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Project Report

# Instrumentation of AC Susceptometer

MSc Semester 4

Unit 512: Project Work

***Ved Rudani*** Roll number: **54**
***Vijay Panchal*** Roll number: **55**

Under the the guidance of:

**Mr. D. B. Patel**

**Dr. U. S. Joshi**

Department of Physics, Electronics and Space Sciences, Gujarat University, Ahmedabad,380009

# Abstract

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# 1. Introduction

This is our master's semester four project report. We have done work on instrumentation for finding magnetic susceptibility of unknown samples. Our final goal is to find phase transitions of samples under change of temperature. Which we didn't do in this project, but will remain for future. In this project report we talked about proper use cases of AC susceptometer and then instrumentation in Experimental setup. We used Primary and Secondary coil setup, thus exploiting mutual inductance to find remaining magnetization of sample under AC susceptometer. Main coils are connected to SR830 LOCK IN amplifiers. Proper Scripts and instrumentation regarding data logging and computer setup is done in our semester 3 project[11][10]. You can visit my github page at https://github.com/vijaypanchalr3/pyinstro and https://github.com/vijaypanchalr3/shotnoise for python code regarding it. We have took extensive help from (Laurent, 2008)[7], (miga, 2007)[8], (bajpai, 1997)[1]. Also, articles by (goldfarb,1986)[6], (chakravarty, 2024)[2] are very useful.

# 2. Why AC susceptometer?

It is better to first address the need of The AC susceptometer, in fact susceptometer in general. Susceptomter is an instrument which measures magnetization of sample respect applied to the field. The need of a susceptometer starts with the importance of magnetic properties of material. Magnetic properties are very important in classification of materials and most importantly its behavior in certain conditions. We will see why susceptometry is important by looking at these two cases.

## 2.1. Classification of material by its magnetic properties

Certainly the importance of classification in fields such as material science, chemistry, geology etc is very important. This classification is done by looking at its magnetization properties. For lay people, certain materials such as iron, nickel are very attractive to magnets but certain materials such as water, mercury are repelled by it. These properties can be deeply understood by looking at its atomic structure and some knowledge of quantum mechanical concepts such as spins of electrons and magnetic moments. By studying magnetic properties which can classify material in certain broad categories.

### 2.1.1. Paramagnetic materials

Most chemical elements and certain compounds show very little magnetization in direction of applied magnetic fields; this material is called paramagnetic materials. This material has very small but positive magnetic susceptibility. Their relative permeability is slightly greater than 1. Paramagnetism is due to unpaired electrons in the materials, so most elements with incomplete orbits have this property. There are theories which try to understand magnetism.

- Curie's law: Paramagnets approximately follow this law which says that magnetism is inversely proportional to its temperature $\chi \propto \frac{1}{T}$.

- Pauli paramagnetism: For some alkali metals and noble metals, conduction electrons are weakly interacting and delocalized in space forming a Fermi gas. For these materials one contribution to the magnetic response comes from the interaction between the electron spins and the magnetic field known as Pauli paramagnetism. [5]

The Bohr–Van Leeuwen theorem proves that there cannot be any diamagnetism or para-

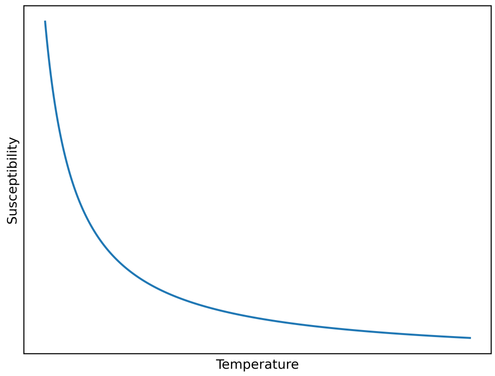


Figure 1: Curie's law which shows inverse temperature relationship

magnetism in a purely classical system.[5]

### 2.1.2. Diamagnetic materials

Diamagnetic materials have opposite magnetization to applied fields. Thus they are repelled by magnets. Diamagnetic properties always exist in material but when compared with strong magnetic properties of the same material it is negligible. They have negative magnetic susceptibility and are very negligible. Superconductors also show diamagnetism, which have very high but negative susceptibility, $\chi \approx -1$ (in SI units). This can be understood by the Meissner effect. Certain theories are made for understanding diamagnetism. They are Lengevin's theory and Landau's theory which we will not go deeper for now.[3]

### 2.1.3. Ferromagnetic materials

Ferromagnetic materials have by far the most visible magnetic effect compared to diamagnetic and paramagnetic materials. These materials are highly attracted to applied magnetic fields, thus having high magnetic susceptibility. Compared to paramagnets and diamagnets, ferromagnets' magnetic susceptibility is easily measured at some degree of accuracy because of its high value. In our project we are focusing on ferromagnets and their phase transitions because of noise factors. Some examples of ferromagnets are iron, nickel, cobalt, LSMO etc.. There's similar classification for ferromagnetic and antiferromagnetic materials, the difference being how spins are aligning. [4]

## 2.2. Magnetic phase transition

By data of magnetic susceptibility we can study magnetic phase transition. With temperature change magnetic properties such as paramagnetism, diamagnetism etc. changes by changing its magnetization property. This is an almost sudden change with temperature. With studying susceptibility data we can pinpoint which temperature change has occurred. This temperature is called Curie Temperature.

