MEASUREMENT METHODS FOR DETERMINATION OF TRANSITION TEMPERATURE OF HIGH-T_C SUPERCONDUCTORS

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Abstract: In this paper contact and non-contact techniques for the measurement of transition temperature of high-T_c superconductors is described. A non contact method has been developed for measurement of transition temperature (T_c) of high-T_c superconductors. This technique is based on the measurement of change in the resonance frequency of a tank circuit with temperature. The applicability of this method in obtaining T_c of a YBCO thin film and a small piece of YBCO bulk sample is demonstrated. This technique can be effectively used for characterizing the samples of irregular shape and small dimensions where it is difficult to make proper contacts required for resistivity measurements.

Index terms: **Transition temperature, Meissner effect, Non contact method, Resistivity, Four probe method, Susceptibility**

I INTRODUCTION

A superconductor shows vanishing resistance and pronounced diamagnetic property below a characteristic temperature called the transition temperature (T_c). The transition temperature is an indicator of the physical properties of different superconductors and accurate measurement of this temperature is critically important in the study of high-T_c superconductors. In a superconductor the resistivity should be zero and the magnetic susceptibility should be negative. At present, the electric transport and ac susceptibility methods are the two methods for the measurement of high-T_c superconducting transition temperature. In a four terminal measurement, the four terminals are collinear and equally spaced. The contacts at the voltage probes become a part of the lead; and since the voltmeter has high input impedance, no significant current will flow in that direction. Hence, we can measure only the potential difference between the voltage contacts. The contact technique is suitable for thin/thick films and bulk samples of regular geometries with a relatively large size. The ac susceptibility method adopts a lock in amplifier to provide ac voltage signal for the primary coil, and then lock-in-amplifier is used to measure the electrical signals of the secondary coil according to the magnetic susceptibility values. The magnetic measurements are non-invasive and are more reliable than resistance measurements.

II MEISSNER EFFECT METHOD

The Meissner effect can be used to measure the critical temperature of superconductors as a function of drive current. The critical temperature of high-T_c superconducting material is measured using liquid nitrogen. According to Lenz's law, a current loop will create a magnetic field and when the magnetic field passes through another coil it creates an induced emf in the wire. When the superconductor is placed in between the coil and reaches critical temperature there will be no induced emf due to the Meissner effect [1]. The critical temperature of the sample can be measured by finding the point where the change in potential in the secondary coil occurs. The receiver coil is connected into a lock-in-detector to eliminate white noise and allow for better measurements. An induced emf was created by the magnetic field from the transmitter coil on to the receiver coil creates a potential change that will be received to find the critical temperature of the sample. When the sample is below its critical temperature it is superconducting and will expel all the magnetic flux, causing the magnetic field in the undriven receiver coil to be very small. When the sample reaches T_c, the magnetic field in the receiver coil will be more comparable to that in the transmitter coil. The critical temperature measurement setup is designed to take advantage of the fact that superconducting material expel all magnetic flux to find the materials accurate critical temperature. Figure 1 shows the circuit diagram of experimental set up for critical temperature measurement.

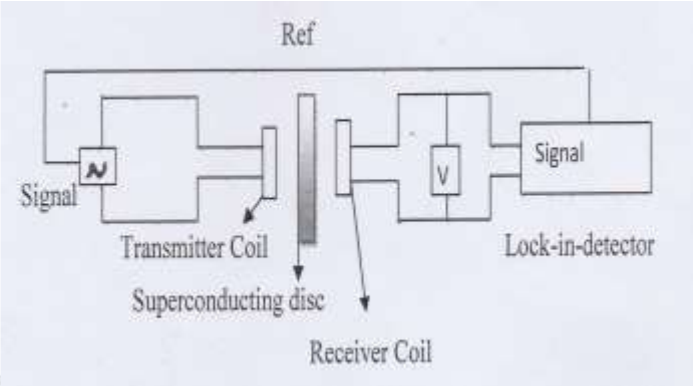

Figure 1. Circuit diagram of experimental set up for critical temperature measurement

III. AC SUSCEPTIBILITY METHOD

Susceptibility technique [2] provides T_c due to the change in magnetization of the sample. The ac susceptibility method avoids contact resistance, eliminates the adverse effects and is a non destructive measurement method. 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. In the absence of a sample, which is usually centered in one of the secondary coils, the detection system should be ideally in equilibrium i.e. the net flux Φ_{net} across the secondary coils is zero. In the presence of a sample the induced magnetization due to the ac primary field $h=h_o e^{-i\omega t}$, will result in an off -balance signal across the secondary coil detection system. A lock-in amplifier (SR530 Stanford, USA) was used for the detection of output voltage from the secondary coil. A. silicon-diode sensor DT-500 was used for temperature sensing. The presence of superconductor inside the coil will produce a change in flux at its superconducting state and thus result in a drop in the induced voltage. The voltage is directly proportional to the magnetic susceptibility. The ac susceptibility methods adopts a lock in amplifier to provide an ac voltage signal to the primary coil, and then lock-in-amplifier is used to measure the electric signals of the secondary coil according to magnetic susceptibility values. The real and imaginary part of the ac susceptibility by induction method was determined. Figure 2 shows set up for the determination of magnetic susceptibility.

