MODIFICATION OF EXISTING CLOSED CYCLE REFRIGERATOR FOR AC SUSCEPTIBILTY MEASUREMENT

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Science in Physics

By

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CERTIFICATE

This is to certify that the dissertation entitled, **"MODIFICATION OF EXISTING CLOSED CYCLE REFRIGERATOR FOR AC SUSCEPTIBILTY MEASUREMENT"** submitted by Sri Sanjaya Kumar Parida, for the award of Master of Science in Physics (2010-12) in the Department of Physics, National Institute of Technology, Rourkela, is a record of authentic work carried out by him under my supervision. To the best of my knowledge, the results embodied in this dissertation have not been previously submitted for any degree in this/any other institute.

Date: **Dr. Prakash Nath Vishwakarma**

DECLARATION

I, Sri Sanjaya Kumar Parida do hereby declare that, this dissertation titled **"MODIFICATION OF EXISTING CLOSED CYCLE REFRIGERATOR FOR AC SUSCEPTIBILTY MEASUREMENT"** is an authentic work done by me in the Department of Physics, NIT Rourkela. I also declare that this report has been neither published before nor submitted to any other institution.

Sanjaya Kumar Parida

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Abstract

An experimental set up for cryocooler based ac susceptometer is designed. Hylum is chosen the former over which, the copper windings are done. The design consists of primary coil, secondary coils and a sample holder. Ac susceptibility is most conveniently measured using the mutual inductance principle. Copper wire (150micron) winding is done on the primary and the secondary coils. The secondary coil is connected in series opposition i.e. if one wound is clockwise, the other is anticlockwise. A Pt100 temperature sensor is put in close proximity with the sample, in order to record the sample temperature accurately. $La_{0.83}Sr_{0.17}MnO_3$ sample is prepared as test sample for measuring the susceptibility.

CHAPTER 1

INTRODUCTION

Science of magnetism back to 600 B.C, when Thales of Miletus had a knowledge that iron ore possessed the properties of attracting small pieces of iron towards it. Magnetism is a property of materials that respond to an applied magnetic field. The magnetization M is defined as the magnetic moment per unit volume. In magnetic materials, sources of magnetization are the electrons orbital angular motion around the nucleus. The magnetic susceptibility is the degree of magnetization of a material in response to an applied magnetic field. The magnetic susceptibility per unit volume is defined as

$$\chi = M/H$$

Susceptibility is a measure of the quality of the magnetic material and is defined as the magnetisation produced per unit applied magnetic field. It is a dimensionless quantity.

1.1 Magnetic behaviour of materials:

The magnetic behaviour of materials can be classified into the following five major groups:

1. Diamagnetism
2. Paramagnetism
3. Ferromagnetism
4. Antiferromagnetism
5. Ferrimagnetism

1. Diamagnetism:

Diamagnetism is a fundamental property of all matter, although it is usually very weak. It is due to the non-cooperative behaviour of orbiting electrons when exposed to an applied magnetic field [1]. Diamagnetic substances are composed of atoms which have no net magnetic moments. When exposed to a magnetic field, a negative magnetization is produced because of the orientation of atomic orbitals, which opposes the direction of the applied magnetic field. So the developed magnetization is opposite to the magnetic field and hence a negative susceptibility. The susceptibility of diamagnetic materials is temperature

independent. Example -He, Ne, Ar, Xe, YBCO, NaCl, etc. The magnetic susceptibility of different diamagnetic material is given in Table 1

Material	Susceptibility($x10^{-5}$)
water	-0.91
carbon	-1.6
silver	-2.6

Table 1

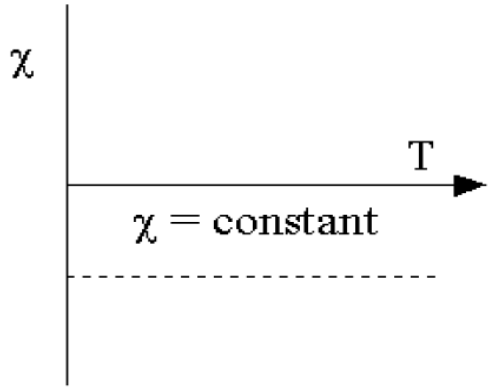


Fig 1.1: susceptibility of Diamagnetic material

2. Paramagnetism:

This class of materials, some of the atoms or ions in the material have a net magnetic moment due to unpaired electrons in partially filled orbitals. Like diamagnetism, the magnetization is zero when no field is applied. In the presence of a field, there is alignment of the atomic magnetic moments in the direction of the magnetic field, resulting in a net positive magnetization and positive susceptibility. Example- Pt, Na.

The magnetic susceptibility of different paramagnetic material is given in Table 2

Material	Susceptibility($x10^{-5}$)
sodium	0.72
magnesium	1.2
aluminium	2.2

Table 2

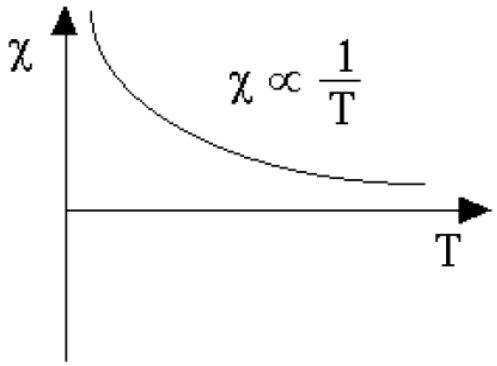


Fig 1.2: susceptibility of Paramagnetic material

3. Ferromagnetism:

A ferromagnetic substance has unpaired electrons. These categories of the materials are having permanent dipole moment. When the applied field is removed, the electrons in the ferromagnetic substance maintain a parallel orientation.