Certain material types have unique phase transition properties. For example, Superconductors are almost perfect diamagnet at their superconducting state and almost none magnetic(for most of the compound superconductors) after critical temperature $T_c$. You can see certain phase relationships in figure 2[9].

## 2.3. AC susceptibility vs DC susceptibility

We can measure susceptibility values in two different ways. First is after applying a DC magnetic field and second is after applying AC magnetic field. Difference is first you will measure $\chi_{DC} = \frac{M}{H}$ where second $\chi_{AC} = \frac{dM}{dH}$. AC magnetic fields will generate loss terms because of changing magnetic fields of the sample.

$$\chi = \chi' + \chi''$$

AC susceptibility will depend on frequency of applied field. Also, if we can measure phase change of applied magnetic field and sample magnetic field, we will see frequency dependent relationships. For example look at figure 3.

After knowing the importance of why it is important to measure magnetic susceptibility we will devise instrumentation techniques to measure magnetic susceptibility at given temperature and

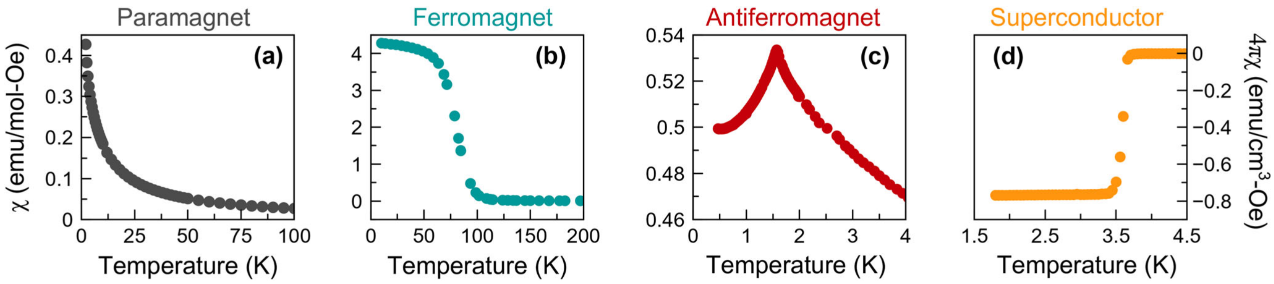


Figure 2: This is phase transition for different materials with magnetic classification. Relations are related with temperature, you can measure curie temperature for different materials which will directly relate to phase transition.

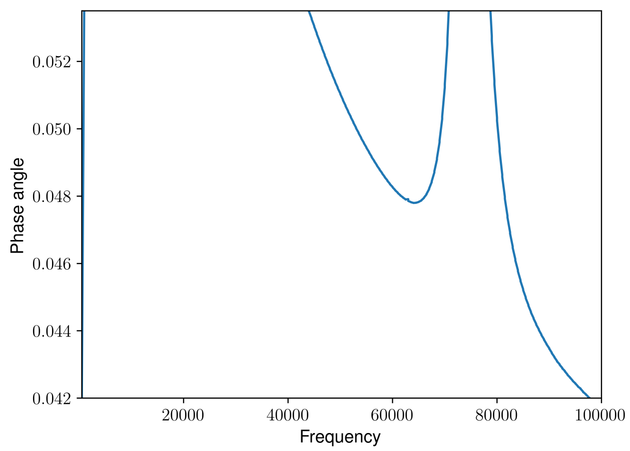


Figure 3: Phase relation with frequency for Iron rod

frequency. This will give insight into magnetic phase transition and sample magnetic properties.

# 3. Experimental setup

Setup was done in such a way that approximately all the instrument's magnetic field cancels out; only remaining is that of sample's magnetization. This can be done by canceling mutual inductance of measuring coils.

## 3.1. Coil assembly

Coils in Setup have two types. One is the primary coil which produces a magnetic field. Second is that of secondary coils which couple with primary and produce mutual inductance. Primary coil is connected to the input signal which is nothing but a reference signal from the LOCK-IN amplifier. This creates a changing magnetic field.

This changing magnetic field is picked up by secondary coils which current inductive current, but they are winded in the opposite direction their current cancels out resulting in zero output signal but when signal is but in any one of these coils generates some residual magnetization and resultant signal. This signal $V_{out}$ is proportional to $\chi$ of the sample at hand. Geometry of coil is given in table 1. Also, figure 4 shows the coil assembly of our setup.

| | Primary Coil | Secondary Coil |
|---|---|---|
| Diameter of wire | 30 gauge | 38 gauge |
| Length of winding | 20 cm | (8.3 + 8.3) cm |
| No. turns | 550 | (450 + 450)[1] |

Table 1: Geometry of coils in our setup

## 3.2. Offset Settings

At the initial stage, we have to offset null between two secondary coils voltage measurement. As a matter of facts we tried to take both our coils to be as symmetrical as possible but inherent human error is there. For this issue we took certain steps,

