

FABRICATION OF AN AC MAGNETIC SUSCEPTIBILITY MEASUREMENT SYSTEM

Felipe Voltaire M. Florento, Norberto Alcantara, Gil Nonato C. Santos

Abstract — The fabrication of an AC magnetic susceptibility measurement system for the characterization of superconductors has been developed. Magnetic susceptibility describes the magnetic characteristic of materials, it is a measure of the ease with which certain materials are magnetized when subjected to a magnetic field. The mutual inductance method and the lock-in technique served as the mechanism for the equipment. The setup consists of a primary coil that is driven by a current to generate a magnetic field inside. Two pairs of secondary coils are wound oppositely inside the primary coil to cancel their mutual inductances. The primary coil is wound using a gauge # 36 magnetic wire with 1105 turns and a resistance of 118.43Ω . The secondary coils were wound with a gauge #40 magnetic wire with 1625 turns and have the resistance of 338.41Ω and 338.63Ω respectively. The presence of a superconductor inside the coil will produce a change in the flux at its superconducting state and thus result in a drop in the induced voltage. This voltage is directly proportional to the magnetic susceptibility. To obtain accurate measurements, it was necessary to phase tune the reference signal with the physical setting of the secondary coils at room temperature. This was done through the help of phasor diagrams. The reference signals used were at 400Hz, 5V at 1.2 kHz, 5V, at 800 Hz, in 5V, 3V and 1V. The resultant vector obtained at 400Hz, 5V was $0.003V \angle 15.69^\circ$ while at 1.2 kHz, 5V was $0.022V \angle -56.31^\circ$ and at 800Hz, 5V was $0.012V \angle -34.99^\circ$. This resultant vector was then adjusted by 180° to be located in the second quadrant. The reference signal's phase setting was adjusted to be either perpendicular to resultant vector or parallel to one of the secondary coil's vector depending on the placement of the phasors. A standard sample was used to determine the efficiency of the apparatus. YBCO sample was used to serve as the standard. The results of the test runs had yielded a curve directly proportional to the real and imaginary parts of the magnetic susceptibility when the adjusted phase setting was along the adjusted resultant vector.

Index Terms— AC Magnetic Susceptibility, Superconductors, YBCO, Lock-in technique, mutual inductance

1 INTRODUCTION

The term “susceptibility” originally came from Lord Kelvin Thomson. He defined the “magnetic susceptibility” [as] the intensity of magnetization acquired by an infinitely thin bar of it placed lengthwise in a uniform field of unit magnetic force. Lord Kelvin specifically made a proper distinction between susceptibility and permeability, whereas the latter was meant to relate the magnetic induction **B** to **H** [11]. Susceptibility, in more general terms, can be referred to as “a parameter giving an indication of the response of a material to an applied magnetic field. The susceptibility is the ratio of the magnetization (**M**) to the applied field (**H**). $\chi = M/H$ [19].

The study of the behavior and response to AC and DC magnetic fields is a long standing area of research and technological interest especially in studying the magnetic properties of high T_c superconductors or HSTCs [4]. This technique is now becoming one of the most common “tools”

used in the search for and the study of high T_c superconductors [16].

Researchers have been fond of studying specimens with the use of this “tool”. Magnetic measurements differ from resistivity measurements in many ways: (1) specimens do not require electrical contacts; (2) A magnetic signal is given at temperatures below T_c , when resistivity = 0, so the magnetic signal can be used to characterize the material at low temperatures; (3) The superconducting volume fraction can be estimated; (4) The signal is given even if the superconducting path is not continuous; (5) The critical current density J_c 's can be measured independently of contacts and of inter-granular weak links [29]. A certain characteristic of a superconductor exhibiting zero resistivity manifests perfect diamagnetic shielding- susceptibility χ is exactly -1.

Magnetic properties are measurable by the use of either AC or DC magnetic fields. An AC measurement involves the sample to be enclosed in a sensing coil driven by an external AC field. A time-varying magnetization is produced in which a second detection coil senses. It passes through a lock-in amplifier which then filters out the noise and detects the target signal [8].

