DESIGN OF A CRYOCOOLER BASED AC SUSCEPTOMETER

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 work in the report entitled **"DESIGN OF A CRYOCOOLER BASED AC SUSCEPTOMETER"** by **Abakash Pradhani, Sanjaya Kumar Parida and Tripta Parida** in partial fulfilment of Master of Science degree in **PHYSICS** at the National Institute of Technology, Rourkela; is an authentic work carried out by them under my supervision and guidance. The work is satisfactory to the best my knowledge.

Dr. Prakash Nath Vishwakarma

Department of Physics

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DECLARATION

I hereby declare that the project work entitled "**DESIGN OF A CRYOCOOLER BASED AC SUSCEPTOMETER**" submitted to the NIT, Rourkela, is a record of an original work done by me under the guidance of **Dr. Prakash Nath Vishwakarma** Faculty Member of NIT, Rourkela and this project work has not performed the basis for the award of any other Degree or diploma/ associate ship/fellowship and similar project if any.

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Date: **Abakash Pradhani**

CONTENTS PAGE NO.

ABSTRACT

An experimental set up for cryocooler based ac susceptometer is designed and fabricated. Hylum is chosen as the former material. Primary and secondary coils are winded over a single hylum former. Secondary coil, a set of two equal and oppositely winded coils, is first winded over the hylum former. Then the primary coil is winded over it. Copper wire (150 micron) is used for winding. The whole set up is then set in the cryocooler (HC-4E, Sumltomo cryogenics). An ac current is supplied to the primary coil from DSP Lock-in Amplifier (Model SR830), which produces the required magnetic field. This magnetic field magnetize the sample and an induced emf in secondary coil is observed. The effect of magnetic field in secondary coil is cancelled by the two oppositely winded coil system. So that the emf induced is only due to the change in moment of sample. This emf is then separated to in-phase and out-of phase voltage component in lock-in amplifier. The in-phase and out-of phase voltage component is converted to in-phase and out-of phase component of ac susceptibility by dividing a voltage factor V_0. Temperature is lowered down to liquid helium temperature in cryocooler and data are recorded using lab view program. Then the in-phase and out-of phase component of ac susceptibility is plotted against varying temperature. This is the background plot for ac susceptometer.

In our project we have chosen $La_{1-x}Sr_xMnO_3$ (LSMO) as sample. Sample is prepared by auto combustion sol-gel method. Pellets are made and cut into rectangular shape. One such rectangular shaped cut sample is carefully placed in the centre of one of the secondary coil. Then the ac susceptibility measurement is done by retaining all the parameter such as applied voltage and frequency constant in lock-in amplifier. Data are plotted and the susceptibility plot with sample is obtained. The susceptibility plot for sample was obtained by deducting the background from the susceptibility plot with sample. The phase change in the LSMO system is studied using the Ac susceptometer.

CHAPTER-1

INRODUCTION

The magnetic properties describe the behavior of any substance under the influence of magnetic field. When we apply a magnetic field, the material gets magnetised. Magnetisation is the net magnetic moment developed per unit volume when a magnetic field is applied and is denoted by M. The magnetisation is different for different materials. Hence this is a measure of the quality of the magnetic materials. Another important parameter used to measure the quality of magnetic materials is the magnetic susceptibility, χ. The magnetic susceptibility χ of a material is the ratio of the magnetization M to the applied field H.

$$\chi = M/H.$$

The above relation is for a time independent or dc applied magnetic field and also called the dc susceptibility, χ_{dc}.

When a time dependent magnetic field is applied,the susceptibility is given as:

$$\chi_{ac} = dM/dH,$$

Where, χ_{ac} is now the differential or ac susceptibility.

These two types of susceptibility will be discussed later. Generally, χ is a dimensionless quantity. The susceptibility can also be defined with respect to unit volume or unit mass or unit molar of substance. If the susceptibility is measured per unit mole of substance, then it is χ_{mol} termed as *molar susceptibility*. Similarly, for volume susceptibility χ_v = M/H. Mass susceptibility $\chi mass = \chi_v /\rho$, and molar susceptibility $\chi mol = M\chi mass = M\ \chi v/\rho$ where ρ is the density. Although B, H, and M must necessarily have the same units, it is customarily to denote in CGS (SI) units, B in gauss (G) or tesla (T), H in Oersted (Oe) = A/m and M in erg/Oe cm^3 or emu/cm^3 (A/m) **[1]**

1.1 Magnetic behaviour of materials

The magnetism in solids arises due to orbital and spins motion of electrons as well as spins of the nuclei. The motion of electrons is equivalent to an electric field which produces the magnetic effects**[2]**. The major contribution comes from the unpaired valence electrons which produces permanent magnetic moments. Hence the magnetic behaviour of any material depends on the electronic arrangement within the atom and the magnitude and sign of susceptibility vary with the type of magnetism, and hence characterises the various magnetic materials. There are five types of magnetism exhibit by different magnetic materials:

1) Diamagnetism,

2) Paramagnetism,

3) Ferromagnetism,

4) Antiferromagnetism,

5) Ferrimagnetism.

1) Diamagnetism:

Diamagnetism is a very weak effect and is observed in materials which do not have any permanent magnetic moments. In a material, it arises due to changes in atomic orbital states induced by applied magnetic field. Diamagnetic materials have a very weak negative susceptibility, typically of order -10^{-6}. That is when a diamagnetic material is placed in a magnetic field, $B < \mu_0 H$.The examples of some materials exhibiting diamagnetism are, Bi, Hg, Ag, Pb, Cu, H_2O, etc. Table.1 gives the diamagnetic susceptibility values for different diamagnetic materials. All materials show diamagnetic behaviour, but in para- and ferromagnetic materials this weak negative value of susceptibility due to diamagnetism is supressed by higher positive positive paramagnetic or ferromagnetic susceptibilities, so that their overall susceptibility is positive. Figure.1 shows the temperature independent behavior of diamagnetic susceptibility.

Materials	χ (10^{-5})
Bi	-16.6
Hg	-2.9
Ag	-2.6
Pb	-1.8
Cu	-1.0
H_2O	-.91

Table 1 χ values for diamagnetic materials

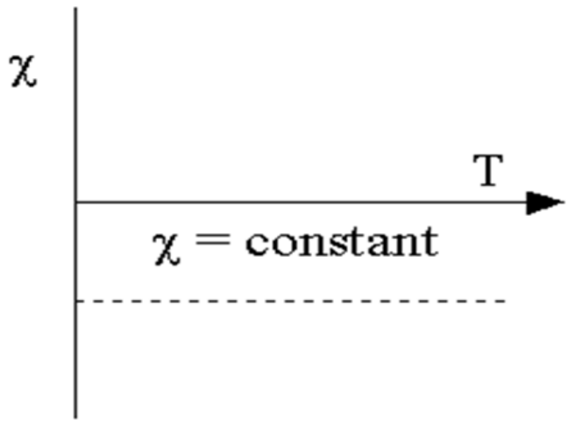


Fig 1.1 shows χ vs. temperature.

2) Paramagnetism:

Paramagnetism occurs in those materials which have permanent magnetic moments. When the magnetic field is not applied, these moments are randomly oriented and no net magnetisation is produced. In a applied magnetic field, these moments start orienting themselves in the direction of field resulting in some net magnetisation in the direction of field. The paramagnetic materials have small, positive and temperature-dependent susceptibility. At liquid helium temperatures (of order 1 K), susceptibilities is of order $+10^{-3}$ or $+10^{-2}$, thus greatly exceeding the small negative susceptibility. The examples of some paramagnetic materials are Na, gaseous nitric oxide, O_2, Mn^{+2}, etc. At room temperature, paramagnetic susceptibilities are much less and are about $+10^{-5}$, barely exceeding the diamagnetic susceptibility. The paramagnetic susceptibility is a function of temperature and decreases with increase in temperature. Figure.2 shows the applied field and temperature dependent behavior of paramagnetic materials.

