

# **Design and fabrication of an experimental set up for ac Susceptibility measurement of**



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**ABSTRACT:** An Experimental set up for ac susceptibility measurement is designed and fabricated. The design consists of a primary coil and a pair of secondary coils. The set-up is based on the phenomenon of “mutual induction”. The sample is directly inserted inside in one of the secondary coil.  $\text{La}_x\text{Sr}_{1-x}\text{MnO}_3$  is prepared by auto combustion sol gel method. Sample was characterized by XRD and it is found to be single phase. The measurement is done with the help of a lock in amplifier which was interfaced with a computer. A sharp rise in susceptibility is observed at 363K.

**Key words:** ac susceptibility, magnetization, mutual induction, LSMO

## 1. INTRODUCTION:

We have designed an ac-susceptometer to measure volume susceptibility. The dc-magnetometer and the ac-susceptometer are two different tools which provide different ways of getting magnetic properties. But both these two techniques depend on sensing coils and are used to measure the variation of the magnetic flux due to magnetic sample. The ac technique detects changes in the magnetization that lead to  $dM/dH$  in the limit of small ac fields, and this is why sometimes referred to as a differential susceptibility.

In the dc measurement, the magnetic moment of the sample does not change with time. Thus, a static magnetic measurement is performed. An ac output signal is detected in a vibrating sample method, but this signal arises from the periodic movement of the sample. In the ac measurement, the moment of the sample is actually changing in response to an applied ac field, allowing the dynamics of the magnetic system to be studied.

Since the actual response of the sample to an applied ac field is measured, the magneto-dynamics can be studied through the complex susceptibility ( $\chi' + i\chi''$ ). The component  $\chi'$  represents the component of the susceptibility that is in phase with the applied ac field, while  $\chi''$  represent the

component that is out of phase. The out of phase component  $\chi''$  is related to the energy losses, or in other words, the energy absorbed by the sample from the ac field. Ac susceptibility has a great application in different fields, like spin glass, superparamagnetism, superconductivity etc.

## 2. DESIGN DETAILS OF THE SET UP:

It consists of primary excitation field coil, a secondary pick up coil, a secondary compensation coil. The base material chosen for the coils is Teflon. The number of turns in primary and secondary both are 1000, with 500 clockwise and 500 anticlockwise in the secondary. The primary coil and two secondary coils should be arranged properly [1]. We put our sample directly in one of the secondary. We can keep the secondary inside and primary outside and vice versa [2]. But in our set up we kept the primary coil outside. Because when we put the secondary outside, comparably less induction occurs. So the former being positioned in between the later couple in order to allow modifications out the outer secondary for calibration. A Pt100 sensor is put inside the secondary, in contact with the sample.



Fig 1. Picture of the ac susceptometer (Above one is the primary coil and below one is the secondary coil wrapped with the Teflon tape)

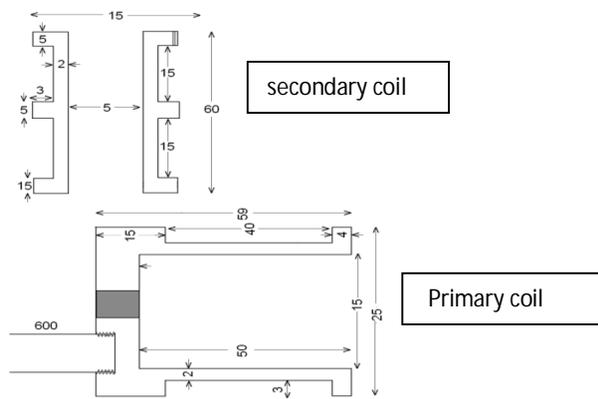


Fig 2. Cross section of an ac susceptometer

**3. SAMPLE PREPARATION:**

$La_{2/3}Sr_{1/3}MnO_3$  (LSMO) is chosen for sample under investigation.  $La_{2/3}Sr_{1/3}MnO_3$  has been the subject of research for more than a decade for its immense applications in different fields as well as the interesting physics in it.

$La_{2/3}Sr_{1/3}MnO_3$  is the derivative of parent compound  $LaMnO_3$ .  $LaMnO_3$  is an antiferromagnetic insulator and Sr doping at La site enhances ferromagnetic interactions in the compound. With increasing Sr content, the compound shows a variety of magnetic and electronic states [6] (fig 3).