[1]symmetrical winding, 450 clockwise and 450 anticlockwise

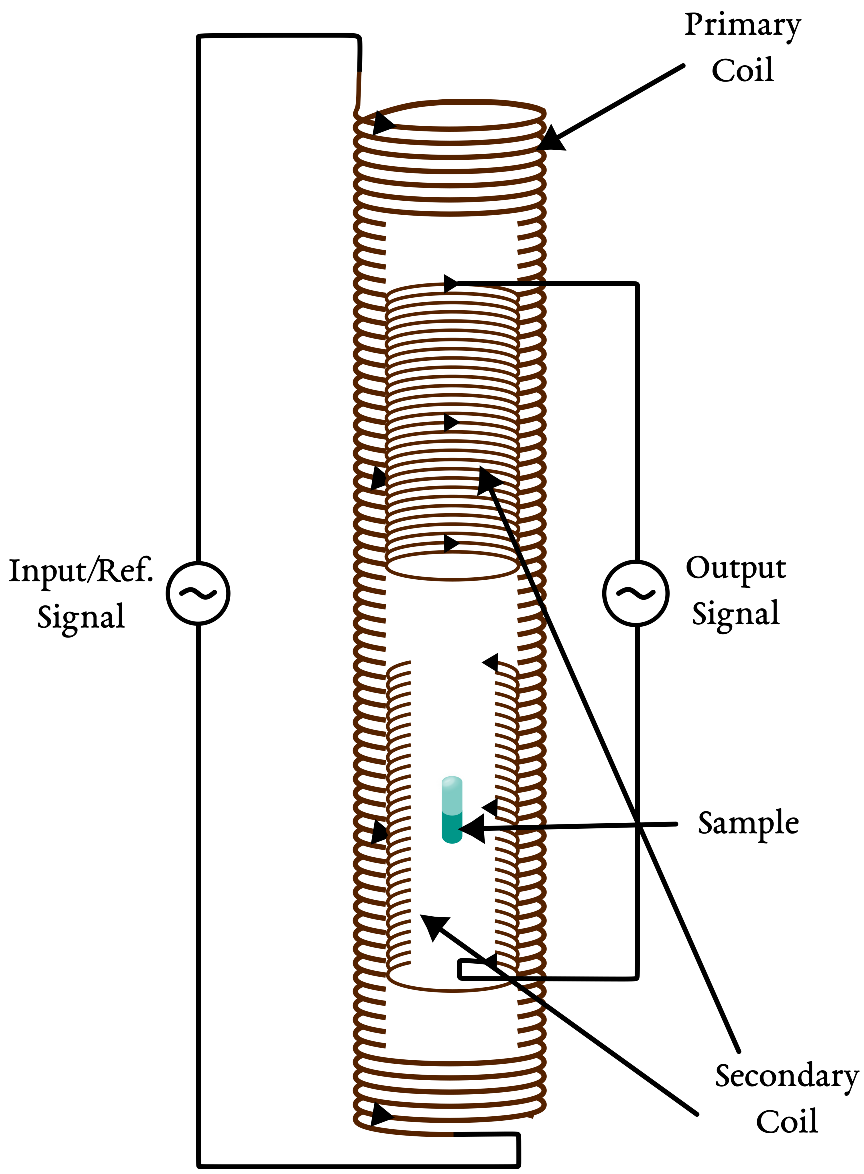


Figure 4: This is the main setup of the experiment. you can see primary coil and two secondary coil winded opposite directions

- Very crudely we take out or wind up our windings. This is to balance both secondary coils to almost perfection. This method is quite clumsy and pain staking. The offset still remains and we can't go beyond the noise limit of the instrument.

- Another thing we can do is that of using some external balancing circuit. Since we have very limited time we can't go down this rabbit hole.

Finally we decided to ignore some offset and subtract it from data of samples. This way is not that effective but we don't have other options.

## 3.3. Calibration

The giant task of our project was to calibrate our handmade instrument. This is also benign by noise of instruments and readings. We made a decision and did this the following way. As it turns out, Calibration can be done in two ways.[7]

- Theoretically, by mutual inductance cancellation. [6]

- Experimentally, by taking some standard susceptibility values of some known and reliable sample.

There is still one catch, what we need is some $\chi_{int}$[7]. What we are measuring is $\chi_{ext}$. What are these different susceptibilities? Well as it turns out the $\chi_{int}$ is what we expect from theoretically,

$$\chi_{int} = \frac{dM}{dH}$$

And

$$\chi_{ext} = \frac{dM}{dH_a}$$

This two values are related by following identity [7],

$$\chi_{int} = \frac{\chi_{ext}}{1 - D\chi_{ext}}$$

This value $\chi_{ext}$ is what we took proportional in the previous topic as LOCK IN's output signal $V_{out}$. If we take the first equation then we can approximate the calibration constant to some extent, we took the second way that is experimental. We took some samples that can give out certain values of output. Before going there, we did the first method. Simplified setup for primary coil is shown in figure 5. Here we neglected the back inductance of the secondary coil.
Simplified circuit makes following kind of differential equation,

$$V_{in} - L\frac{dI}{dt} - IR = 0 \tag{1}$$

Where, $V_{in}$ is nothing but $V_{ref}$ from the LOCK IN amplifier. It is in the following form $V_{in} = V_0 e^{i(w_r t + \phi_r)}$, $w_r$ is applied frequency, $\phi_r$ is taken as zero from input. $I$ is currently in the primary, this parameter is very important and gives applied magnetic field strength $H_a$. $L$ and $R$ are both electrical parameters of the primary coil, respectively self-inductance and resistance. Solution of equation 1 gives current in primary at instance of time,

