A Cryostat for the Measurement of Hall Effect and Magnetic Susceptibility between 77 – 300 K

[1] *MR. S .R .PARIKH*, [2] *PROF. H. A. SHUKLA*

[1]**M.E.[Cryogenics Engg.] Student, Department Of Mechanical Engineering, L.D. College Of Engineering , Ahmedabad**

[2] **Asst.Professor, Department Of Mechanical Engineering, L.D. College Of Engineering , Ahmedabad**

Shaival01@gmail.com, shuklahardik2@gmail.com

***ABSTRACT*: —** **A** cryostat is described for the measurement of Hall Effect and magnetic susceptibilities at temperatures from 77–300 K in one single experiment. The susceptibility is measured by the A.C. mutual inductance method. The sample, kept cold during the whole experiment, the small double walled cryostat directly immersed in a liquid nitrogen bath. The sample holder is prepared by hylum material Where primary and secondary winding is done for measurement of magnetic susceptibility and separate sample holder of AISI 304 using for measuring Hall Effect where the magnet is used. With the aid of an electrical heater and a temperature sensor of PT-100 is used to measure temperatures between 77 – 300 K can be produced and measured with an overall precision better than other .The sample is held so that its position can be adjusted inside the coils or removed completely at any temperature.

Keywords— **Cryostat , Magnetic susceptibility Hall Effect**

I: INTRODUCTION

In a study of the magnetic properties and Hall Effect of some Cuprates material like YBCO and nickel salts and other sample made in our laboratory, it was convenient to have a cryostat capable of measuring magnetic susceptibilities at any temperature Between 77- 300 K without changing samples or the measuring system. It was also necessary to be able to move the sample at any temperature to compensate for the spurious variations of the mutual inductance due to the measuring coils (McKim and Wolf 1957). For measuring the Hall Effect provided the magnet whose magnetic filed is up to 0.7 T at outer vessel at a room temperature.

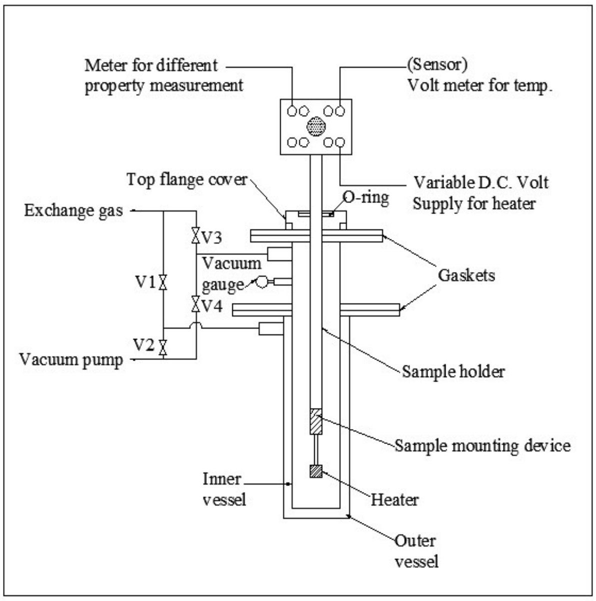
We then designed a cryostat in which a small Dewar Inside placed the susceptibility measuring coils, both immersed in a bath. Inside this Dewar, gases separately admitted for purging purpose, and liquefied in small quantities by means of the external bath to cool the sample. **A** heater and a PT 100 Sensor Were used for the measurements between 77- 300 K

This apparatus was very convenient since it permitted precise measurements to be performed in the whole temperature range in a relatively short time, and also very rapid changes to samples without having to warm the cryostat.

Recently a lot of interest has been shown in the properties of amorphous magnetic materials (Handrich and Kobe 1970,Kaneyoshi 1973, Slechta 1975). Unfortunately it has been found that if amorphous films of the pure transition elements are required they can only be prepared in thin films of 200 **A** or less (Bennett and Wright 1972). The small bulk of these films and their method of preparation, normally the condensation from the vapour phase onto a cold (-4K) glassy substrate, make conventional magnetic moment measurements difficult to perform. However it has been known for some time that the galvanomagnetic properties of ferromagnetic materials are directly related to their magnetic properties (Jan 1957). In an attempt, therefore, to obtain data,if indirectly, on the magnetic properties of these films it was decided to proceed with a series of measurements of their galvanomagnetic properties (Whyman 1974, Whyman and Aldridge 1975). This note describes the cryostat used in these measurements.