Superconducting quantum interface devices, SQUID magnetometers, and vibrating sample magnetometers, VSM, make use of this method- measuring the magnetic moment of the material, and are widely used nowadays [8]. However,

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- Felipe Voltaire M. Florento, Bachelor of Science in Physics, De La Salle University -Manila Philippines
 - Norberto Alcantara, Master of Science in Physics, De La Salle University- Manila, Philippines
 - Gil Nonato C. Santos, Doctor of Philosophy in Materials Science and Engineering, De La Salle University-Manila, Philippines. E-mail: Gil.Santos@dlsu.edu.ph

an AC susceptometer is quite different from these instruments. The use of the lock-in technique is able to measure susceptibility under very small AC magnetic fields, in the nano- range, with or without a DC bias field. Complex susceptibility, composed of the real component χ' , and the imaginary component χ'' , can be separated and serve as a function of frequency and temperature [12]

This paper presents a design of an AC susceptometer employing local facilities. The researchers have used a system of two identical sensing coils (secondary) oppositely wound to detect the variation in flux due to the sample positioned in the center of one of them- the mutual inductance method. The lock-in technique was used to register the sample's induced magnetic response sensed by the pick-up coil. Measuring the temperature dependence of the absolute magnetic susceptibility is employed to characterize the magnetic properties of high T_c superconductors. Results of the magnetic characteristics of our specimens from the AC data are then discussed.

2 EXPERIMENTAL SECTION

A typical AC-susceptometer for measuring the magnetic susceptibility of non-ferromagnetic material is often consisting [16, 17] of a primary excitation field coil, a secondary pick-up coil, and a secondary compensation coil (known as three-coil system). The design described in the following paragraphs was built for low-frequency AC susceptibility measurements using local facilities.

The application of the mutual inductance technique in this assembly involved the coiling of two secondary coils in opposition on an "I"-shaped inset made of industrial plastic. Thin magnetic wires were used in the coiling to minimize resistance and to increase sensitivity. It was then covered with teflon and then with aluminum coil wound with stranded copper wire to reduce their capacitive coupling and to achieve thermal good stability as well [29]. It was then covered by another layer of teflon. The primary coil was wound over this, which was covered by another layer of teflon and aluminum. The DC bias field coil then followed which was also covered by layers of teflon and aluminum. Details are given in Table 1.

Table 1. Characteristics of the coil assembly

	DC Bias	Primary	Secondary	
			A	B
Wire gauge	#30	#36	#40	#40
Number of turns	535	1105	1625	1625
Resistance (Ω)	15.34	118.430	338.41	338.63

This assembly was then protected by a hollow cylinder in order to prevent contact with the wires and thus minimize changes in mutual inductance. The cross sectional diagram of the system showing the arrangement of the coils is shown in Figure 1 while the actual assembly shown in Figure 2.

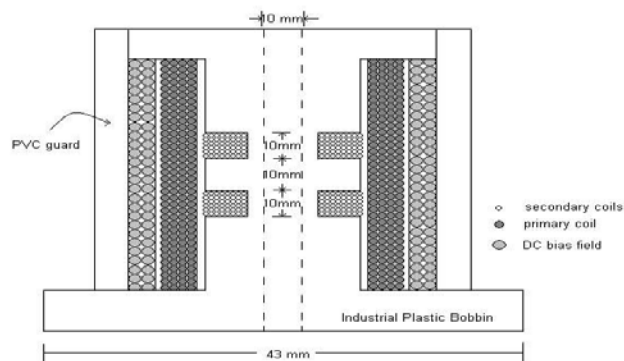


Figure 1. Coil Assembly. The cross sectional representation of the coil configuration.

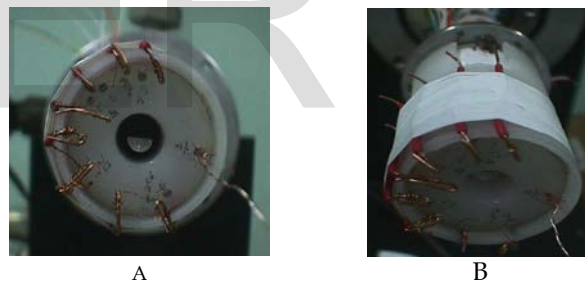


Figure 2. (A) and (B) shows the actual setup mounted on the coldhead.