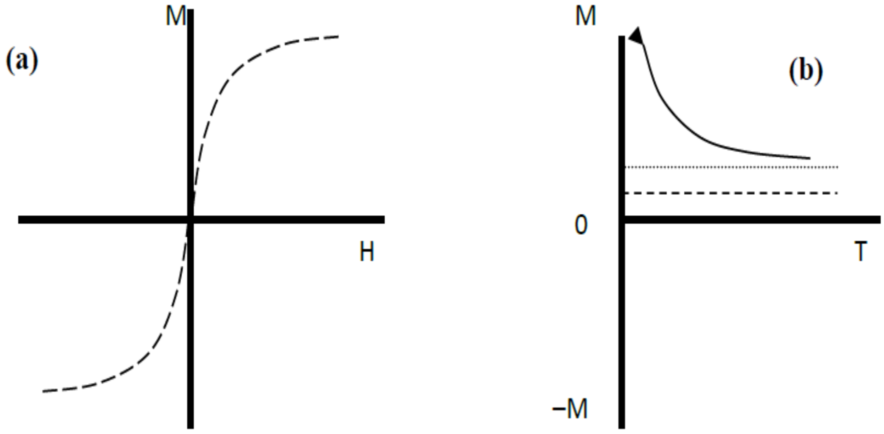


Fig 1.2 shows a) M vs. H and b) M vs. temperature plots for paramagnetic materials [2].

3) Ferromagnetism:

Like paramagnetism, ferromagnetism is also associated with the presence of permanent magnetic dipoles, but the moments of adjacent atoms are aligned in a particular direction even in the absence of applied magnetic field. Thus a ferromagnetic material exhibits a magnetic moment in the absence of a magnetic field. The magnetisation existing in absence of an applied magnetic field is called the spontaneous magnetisation. Ferromagnetic materials have a number of small regions called domains which are spontaneously magnetised. Thus ferromagnetic materials possess a large and positive value of susceptibility, typically of order $+10^3$ or 10^4 or even greater. The examples of ferromagnetic materials are the elements such as Fe, Co, Ni, Gd, and a number of alloys and oxides such as MnBi, MnAs, CrO_2, etc. The ferromagnetic susceptibility of a material is quite temperature sensitive. Above a temperature known as the Curie temperature, the material loses its ferromagnetism, and it becomes merely paramagnetic. The effect of applied magnetic field and temperature on magnetisation of ferromagnetic materials is shown in fig 1.3 below.

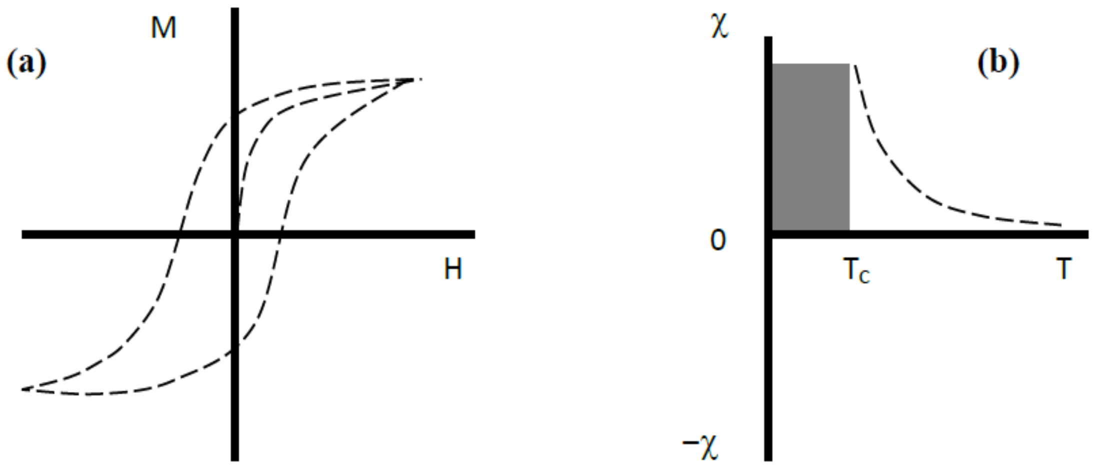


Fig 1.3 shows (a) the M-H loop and (b) χ vs. temperature behavior of ferromagnetic materials. BelowCurie temperature (Tc) the material is ferromagnetic and above Tc the material behaves as paramagnet.

4) **Antiferromagnetism:**

It involves materials in which the atoms or ions or molecules have a permanent dipole moment and the crystals have domain structure, but alternating ions within a domain have their magnetic moments oriented in opposite directions, which cancels each other's effect and domain as a whole has zero magnetization, or zero susceptibility. This type of magnetism was first observed in MnO crystals. In the absence of applied magnetic field, the neighbouring magnetic moments cancel each other and the material has no magnetisation. When a field is applied, a small magnetisation appears in the direction of the field and it increases with increase in temperature. Such behaviour is the characteristics of antiferromagnetic materials. The magnetisation is maximum at a critical temperature $T_{N,}$ called the Neel temperature. Above Neel temperature, the material loses its antiferromagnetism, and it becomes merely paramagnetic. The effect of applied magnetic field and temperature on magnetisation of antiferromagnetic materials is shown in fig 1.4 below.

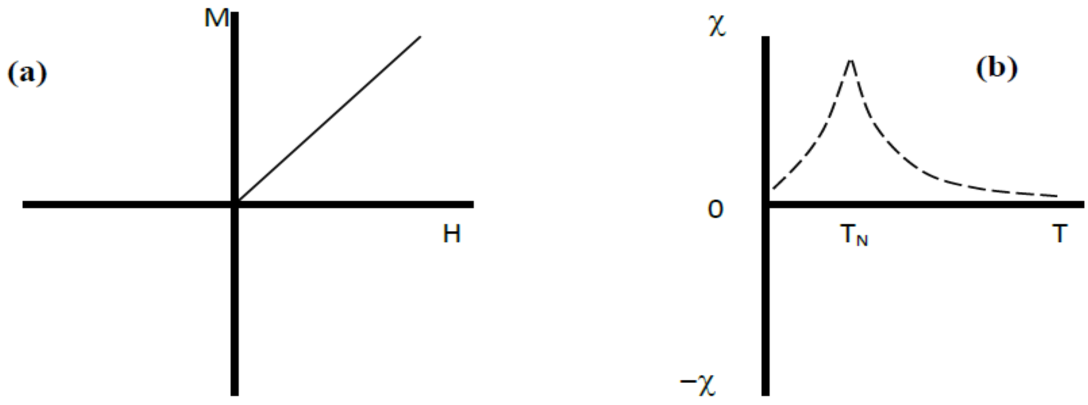


Fig 1.4 shows (a) M-H curve and (b) M-T response of antiferromagnets, Below Neel temperature(T_N) the material is antiferromagnetic and above T_N it is paramagnetic.

5) Ferrimagnetism:

Ferrimagnetism is similar to antiferromagnetism but the adjacent moments are unequal in magnitude and hence complete cancellation of moment does not take place. Examples, Fe_3O_4, $MgAl_2O_4$, etc. A ferromagnetic material resembles a ferromagnetic as both possess spontaneous magnetisation and exhibit hysteresis and identical magnetic properties.