Below the Curie temperature, the Ferro magnet is ordered and above it, disordered. The saturation magnetization goes to zero at the Curie temperature. It contains spontaneous magnetic moment – a magnetic moment even in zero applied magnetic fields. The Curie temperature of some ferromagnetic material is given table 3:

Material	Curie temp.(^{0}C)
Iron	770
cobalt	1130
nickel	358

Table 3

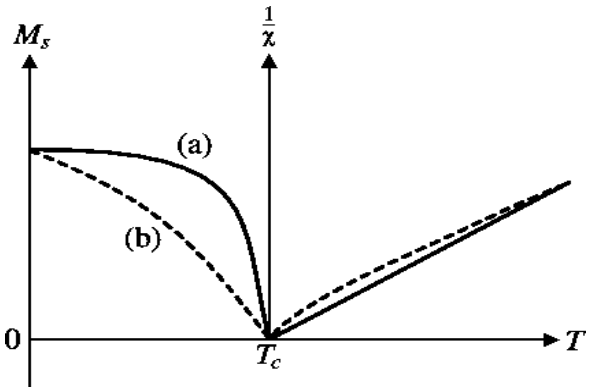


Fig 1.3: Ferromagnetic behaviour

4. Antiferromagnetism:

In anti-ferromagnetism is the behaviour of susceptibility above the critical temperature, called the Néel temperature (TN). Above TN, the susceptibility obeys the Curie-Weiss law for paramagnets but with a negative intercept indicating negative exchange interactions. Antiferromagnets have a zero net magnetic moment Example- NiO, MnO, FeCl2, etc.

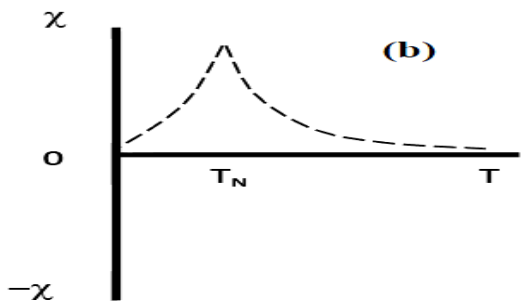
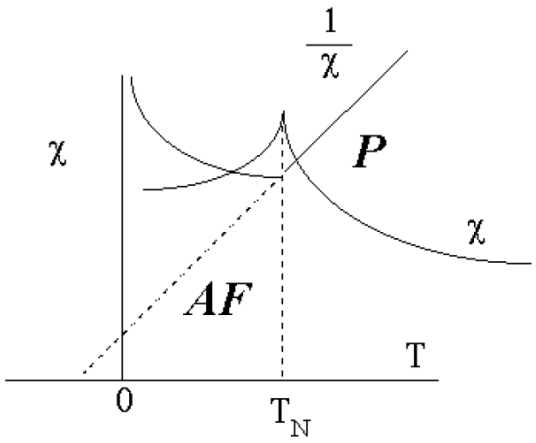


Fig 1.4: Antiferromagnetism

5. Ferrimagnetism:

In ferrimagnetism, the magnetic moments of the sub lattices are not equal and result in a net magnetic moment. The molecular field on each sub lattice is different. The spontaneous magnetizations of the sub lattices will in general have quite different temperature dependence. The magnetic susceptibility of ferrimagnetism does not follow the Curie-Weiss law. However, Ferro- and ferrimagnetism have very different magnetic ordering**.** Ex- Magnetite is a well-known ferromagnetic material.

Figure 1.5: shows the ferrimagnetic material. Below Neel temperature (T_N) the material is antiferromagnetic and above T_N it is paramagnetic



1.2 Ac susceptibility:

In AC magnetic measurements, a small AC drive magnetic field is superimposed on the DC field, causing a time-dependent moment in the sample. As long as the AC field is small, the induced AC moment is

$$\chi_{AC} = (dM/dH)$$

$\chi = dM/dH$ is the slope of the M(H) curve, called the susceptibility.

A sample is subject to a small alternating field, $H(t) = H_{ac} \sin\omega t$ produced by a primary coil, and the resulting emf induced in a secondary coil wound around the sample is detected. This emf is directly proportional to the time derivative of the magnetization of the sample. One advantage of the AC measurement is that measurement is very sensitive to small changes in M(H) .

1.3 Different methods to measure susceptibility:

1.3.1 Vibrating sample method:

The basic principle is the flux changing in a coil. A sample is vibrated near the coil. The shape of the sample is usually spherical or small disc is connected to the end of a rod. A mechanical vibrator is fixed at the other end of the rod. When a magnetic field is applied, it induces some magnetization in the sample. This voltage difference is proportional to the magnetic moment of the Sample. It is a very versatile and sensitive. It can be applicable for both weak and strong magnetic substances [1].

1.3.2 Magnetometer method:

Actually it is very old method and simplest also. The basic principle behind this is a static measurement of the external field of the specimen by means of the deflection produced by the field in the angular position of a suspended magnet. The field applied to the specimen is supplied by a solenoid. In this method the deflection of the magnet measures the magnetization of the entire specimen averaged its volume. Here the shape of the specimen is ellipsoidal. This method is also very sensitive.

1.4 Method to measure ac susceptibility:

1.4.1 Ballistic Method:

This is an ac method to measure susceptibility. Here a search coil is wound on the centre of the specimen, which in turn is placed in the centre of a solenoid in the air gape of an electromagnet. The search coil and the solenoid are connected to the flux meter and magnetizing circuits, respectively. If the applied field is Ha and the demagnetization field is H_d then the true field is

$H= H_a-H_d = H_a-N_dM$

This value of H applies only to the centre of the specimen. Applying the value of the magnetic induction B we get,

$H=H_a- (B-Ha)/(4\pi/N_d)-1$

Here N_d=Ballistic demagnetization factor

1.5 DIFFERENCE BETWEEN AC AND DC SUSCEPTIBILITY:

In a dc magnetization the magnetic moment 'M' is measured for some applied dc field, H_{dc} .

The dc or static susceptibility is thus given by:

$\chi_{dc} = M / H_{dc}$

The ac method directly gives the susceptibility χ_{ac} , when an alternating current (ac) is applied. χ_{ac} =dM /dH .

In AC magnetic measurements, a small AC drive magnetic field is superimposed on the DC field, causing a time-dependent moment in the sample. The field of the time-dependant moment includes a current in the pickup coils, allowing measurement without sample notion. Thus one measures change in magnetization ΔM for a given change in magnetic fields ΔH. Whereas in the DC method, H remains constant.

Thus, the AC susceptibility is actually the slope (dM/dH) of the magnetization curve (M versus H curve). The AC technique detects changes in the magnetization the lead to dM/dH in the limit of small AC fields, and this is why sometimes referred to as a differential susceptibility. This is the fundamental difference between the AC and DC measurement techniques.