The sample holder was made of an aluminum rod, 5 mm in diameter cut in half and 4.5 cm long protruding from a base as shown in Figure 3. The sample is put in place in one of the secondary coils in the flat part of the rod by tying it with a dental floss. A silicon diode thermocouple (DT-470) is in contact with the base of this holder and is as close as possible to the sample. The contribution of the sample holder designed in this manner to the induced voltages is minimal and temperature dependent. The holder permits the use of rectangular samples with maximum dimensions of 10mm x 6 mm x 4 mm.



Figure 3. The sample holder

The basic setup of the AC susceptometer is shown in Figure 4. The primary coil is driven by a function generator. The voltage drop across a resistor situated in series with the primary coil serves as the reference of the SR830 lock-in amplifier. The resistor is of the same scale with the resistance of the primary coil. This eliminates changes in the resistance of the primary coil when the temperature drops below 100K. The induced voltages on the secondary coils were connected to the differential input of the SR830 which is used as an off-balance meter. One of the main critical experimental points of the present setup is the temperature measurement, which should be considered seriously. During the measurement the coil temperature changes with that of the sample by immersing in liquid helium inside a closed cycle refrigerator. A silicon diode thermocouple (DT-470) was used to measure the sample temperature and is connected to a Lakeshore 330 Temperature Controller. Also it was found a good idea to coat both the sample and the thermocouple with a thin layer of vacuum grease, which acts as a good thermal contact agent. 2.2K Ω resistors were also placed in series with the secondary coils to compensate for phase differences.

3 RESULTS AND DISCUSSION

The efficiency and effectiveness of the system is determined through the calibration runs with a standard material and tests of other non-standard materials. There were also preliminary tests to determine the accuracy of the coil configuration.

The measurement of the resistance of each of the coils was necessary in phase adjustment when viewing through the help of phasor diagrams. The measurement of the resistance for the secondary coils is necessary to ascertain if rewinding needs to be done. The setup for obtaining the resistance is made by applying a current source to the coil and is compensated by a decade resistance box. Figure 5 shows the schematic diagram of this setup. Measurement of the voltage across the coil for each change in the current is noted down. The resistance is calculated after and is averaged. This was done for all the coils. Table 2 shows the average resistance of the coils.

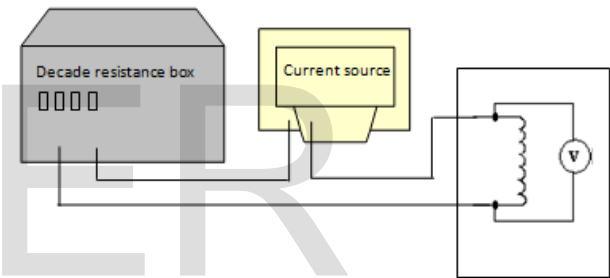


Figure 5. Schematic diagram of the setup done in resistance measurement

Table 2. The average resistances of the coils

	Resistance (Ω)
Secondary A	338.476
Secondary B	338.963
Primary Coil	118.429
DC Bias Field Coil	20.926

Given the resistances of the secondary coils, we can see that their difference $R_{A-B} = 0.487\Omega$. With such difference, it becomes necessary to place resistors in series with these pair of coils. The resistance must be able to sweep the phasors in alignment to minimize errors.