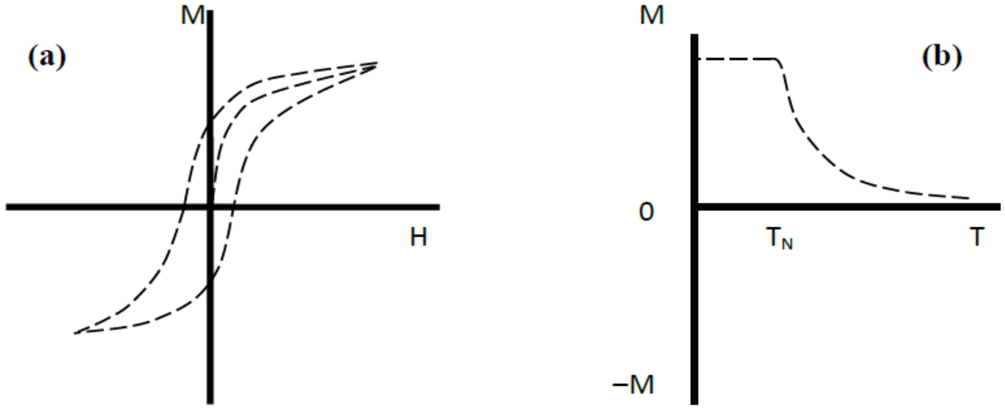


Fig 1.5 shows the (a) M-H loop and (b) M-T response of ferrimagnetic material.

1.2 TYPES OF SUSCEPTIBILITY

As mentioned earlier there are two types of susceptibility: ac susceptibility and dc susceptibility. Both have their own significance and application. The difference between ac and dc susceptibility is discussed below.

DIFFERENCE BETWEEN AC AND DC SUSCEPTIBILITY

Mainly there are two methods used to measure susceptibility of a material, i.e., ac and dc method. In dc method, the magnetisation M is measured which can be converted to susceptibility χ using relation χ_{dc}= M/H, where H is the applied magnetic field. In ac method, the ac susceptibility, χ_{ac}= dM/dH, is directly measured when an alternating current (ac) is applied. The ac and dc methods are two entirely different tools that provide different ways of examining magnetic properties. The variation in the magnetic flux due to magnetic sample is measured in both of these techniques.

In dc magnetic measurements the equilibrium value of the magnetisation of a sample is determined. A dc magnetic field is used to magnetise the sample and the magnetic moment of the sample is measured. The moment can be measured by force, torque or induction techniques. The induction technique is the most common in modern instruments. In Inductive measurements the sample is moved relative to a set of pickup coils. In conventional inductive magnetometers, the voltage induced is measured by the moving magnetic moment of the sample in a set of copper pickup coils. For ac magnetic measurements Inductive magnetometers can also be used.

In dc magnetic measurements the magnetic moment does not change with time, whereas in ac magnetic measurement the moments change their direction with response to the applied ac field. The dynamics of the magnetic system can be studied by ac susceptibility measurement. The frequency dependence of complex ac susceptibility can be studied which provide important information about the superconducting properties and ac losses in polycrystalline high T_c superconductors. The transition temperatures of both inter- and intragranular regions can be found out using ac measurement technique [3].

1.3 SUSCEPTIBILITY MEASUREMENTS

Different methods are used for the measurement of ac and dc susceptibility. The different methods are as follows.

EXTRACTION METHOD:

In this method we measure the flux change in the search coil due to the removal or extraction of specimen from the applied magnetic field[1]. The magnetic flux is measured for two cases. First, when both search coil and specimen are present in the magnetic field and then when the specimen is suddenly removed from the magnetic field. The difference of magnetic flux in the two measurements gives the magnetisation of the sample. The flux change does not involve H, resulting in the higher sensitivity.

VIBRATING SAMPLE MAGNETOMETER:

This is another technique of susceptibility measurement. It is based on the flux change in a coil when a magnetized sample is vibrated near it **[4]**. A small disk shaped sample is attached to the end of a nonmagnetic rod, the other end of which is fixed to a mechanical vibrator. An alternating emf is induced in the detection coils due to the oscillating magnetic field of the moving sample. The small alternating current is then amplified using a lock-in-amplifier. The magnetic moment of the sample, hence magnetisation M of the sample is measured.

ALTERNATING FIELD GRADIENT MAGNETOMETER (AFGM):

In this method, the sample is mounted at the end of a fiber. Then the sample is subjected to a dc field plus an alternating field gradient. A coil pair is used to produce the alternating field gradient which produces an alternating force on the sample. This force causes the sample to oscillate and flexes the fibre. If the frequency of vibration is equal to the resonant frequency of the system, the amplitude of vibration increases by the quality factor Q of the vibrating system. A piezoelectric crystal is used to generate voltage proportional to the vibrational amplitude. This voltage is proportional to the sample moment.

1.4 Application of AC Susceptibility

It has a great application in different fields, like spin glass, superparamagnetism, superconductivity etc. A brief discussion is given below.

(I) SPIN-GLASS

Spin-glass behaviour is usually characterized by AC susceptibility [5]. Spin-glass system is a nonmagnetic lattice populated with a dilute, random distribution of magnetic atoms. Below a critical temperature T_f known as freezing temperature, a metastable frozen state appears without the usual magnetic long range ordering above which paramagnetic state is observed.The freezing temperature is determined by measuring χ' vs temperature, a curve which shows a cusp at the freezing temperature. The most studied spin-glass systems are dilute alloys of paramagnets or ferromagnets in nonmagnetic metals. The cusp in the χ' vs temperature curve depends on the frequency of the ac susceptibility measurement. This behaviour is not found in any other magnetic system and therefore confirms the spin glass phase.

(II) SUPERPARAMAGNETISM

The characterization of small ferromagnetic particles which shows superparamagnetism can be done by ac susceptibility measurements. Consider a system where there is a distribution of small ferromagnetic particles in a non-magnetic matrix and assumed that the particles are separated far enough so that the interparticle interactions are neglected. If ferromagnetic particles are small enough, they will be single domain. Above the blocking temperature T_B, the system behaves as a paramagnet. But in this case the independent moments are not atomic moments but large groups of moments, each group inside a ferromagnetic particle [6]. . The system is therefore called superparamagnet and the phenomenon is termed as superparamagnetism.

(III) SUPERCONDUCTIVITY

The physics of superconductivity is well studied by AC susceptibility measurements. The frequency dependence of complex ac susceptibility provides important information about the superconducting properties and ac losses in polycrystalline high T_c superconductors. Meissner effect is considered as the fingerprint in superconductivity. Whether the Meissner effect is a surface or bulk phenomenon can only be determined by ac susceptibility measurement. Moreover, the presence of multi T_C, irreversibility line, critical current density, intergranular and intragranular contribution is also studied by ac susceptibility measurements **[7]**. In normal state the susceptibility of superconductors are very small, but in superconducting state they show perfect diamagnetism, χ=-1. Hence, the onset of a significant nonzero susceptibility is considered as the superconducting phase transition temperature.

The above examples give a brief introduction to the wide applicability of AC magnetic measurement. Many important material properties require characterization by this technique. The various advantages of ac susceptibility over dc susceptibility installed a motivation to design and fabricate an ac susceptometer.

1.5 DIAGRAM OF A TYPICAL SUSCEPTOMETER

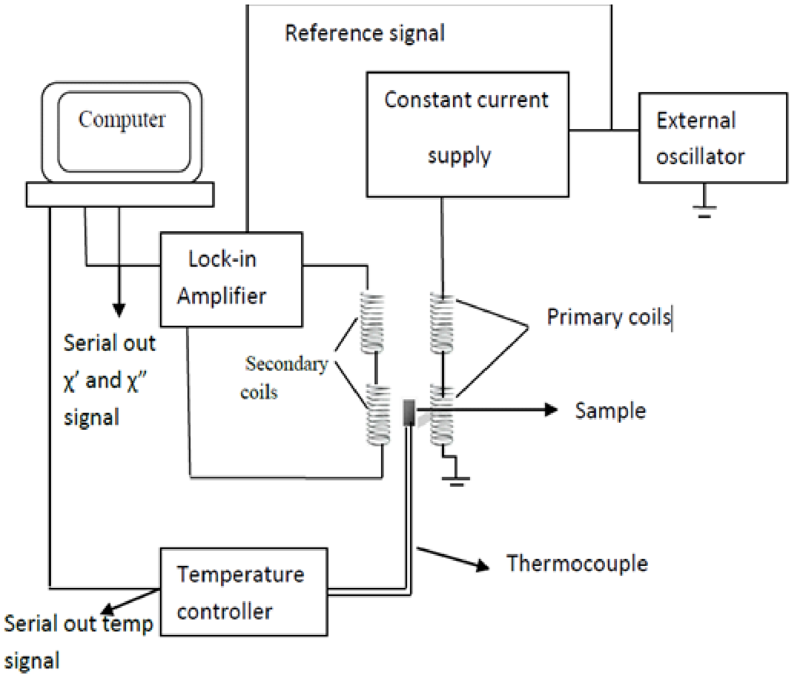


Fig 1.6 shows the block diagram of an ac susceptometer [8]

