 ohm) is connected to square shaped connection pads at both ends. The wide square shape of connection pads make their resistance negligible as compared to platinum film resistance.

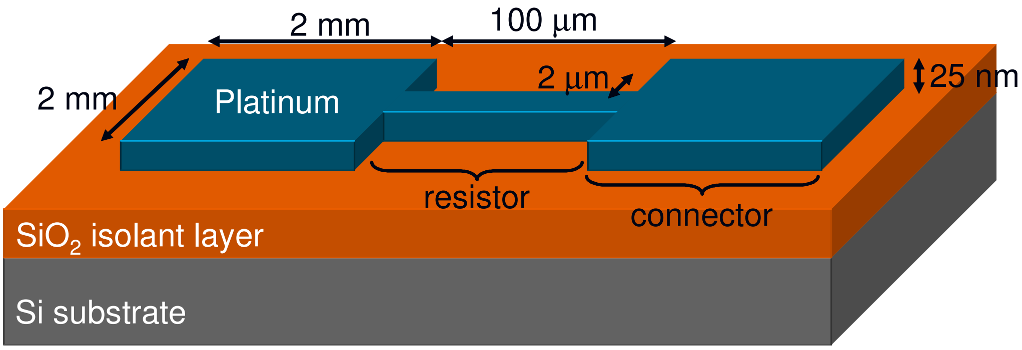


Figure 8: Shape of Sample Resistor

It has been found that about four resistors can be placed on a substrate of 2 x 2 cm. The substrate used in this experiment is a silicon wafer which has been oxidized to avoid diffusion of Pt in Si and electrical conductivity.

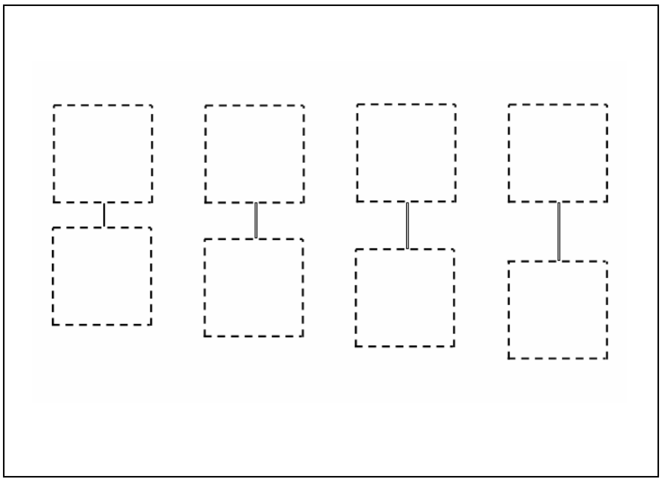


Figure 9: Block design with four resistors

5.4 Design Mask

A design mask was prepared after finalizing the geometries of the sample resistor. A number of samples for the resistors with different thicknesses were designed.