1.6 WHY AC SUSCEPTIBILITY?

1.6.1 SPIN GLASS:

Spin-glass behaviour is usually characterized by AC susceptibility. In a spin-glass, magnetic spins experience random interactions with other magnetic spins, resulting in a state that is highly irreversible and metastable. This spin-glass state is realized below the freezing temperature, and the system is paramagnetic above this temperature. The most studied spin-glass systems are typified by $Cu_{1-x}Mn_x$. The freezing temperature is determined by measuring χ' vs. temperature where a cusp appears at the freezing temperature. The AC susceptibility measurement is particularly important for spin-glasses, because the freezing temperature cannot be extracted from specific heat.

1.6.2 SUPERPARAMAGNETISM:

AC susceptibility measurements are an important tool in the characterization of small ferromagnetic particles which exhibit superparamagnetism. This theory was explained by Neel and Brown. In this theory, the particles exhibit single-domain ferromagnetic behaviour below the blocking temperature T_B, and are superparamagnetic above T_B .

1.6.3 MAGNETIC PHASE TRANSITIONS:

The low frequency susceptibility measurement behaviour is similar to that of dc measurement**.** χ diverges at the critical temperature of a ferromagnetic phase transition. Critical exponents characterize the nature of the divergence as a function of temperature and DC applied field.

1.6.4 SUPERCONDUCTIVITY:

AC susceptibility is the standard tool for determining the physics of superconductors as well as in measuring critical temperature. In the normal state, superconductors have a small susceptibility. In the fully superconducting state, the sample is a perfect diamagnetic and so $\chi' = -1$. The nonzero χ' is taken as the superconducting transition temperature.

So, that ac susceptibility has many advantages over dc susceptibility. It is the reason for choosing ac susceptibility.

1.7 Motivations:

Our senior batch students have made an experimental set up for ac susceptibility measurement with Teflon having a primary coil, a secondary coil and a sample holder. They got a sharp rise in susceptibility is observed at 90^0C (363K) i.e. a high temperature Susceptibility measurement. This gave us motivation to design a cryocooler based ac susceptometer with hylum that can perform an experiment at very low temperature i.e. below liquid nitrogen temperature in closed cycle refrigerator system (CCR).

CHAPTER 2

LITERATURE REVIEW

M. Nikolo *et al*., Ph.D. thesis, University of Colorado, Boulder (1991):

- ✓ In the dc measurement, the magnetic moment of the sample does not change with time.
- ✓ An ac output signal is detected but this signal arises from the periodic movement of the sample and therefore it does not represent the ac response of the sample itself.
- ✓ On the other hand, in an ac measurement the moment of the sample is changing in response to an applied ac field. Thus the dynamics of the magnetic system can be studied.
- ✓ This method also offers the opportunity to determine the frequency dependence of the complex susceptibility which leads to information about relaxation processes and the relaxation times of the magnetic systems studied.

M. Nikolo *et al*., (1994):

- ✓ This inductive method presents a rapid means of measuring samples without the need for current or voltage leads.
- ✓ The measurement of ac susceptibility can provide important information about the superconducting properties.
- ✓ The imaginary part of the complex susceptibility can be used to probe the nature of the coupling between grains.

R. B. Goldfarb, V. Minervini *et al.*, Rev. Sci. Instrum. 55, 761-764 (1984):

- ✓ The principle of measuring ac susceptibility involves subjecting a sample to a small alternating magnetic field. The flux variation due to the sample is picked up by a sensing coil surrounding the sample and the resulting voltage induced in the coil is detected.
- ✓ This voltage is proportional to the time derivative of the sample's magnetization as will be shown here. Using the concept of mutual inductance one can derive an expression for X in terms of directly measurable quantities.

H. Scofield *et al*., Am. 1. Phys. 62, 129-133 (1994):

- ✓ The lock-in amplifier acts as a discriminating voltmeter. It measures the amplitude and the relative phase of an ac signal. Here, a reference signal having the same fundamental frequency as the desired measured signal and having a fixed phase relationship with it is provided.
- ✓ The lock-in amplifier acts as though it were a band pass filter of enormously high *Q* with its center frequency at the desired signal frequency.
- ✓ The output of a lock-in amplifier is a magnified dc voltage proportional to its synchronous ac input signal.

M. Nikolo et al., (1994):

- ✓ The most useful features of the ac susceptometer is that both the real or in-phase component *X'*, and the imaginary or out-of-phase component, *X'*, can be measured.
- ✓ To measure both components, the lock-in detector requires a reference signal at the same frequency and in phase with the current from the ac current source.
- ✓ If the temperature of the primary coil changes, the phase angle will vary with the changing resistance of the coil and the lock-in amplifier should be referenced to the voltage drop across a resistor, placed outside the cryostat, in series with the primary coil.
- ✓ On the secondary circuit, the input impedance of the lock-in amplifier is large enough to make any change in the coil resistance insignificant.
- ✓ The voltage signal across the secondary coils (inductive circuit) is to a first order 90° out of phase with the reference voltage signal, which in turn is in phase with voltage signal across a precision resistor in series with the driving primary coil.

S. Ramakrishnan , S. Sundaram, R. S. Pandit, and G. Chandra *et al.*, J. Phys. E 18, 650 (1985):

- ✓ Frequency and field dependence of real and imaginary parts of ac susceptibility has become an established tool for investigating magnetic materials.
- ✓ In this technique, the change in the mutual inductance of a primary and two identical but oppositely wound secondary's is measured when a sample is placed in it. This change is a measure of the susceptibility of the sample.

A. F. Deutz, R. Hulstman, and F. J. Kranenburg *et al.*, Rev. Sci. Instrum. 60, 113 (1989):

- ✓ One of the major experimental problems is that of the offset voltage in the secondary coils even in the absence of the sample. Various instruments differ in the offset nullification techniques.
- ✓ For instance, in some of the setups, a precision variable mutual inductance is adjusted to balance the bridge or ratio transformers and Kelvin–Varley voltage dividers are used to nullify the offset.
- ✓ The aim of the present work is to construct an inexpensive ac χ setup for an automated and simultaneous measurement of ac χ as well as its harmonics detected accurately in real and imaginary parts.