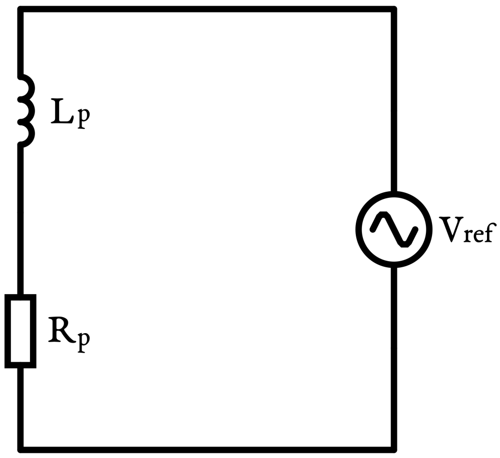


Figure 5: This is a simpler circuit of coil assembly in our setup. we are neglecting effect of second coil

$$I_{real} = \frac{V_0\left[\frac{R}{L}cos(w_r t + \phi_r) + w_r sin(w_r t + \phi_r)\right]}{L(\frac{R^2}{L^2} + w_r^2)} + Ce^{-\frac{R}{L}t} \tag{2}$$

$$I_{imag} = \frac{\left[\frac{R}{L}sin(w_r t + \phi_r) - w_r cos(w_r t + \phi_r)\right]}{L(\frac{R^2}{L^2} + w_r^2)} \tag{3}$$

If we take $\phi_r = 0$ and $V_0 = 1V$ which signifies that total input phase and amplitude of reference voltage from LOCK IN amplifier is 0 deg and 1 V. Also, $C$ is an integral constant.

$$I_{real} = \frac{\frac{R}{L}cos(w_r t) + w_r sin(w_r t)}{L(\frac{R^2}{L^2} + w_r^2)} + Ce^{-\frac{R}{L}t} \tag{4}$$

$$I_{imag} = \frac{\frac{R}{L}sin(w_r t) - w_r cos(w_r t)}{L(\frac{R^2}{L^2} + w_r^2)} \tag{5}$$

After taking condition that initial current is zero,

$$C = \frac{-R}{L^2(\frac{R^2}{L^2} + w_r^2)} \tag{6}$$

$$I_{real} = \frac{\frac{R}{L}cos(w_r t) + w_r sin(w_r t) - \frac{R}{L}e^{-\frac{R}{L}t}}{L(\frac{R^2}{L^2} + w_r^2)} \quad (7)$$

From this current magnetic field strength inside primary coil and coupled by secondary coil is as following

$$H_a(t) = cNI(t)$$

Which is measured in $A/m$, here $c$ is coupling constant for secondary coil. Since, the magnetic field inside the coil is not homogeneous, finding this parameter is very difficult, also if we find it then we get averaged out readings as whole coil. Magnetic field inside coil $B = \mu_0 H_a$. Also, flux inside secondary coil is as following,

$$\Phi = \mu_0 H_a n \pi a^2$$

Hare, $n$ and $a$ are geometrical parameters of the coil, $n$ is the number of turns of a single secondary coil and $a$ is the diameter of the secondary coil. We took the secondary coil in the centre of the primary coil as possible.

$$\Phi = MI$$

$$\frac{d\Phi}{dt} = M\frac{dI}{dt}$$

Here, $M$ is a mutual inductance parameter, which only depends on setup and sample placement. Final, voltage measurement from our secondary coil is only dependent is only LOCK IN readings [7],

$$V_{out} = \alpha H_a f V_{sample} \chi_{ext}$$

Here, $\alpha$ is calibration constant, which we have to determine. Theoretically, We know $H_a$, $V_{sample}$ (volume of sample: we took some small cylindrical capsule), $f$, $\chi_{ext}$ then we can find $\alpha$ directly. But knowing this parameter and their exact place in the instrument is very difficult. This is problematic. So, we turn to the second method and fix the orientations and parameters, put some sample and compare its reading calculated by their known magnetic susceptibility.
For calibration purposes we took four samples and their readings of output voltages. These are 1. $Fe_2O_3$, 2. Nickel, 3. LSMO (Lanthanum strontium manganite), 4. $Gd_2O_3$. For characterization purposes $Fe_2O_3$ and $Gd_2O3$ are paramagnets where Nickel and LSMO are Ferromagnets. Problem we faced in our measurement is that our instrument's sensitivity is very low and sample weight is very small. As a result paramagnetic substances have feeble signals which are not very reliable compared to ferromagnets like Nickel and LSMO. The data are shown in figures 6, 7, 8 and 9 where you can see why ferromagnets signal is reliable.