The structure of  $LaMnO_3$  is perovskite type orthorhombic structure. With increasing Sr content, the lattice parameter changes accompanying structural transformation from orthorhombic to rhombohedral. The tolerance factor which is a measure of microscopic distortion from the ideal perovskite structure ( $t=1$ ) of the form  $ABO_3$  (fig 4).

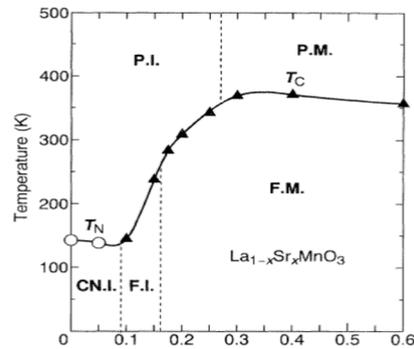


Fig.3 Magnetic Phase diagram of  $La_xSr_{1-x}MnO_3$ . Open circles and filled triangles are Neel temperature ( $T_N$ ) and Curie temperature ( $T_C$ ) respectively. The abbreviations mean paramagnetic insulator (PI), Paramagnetic metal (PM), Spin canted insulator (CNI), Ferromagnetic insulator (FI) and ferromagnetic metal (FM) [6]

The Sr doping in  $LaMnO_3$  not only affects the magnetic and structural properties, but the electrical properties also undergo a profound change. The parent compound  $LaMnO_3$  is an insulator and Sr doping brings metallicity in the compound. From the magnetic phase diagram and electrical conductivity data, it is evident that for Sr doping more than 20%, the sample becomes Ferromagnetic metal, with Curie temperature systematically shifting to higher temperatures.  $La_{2/3}Sr_{1/3}MnO_3$  has Curie temperature, ( $T_c$ ) above room temperature, about 380 K and a large magnetic moment at room temperature. Since its transition temperature  $T_c$  is also above room temperature, these materials find immense application in various fields.

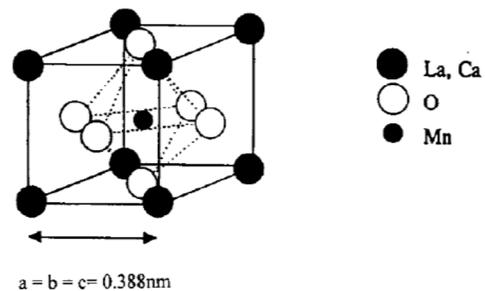


Fig4. Perovskite structure of LSMO at room temperature

Here we report on the preparation of nanoparticles  $\text{La}_{2/3}\text{Sr}_{1/3}\text{MnO}_3$ . Phase formation, crystal structure and particle size were studied by X-ray diffraction (XRD).

Nanoparticles of LSMO are prepared by auto combustion sol-gel method. The precursors chosen for the synthesis are  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{Sr}(\text{NO}_3)_2$  and  $\text{MnCO}_3$ . Stoichiometric amounts are taken as 0.67% of 0.02mole, 0.33% of 0.02mole, 0.02 moles respectively. Glycine was taken as a combustion agent. It is notified that the ratio of glycine: metal nitrate is 1:1.

The reactants were dissolved into the distilled water and stirred for 30 minutes continuously. The solution is now colorless. Then glycine is added into the solution. The solution was then heated with constant stirring at  $80^\circ\text{C}$  to evaporate the excess solvent. After 3 hours of heating the solution converted to a viscous gel. Then after burning due to glycine agent the gel is converted to the black powder. The gel was dried at  $250^\circ\text{C}$ . After collecting the powder, calcination is done at  $600^\circ\text{C}$  for 2 hours. The furnace heating rate is maintained  $4^\circ\text{C}/\text{minute}$  during calcination. After cooling the sample is collected from the furnace and is grinded in an agate-motor. The grinded powder is now ready for necessary characterization. And after calcination sintering has done at  $825^\circ\text{C}$  for 5 hours. The sample is pressed into the pellet.