CHAPTER 3

EXPERIMENTAL SET UP:

3.1 Review of Mechanical assembly:

W. L. Pillinger, P. S. Jastram and J.G. Daunt *et al*., Rev. Sci. Instrum 29,159 (1958):

The length of the glass cryostat is around 300 mm. The lower tail of the glass cryostat is a double-walled cylinder of inner diameter 7 mm and length 200 mm. It is dipped into liquid N_2. The sample holder is a sapphire plate of length 70 mm and width 4 mm. It is attached to a hollow stainless steel rod through a non-metallic hylum a fiber impregnated polymer extension of 60 mm length. The sample holder moves up and down through the Wilson seal inside the glass cryostat.

Primary and a set of secondary coils are wound on separate hylam formers. Hylum was chosen for its thermal and mechanical properties. We have made two sets of the coil system. One major problem is that offset voltage, which cannot be changed by unwinding the few secondary turns, once the coil set is wound. The problem is more pronounced with the secondary coils having a large no. of turns. The uniformity and tension of the windings in the primary as well as secondary coils cannot be identical; hence the individual inductive and capacitive coupling of secondaries with the primary is different. From literature review, the diagram of typical ac susceptometer is given below-

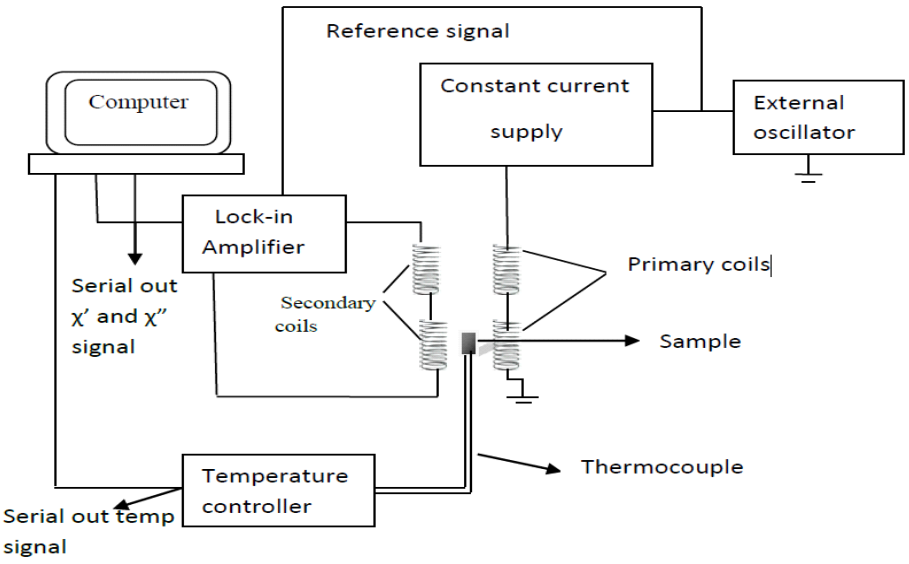


Fig 3.1: Typical ac susceptometer [11]

3.2 Cross-Section view of our 1st design:

3.2.1 Design of secondary coil:

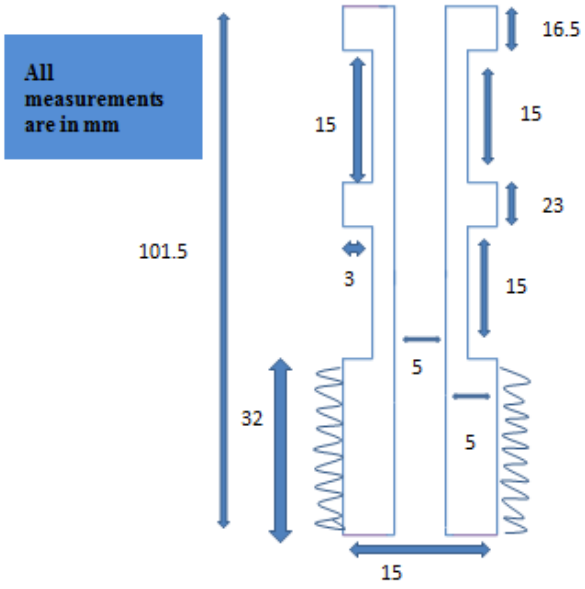


Fig 3.2: schematic view of secondary coil

Fig 3.3: secondary coil made up of hylum

3.2.2 Design of Primary coil:

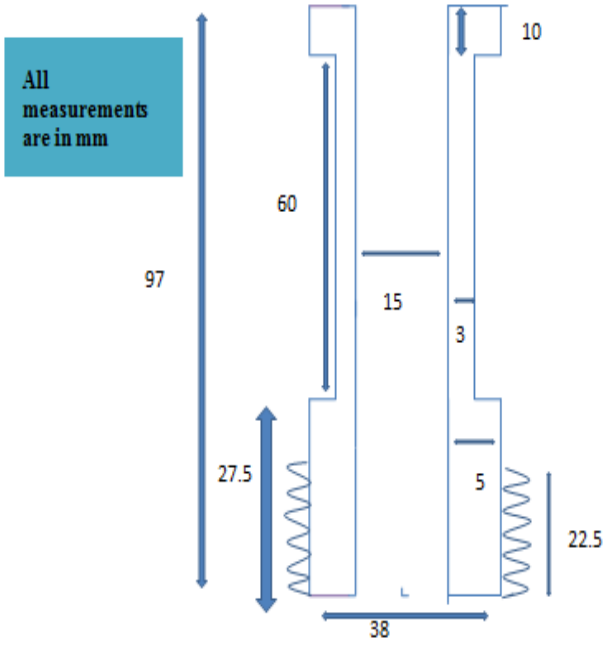


Fig 3.4: schematic view of primary coil

Fig 3.5: Primary coil made up of hylum

Copper wire (150 micron) is wounded on the primary and the secondary coils. For the 1st design, the number of turns in primary coil is 3300 and in secondary coil it is 1400 in which 700 turns is clockwise and another 700 turn is anticlockwise. The material chosen for the design is Hylum for its thermal conductivity. A Pt100 temperature sensor is put inside the secondary coil that touches to sample. This design was dipped in liquid nitrogen for achieving lower temp. But the dimension of the previous design is modified to fit in Closed Cycle Refrigerator to attain the liquid Helium temperature. Hence we modified the design.