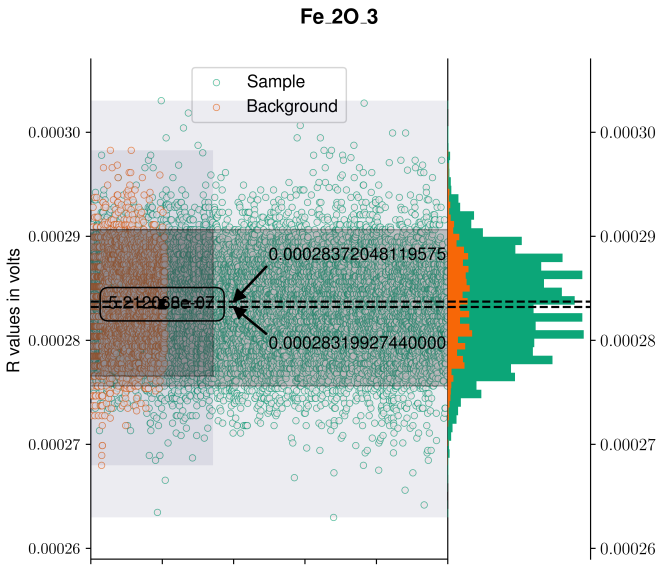


Figure 6: Plot of absolute values of voltage $Fe_2O_3$ and it's Background. You can see difference is $5.212068 \times 10^{-7}$

Also values for Nickel and LSMO. You can see stark differences.
The values that we found in this data is that voltage differs relative to background(because we couldn't make it zero). These values are shown in table 2.

| Samples | absolute voltage values in V |
|---|---|
| $Fe_2O_3$ | $5.212068 \times 10^{-7}$ |
| $Gd_2O3$ | $3.543178 \times 10^{-7}$ |
| Nickel | $2.711438 \times 10^{-4}$ |
| LSMO | $2.257564 \times 10^{-4}$ |

Table 2: Absolute value reading after subtracting from background readings

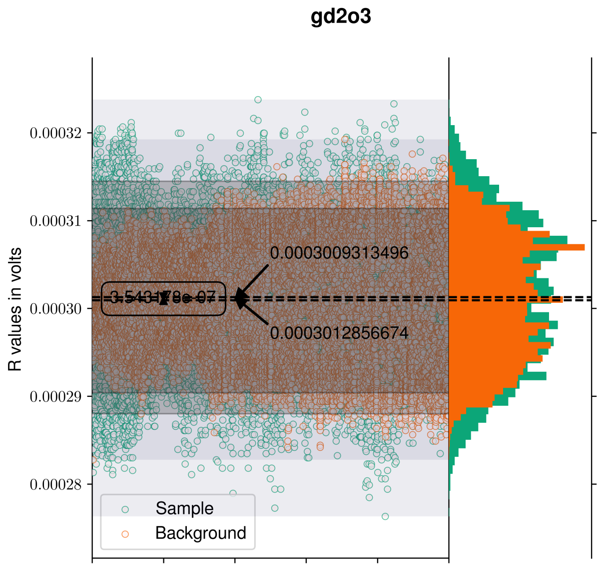


Figure 7: Plot of absolute values of voltage $Gd_2O_3$ and it's Background. You can see difference is $3.543178 \times 10^{-7}$

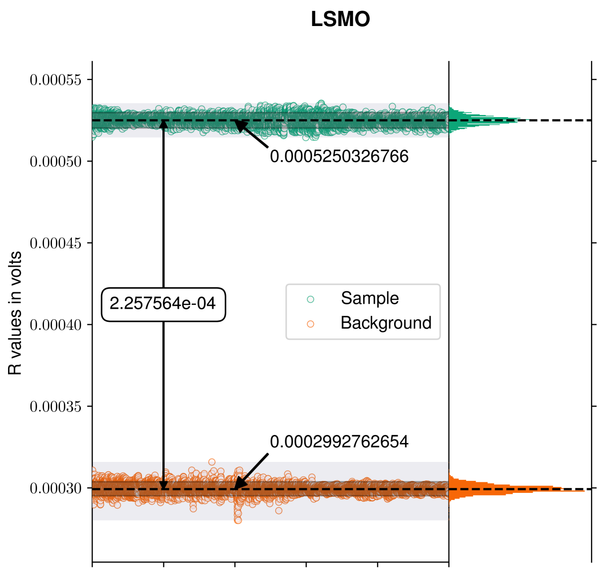


Figure 8: Plot of absolute values of voltage $LSMO$ and it's Background. You can see difference is $2.257564 \times 10^{-4}$

Now the big problem is that Susceptibility value of ferromagnets is too dependent on experiment parameters (magnetic field strength, frequency, temperature etc). Compared to it, paramagnets have almost constant value of magnetic susceptibility. As a consequence we could not find exact values of this material. We could only rely on approximate values of it.