The above set up is from "M I Youssif,A. A. Bahgat and I.A Ali, *Egypt. J. Sol,* **23**, 231 (2000)".In the above figure, the schematic diagram of an ac susceptometer designed by [8] is shown. It consists of primary coil for producing magnetic field, a set of equal but oppositely winded secondary coils. One of the secondary coil functions as the pickup coil to sense the induced emf by the sample and the other functions as compensation coil. In this article, four coil systems are used for susceptibility measurement. Two identical primary coils of 120 turns each and two oppositely winded secondary coils of 600 turns each are winded around a hollow cylinder of 1.2 cm in height [8]. The two identical primary coils generate the ac magnetic field. An ac current source and an external oscillator were coupled together which allows to measure the ac susceptibility over a wide range of field amplitude, temperature and frequency. The signal from the pickup coil goes to a computer controlled lock in amplifier where the in-phase and out of phase components of complex ac susceptibility are separated.

CHAPTER-2

DESIGN AND FABRICATION OF AC SUSCEPTOMETER

2.1 Cross-Sectional view of the two designs:

We have made two set ups. The cross-sectional view of 1st set up is shown in fig 2.2.

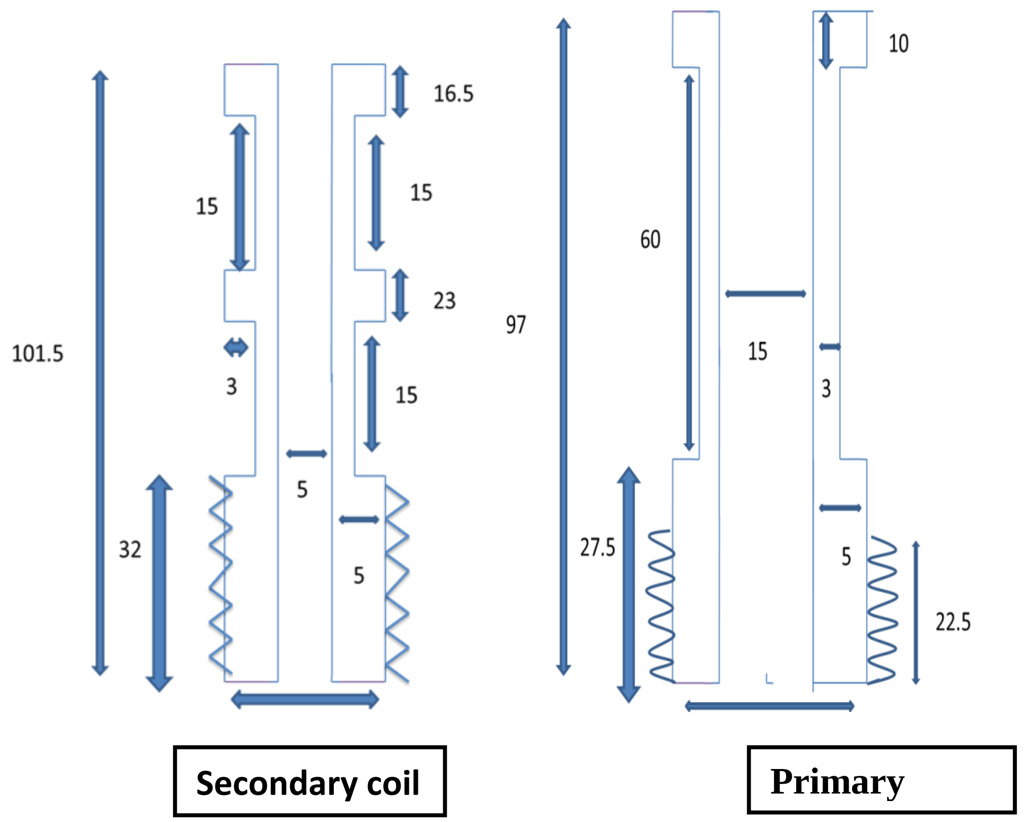


Fig 2.1 all parameters are in mm. Cross sectional view of the design 1.

The figure 2.1 shows the cross sectional schematic design of the primary and secondary coil of our 1stset-up. The material chosen for all this is hylum because of its high thermal conductivity and low electrical conductivity which can avoid eddy current. After designing, copper wires (150 micron diameter) are winded over the groove made for primary and secondary coils. The number of turns in the primary coil is 3300 turns. The secondary coil is a two coil system and the coiling area is separated by a distance of 23mm. It has 500 turns in clockwise direction and another 500 turns in counter-clockwise direction. The secondary coil is inserted into the primary coil. The secondary coil is made hollow, inner diameter of which is 5 mm. The sample is rested at the centre of one of the secondary coil. A Pt100 temperature sensor is also inserted inside that space, in contact with the sample, for local temperature recording.

Motivations for design 2:

For achieving lower temperature the 1st set-up is dipped in liquid nitrogen. Then with the help of lock-in-amplifier the in-phase and out-of-phase components of voltage are recorded corresponding to different temperature measured by pt100. Hence, with the help of 1st set up we are able to measure the ac susceptibility between the temperature ranges from room temperature (300 K) to liquid nitrogen temperature (77 K). The installation of the Closed Cycle Refrigerator (CCR) in our lab has motivated us to design a cryocooler based ac susceptometer. The advantage of fitting our set-up in cryocooler is that we can lower the temperature down to liquid helium temperature (4 K).For second design we have reduced the dimensions of primary and secondary coil system so that it could be fitted in cryocooler. The numbers of turns in primary and secondary coils are adjusted according to it. The number of turns in the primary coil is 3000 and in secondary the number of turns is 1800 clockwise and 1800 anticlockwise. In our set-up the primary coil is winded outside and the secondary coil is winded inside. Because, comparably less induction occurs when secondary is winded outside. The cross-sectional view of 2nd set up is shown in fig 2.2.

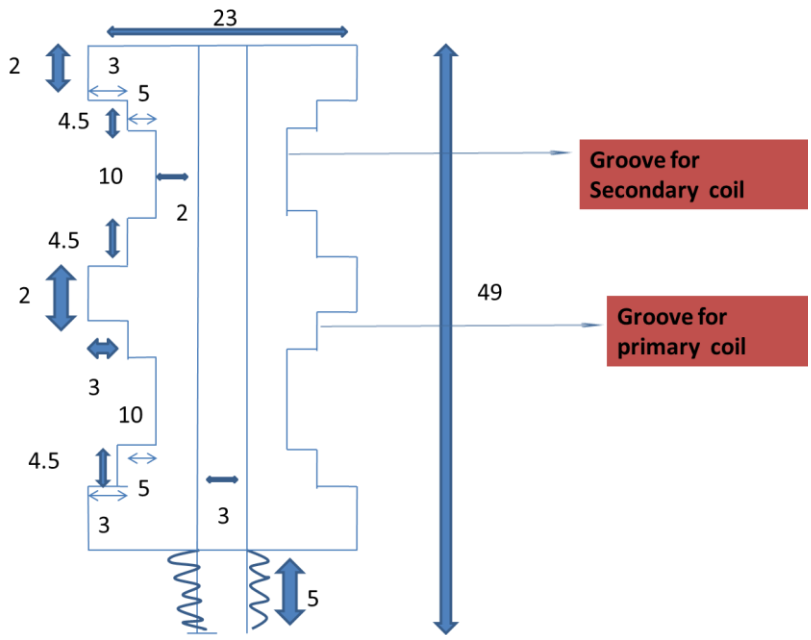


Fig 2.2 all parameters are in mm. Cross sectional view of the design 2.