3.3 Modified design for CCR:

3.3.1 DESIGN OF SECONDARY AND PRIMARY COIL:

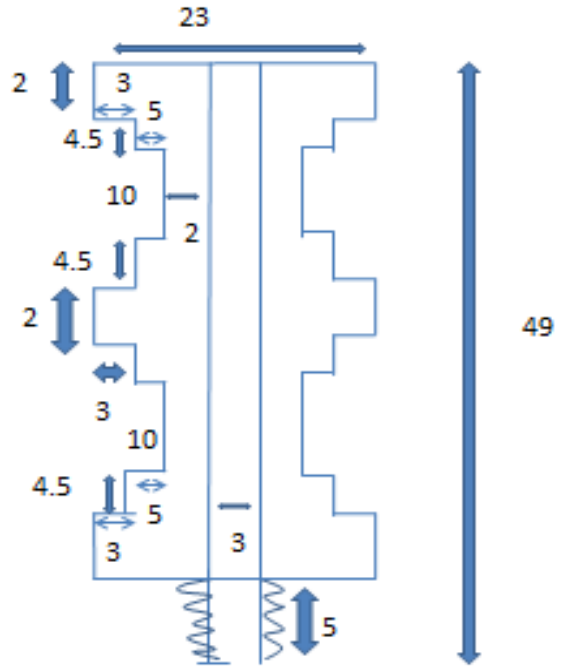


Fig 3.6: schematic view of secondary and primary coil

Fig 3.7: design of susceptometer mounted on CCR

Copper wire (150 micron) is also wounded on the primary and the secondary coils. Here the secondary coil groove is made below the primary coil groove. The number of turns in primary coil is 3000 i.e. 1500 each in same direction and in secondary coil it is 3600 in which 1800 is clockwise and another 1800 is anticlockwise. A Pt100 temperature sensor is put inside the secondary coil that touches to sample. Lock-in amplifier is used to detect and measure very small AC signal. After that we performed our experiment.

3.4 Experimental set up figure:

Fig 3.8: CCR

Fig 3.9: compressor

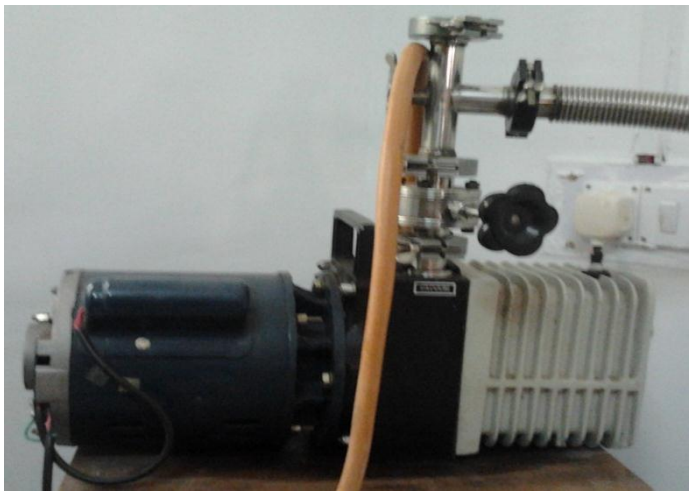


Fig 3.11: Vacuum pump

Fig 3.10: Lakeshore 331 temp. controller

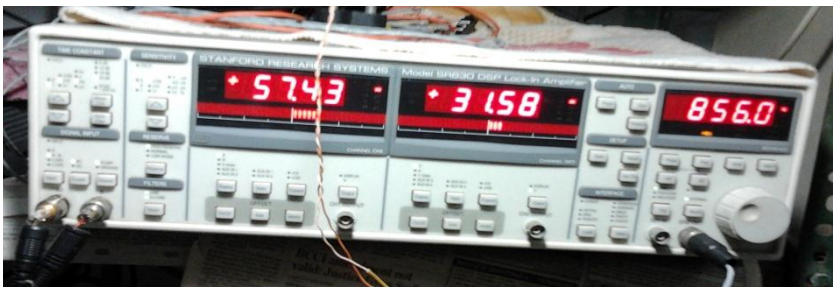


Fig 3.12: Lock-in-amplifier

3.5 Magnetic field at the centre of the solenoid:

We have used the formula for the solenoid H= Ja F (α β)

Where

$$F(\alpha,\beta) = \frac{\mu\left(1+\sqrt{\alpha^2+\beta^2}\right)}{\left(\alpha+\sqrt{\alpha^2+\beta^2}\right)}$$

where α = b/a and β=*l*/a are the inner and outer radius of the solenoid. 2*l* is the length of the solenoid.

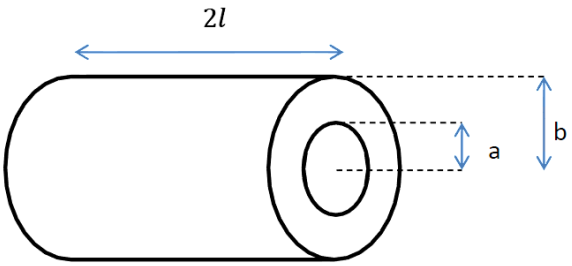


Fig 3.13: solenoid and its dimensions

Current density is J = I/A Where I= total current passing through the solenoid and A= total cross-sectional area offered to the current flow, {2*l*(b-a)} . In this method the change in flux dφ is measured as induced voltage V(t) in the secondary coil.

The measured rms voltage V across the two secondary coil is :-

$V(t) = -d\emptyset/dt$

The magnetic flux through the N turn oppositely wound coils of radius a is :-

$\emptyset = \pi\ 2(t)\mu_0$

With M(t) denoting the magnetic induction inside the sample averaged over the volume V.

So, V(t) = $-\mu_0\ \pi\ ^2NdM(t)/dt$

But for complex susceptibility χ_n' and χ_n'' one can do a Fourier expansion of M(t)

M (t) = Σ Hac (χ_n' cos nωt + χ_n" sin nωt)

Putting M(t) in the equation of V(t) ,we get,

V(t) = V_0Σ n (χ_n' sin nωt – χ_n" cos nωt)

Where $V_0 = \mu_0 \pi\, 2\omega N H_{ac}$

The real and imaginary component of susceptibility χ_n' and χ_n" are determined directly from M(t) through the relationship

$$\chi_n' = \frac{1}{\pi H} \int_0^{2\pi} M(t) \sin(n\omega t) d(\omega t)$$

$$\chi_n'' = \frac{1}{\pi H} \int_0^{2\pi} M(t) \cos(n\omega t) d(\omega t)$$

Here H is alternating magnetic field(H_{ac}). n=1 denotes the fundamental susceptibility where n= 2,3,4… etc are the higher order harmonics associated with non linear terms in χ .