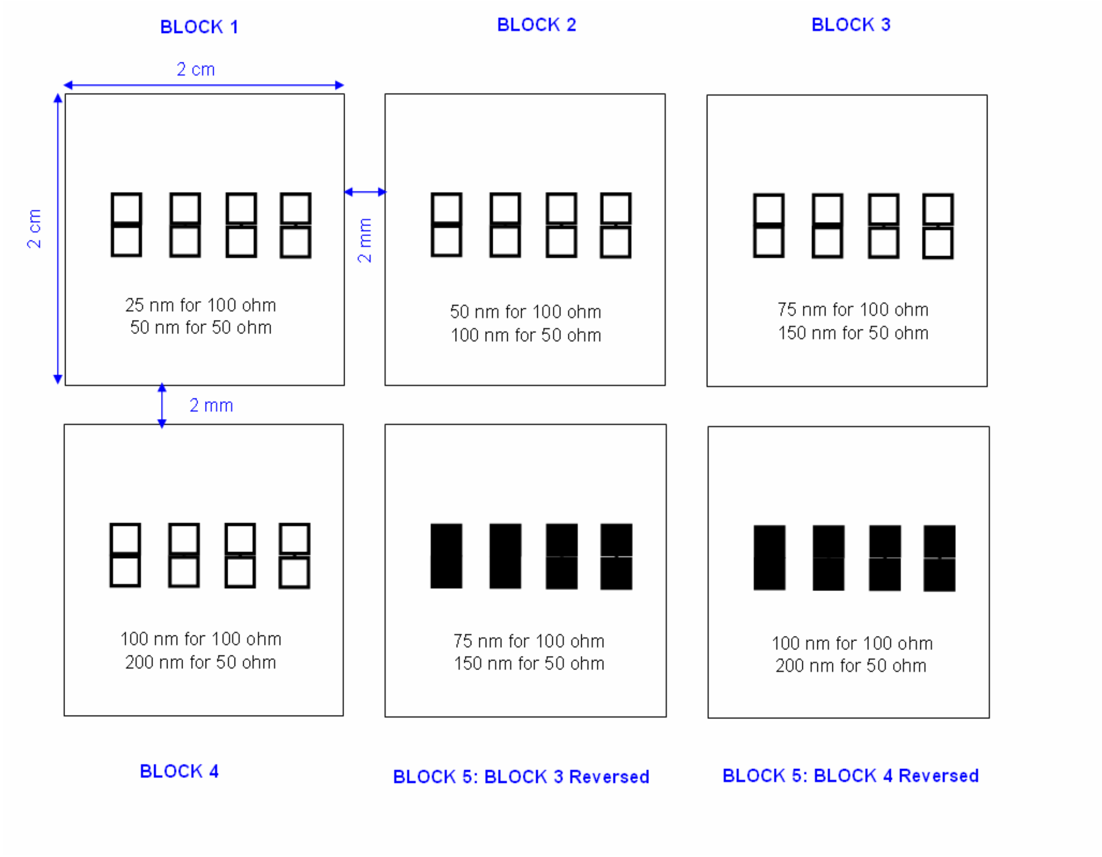


Figure 10: Design Mask

The design mask shown in the above figure has six 2 x 2 cm blocks with 4 resistors in each block. The thickness for the resistors in each block (from block 1 to block 4) is different. For example, in order to get a resistance of 100 ohms in block 1, the thickness is 25nm and it is 50nm for 50 ohms resistance. Similarly, thickness of 50 nm in block 2 gives a resistance of 100 ohms whereas 100 nm in the same block gives a resistance of 50 ohms.

The design mask has been prepared for Chemical Vapour Deposition (CVD) process. The process is used to deposit platinum on a substrate using the design mask. The thickness can be varied during the CVD process. First four blocks are designed for the deposition technique known as lift-off (creates patterns on the wafer surface through additive processes) whereas Block 5 and Block 6 (which are same as Block 3 and Block 4 respectively) are designed to use with technique called etching (creates patterns on the wafer surface through subtractive processes).

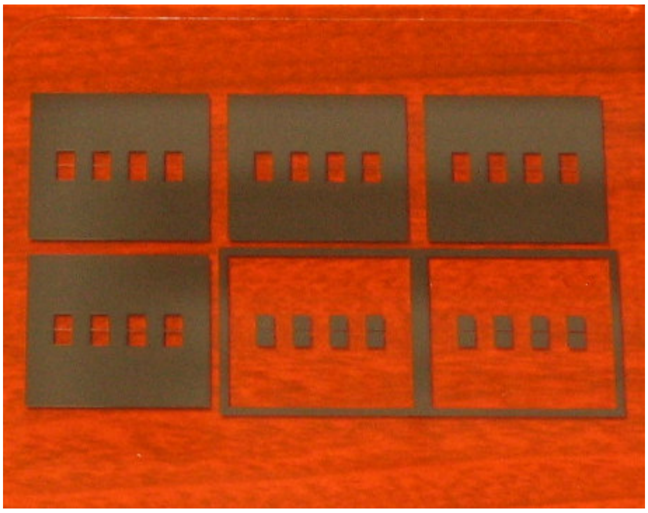


Figure 11: Picture of the mask

6 CONCLUSION

The circuits for time-resolved temperature measurements were carefully designed and implemented keeping into account all the possible constraints. These constraints range from electrical principles to the geometry of the sample resistor. Firstly, the sample resistor will be calibrated in a furnace to get the temperature/resistivity profile. It has been planned to run a series of experiments on nanosecond, picosecond and femtosecond laser pulses. The resistor will then be connected to the temperature measurement device and irradiated with the pulsed laser. The changes in resistance will be recorded with the help of an oscilloscope and then the changes in temperature will be deducted from the calibration. The results anticipated for nanosecond laser pulse irradiation are shown in the figure. [1]

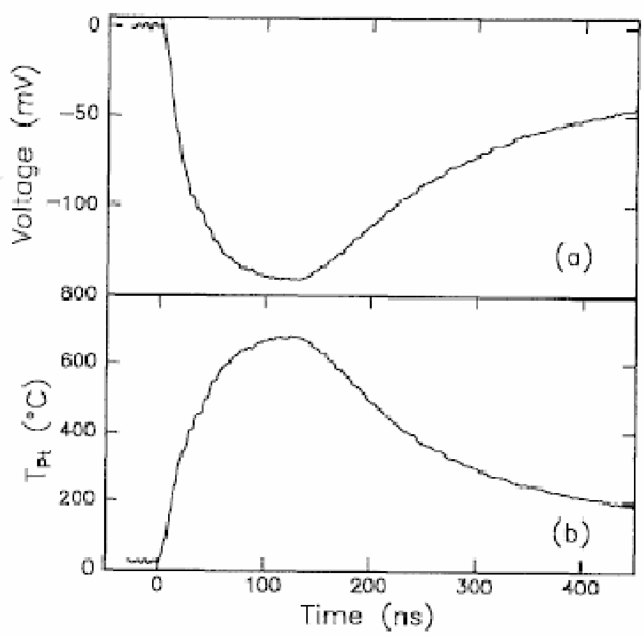


Figure 12: Anticipated Graph for Nanosecond Laser Pulse Irradiation (taken from Aziz's research paper on time-resolved temperature measurement)

7 REFERENCES

1. *D. P. Brunco, J. A. Kittl, C. E. Otis, P. M. Goodwin, M. O. Thompson and M. J. Aziz,* Time-resolved temperature measurements during pulsed laser irradiation using film metal thermometers (1993)
2. *J. A. Kittl, R. Reitano, M. J. Aziz, D. P. Brunco and M. O. Thompson,* Time-resolved temperature measurements during rapid solidification of Si-As alloys induced by pulsed-laser melting (1992)
3. *M. Avrekh, O. R. Monterio, I. G. Brown,* Electrical resistivity of vacuum-arc-deposited platinum thin films (1999)