GUJARAT UNIVERSITY

Estd. 1949

Project Report

Instrumentation of AC Susceptometer with Determination of the AC magnetic Susceptibility and finding magnetic phase transitions of unknown samples

MSc Semester 4

Unit 512: Project Work

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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. We got needed help from the department of physics's condensed matter laboratory, Dr. Utpal Joshi, Swati Pachauri. Lock-In amplifier that we got access from Utpal sir and it was a necessary component in our project.

In this project we did first to design and improvise our instrument, which is an AC susceptometer. Major problem faced by such a setup by non-expert is the problem of noise. We gave our best time for this to get as minimum noise as possible. This was a very big task for us. Another thing which made us think is that of getting offset null for AC susceptometer. We did this by changing the coil's parameter and relative orientation of the coils with each other. Final giant in our project was calibration, as it turns out our instrumentation has some noise floor and for samples, especially that of paramagnetic, have very feeble magnetization which is hard to detect with our setup. So, we choose ferromagnetic samples such as nickel, LSMO and Gd_2O_3. Problem with these samples was that their magnetic susceptibility is highly dependent on conditions of measurement, so we can't find its absolute values like paramagnetic samples. This problem seems to be big for calibration of our instrument. Finally we have tried and successfully calibrated with some accuracy. Final goal was to find temperature dependent magnetic susceptibility plots and find curie temperature where magnetic phase transition happens. This task does not need very accurate calibration and is possible with our instrumentation.

2. Theoritical concepts

We should know little about some concepts of magnetism. Magnetism in loops is profoundly due to relative motions of charge particles. This is related to concepts of relativity (especially Einstein's special relativity). As we know moving charge creates a magnetic field which curls around movement direction. These magnetic fields get accumulated with a number of loops which relate to our instrumentations. Also, changing velocity (relates directly to changing current) creates a changing magnetic field.

There's another type of magnetism which differs in some minor differences. This is related to the material's magnetic moments. As we know atoms have magnetic moments related with two momentums,orbital angular momentum and spin angular momentum. This degrees of freedom of electrons (as by product relates to atoms) creates magnetic properties of materials.

First evident by Stern and Gerlach

A magnetic susceptibility of a material can be defined as the amount of a material gets magnetised , when it is placed in an external magnetic field . In other words , an amount of magnetization of a material occurs when it is placed in external magnetic field .

An expression for magnetic susceptibility is given by ,

X = M / H

Where , M= Magnetic susceptibility H= Applied/External magnetic field

There are two other measures of susceptibility:

1. MAGNETIC MOLAR SUSCEPTIBILITY (Xm)

2. MASS MAGNETIC SUSCEPTIBILITY (X)

Xm = M X

Where, X = Xv / , = density (kg/m^3)

Magnetic susceptibility is a factor that indicates the magnetic behavior of a material.It gives an idea about a material that it can be attracted or can be repelled.

CLASSIFICATION OF MATERIALS BASED ON THEIR MAGNETIC PROPERTIES :-

DIAMAGNETIC MATERIAL :

A magnetic materials which aligns its domains or field lines against the applied magnetic field are known as diamagnetic materials.

These materials are strongly repelled by the magnets .

As these materials gets magnetize in opposite side of applied field , they have a small amount of magnetization .

Example :- water, tin , mercury ,etc.

They have magnetic susceptibility $X < 0$, negative value of magnetic susceptibility.

2 . PARAMAGNETIC MATERIAL :

A magnetic materials which aligns its domains or field lines with the applied field are known as paramagnetic materials .

These materials are weakly attracted by the magnets and also they are temperature dependent .

Example :- aluminium , alkaline earth metals , etc.

They have magnetic susceptibility $X > 0$, positive value of magnetic susceptibility.

3 . FERROMAGNETIC MATERIAL :

A magnetic materials that are highly gets magnetized in an external magnetic field are known as ferromagnetic materials .

These materials are highly attracted by the magnets .

Example :- iron, cobalt, nickel , etc .

They have magnetic susceptibility $X > 1$, always higher value of magnetic susceptibility.

There are further classification of materials based on their magnetic properties can be done as :

a) Anti-ferromagnetic materials

b) Ferrimagnetic materials

These two types are not discussed in detail because they are not in context to our project work .