II : Description of the apparatus

A simplified diagram of the cryostat is shown in figure 1. The Cryostat is a made double wall SS-304 tube with the space between the walls permanently evacuated.



Fig–1 . Cryostat for magnetic susceptibility and Hall Effect measurement

The internal wall is much longer than the external, thus providing an evacuated. During the whole experiment, is external a small glass Dewar immersed in a liquid nitrogen bath. The measuring coils placed inside the internal Dewar. In which the sample can be inserted into the sample holder. With the aid of an electrical heater and PT 100 Sensor, temperatures between **77** and 300 K can be produced and measured with an overall precision. The sample is held so that its position can be adjusted inside the coils or removed completely at any temperature.

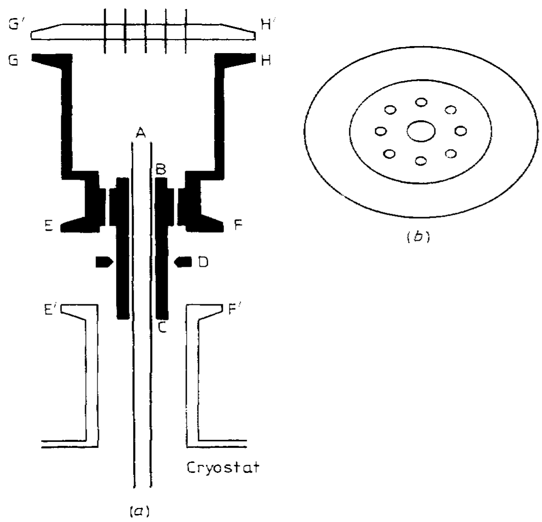


Fig 2. Device for the supporting the sample bar in cryostat.

The upper end of the hollow alloy or S S bar **(A),** through which the electrical leads of the sample pass, is inserted in the tube (BC) and fastened in position by two series of three small Allen screws (D) placed at 120^0 to each other.

The base (EF) of the device allows a quick fitting to the flange (E'F') on top of the cryostat. The vacuum-tight connection between EF and E'F' is achieved by the interposition of a centring circlet with O-ring and is fastened by a clamping ring,. In a similar way, the top (GH) of the device is closed vacuum-tight by a current lead through flange (G'H').

If the standard components mentioned above are not available, or they do not have the proper dimensions, the two ends (EF) and (GH) of the device can be easily redesigned to fit vacuum-tight on the rest of the cryostat and to close at the top with a flange, by the use of O-rings and vertical screws going through the flanges. In the case of a home-made flange (G'H'). vacuum- tight seals for electrical leads through it can be easily constructed in the way described by McKinnon (1968) and Rose-Innes (1973).

As shown in figure 2(b). Several holes are made around the tube BC connecting the interior of the device with the inner chamber of the cryostat.

The magnetic susceptibility is measured by an A.C. mutual inductance bridge similar to that described by Jastram et al. (1959). The secondary measuring coil is wound outside the primary and is made in three sections: the principal winding is at the centre; at each end there is a compensating section built in such a way that the mutual inductance of the empty coil system at liquid nitrogen temperature is below 10 **μH.** The details of the construction of the coils are described by Vargas and de Oliveira (1969). 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 800 and in secondary coil it is 900 in which 450 turns is clockwise and another 450 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. Lock-in amplifier is used to detect and measure very small A.C. signal. After that we performed our experiment. When measure the Hall Effect that time remove the primary and secondary windings and put magnet of the outside of outer vessel.