For this set-up we have designed, the value of a= 8.5mm, b = 11.5mm and l= 22mm

Putting these values we get , $\alpha = 1.35$ and $\beta = 2.588$

So, the value of $F(\alpha,\beta) = 0.3186$

So, the average current density $J = I/A$

Where $A=2l(b-a) = 132 \times 10\text{-}6m2$

So, J= 37.87 A/M2

So, B= $1.289*10^{-7}$ Tesla

$V_0 = \mu_0\, \pi a^2 \omega N H_{ac}$

$V_0 = \pi \times (8.5 \times 10^{-3})^2 \times 2\pi \times 856 \times 3000 \times 1.289*10^{-7}$

$= 4.72 \times 10^{-4}$ volts

CHAPTER 4

SAMPLE PREPARATION

4.1 CHOICE OF SAMPLE:

Lanthanum strontium manganite (LSMO) is an oxide ceramic material. The general formula is $La_{1-x}Sr_xMnO_3$, where *x* describes the doping level of strontium. *x* is usually in the range of 10-20%. It has a perovskite crystal structure (ABO_3). In the crystal, the 'A' sites are occupied by lanthanum and strontium atoms, and the 'B' sites are occupied by manganese atoms.

4.2 PROPERTIES OF LSMO:

LaMnO3 is an intrinsic p-type conductor. Electronic conductivity is enhanced by substitution of the La^{3+} site with strontium. When a L^{3+} ion at the A-site is replaced by a Sr^{2+} ion, an electric hole is formed and causes increase in electrical conductivity. The electronic conductivity depends on the preparation and crystalline structure of the LSMO sample. D'Souza and Sammes determined for LSMO at room temperature that similar compositions of LSMO do not always show low strength. Since strength is sensitive to processing[13]. It is possible to obtain a wide range of strength values for the same composition.

4.3 APPLICATIONS OF LSMO:

- LSMO is primarily an electronic conductor.
- This material is commonly used in as a cathode material in commercially produced solid oxide fuel cells (SOFCs).
- LSM behaves like a half-metal and use in spintronic devices.
- LSMO motors used in oil and drilling industries.
- This figure 4.1 shows the temperature dependence of resistivity of LSMO. No ferromagnetic phase transition is observed for concentration x ≤ 0.05. It shows a non-

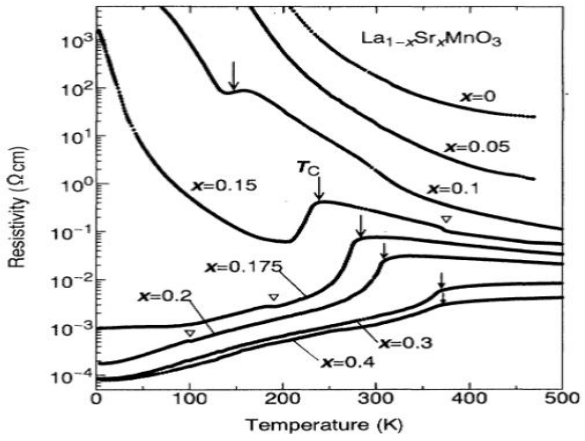


Fig 4.1 Temperature dependence of resistivity for LSMO crystal [13]

metallic behaviour for x ≤ 0.2. With the increase in "x", it shows a metallic behaviour with the shifting of Tc towards high temperature [13].

- Below the magnetic transition temperature, three phase are present spin-canted anti ferromagnetic insulator (CNI) in low doped region (x < 0.1), a ferromagnetic insulator (FI) in the region 0.1 to 0.15 and a ferromagnetic metal (FM) in high doped region x > 0.15. Above the magnetic transition temperature (Tc and TN) the non-metal to metal transition takes place at x=0.3 [13].

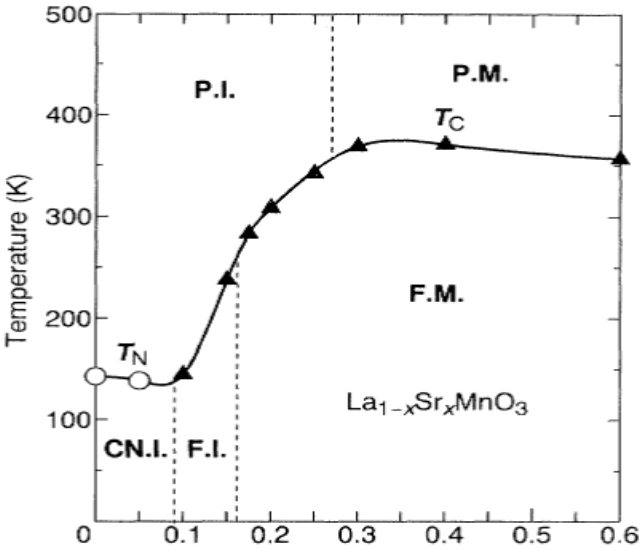


Fig 4.2: Electronic phase diagram of LSMO [13]

4.4 LSMO PREPARATION:

There are many conventional methods to prepare LSMO. These are

- Sol-gel method
- Solid-state method
- Pulsed laser deposition method
- Metal organic deposition technique
- Spray dryer method
- Co-precipitation method

The components corresponding to the formula $La_{0.83}Sr_{0.17}MnO_3$ is prepared by sol-gel method. The precursors used are Manganese carbonate ($MnCO_3$), Lanthanum nitrate $(LaNO_3)_3$, Strontium nitrate $(Sr(NO_3)_2)$ and the fuel used is glycine. The calculation of the given mixture is given the table below. They were taken in different molar ratio to prepare a nominal stoichiometry. At first a starting solution of nitrate precursors are separately made by mixing $MnCO_3$ with minimum amount of concentrated nitric acid (HNO_3) and $La(NO_3)_3$ and $Sr(NO_3)_2$ in distilled water. Glycine was taken as a combustion agent. All these solutions were mixed and to this resultant solution glycine is added in stoichiometric proportion. The solution was then heated with constant stirring at

100 °C to evaporate the excess solvent. After 3 hours of heating the solution converted to a viscous gel. Then after burning the gel is converted to the black powder. The gel was dried at 250 °C. After collecting the powder, calcination is done at 770°C for 3 hours. After cooling the sample is collected from the furnace and is grinded in a agate-mortar pestle. This obtained powder is then grinded for 2 hrs. This grinded powder is then pressed into pellet and then sintered at 770^0C for 3 hrs. Hence the sample was ready.

