Development & Performance of Cryostat for Measurement of Magnetic Properties

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Abstract— A Simple homemade cryostat for measurement of magnetic properties is described. The magnetic susceptibility is measured by a.c. mutual inductance method. The temperature from 77-300K are made using lock-in-amplifier, a PC and some suitable electrical circuits. The sample is placed on copper strip and whole double walled cryostat is dipped in a bath of liquid nitrogen. The material for coil bobbins and sample holder is selected hylum for its good thermal and mechanical properties.

Key words: Cryostat, susceptibility, coil system

I. INTRODUCTION

A versatile cryostat includes simple experiment set up for measurement of magnetic properties. Variety of Samples like ferromagnets, antiferromagnets, superconductors etc. can be measured with least cost and minimum difficulty. One sample is measured at one time and can not move it position during measurement. It is mandatory that area surrounding the sample and space inside the vessel must be at vaccum condition.Vacuum ports are connected to rotary pump for maintaining vacuum throughout the measurement for better result.

Sample is placed inside the coil assembly.Much care is taken in designing a coil a s it is a heart of set up. There is a single primary coil to which ac signal is applied and two secondary coil in series opposition. Here sample is placed at centre of one of the secondary coil, Winding on both the coils should be such that output voltage across the secondary coil combination should be zero when no sample is present.[1,2]

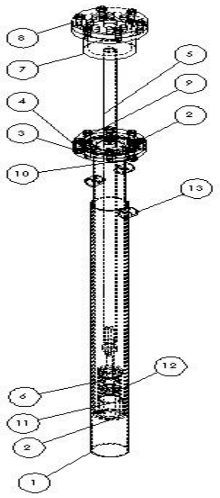
Whole set up is immersed in the bath of liquid nitrogen. It is able to measure properties at any temperature between 77-300K without changing the position.[3,4,5]. Eight connections 2-2 for primary &secondary, 2-2 for thermometer & heater are attached with the set up. 1Ω reistance is attached with primary coil. Platinum resistancethermometer is attached with sample for measurement of temperature. All measurements are performed using lock-in-amplifier which can measure readily higher harmonics.

II. DESCRIPTION OF APPARATUS

We have used a Stanford Research Systems lock-in-amplifiers, model SR-830 for this set up. This LIA has a precision sine wave oscillator and four 16 bit analog-digital inputs as well as four digital-analog outputs having 1mV resolution. The whole assembly consists of following parts.[6]

A. Cryostat

A schematic design of cryostat is shown in fig.1.



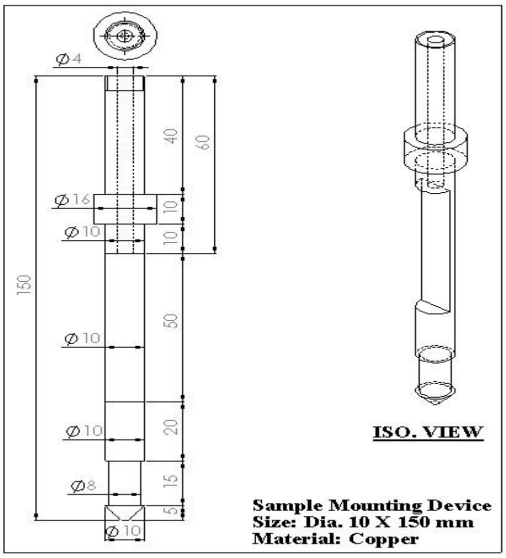
ITEM NO.	PART NUMBER	QTY.
1	Outer Vessel	1
2	Inner Vessel	1
3	Flange for Inner Vessel	1
4	Flange for Smaple Holder	1
5	Sample Holder Pipe	1
6	Sample Mounting	1
7	Cup	1
8	Cup Flange	1
9	ISO 4762 M6 x 16 --- 16N	14
10	Hexagon Nut ISO - 4034 - M6 - N	6
11	Primary Coil	1
12	Secondary Coil	1
13	Collar	3