III. Test Procedure

A.C. susceptibility measurements were performed using inductance method consisting of primary and secondary coils. The primary coils (wounds in same direction) are coaxially wound on secondary coils (two sections connected in series but of opposite windings). The primary coil was connected to a Lock-In- Amplifier. A low frequency (10-300 Hz) sine wave current was generated using a signal generator and was fed to the primary coils as well as to the Lock-In-Amplifier as a reference signal. The alternating current produced an alternating magnetic field inside the primary coils, which induced voltage in the secondary coils. With no sample in the coil, the mutual inductance of the combined coils is zero. The signal from the secondary coil was fed back to the **Lock**-In-Amplifier for phase sensitive detection. Hence when a superconducting sample is placed in one of the sections of the secondary coils, any change in the magnetization of the sample (and hence the susceptibility) corresponds to a change in the emf induced in the secondary coil which is measured by the Lock-In-Amplifier. AC susceptibility measurements have been employed to qualify the suitability of materials as superconductors.

AC susceptibility is one of the fundamental measurements used for characterizing magnetic samples. In particular, the temperature variation of the AC susceptibility is especially useful in identifying phase transitions. Most low-temperature measurements of AC susceptibility make use of liquid cryogens.

A set of mutual inductance coils Usually, there is a single primary outer coil to which an A.C. signal is applied, and two secondary coils in series opposition. In such an arrangement, the output voltage across the secondary coil combination should be zero when no sample is present, since the effect of direct coupling between the primary and secondary coils is to induce two opposing signals which should cancel in a balanced system. However, when a sample is placed at the center of one of the secondary coils, an AC signal, corresponding to the sample response, should be observed.

There are a number of problems associated with the use of cold finger cryostat as a cooling system for such a coil arrangement: (i) A physical thermal link between the cryostat cold finger and the sample is necessary to cool the sample. However, the presence of such a link may itself contribute to the measured susceptibility. This is due not only to the link's own magnetic response but also, if the link is metallic, to induced Eddy currents. (ii) Control and monitoring of the sample temperature may be adversely affected by thermal gradients. (iii) Thermal gradients may also cause differential expansion in the coil formers, changing the relative inductance of the coils. Thus, the background signals in the two secondary coils will no longer balance, and a spurious signal will be added to the measured response.

Data logging system for AC magnetic susceptibility measurements (77-300 K)**.**system suggested the semi-automated apparatus for the simultaneous measurement of various AC magnetic properties in low applied fields (1 kA $^{m-1}$) over the temperature range 77 -300 K is described. The system is based on a variable temperature cryostat and a high resolution data logger capable of measuring ten signals to 1 µV resolution. In these measurements the sample forms the core of a transformer with phase sensitive detection (PSD) of the secondary induced voltage being used to determine. The capability of the system is demonstrated by measurements obtained on the terbium crystal for which the secondary coil was wound directly on to the sample with the primary being wound around a nylon former in which the sample was a tight fit. Hold-times of 48 hr and 8-16 hr were obtained with liquid nitrogen and helium as the coolants respectively. A block diagram of the system, which consisted of a Keithley data logger and programmable calculating unit (PCU), a teletype, a Data ways Interface and a Hewlett-Packard 9830 desk-top calculator, is shown in figure 3.

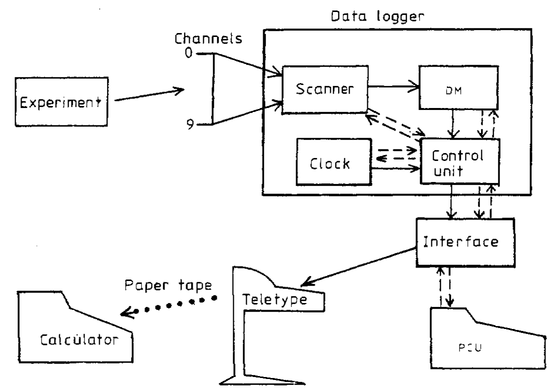
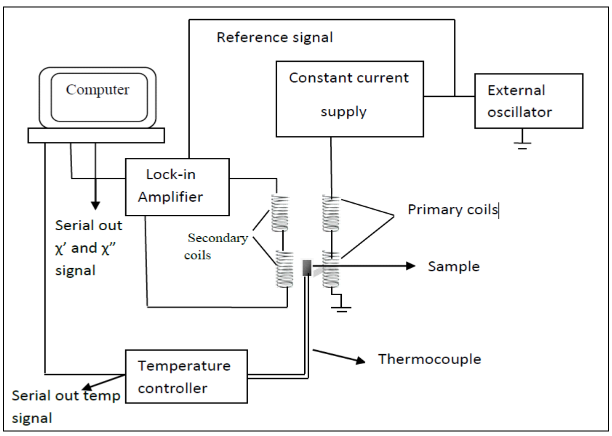