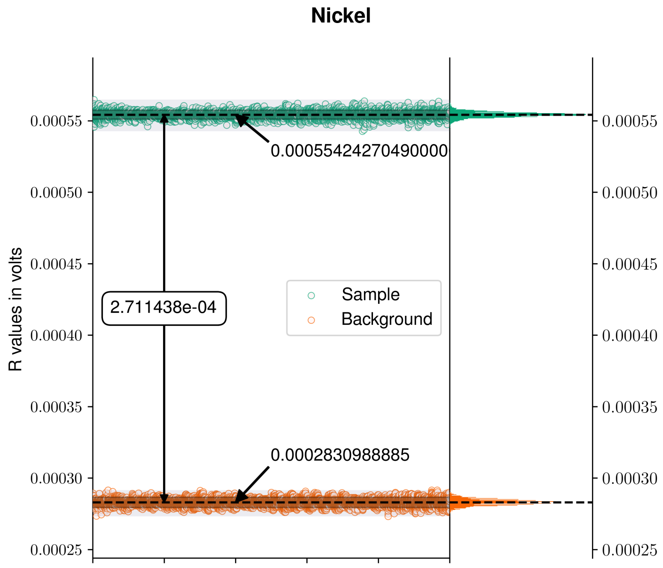


Figure 9: Plot of absolute values of voltage Nickel and it's Background. You can see difference is $2.711438 \times 10^{-4}$

Also, we found $V_{in}$ and $V_{out}$ trends, which should directly give connection to magnetic susceptibility. This are that reading in figure 10,

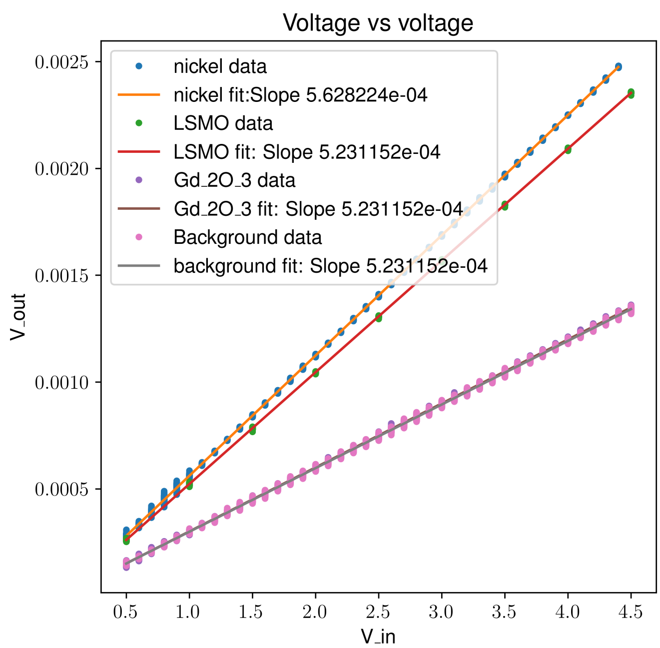


Figure 10: Different samples data for $V_{out}$ vs $V_{in}$

| Samples | Slope |
|---|---|
| Nickel | 5.62822393e-04 |
| LSMO | 5.23115219e-04 |
| $Gd_2O_3$ | 2.99442997e-04 |
| Background | 2.97732056e-04 |

Table 3: Slopes readings of different samples $V_{out}$ vs $V_{in}$

This gives slopes of different samples and we can compare it to find ratios of different $\chi_{ext}$s. This relation should look like the following, $V_{out} \propto V_{in}$.

$$V_{out} = \tau \chi_{ext} V_{in}$$

Here, $\tau$ is some constant. From the data and above equation,

$$Slope \propto \chi_{ext}$$

$$\frac{Slope^{(LSMO)}}{Slope^{(nickel)}} = \frac{\chi_{ext}^{(LSMO)sample}}{\chi_{ext}^{(nickel)sample}}$$

Here, $\chi_{ext}^{sample} = \frac{W\chi_{ext}}{W_a}$, where $W$ and $W_a$ are weight and atomic weight.

$$\frac{5.23115219e-04}{5.62822393e-04} = \frac{(0.2906g)\chi_{ext}^{(LSMO)\times(58.693)}}{(0.1942g)\chi_{ext}^{(nickel)} \times (226.45)}$$

$$1.075905 = \frac{\chi_{ext}^{(LSMO)} \times 17.0562}{\chi_{ext}^{(nickel)} \times 43.9766}$$

$$2.77404 \times \chi_{ext}^{nickel} = \chi_{ext}^{(LSMO)}$$

For nickel we took magnetic susceptibility about $\chi_{int}^{(nickel)} = 0.004423 m^3/mol$.
If we put these values in equation 3.3. This gave us a value of $\alpha = 11.82249$. This value is relatively high, which signifies sensitivity of our instrument. Compare it to values of some other commercial and also from other experiments, we have very $\frac{1}{\alpha} = 0.08485 Am^2V^{-1}s^{-1}$ is low compared to commercials around $2.1546 Am^2V^{-1}s^{-1}$ and from one experiment $11.24 Am^2V^{-1}s^{-1}$[7].

## 3.4. Noise

Noise factor in our instrument is very high. For Example the $R$ values which are absolute values of voltage ($\sqrt{X^2+Y^2}$) have relatively less noise compared to phase measurement. These noise values can't be ignored since our measurements will have a relatively low number of measurements and the signal will be very feeble.
LOCK IN in our setup is a very low noise instrument. This is a good thing to start with but as we built our instrument this factor is not very important. Instrument noise in our setup is very high for coils and other things. We take certain steps to reduce it, for example we made Faraday's cage like structure from aluminum foil to reduce external interference.
After doing all this we have very significant noise in our setup. Take a comparison between $R$ values of samples and phase measurement of the system. Compare this figures 11, 12, 13 and 14 to the $R$ measurement given on this figures 6, 7, 8, 9.