Fig.1: Cryostat for measurement of magnetic properties

The length of SS cryostat is around 800mm.The sample holder tube is inserted into the inner vessel. The height of the inner vessel is 600mm.It's outside diameter & wall thickness are 26mm & 1.5mm respectively. Inner vessel is surrounded by outer body with 2.5mm clearance between two. The height of the outer vessel is also 600mm. It's outside diameter & wall thickness are 32mm & 4mm respectively. A hole is provided between two vessels for evacuation. Two holes are provided with inner vessel for vacuum pump & vacuum gauge. Two flanges are also attached with set up, each for inner vessel and sample holder tube. At the top side, cup is provided for facilitating eight different connection. Flanges are fitted by nut & bolt arrangement & vessels are fabricated by arc welding process. Material for vessels are selected SS-304.The evacuated SS cryostat along with the coil system is dipped into a cryocan of liquid nitrogen.

B. Sample Holder

The details of sample mounting arrangement is shown in fig.2.Sample holder is a copper piece of length 150mm &diameter 10mm.



It is extended to some dimensions for fitting purpose with sample holder tube. The sample holder tube has SS-304 material with outside diameter 12mm.It's length is selected 700mm.

C. Coil System

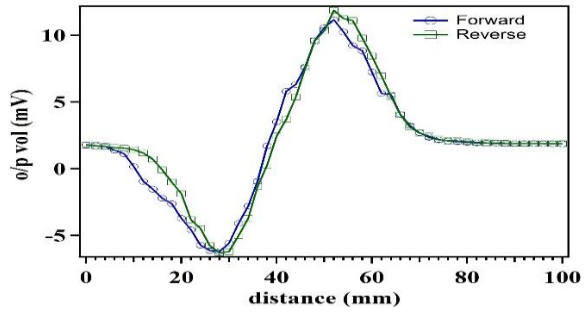
The a.c. inductance method measures the change in mutual inductance between two secondary and one primary coil. The mutual inductance is made close to zero to obtain high sensitivity. The secondary coils are kept inside the primary coil for maximum filling factor for secondary coil and the sample. The coil bobbin is made of hylum material. Hylum is chosen for its thermal and mechanical properties. The inside & outside diameters of secondary coil is 10mm & 15mm respectively. The inside & outside diameters for the primary coil is 17mm & 20mm respectively. The coils are wound by insulated copper wire of standard size of 36 gauge for primary & 44 gauge for secondary coils. The length of primary & secondary coils are 57 & 10 mm each respectively. The number of turns for primary coil is 1000& for secondary 2 x 3000.So,3000 turns are selected for clockwise and similar turns are selected for anticlockwise direction.

III. CALIBRATION

Calibration of cryostat is carried out with the help of lock-in-amplifier device. For calibration, we have taken a strong ferromagnetic material e.g. a small diameter bolt in this case. By inserting the ferromagnetic material (Bolt) inside the coil assembly, a strong sine wave should be generated which shows that cryostat is ready to work and no other disputes regarding electric connection and others are eliminated. So, we inserted bolt inside the coil assembly by gripping the strip and generate graph of voltage v/s distance which should be a sine wave.

Results are obtained by using LIA.Two connections of primary and two of secondary are connected with the lock-in-amplifier as shown in fig. with help of crocodile pins. By inserting bolt 2mm down voltage is shown in right hand side of LIA which is shown in fig. The strip is marked with lines with 2 mm markings. The last terminal of first coil for secondary coil and the first terminal of second coil for secondary coil is made short. Details of resistances of primary and secondary is mentioned below:

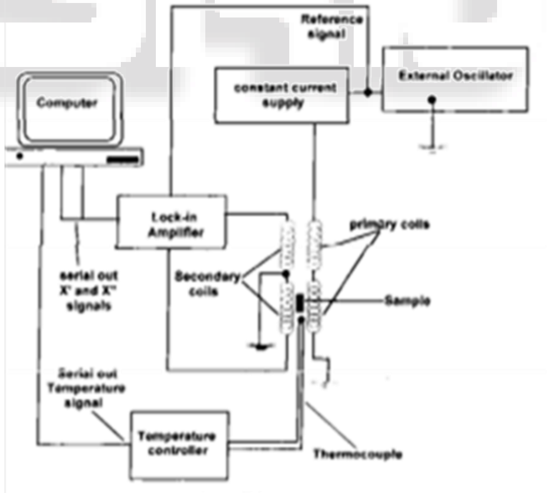
The resistance of primary coil : 51Ω
The resistance of secondary coil : 1003 Ω
The resistance of PT 100 : 113 Ω
Frequency : 223 Hz.
Calibration curve is shown as under.