Fig. 3 - Block Diagram of Data Logging System

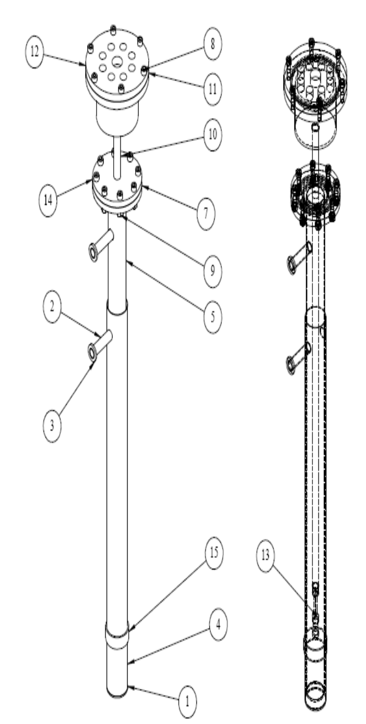


The system is based on a programmed measurement cycle which was activated every one to five minutes during an experiment lasting 20 h and has the capacity to measure signal voltages (1 μV-30 V) on ten channels. Data logger which consists of the following components: a low voltage scanner; a digital multimeter (DM); an interval time/clock and a control unit. Operation of the system was controlled by a program in the PCU which activated the measurement cycle at fixed time intervals. Although operating parameters such as the number of channels to be measured and the time delay between the measurement of each channel were set before the experiment commenced, the program could be interrupted and altered between measurement cycles. A method of assessing the effects of an average demagnetising factor on x and S measurements on irregularly shaped samples is presented. Plot graph temperature vs susceptibility.

IV. Design Procedure

The main requirements of specification or design data available for cryostat are as under.

(1) The cooling media for producing cryogenic temperatures is liquid nitrogen.
(2) The capacity of cryocan for coolant LN_2 is 35 liters.
(3) The mouth piece hole of LN_2 container is 50 mm diameter.
(4) The sample lengths of aluminium wire and copper wire is minimum 10 meters for getting accurate and precise reading.
(5) There should be minimum heat leak to atmosphere through LN_2 vessel and cryostat.
(6) There should be minimum liquid nitrogen consumption as it is costly item.
(7) There should be sufficient space for sample mounting with insulation between other parts and whole length of sample.
(8) There should be provision for heating the space of cryostat with variable heat rate with low voltage.
(9) There should be provision of measurement of temperature of sample space.
(10) The experiment should be done with minimum time and cost.
(11) The cost of cryostat should be as low as possible.



ITEM NO.	PART NUMBER	QTY.
1	Blind for base_r	1
2	Valve Pipe	3
3	Coller	3
4	Outer vessel_1	1
5	Inner vessel_1	1
6	Blind for base_1_r	1
7	Flange for inner vessel_1	2
8	ISO 4762 M6 x 20 --- 20N	14
9	Hexagon Nut ISO - 4034 - M6 - N	8
10	Sample Holder Pipe1	1
11	Cup	1
12	Cup Cover	1
13	Sample Holder	1
14	Flange for inner vessel_1!	1
15	Magnet	1

Fig. 4 - Assembly drawing of hall effect and magnetic susceptibility measurement cryostat

V. Conclusion

A simple sample-inverting cryostat for Hall resistance measurements and magnetic susceptibility has enabled us to measure the Hall resistance easily and precisely at temperatures from 300 K in magnetic fields up to 0.7 T and magnetic susceptibility is measured by lock in amplifier in temperature range upto 77-300 K various cuprates materials for analysis superconductivity of material.

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