WHAT DO YOU MEAN BY AN AC MAGNETIC SUSCEPTIBILITY ?

AC magnetic measurements are taken by applying AC field to the samples and resulting AC magnetic moment is measured i.e., induced by changing magnetic flux by applied AC field .

This results in the different values of magnetic susceptibility for different values of magnetic flux arises from the different values of AC field.

In order to understand AC magnetic susceptibility , first we have consider measurements at low frequencies , where the measurements is almost equals to the DC susceptibility.In this case the absolute value of magnetization is calculated i.e.,

$$X = M / H$$

In case of AC susceptibility, the continuously varying value of magnetization , hence varying value of susceptibility i.e.,

$$Xac = (dM / dH)$$

AC susceptibility is often referred as dynamic susceptibility.AC measurements are very sensitive to the small changes in the values of magnetization .

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 cancelling 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 pick up by secondary coils which current inductive current, but they are winded 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 χ of the sample at hand. Geometry of coil is given in table 1. Also, **??** shows 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,

- 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.[1]

- Theoretically, by mutual inductance cancellation. ((((ref 31)))

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

There is still one catch, what we need is some χ_{int}[1]. What we are measuring is χ_{ext}. What are these different susceptibilities? Well as it turns out the χ_{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 (((ref camb paper))),

[1]symmetrical winding, 450 clockwise and 450 anticlockwise

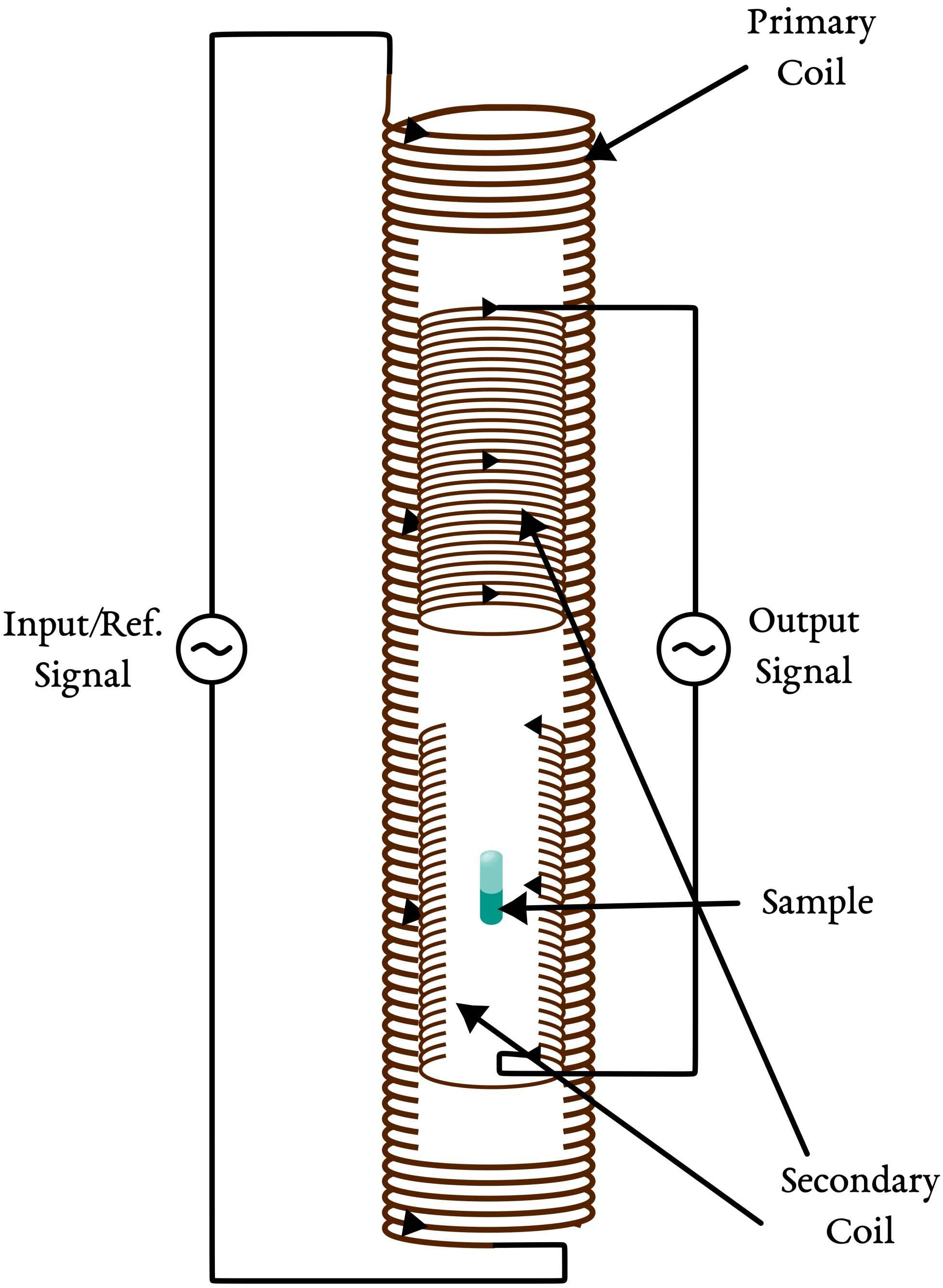

Figure 1: This is main setup of experiment. you can see primary coil and tweo secondary coil winded opposite directions

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

This value χ_{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 **??**. Here we neglected the back inductance of the secondary coil.
Simplified circuit makes following kind of differential equation,

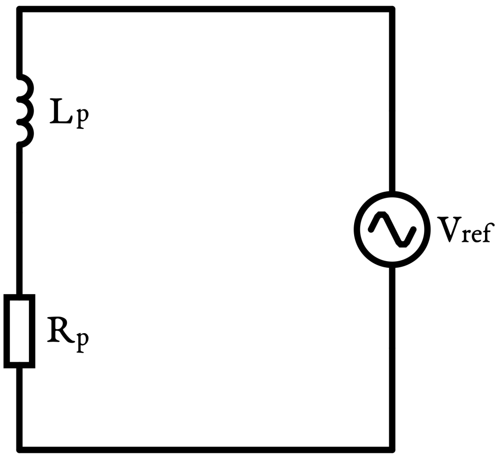

Figure 2: This is simpler circuit of coil assembly in our setup. we are neglecting effect of second coil

$$V_{in} - L\frac{dI}{dt} - IR = 0 \quad (1)$$

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, ϕ_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,

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

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

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.

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

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

After taking condition that initial current is zero,

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

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

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 ((ref camb paper)),

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

Here, α 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, χ_{ext} then we can find α 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 **??**, **??**, **??** and **??** where you can see why ferromagnets signal is reliable.

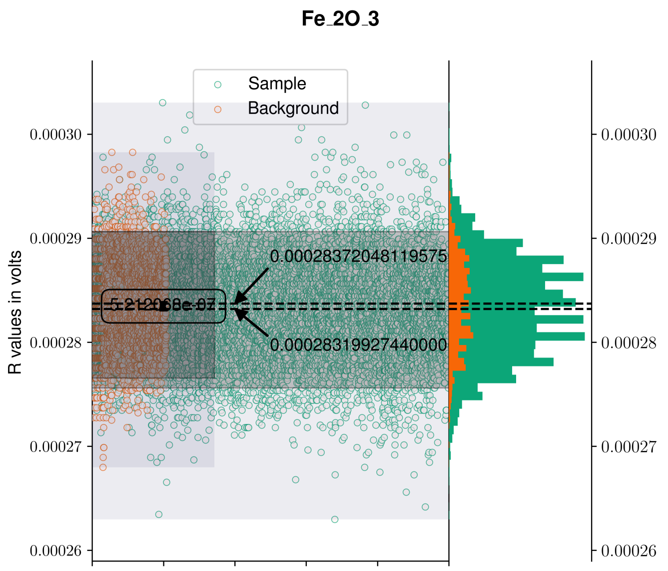

Figure 3: Plot of absolute values of voltage Fe_2O_3 and it's Background. You can see difference is 5.212068×10^{-7}