2.2 Magnetic field at the centre of the solenoid:

The magnetic field at the centre of the solenoid is given by

$H_{ac} = J\,a\,F(\alpha, \beta)$

Where F(α, β) is the field factor which depends on the cross-sectional shape of solenoid.

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

Here $\alpha = b/a$ and $\beta = l/a$, where a and b are the inner and outer radius of solenoid respectively and 2l is the length of the solenoid.

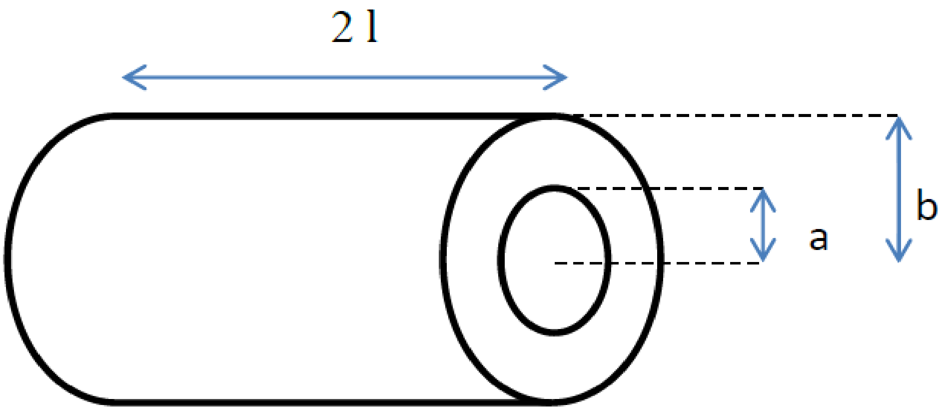


Fig 2.3 Dimensions of solenoid

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 given by {2l(b-a)}.

In this method the change in flux with time is measured as induced voltage V(t) in the secondary coil as: $V(t) = - d\emptyset/dt$

The magnetic flux through the N turn of radius a is

$\emptyset = \pi a^2 N M(t) \mu_0$

So, we have, $V(t) = - \pi a^2 N M(t) \mu_0 / dt$

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

$$M(t)=\sum_1^{\infty} Hac[\chi n' \cos n\omega t + \chi n'' \sin n\omega t]$$

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

$V(t)= V_0 \sum_1^{\infty} n[\chi n' \sin n\omega t - \chi n'' \cos n\omega t]$

Where $V_0= \mu_0 \pi\ 2\omega N Hac$.

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 (Hac). n=1 denotes the fundamental susceptibility while n= 2, 3, 4… etc are the higher order harmonics associated with non-linear terms in χ.

For the 2nd set-up we have designed, the value of

a = 8.5mm, b = 11.5mm and l = 22mm.

Putting these values we get,

α = 1.35 and β = 2.588

So, the value of F(α,β) comes to

F(α, β) = 0.3186

The calculated amount of current passing through the solenoid is

I= 5 mA.

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

Where, A is the total cross-sectional area offered to the current flow,

A = 2l (b–a) =132 x 10^{-6} m2

The value of J comes to J = 37.87 A/m^2.

So, $B = \mu_0 H_{ac} = \mu_0 Ja\ F(\alpha,\beta)$ = 1.289 x 10^{-7} Tesla

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

V_0 = 4.72 x 10^{-4} volts.

2.3 Experimental set up at low temperature:

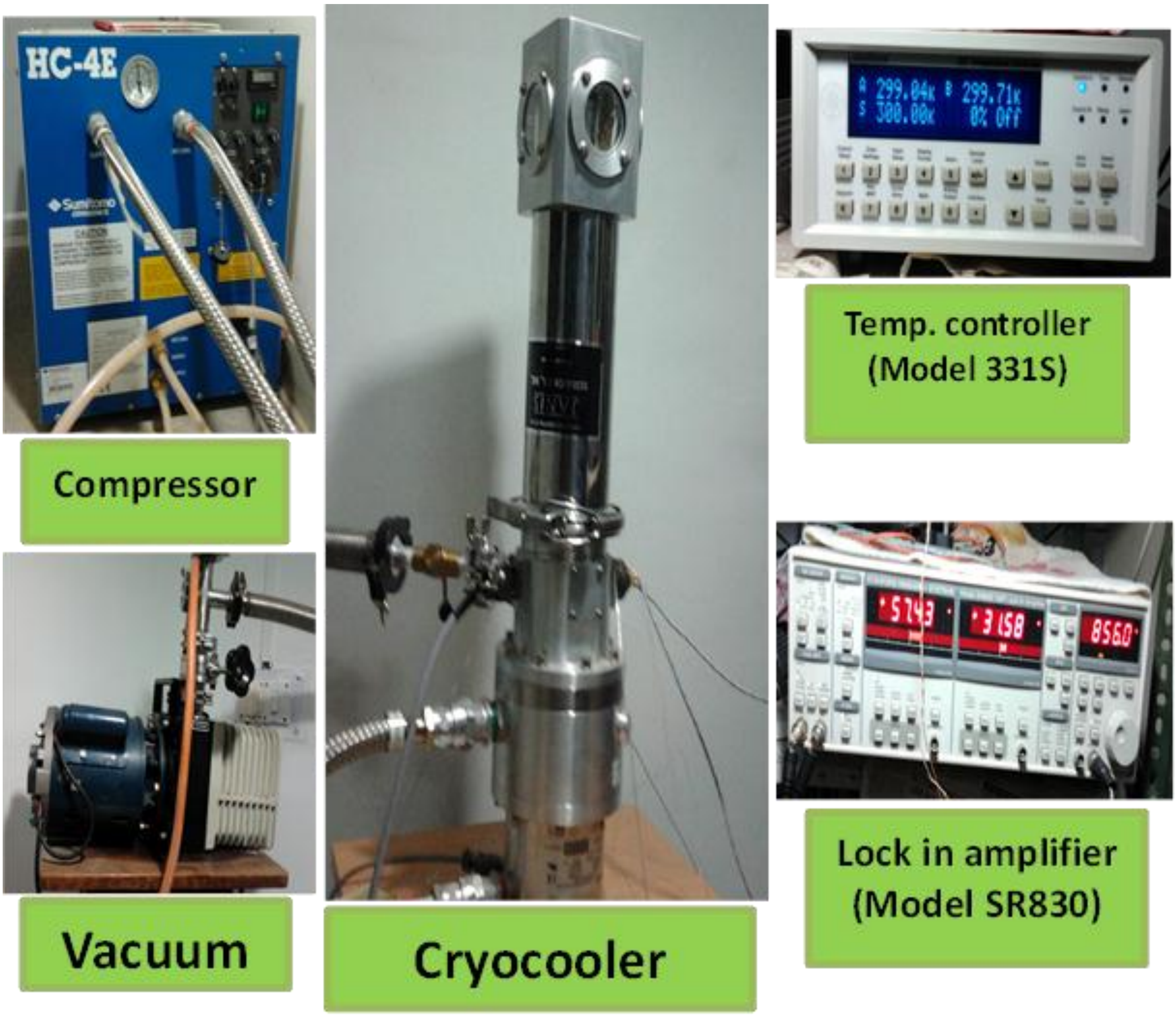


Fig 2.4 Experimental set up at low temperature

CHAPTER-3
Sample Preparation

3.1 Sample Selection

In our project we have chosen $La_{1-x}Sr_xMnO_3$ as sample. $La_{1-x}Sr_xMnO_3$ has immense applications in different fields of physics. It is the derivative of parent compound $LaMnO_3$. Sr doping in antiferromagnetic insulator $LaMnO_3$ at La site enhances ferromagnetic interactions in the compound. $LaMnO_3$. Different magnetic and electronic states were observed with increase in Sr concentration in the compound (Fig 3.1).