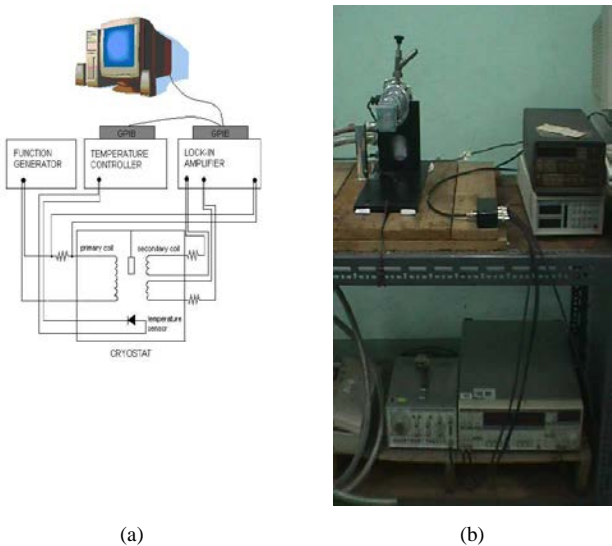
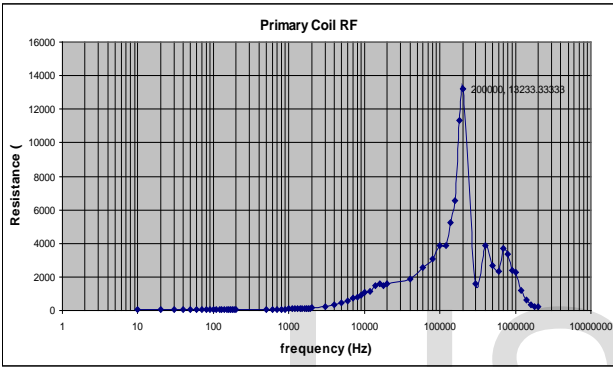


Figure 4. Operational setup (a)schematic diagram. (b) actual setup of the apparatus.

The resistance of the primary coil at varying frequencies was measured. This was necessary to determine the frequency range it can be able to handle without changing the magnetic field prevalent around the coil. The applied magnetic field and the excitation frequency are related by the following equation:

$$B_{app} \text{ (T)} = \frac{\mu_0 n V_o e^{-i(\omega t - \phi)}}{\sqrt{(R_o + \alpha T)^2 + \omega^2 L^2}}$$

The generated graph shows the permissible range for the coil configuration. For frequencies beyond the peak as shown in graph 1, measurements are no longer accurate.



Graph 1. Resistance vs. frequency graph of the primary coil

The tuning of the system involves determination of the phasors of the secondary coils. This, as stated above is done at a zero loss state, which is normally at room temperature. The proper phase setting was measured at different reference frequencies: at 400Hz, 800Hz and 1.2kHz. The measured magnitude R and phase θ are shown in table 3.

Table 3. the magnitude and phase of the secondary coils.

	400Hz		800Hz		1.2kHz	
	R (V)	θ (deg)	R (V)	θ (deg)	R (V)	θ (deg)
Secondary A	0.142	-45.12°	0.464	-71.50°	0.785	-85.91°
Secondary B	0.140	-46.08°	0.454	-72.00°	0.766	-86.64°

Table 4. The magnitude and phase of the resultant vector.

	400Hz		800Hz		1.2kHz	
	R (V)	θ (deg)	R (V)	θ (deg)	R (V)	θ (deg)
R _{A-B}	0.0031	-15.69°	0.012	-34.99°	0.0216	-56.31°

The measured phase for secondary B is adjusted by adding 180° because it was measured in the single mode of the lock-in amplifier whereas it should have been in the opposite direction due to its opposition winding with secondary A. the resultant vector R_{A-B} is then calculated and plotted on the phasor diagram. Calculations showed that at 800Hz, R_{A-B} = 0.012V and that its phase θ = -34.99. The two methods are then incorporated during the calibration runs to determine the proper phase setting. The first method suggests setting the reference phase perpendicular to the phase of the reference. This would either be 55.01° or at -124.99°. The second method suggests setting the reference phase parallel to either secondary vectors, which would be either -71.50° or 107.50°. The same measurements were done at 400 Hz and 1.2 kHz where the values are seen in table 4 above. The phasor diagram is shown in Figure 6 below to illustrate the method.

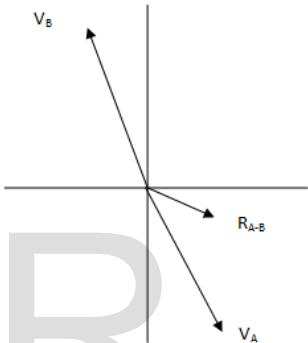
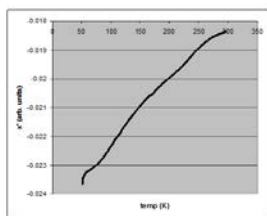
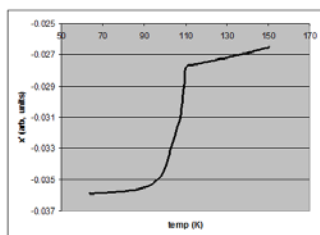


Figure 6. Phasor diagram of the secondary coils.