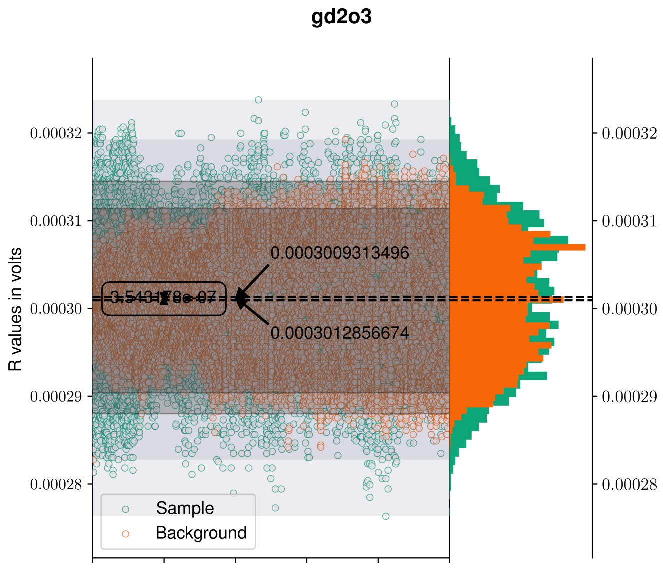

Figure 4: Plot of absolute values of voltage Gd_2O_3 and it's Background. You can see difference is 3.543178×10^{-7}

Also values for Nickel and LSMO. You can see stark differences.

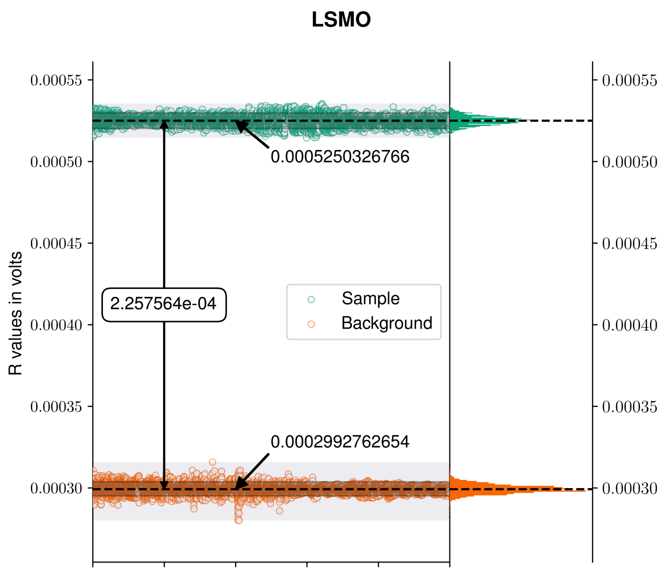

Figure 5: Plot of absolute values of voltage $LSMO$ and it's Background. You can see difference is 2.257564×10^{-4}

The values that we found in this data is that voltage differs relative to background(because we

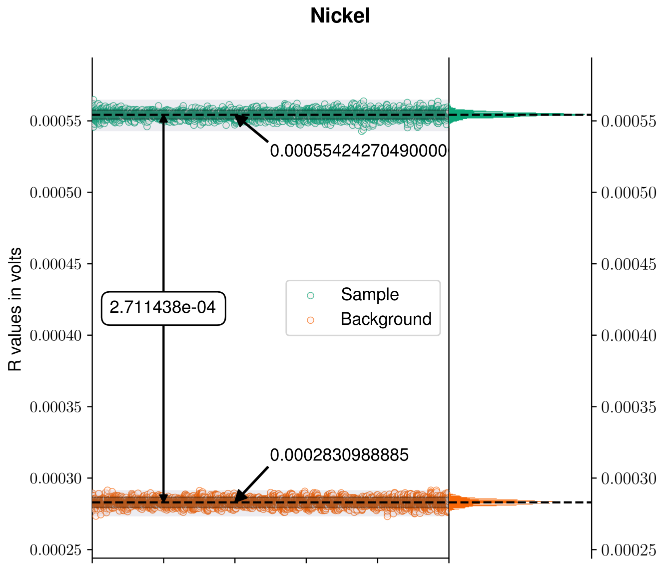

Figure 6: Plot of absolute values of voltage Nickel and it's Background. You can see difference is 2.711438×10^{-4}

couldn't make it zero). These values are shown in table 3.3.