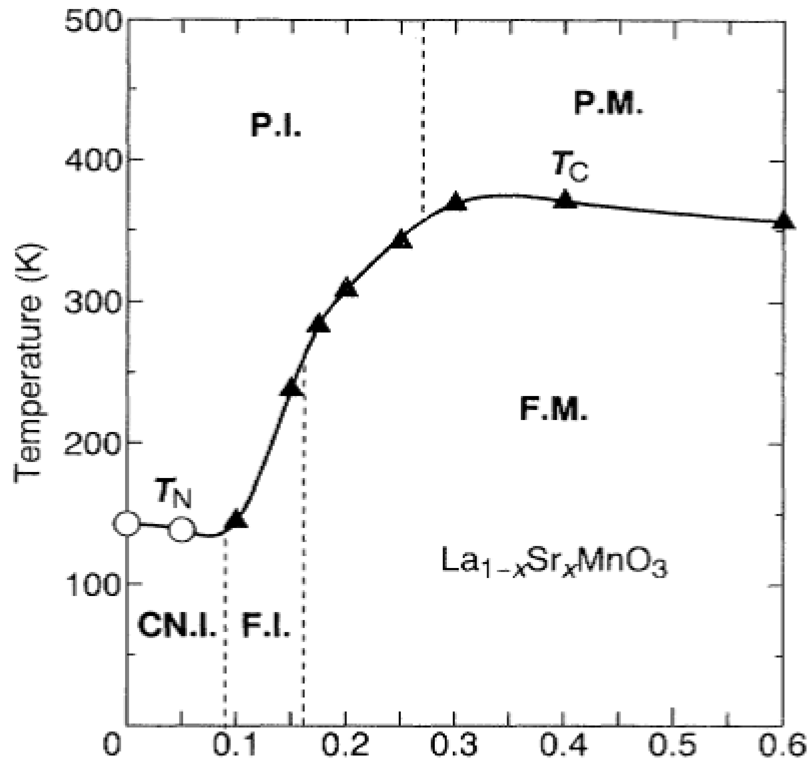


Fig 3.1 Magnetic Phase diagram of La-Sr-MnO_3. Open circles and filled triangles are Neel temperature (T_N) and Curie temperature (Tc) respectively. The abbreviation mean paramagnetic Insulator (PI), Paramagnetic metal (PM), Spin canted insulator (CNI), Ferromagnetic insulator (FI) and ferromagnetic metal (FM) [9].

3.2 Different techniques involved in preparation:

The citrate-gel route:

In this technique the precursors taken are in acetate form. Distilled water is used to dissolve the metal acetates. Solution is then mixed with citric acid in 1:1 ratio. The solution is then heated to 80 °C for 2 hours so that it becomes gel. The gel is further heated to 400^0C to form foam like powder. It is then decomposed to give very light, homogenous, black-colour flakes of extremely fine particle size [10].

Sol gel combustion method:

In this technique the precursors taken are in acetate form. Distilled water is used to dissolve the metal acetates. To this solution, some fuel is added to initiate combustion. We have followed this technique for our sample preparation and detail of this technique is discussed in LSMO preparation part [11].

Spray drier method

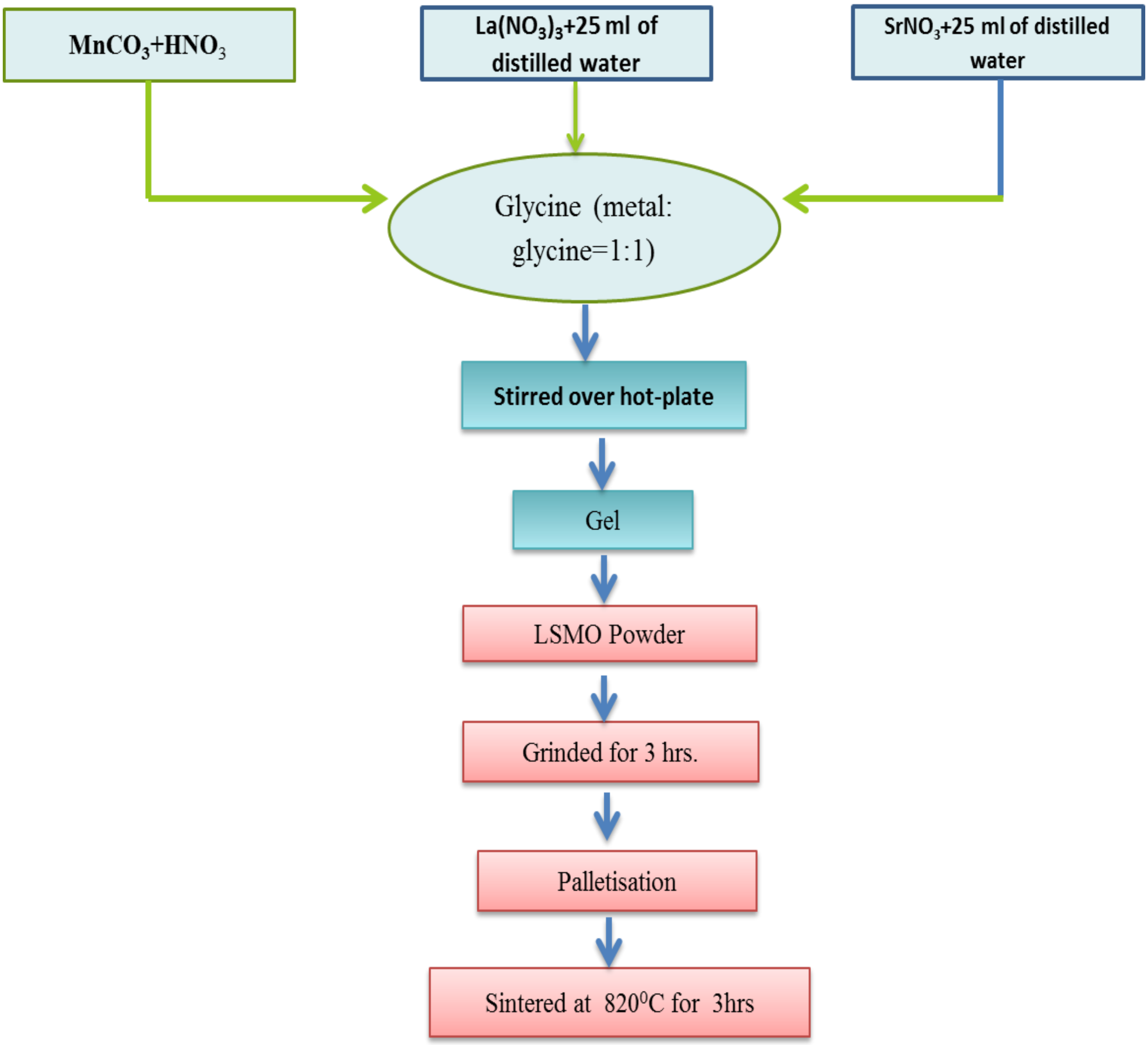
Aqueous solution of lanthanum, strontium and manganese nitrates is mixed in the desired stoichiometric ratios. In a spray drier, the above solution was spray-dried. The liquid fed into the spray drying is converted into a uniform powder. Atomization of product is started and a spray of fine droplets is fed into the drying (120^0C) chamber. In heated gas stream, the fine droplets sprayed into the chamber become suspended. Then evaporation of droplets Is done and is dried to spherical powder. Dried powder is separated from the gas stream and connected in the base of the drying cyclone chamber [11].

3.3 LSMO Preparation:

LSMO powders are prepared by auto combustion sol-gel method. $La(NO_3)_3.6H_2O$, $Sr(NO_3)$ and $MnCO_3$ are chosen as precursors for the synthesis. Stoichiometric amounts are taken as 0.83% of 0.01mole, 0.17% of 0.01mole, 0.01 mole respectively. Combustion agent used in synthesis is Glycine. The ratio of glycine: metal nitrate is 1:1.