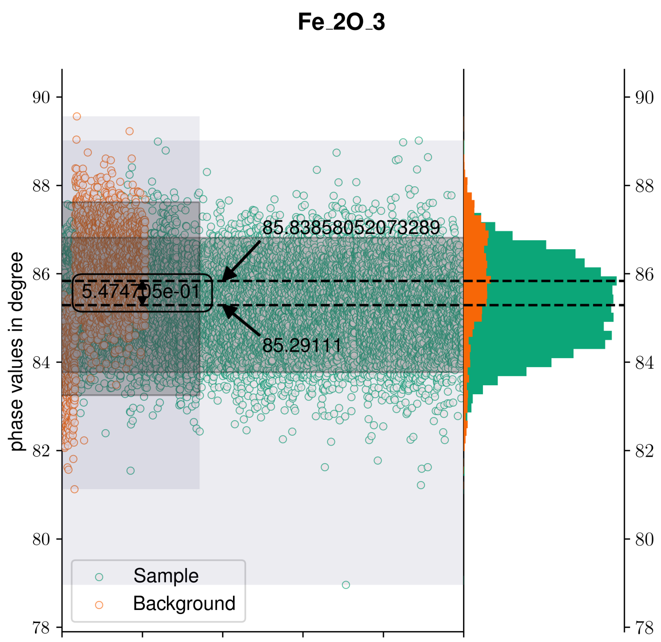


Figure 11: Plot of phase values in degree $Fe_2O_3$ and it's Background

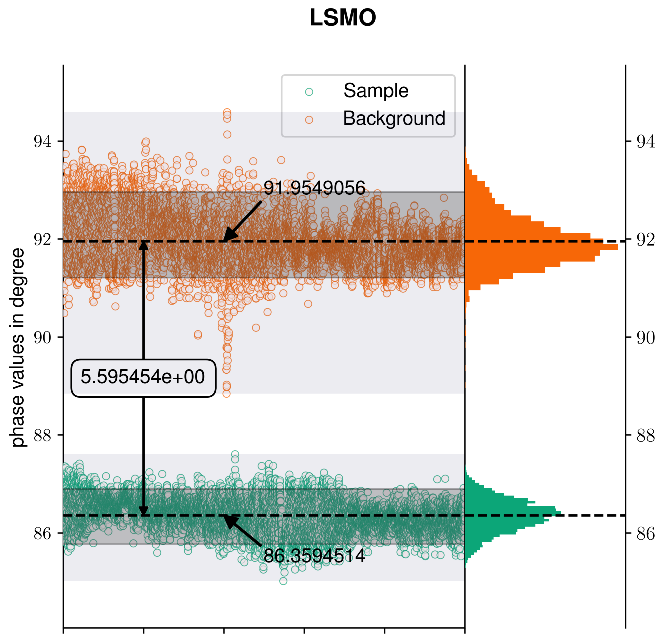


Figure 12: Plot of phase values in degree $Gd_2O_3$ and it's Background

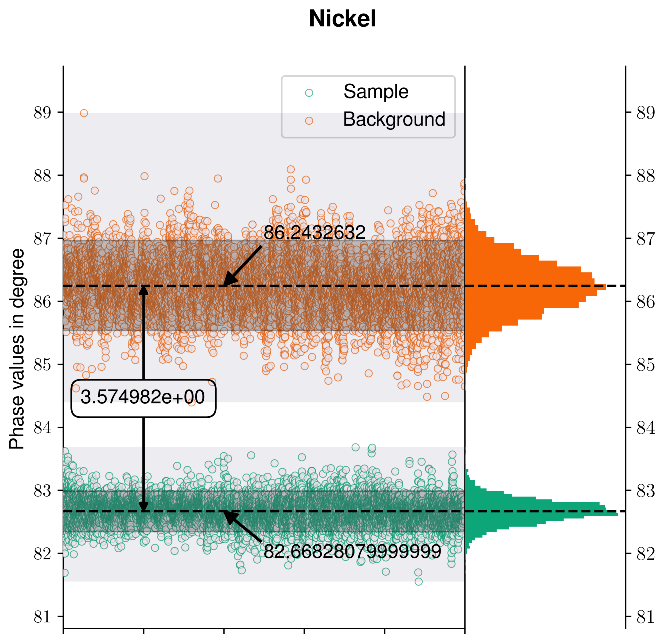


Figure 13: Plot of phase values in degree $LSMO$ and it's Background

# 4. Future work and extension for setup

As you see earlier we got foundational work done for our AC susceptometer instrument. Now, this