IV. TEST PROCEDURE

AC susceptibility is one of the fundamental measurements used for characterizing magnetic samples. Most low-temperature measurements of AC susceptibility make use of liquid cryogens.

The basic principle of the testing is based on the mutual inductance method. A single primary coil to which an AC signal is applied and two secondary coils wounded in opposite direction put inside the primary coil. In such an arrangement, the output voltage across the secondary coil should be zero when no sample is present, since the effect of coupling is to induce two opposing signals which should cancel in a balanced system. When a sample is placed at the centreof one of the secondary coils, sample response should be observed. [7,8]



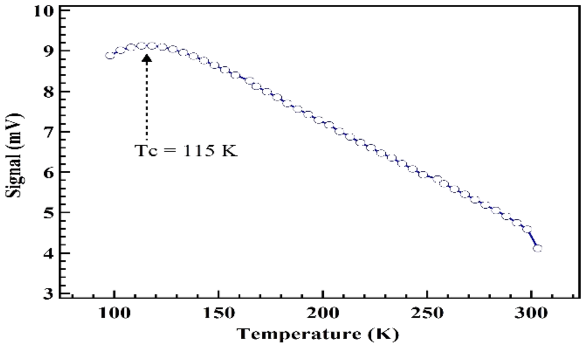
There are number of problems associated with the use of cold finger cryostat as a cooling system for such a coil arrangement: (i) A physical thermal link between the cryostat cold finger and the sample is necessary to cool the sample. However the presence of such a link may itself contribute to the measured susceptibility (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.

The lock in amplifier has a major role in testing procedure. A lock-in-amplifier is a device which is useful for measuring the amplitude & phase of a signal. The output will typically a DC voltage which is proportional to input amplitude. The device has two inputs as shown in fig. One is a input signal that is to be measured and the other is a reference. The reference should have the same frequency as the input. This signal is usually a syncsignal originating from the same source as the input signal. The input signal is first amplified in the amplifier. The gain of this amplifier is adjustable and is used to control the sensitivity of lock in. The reference signal is first led through a sine former. This is a block consisting of many components which transforms the signal to ensure it has sinusoidal form and a specific predetermined amplitude.

The step by step procedure is listed below. First of all open the cryostat sample holder by removing the nuts and placed in a proper place.Insert the coil assembly such that the windings should not affected. Remove four connections from the inner vessel & check connection continuity with the help of multimeter. Fixed the pallet on sample mounting device with the help of double side Teflon tape. Attach a Pt-100 sensor around the pallet for sensing temperature. Connections of sensor will be come out from the top of the flange.Place the sample holder on flange and close the cryostat.Create vacuum between the walls with the help of rotary vacuum pump. After sufficient vacuum created, close the valve and remove pump.Connect temperature measurement device with top connections by soldering.Primary and secondary connections will wind into crocodile pins through which it will go into the lock-in-amplifier.Connect rotary pump on connection for create vacuum. After 30 minutes, put the cryostat in cryocan. Close the gap between cryocan and cryostat by suitable arrangement and reduce evaporation of liquid nitrogen.Between 5 degree interval, measure voltage using LIA. Temperature will cross up to -180̊ .After reach to above temperature, it will finish the experiments. Close all connections & remove cryostat from cryocan. Put it into room temperature & wait until temperature come near the room temperature. Do not stop the rotary pump unless frosting inside the cryostat will occur due to lower temperature of liquid nitrogen.

V. RESULTS & DISCUSSION

We have measured the ac $\chi$of the ferromagnetic material $BiMNO_3$.We have measure voltage every 5 degree temperature drops. Graph of voltage v/s temperature is shown in fig.



Result shows that curie temperature of the sample $BiMnO_3$ is at 115 K.

VI. SUMMARY AND CONCLUSIONS

A low cost and accurate cryostat for magnetic susceptibility measurements has been designed. The cryostat has many silent features such compactness and ease of operation, fast cooling rate down to 77 K, better compatibility with measuring electronics etc. Variety of samples such as bulk and thin films can be magnetically characterized using this cryostat.

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