Then the characterization is done. By XRD Phase formation (fig 5) and crystal structure of the powders were checked by using  $\text{Cu K}\alpha$  radiation source in the  $2\theta$  scan range from  $20^\circ$  to  $80^\circ$ . From the X-ray peak width the average particle sizes of the samples were estimated by using the William Hall method. The XRD data is taken in the  $2\theta$  range of  $20^\circ$  to  $60^\circ$ . In this range, five prominent peaks are observed. All peaks could be indexed to the respective (hkl) planes of LSMO having lattice parameters  $a = 5.484 \text{ \AA}$ ,  $b = 5.534 \text{ \AA}$  and  $c = 7.791 \text{ \AA}$ , space group  $\text{P}^{2/c}$  [8]. No extra peaks were found, indicating that the sample is single phase. The pellet is cut into a small piece of rectangular shape and wrap in a Teflon tape to use in ac

susceptometer in high temperature. Finally the ac susceptibility was measured with in-house developed susceptometer.

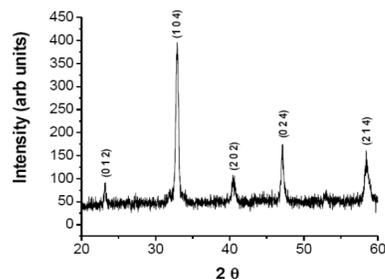


Fig5.XRD analysis of LSMO

#### 4. AC SUSCEPTIBILITY MEASUREMENT OF LSMO:

The sintered sample was cut into cylindrical shape weighing 0.014 gm. The rectangular shaped sample was wrapped with Teflon tape and carefully placed at the centre of one of the coils of secondary coil. Sufficient care is taken in placing the sample in the middle of the coil in order to avoid the edge effect. The whole set up is placed inside the furnace for high temperature susceptibility measurement.

First the setup is heated till  $185^\circ\text{C}$  and we cool the system gradually up to room temperature. Magnetization measurements were done at the time of temperature rising as well as cooling. The x-component (in-phase) and the y-component (out-of-phase) component of the signal are recorded as a function of temperature. These x and y component is then converted in volume susceptibility by dividing with parameter  $v^0$ . The variation of in-phase ( $\chi'$ ) and out-of-phase ( $\chi''$ ) susceptibilities with temperature is shown in Fig 6. As the sample is cooled down from high temperature, the susceptibility is almost constant having very low value. Around  $90^\circ\text{C}$ , the susceptibility starts rising sharply and till the lowest temperature of measurement, no saturation is observed. The sharp rise at  $90^\circ\text{C}$  in susceptibility indicates ferromagnetic ordering below this

temperature. The rise in susceptibility is similar for both in-phase and out-of-phase components of susceptibility. This reflects ferromagnetic nature of LSMO, as per literature. For comparison, the susceptibility data is compared with ref. [3]. The ref data scale is in Kelvin where as our data scale is degree centigrade. In Kelvin scale, 90°C to 363K. It can be seen that our data is in good agreements with that in ref [3]

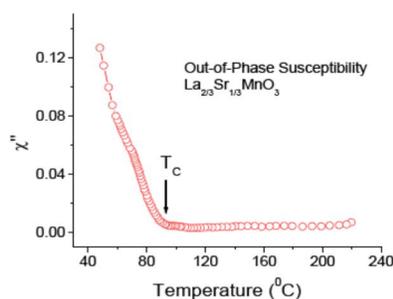


Fig.6a out of phase susceptibility vs. temperature at high temperature measurement

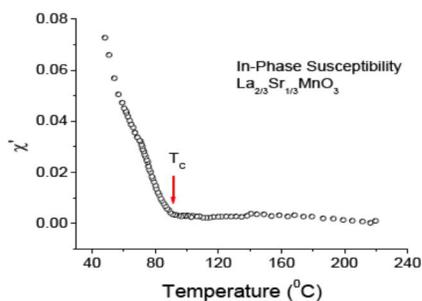


Fig.6b In phase susceptibility vs. temperature at high temperature measurement

## 5. CONCLUSION:

From our experiment we conclude that The XRD peak for LSMO matches accurately with the report results. After getting the Susceptibility value when we plot it with temperature we get the transition of LSMO in accurate position (364K).

To the best of our knowledge, this is the first time report on ac susceptibility experimental measurement of LSMO by a homemade susceptometer.

## 6. ACKNOWLEDGEMENT:

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