The calibration runs were performed using a suitable phase setting to obtain the magnetic susceptibility. From phasor analysis, the suggested phase setting was 90° ahead of the adjusted resultant vector. If the test run would yield incorrect data, the other phase settings would be tested. This is necessary because incorrect phase settings will lead to vector addition or subtraction of the real and imaginary parts- each projecting a shadow to the other. The data acquired from the runs are shown in the susceptibility vs. temperature graphs below.



GRAPH 2. BACKGROUND SIGNAL OF THE IN PHASE COMPONENT GENERATED BY THE FUNCTION GENERATOR WITHOUT ANY SAMPLE PRESENT



GRAPH 4. X' OF YBCO AT REFERENCE SIGNAL OF 800HZ, 5V AT 103° PHASE SETTING.

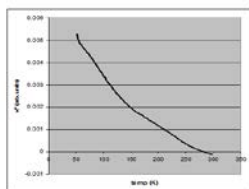
The background signal as shown in graphs 2 and 3, was obtained in order to shift out the noise generated by the secondary coils. Graphs 4 and 5 are the measurements taken at 800Hz, 5V at reference phases 103° .

The in-phase component, or \div' , shows the behavior of YBCO under a magnetic field at varying temperatures. The transition temperature is clearly indicated by the sharp drop seen in the graph. There is some error though, seeing that the drop occurs between 110K and 100K at some of the graphs, wherein one must take note that YBCO has a $T_c \approx 93K$. The error was due to the uncalibrated silicon diode sensor, which still needs to be fit to a curve.

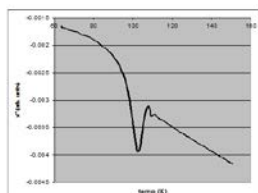
The out-of-phase component, or \div'' , is interpreted as the hysteretic loss of the material. The two peaks occur at the start of the transition temperature in the in-phase component, which tells us that the data acquired is synchronized.

4 CONCLUSION

An AC Magnetic Susceptibility Measurement System is fabricated through the use of local facilities and simple materials. The assembly consisted of a primary coil, which was made of gauge #36 magnetic wire, and two oppositely wound secondary coils, with magnetic wire of gauge #40. A DC Bias Field of magnetic wire #30 was added to allocate for future studies. A function generator feeds the magnetic field through the primary coil and changes in the flux is detected by the secondary coils connected to the differential mode of the lock-in amplifier. The phasors of the secondary coils were determined and were used to align the reference phase at 1200Hz, 800 Hz and 600Hz and with an amplitude of 5V to obtain an accurate result. Certain modifications on



GRAPH 3. BACKGROUND SIGNAL OF THE OUT-OF-PHASE COMPONENT GENERATED BY THE FUNCTION GENERATOR WITHOUT ANY SAMPLE PRESENT.



GRAPH 5. X'' OF YBCO AT REFERENCE SIGNAL OF 800HZ, 5V AT 103° PHASE SETTING.

the phase were necessary after each and every run to discern any shadows present in the system.

The differential voltage across the two secondary coils are measured with changing temperature. This voltage is known to be directly proportional to the magnetic susceptibility and is used to represent the latter. Thus, the use of arbitrary units to represent the susceptibility.

In conclusion, the apparatus that has been calibrated is suitable for measurement of the magnetic susceptibility of superconductors at various frequencies. The system is able to obtain the desired data, though it is necessary to phase tune the system for different frequencies and for different materials.