Calculation for $La_{0.83}Sr_{0.17}MnO_3$:

Compound name	Weight in gram	Total weight taken (gm)
$MnCO_3$	114.95	1.1495
$La(NO_3)_3$	433.02	3.594066
$Sr(NO_3)_2$	211.63	0.359771
Glycine	75.07	1.5014

4.5 Flow chart for sample preparation:

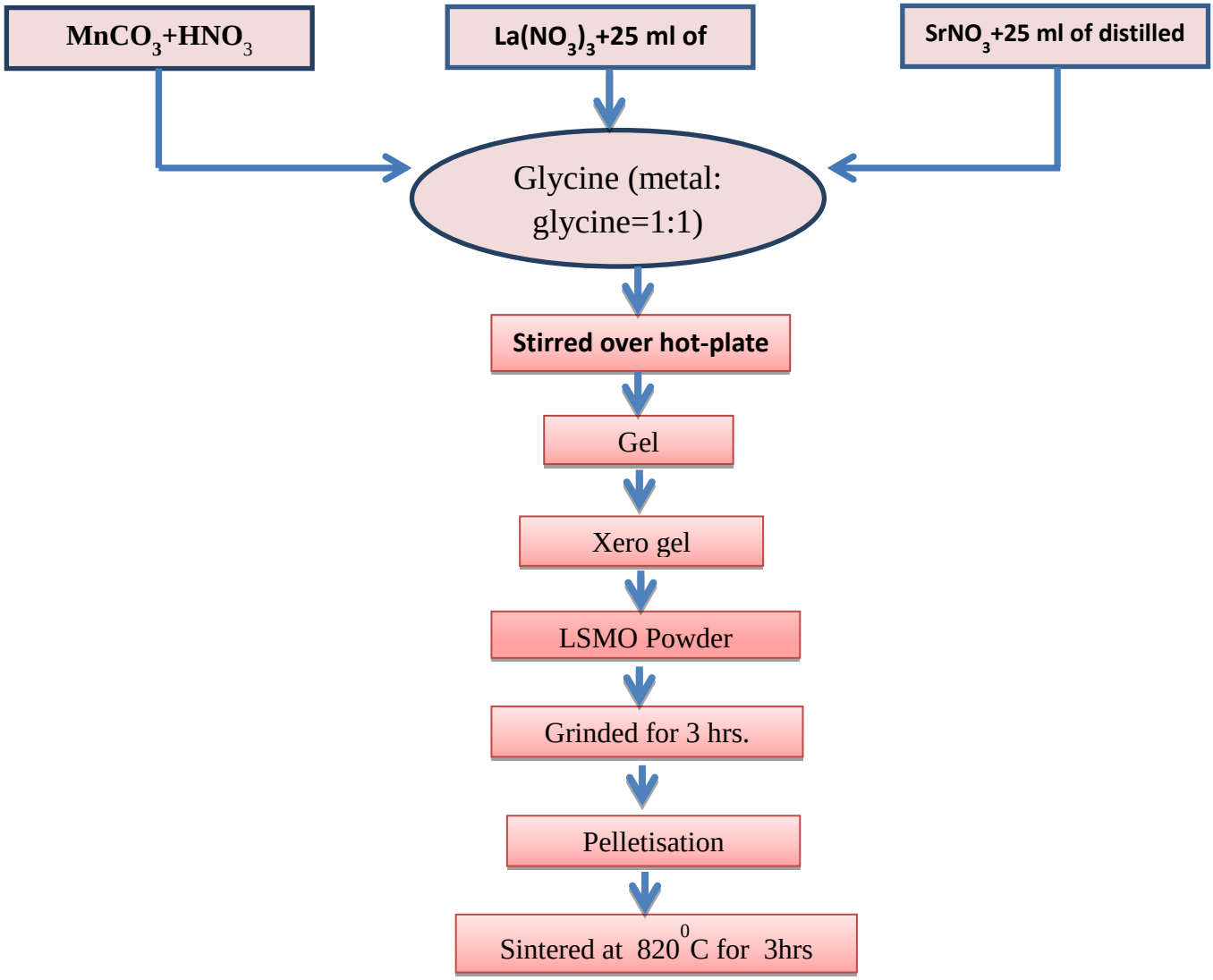


Fig 4.3: Flow chart for sample preparation

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Ac Susceptibility of the LSMO:

The sample was cut into rectangular shape (7mm) and carefully placed at the centre of one of the secondary coil. Care is taken in placing the sample in the middle of the coil in order to avoid the edge effect. The set up is cooled down to 10K. The x- component (in phase) and y-component (out of phase) of signal are recorded by lock in amplifier as a function of temperature. These x and y component is then converted in volume susceptibility by dividing with parameter v_0. Around 180 K, the susceptibility starts rising sharply and till the lowest temperature of measurement no saturation is observed. The rise is similar both for in-phase and out-of-phase components of susceptibility. This reflects ferromagnetic nature of LSMO, as per literature review.