The reactants are dissolved in distilled water and continuously stirred till a colourless solution is obtained. Then glycine is added to the solution and is then heated with constant stirring at 80^0c to evaporate the excess solvent. The viscous solution after 3 hour of heating became a viscous gel. The gel converted to the black powder after burning due to glycine agent. The powder is collected and grinded for 3 hours. Then it was calcined at 600^0c for 2 hours. The furnace heating rate is maintained at 4^0c/minute. After cooling the sample was collected and grinded for another 3 hours. Pellets are made from the sample and sintered at 820^0c for 3 hours. After cooling the pelletes are collected. Pellets are cut into small rectangular shaped pieces and wrapped in Teflon tape for use in ac susceptometer in low temperature. Finally susceptibility measurement is done using ac susceptometer already fabricated.

3.4 Flow chart to prepare LSMO:



Chapter 4

RESULT AND DISCUSSION

4.1 AC SUSCEPTIBILITY OF LSMO:

The sintered pellets are cut into rectangular pieces and wrap with Teflon tape. One of the rectangular pieces is then carefully placed at the centre of one of the secondary coilin order to avoid the edge effect. The set up was cooled down in cryocooler. The set-up is cooled down to 10 K and then allowed to heat up normally to room temperature. The x-component (in phase) and y- component (out of phase) of signal are then recorded by lock in amplifier as a function of temperature. These x and y component is then converted to volume susceptibility by dividing with parameter $v_0$calculated earlier. Finally we are getting the in-phase and out-of-phase components of complex ac susceptibility and are plotted against temperature.

The variation of in-phase (χ') and out-of-phase (χ") susceptibilities for with LSMO sample and without sample (Background) with temperature is shown in Fig 4.1 and Fig 4.2 respectively.Then the background data was deducted from the susceptibility data with sample to get the in phase and out of phase components of ac susceptibility due to the $La_{0.83}Sr_{0.17}MnO_3$ sample. Results are shown in Fig 4.3 and Fig 4.4.

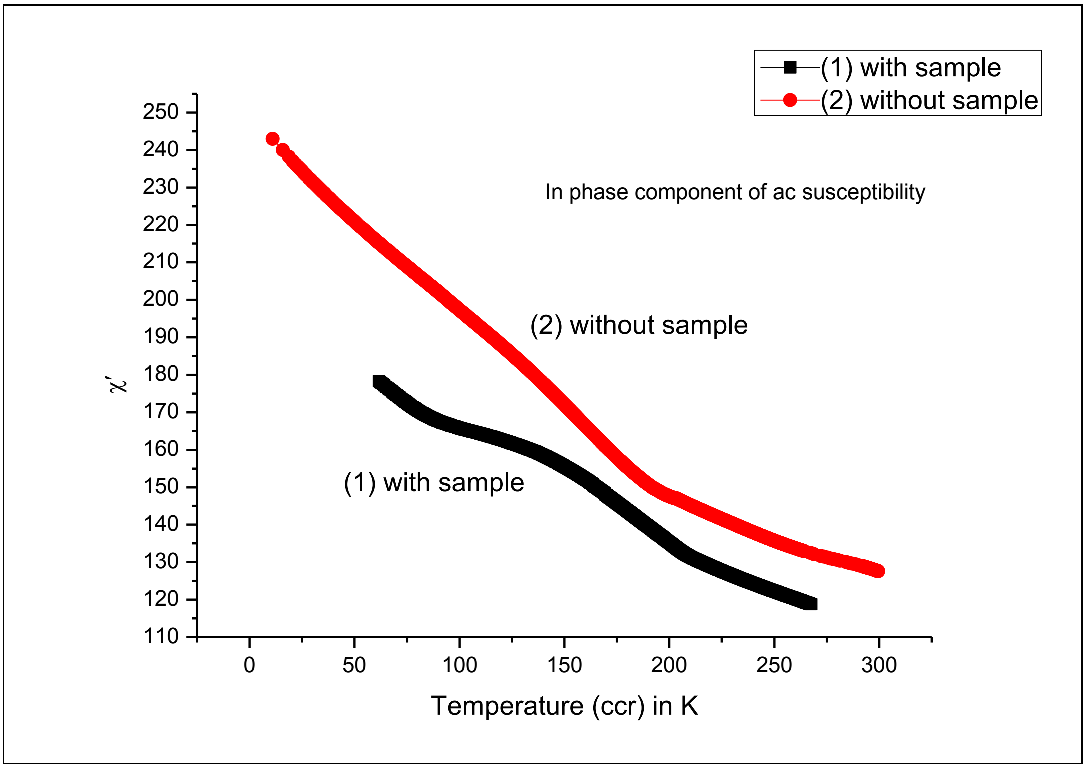


Fig 4.1 shows the in-phase component ofac susceptibility for both with sample and without sample.

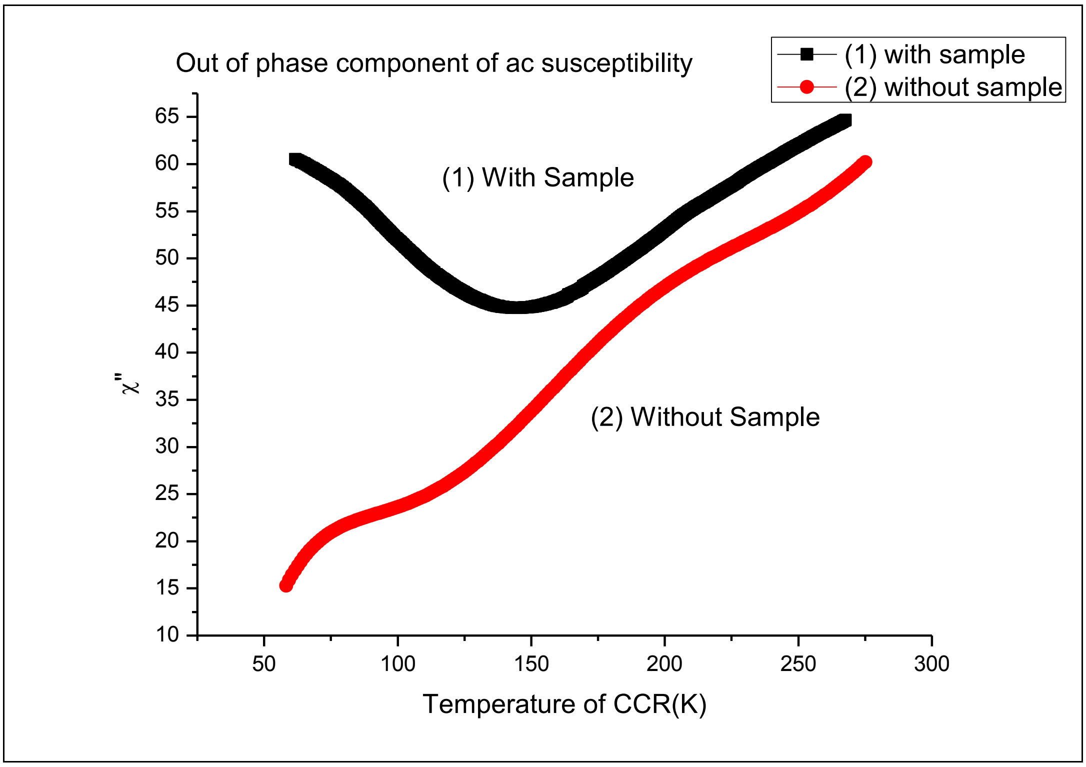


Fig 4.2 shows the out-of-phase component of ac susceptibility for both with sample and without sample.