Samples	absolute voltage values in V
Fe_2O_3	30565445
Gd_2O3	20454545
Nickel	457845454
LSMO	454544544

Table 2: Absolute value reading after subtracting from background readings

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

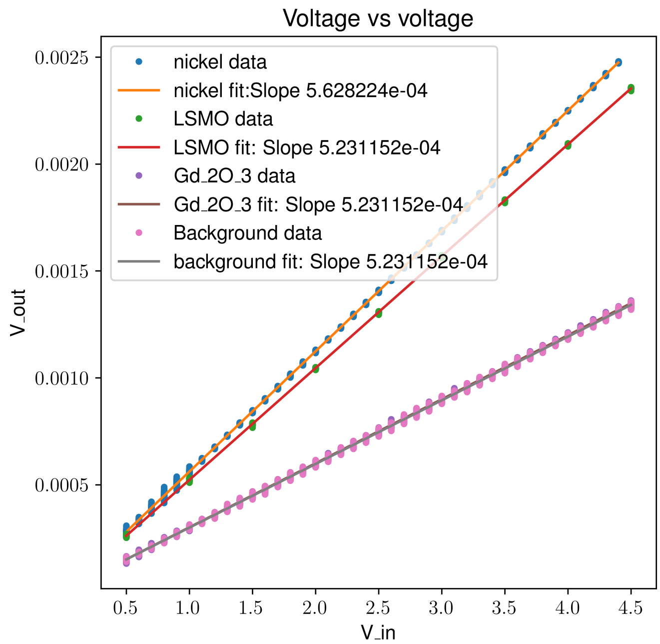

Figure 7: Different samples data for V_{out} vs V_{in}

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 χ_{ext}s. This relation should look like the following, $V_{out} \propto V_{in}$.

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

Here, τ 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 ((reference)). 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 $11.24 Am^2V^{-1}s^{-1}$[1].

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 aluminium 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 3.4, **??**, **??** and **??** to the R measurement given on this figure **??**, **??**, **??**, **??**.

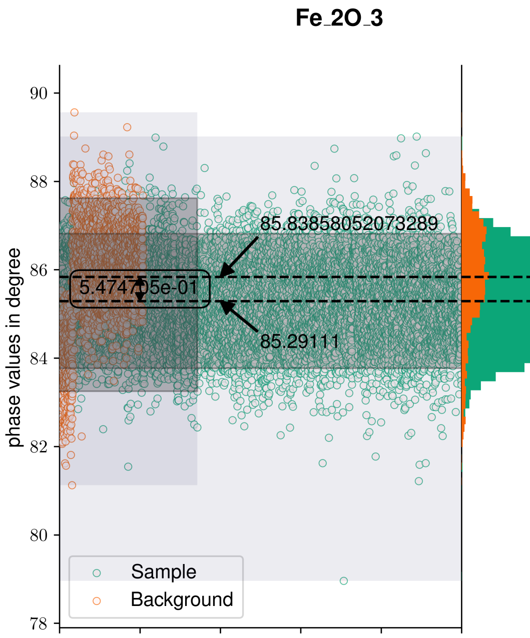

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Table 4: Plot of phase values in degree Fe_2O_3 and it's Background