REFERENCES

- [1] Bernaldez, F. L., Molina, K. L., Sarmago, R. V. (2001) Investigation of Phasor Behavior of High Tc Bulk Superconductors by AC Susceptibility Measurements. Proceedings of the 19th SPP Philippine Physics Congress (pp 407-410) Manila.
- [2] Campbell A.M, IEEE Trans. Appl. Supercond. 5, 682 (1995).
- [3] Campbell, A. C., (1991) DC Magnetization and Flux Profile Techniques. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 129-158) New York.
- [4] Civale L., Worthington T.K., Krusin-Elbaum L. and Holtzberg F., (1991). Non-Linear A.C. Susceptibility Response Near the Irreversibility Line. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 313-322) New York.
- [5] Civale L., Worthington, T. K., Krusing-Elbaum, L., Holtzberg F. (1991) Non-Linear AC Susceptibility Response Near the Irreversibility Line. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 313-332) New York.
- [6] Colorado Superconductors Institute (April 3, 2002). Magnetic Susceptibility[On-line]. Available: <http://www.user.qwest.net/?csconductor>
- [7] Couach, M., and Khoder, A.F. (1991). AC Susceptibility Responses of Superconductors: Cryogenic Aspects, Investigation of Inhomogenous Systems and of the Equilibrium Mixed State. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 25-48) New York.
- [8] Crangle J., Solid State Magnetism, Edward Arnold, Great Britain (1991), pp 57-65.
- [9] Dodrill, B.C., (1990) AC and DC Magnetization Studies in Solid State Magnetism: A Synopsis. Lakeshore's Series 7000 Magnetic Measurement Solutions. Ohio
- [10] Drobac, D., and Marohnic, Z., Multipurpose Measuring Device based on AC Susceptometer. (2000)
- [11] Goldfarb R.B., Lelental M., and Thompson C. A., (1992). Alternating-Field Susceptometry and Magnetic Susceptibility of Superconductors. (LSCI) Plenum

- Press, London, England.
- [12] Gomory F, Supercond. Sci. Technol 10, 523 (1997).
 - [13] Griffiths, D. J., Koch, C. C. and Charlesworth J. P., Phil. Mag. 33, 505 (1976).
 - [14] Hein R. A., and Falge R. L. Jr., Phys. Rev. 123, 407 (1961).
 - [15] Ishida T., and Mazaki H, Phys. Rev. B 20, 131 (1979).
 - [16] Khoder A.F., and Couach M., (1991) Early Theories of χ' and χ'' of Superconductors: The Controversial Aspects. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 213-228) New York.
 - [17] Korean Research Institute of Standards and Science (April 3, 2002). Biomagnetism[On-line]. Available: <http://www.kriss.re.kr/quantum/supercon/bio.html>
 - [18] Krause, J. K., and Bergen, J. R., (1990) Understanding Magnetic Measurement Techniques. High Tc Res.
 - [19] Lakeshore IDEA Field Control User's Manual, (1990) Lakeshore Cryotronics, Inc., Ohio.
 - [20] Ling, X. X., and Budnick, J., (1991) AC Susceptibility Studies of Type II Superconductors: Vortex Dynamics. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 377-388) New York.
 - [21] Martien D., (April 3, 2002) Quantum Design. Introduction to: AC Susceptibility. [On-line]. Available: <http://www.qdusa.com>
 - [22] Maxwell E. & Strongin M., Phys. Rev. B 20, 131 (1963).
 - [23] Mermin, David & Ashcroft, Neil. (1976). Solid State Physics. Philadelphia: Holt Rinehart & Winston.
 - [24] Nikolo, M., Hermann, A. M., (1991) High Field AC Susceptometer Design for Measurements of Superconducting Single Crystals and Results. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 553-560) New York.
 - [25] Rillo, C., Lera, F., Badia, A., Angurel, L.A., Bartolome, J., Palacio, F., Navarro, R., and van Duynveldt, A. J., (1991) Multipurpose Cryostat for Low Temperature Magnetic and Electric Measurements of Solids. Magnetic Susceptibility of Superconductors and Other Spin Systems (pp 1-24) New York.
 - [26] Rynyan W. R., Semiconductor Measurements and Instrumentation: McGraw-Hill Books Company – USA.
 - [27] Stoppard O. and Guban D, Physica C 241, 375 (1995).
 - [28] Van Duynveldt, A. J., AC Susceptibility Studies in Solid State Magnetism. Leiden University
 - [29] Youssif, M. I. & Bahgat, A. A. (2000). AC Magnetic Susceptibility Technique for the Characterization of High Temperature Superconductors. Egypt J. Sol., Vol 23, no. 2. (Mansoura University & Al-Azhar University)