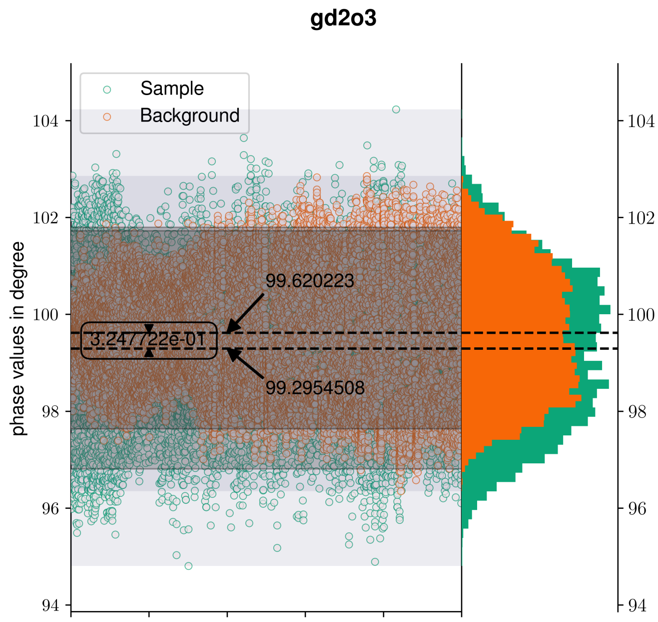


Figure 14: Plot of phase values in degree Nickel and it's Background

setup can be used measuring susceptibility values at particular frequencies and temperatures. We can extend this with an external setup for temperature sweeps. We have designed that setup but not yet implemented it because of a lack of time in semester work.

## 4.1. Temperature controlling and measuring setup

Essential constraints for temperature control and measurement is as follows,

- Temperature must be uniform throughout the sample.
- Temperature must be properly controlled under time of measurement.
- When measuring temperature it must measure the exact temperature of the sample.
- It must not affect measurement of susceptibility which implies that measurement and control unit must be very less magnetic compared to sample.

With this constraint we have designed the setup. We need the first heater. For that we have a temperature control unit with PT100 as feedback sensor. From the 4th constraint we need very little interference, for that we can use ceramic (non ferrite). One rod of ceramic is connected with a sample inside the instrument and the other end with heater wire (nichrome wire) outside the instrument. Of Course this will affect heat flow and we will end up with a gradient of temperature levels. This will compensate for the slow heat rate (few celsius per minutes) from the heater. Still this will affect temperature levels at sample. These problems can be solved by using another temperature sensor very close to the sensor and a small sample size (capsule size). Another sensor will help us to take data effectively without creating problems with the heater.

## 4.2. One coherent system for automatic measurement

As in the previous section we have discussed how temperature setup can be done. This temperature setup must be in sync with measurement of susceptibility. For this we need one system where we can have timed data and simultaneous data of different measurements. We have devised a plan for doing that. We need one microprocessor and ADC for the temperature sensor. This microcontroller will be coupled with a computer. Also as we discussed previously, SR830 interfaces directly to the computer. This data stream will be connected to one frame computer with simultaneous measurement readings coming from both places. For temperature measurement we took PT100 Sensor. This sensor is then connected with an ADC converter and porting mechanism to the microprocessor. We will be using a microprocessor as Arduino Uno. We will be using ready-made porting and an ADC converter for Arduino.[2] With Arduino's capability to easily connect to a mainframe computer, we will use it as an intermediate step in taking data. You can say the whole plane in the Block diagram shown in figure 15.

[2]MAX31865

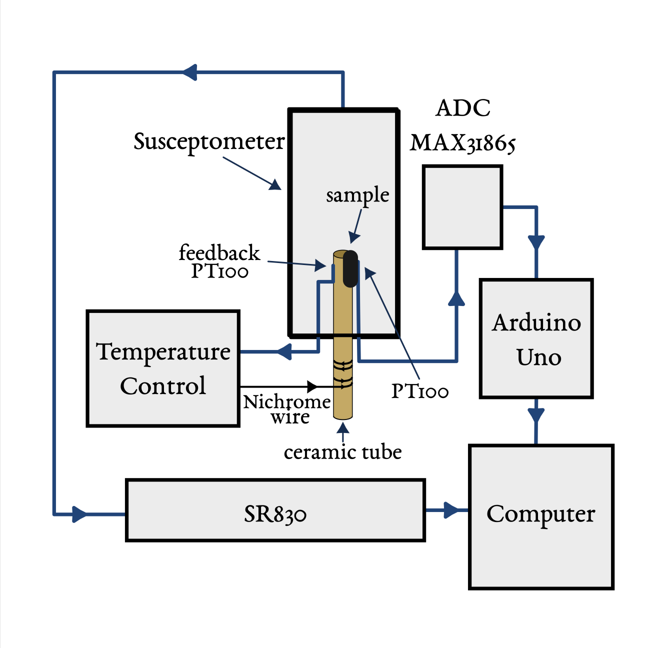


Figure 15: Final setup would look like this with temperature and susceptibility data

# 5. Results and Conclusion

We have made an AC susceptometer for measuring magnetic properties of material. These instruments can measure magnetic susceptibility automatically of a sample and with extended instrumentation as we talked in Experimental setup is capable of measuring magnetic properties in temperature sweeps. This can help tremendously in finding magnetic phase transition. We didn't find that here but we find magnetic susceptibility of some of the samples.

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