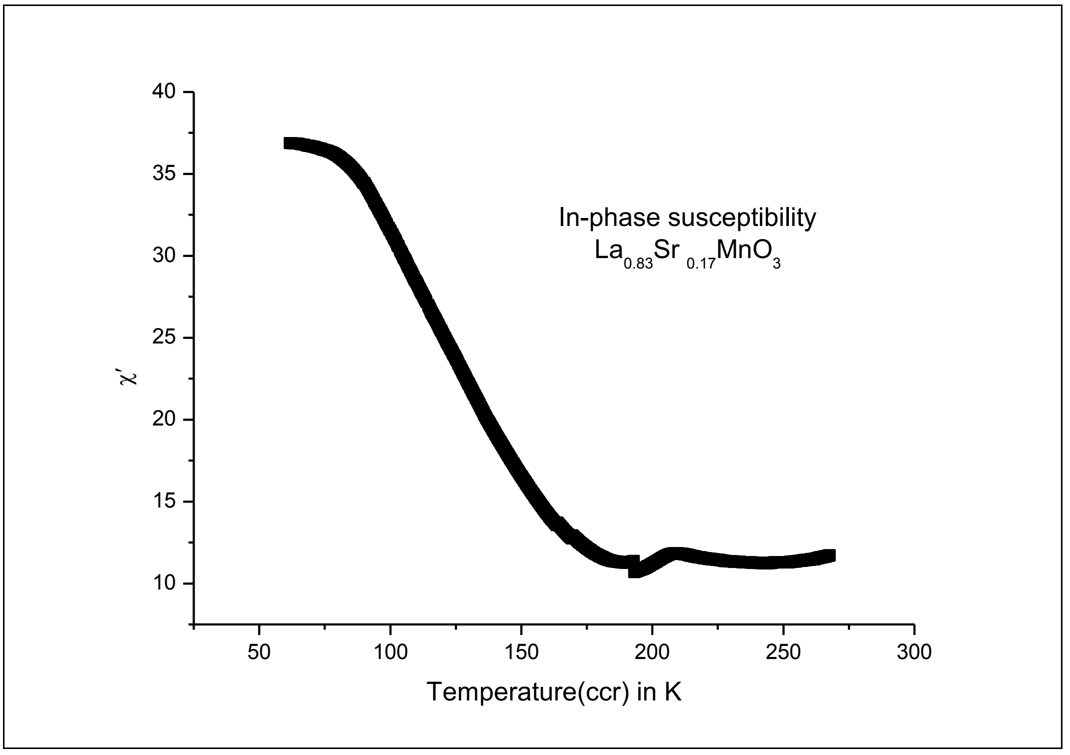


Fig 4.3 shows the in-phase component of ac susceptibility for $La_{0.83}Sr_{0.17}MnO_3$ sample, obtained after subtracting the background.

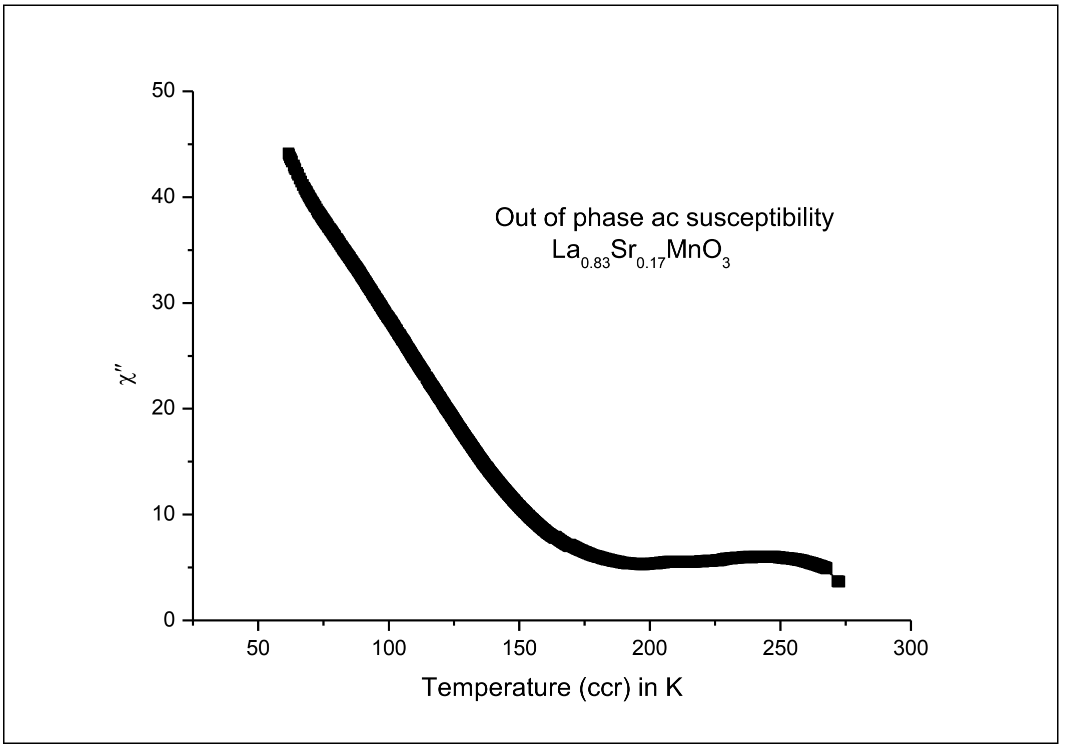


Fig 4.4 shows the Out of phase component of ac susceptibility for $La_{0.83}Sr_{0.17}MnO_3$ sample, obtained after subtracting the background.

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. Fig 4.2 reflects that the transition temperature of the $La_{0.83}Sr_{0.17}MnO_3$ sample is around 180 K. According to the literature survey of LSMO sample the transition temperature is around 260 K. This is due to the fact that the real part of ac susceptibility, χ' has been plotted against the temperature measured by the cryocooler temperature sensor as mentioned in the plot. And there is a temperature difference between the temperature measured by cryocooler temperature sensor and the sample temperature. The cryocooler temperature sensor basically measures the temperature of the cryocooler base whereas the sample is mounted inside the hylum former. The difference in temperature observed between cryocooler base and sample is due to the following facts. The thermal conductivity of hylum material is not so good. Another reason may be that we the use of vacuum in cryocololer. As vacuum does not allowed the flow of heat from the sample to the cryocooler base, the sample remained at a higher temperature than cryocooler base. Due to the above mentioned facts the transition is observed at 180 K instead of 260 K.

4.2 CONCLUSION:

We have made the susceptometer twice. In the 1st set up there are two parts, one is primary coil and another is secondary coil. This set up was cooled by dipping it in liquid nitrogen. In motive of designing a cryocooler based ac susceptometer 2nd set up was designed and set in the cryocooler. Hylum is chosen as the former material. Primary and secondary coils are winded over a single hylum former. Secondary coil, a set of two equal and oppositely winded coils, is first winded over the hylum former. Then the primary coil is winded over it. Copper wire (150 micron) is used for winding. The whole set up is then set in the cryocooler (HC-4E, Sumltomo cryogenics). An ac current is supplied to the primary coil from DSP Lock-in Amplifier (Model SR830), which produces the required magnetic field. This magnetic field magnetize the sample and induced an emf in secondary coil is observed. The effect of magnetic field is cancelled by the two oppositely winded coil system. So that the emf induced is only due to the change in moment of sample. This emf is then separated to in-phase and out-of phase voltage component in lock-in amplifier. The in-phase and out-of phase voltage component is converted to in-phase and out-of phase component of ac susceptibility by dividing a voltage factor V_0. Temperature is lowered down to liquid helium temperature in cryocooler and data are recorded using lab view program.

LSMO sample was prepared by auto combustion sol-gel method. After preparing pellets they are cut into small rectangular shaped pieces. One of such piece is inserted in secondary coil and Ac susceptibility measurement is done.

After getting the susceptibility values, when we plot it against temperature recorded by the cryocooler sensor, we get the transition of $La_{0.83}Sr_{0.17}MnO_3$ around 180 K. There is some deviation in transition temperature. The reason is discussed in the result discussion part.

The ac susceptometer is working properly, but there is need of overcoming the problem of the difference in temperature arises between cryocooler and sample. Therefore some modifications are still required to our ac susceptometer for better results.

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