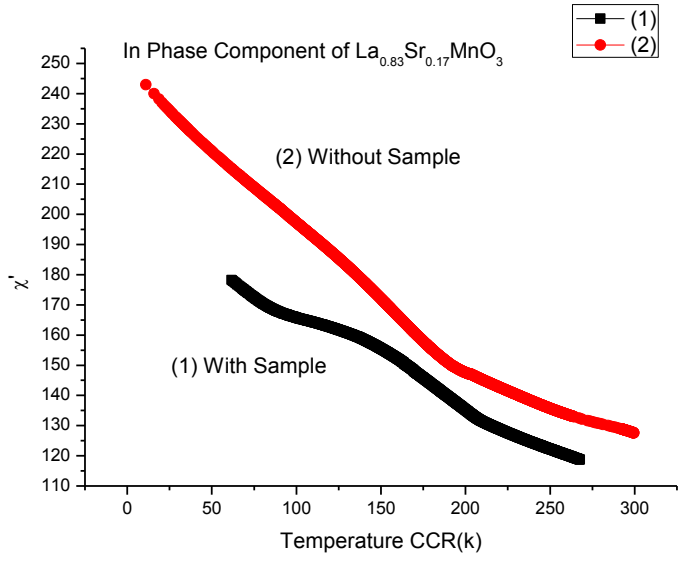


Fig 5.1: In-phase susceptibility of $La_{0.83}Sr_{0.17}MnO_3$ with & without sample

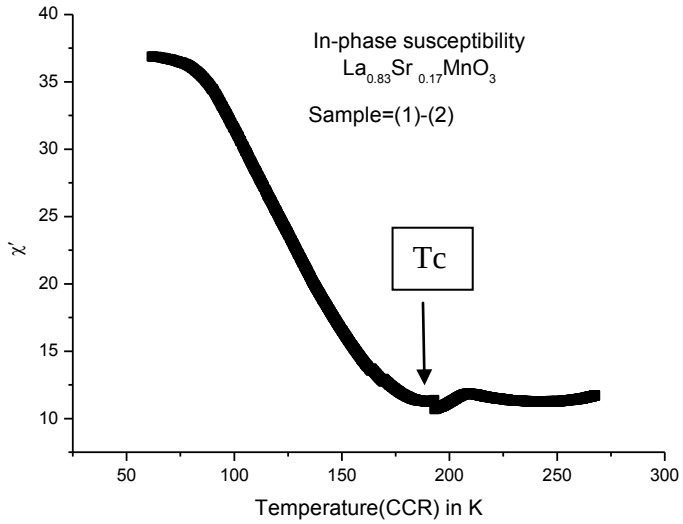


Fig 5.2: In-phase susceptibility of $La_{0.83}Sr_{0.17}MnO_3$ with T_c 180K

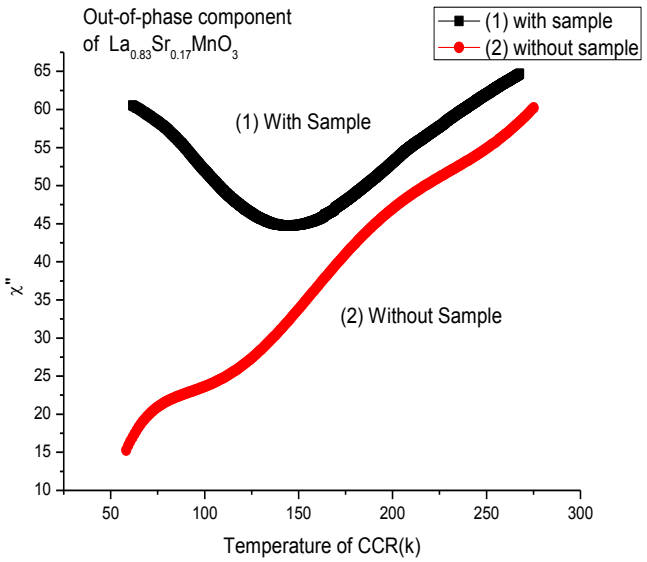


Fig 5.3: Out-of-phase susceptibility of $La_{0.83}Sr_{0.17}MnO_3$ with & without sample

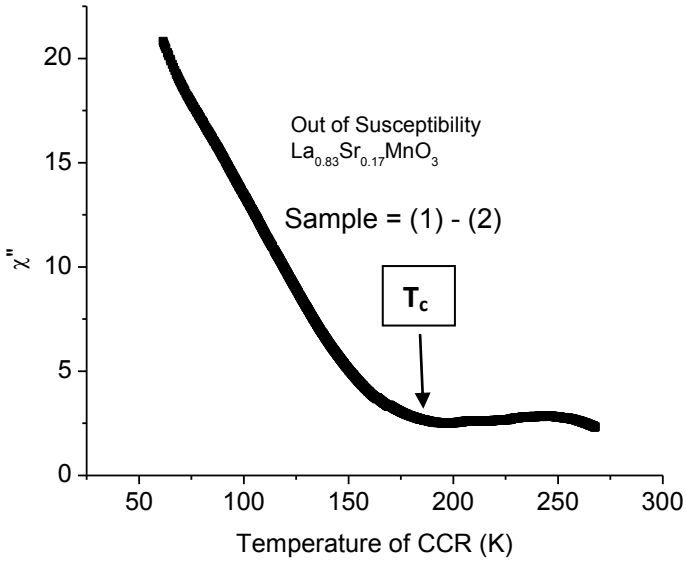


Fig 5.4: Out-of-phase susceptibility of $La_{0.83}Sr_{0.17}MnO_3$ with T_c 180K

From above experimental figure, we got the transition temperature of the $La_{0.83}Sr_{0.17}MnO_3$ sample around at 180K. At this temperature, the susceptibility is rising sharply with decreasing the cryocooler temperature. It is observed in both in-phase and out-phase susceptibility component. As per literature review, the transition temperature of $La_{0.83}Sr_{0.17}MnO_3$ is 260K. However we observed it at ~180K. This is due to the temperature difference between cryocooler temperature and the sample temperature. Another reason is that vacuum does not allow the heat flow from sample to the cryocooler base. This is why the transition temperature of $La_{0.83}Sr_{0.17}MnO_3$ is found around at 180K.

CHAPTER 6

CONCLUSION

A cryocooler based ac susceptometer has been successfully designed and fabricated. The former material chosen is Hylum. The design consists of a primary coil, a secondary coil and a sample holder. Copper wire (150micron) winding was done on the primary as well as the secondary coil. The secondary coil is connected in series opposition i.e. if one wound is clockwise, the other is anticlockwise.

The set-up is based on the principle of "mutual induction". A Pt100 temperature sensor is put in close proximity with the sample, in order to record the sample temperature accurately. $La_{0.83}Sr_{0.17}MnO_3$ sample is prepared as test sample for measuring the susceptibility.

After preparation, the sample was cut into rectangular shape (7mm) and carefully placed at the centre of one of the secondary coil. The sample was placed very carefully in the middle of the secondary coil in order to avoid the edge effect. The set up was cooled down to 10K. After getting the Susceptibility value when we plot it with temperature we got the transition of LSMO is 180K.

Hence my set up was working properly and the sample which I have made was also properly made.

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