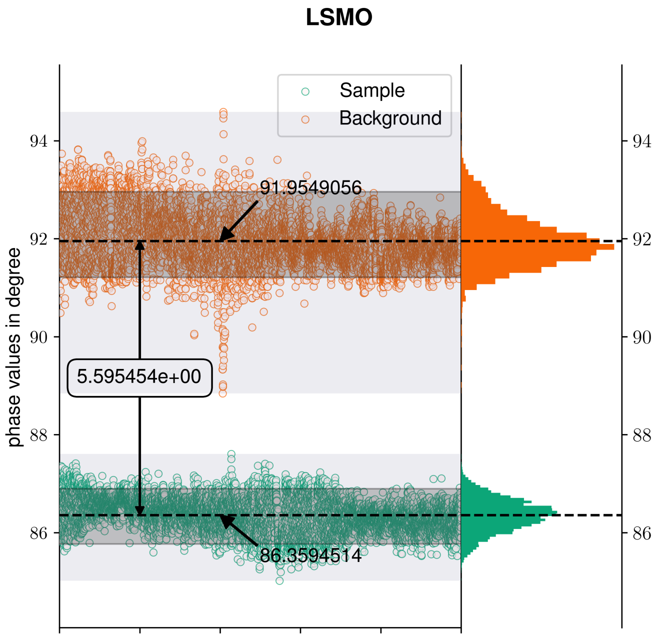

Figure 8: Plot of phase values in degree Gd_2O_3 and it's Background

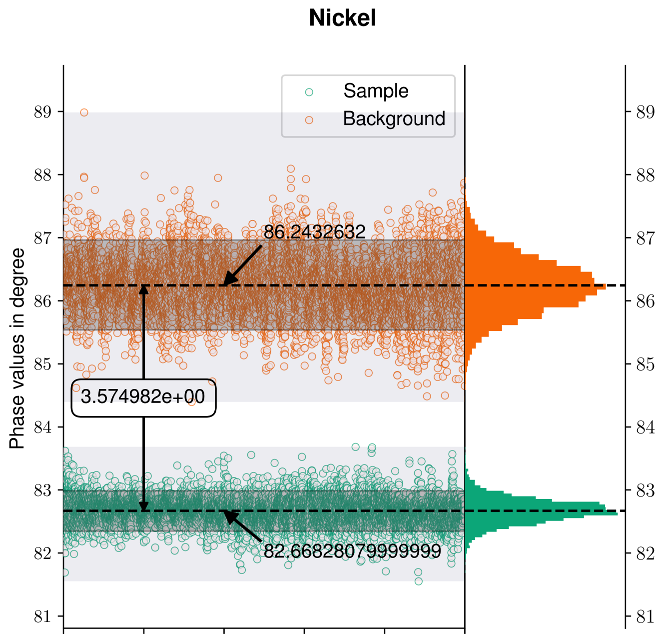

Figure 9: Plot of phase values in degree *LSMO* and it's Background

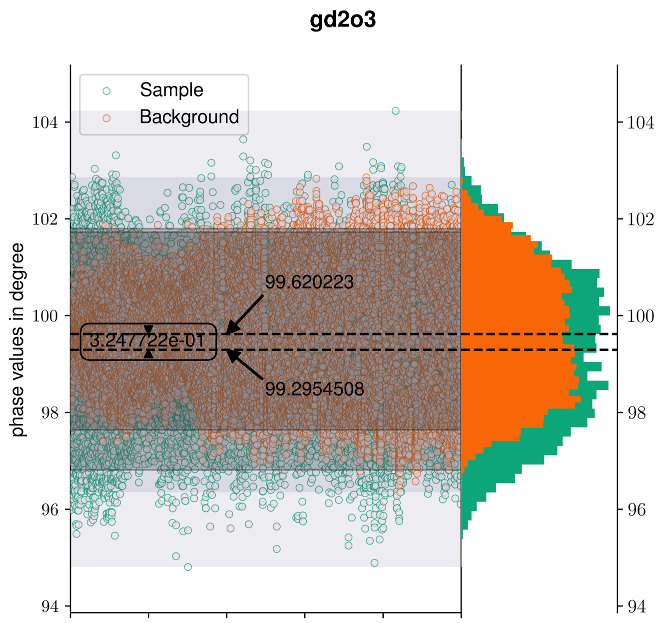

Figure 10: Plot of phase values in degree Nickel and it's Background

4. Results and Analysis

Our result is partitioned in partitions related with steps we took in our project. This is MSc project that's why we wrote this way. First part is the initial plots which shows how our instrument works. This shows position-dependent graphs of ferro-magnetic substances (Iron). This shows some sinusoidal curve which is logical and we can find maximum magnetization at a certain distance into secondary coils. This is that curve,

5. Conclutions

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References

[1] P Laurent, J. Fagnard, B Vanderheyden, Haribabu Nadendla, David Cardwell, Marcel Ausloos, and P. Vanderbemden. An ac susceptometer for the characterization of large, bulk superconducting samples. *Measurement Science and Technology*, 19, 05 2008.

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