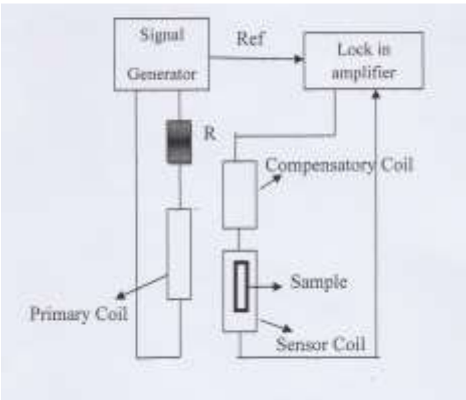

Figure 2. Schematic diagram of ac susceptometer.

The magnetic manifestation of zero resistivity is that a material is a superconductor if it exhibits perfect diamagnetic shielding i.e., its susceptibility χ is exactly -1. Therefore, ac susceptibility measurement can be used to determine critical temperature. The complex susceptibility can be defined as

$$\chi = \chi' + i\,\chi''$$

χ' is related to measure of flux exclusion (diamagnetism) due to induced shielding currents (elastic susceptibility) while χ'' represent magnetic losses due to the movement of flux lines i.e. the energy absorbed in the sample (viscous susceptibility). In case of high-T_c superconductors, the viscous susceptibility reflects ac losses. For a good superconducting sample, there is always a low temperature region where χ' can reach –1 in a small ac field. At a given low temperature, with increasing H, χ' increases from –1 to a stable value. At the same time χ'' increases from zero to a maximum value, before χ' reaches its stable value. This phenomenon is general for high-T_c superconductors. Figure 3 shows ac susceptibility ((χ' and χ'').for perfectly diamagnetic sample

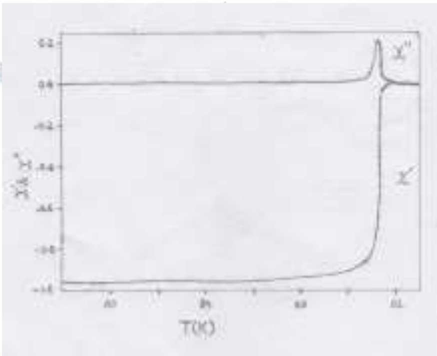

Figure 3. Variation of ac susceptibility ($\chi'$and χ'') with temperature for YBCO pellet

IV FOUR-PROBE DC METHOD

Resistivity measurements on high-T_c superconductors are usually carried out using the four probe technique. In standard four-probe dc method (figure 4), four leads are attached to the superconductor in a linear configuration. Outer two leads are meant for passing current in the specimen and inner two leads for measuring potential drop between the two voltage terminals. Each current lead and each voltage lead is distinct and there is no overlapping of electrical contacts. For making the contacts to the superconductor, high conducting silver paste was used successfully with good reproducibility. A small current (~10μA) is passed between the two current points in order to avoid sample heating. A Keithley constant current source (Keithley 224) was used for passing current through the specimen. The voltage developed was measured using digital nano-voltmeter (Keithley, 181). A platinum resistance thermometer (PRT) was used to read the temperature. A digital multimeter (Keithley 195A) was used to measure the temperature of the PRT. The sample is first cooled to liquid nitrogen temperature. The R-T data is recorded while slowly raising the temperature of the film above liquid nitrogen temperature. The temperature interval between two subsequent readings is generally 5K between 300K to 130K and 2K between 130 K to 100K and 1K till the zero resistance is achieved. The experimental values of resistance as a function of temperature are recorded automatically through the computer interfacing.

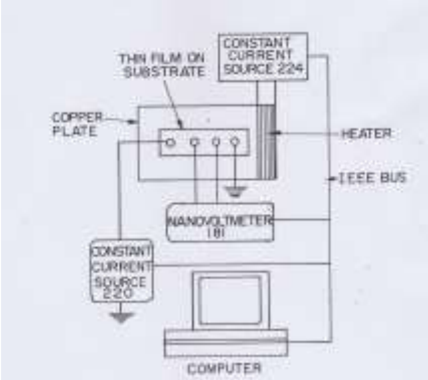

Figure 4. Experimental set up for recording and plotting R-T curve by standard four-probe dc method

V RESONANCE FREQUENCY METHOD

This technique is based on the measurement of change in the resonance frequency of a tank circuit with temperature. Figure 5 show the schematic of the experimental set up used for measuring resonance frequency of the tank circuit.

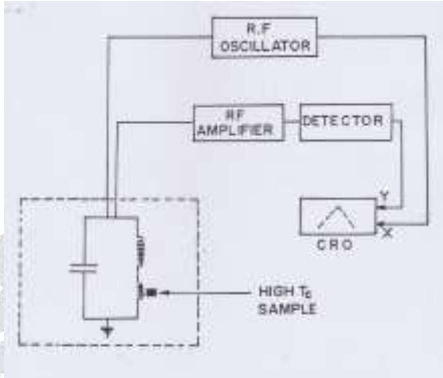

Figure 5. Schematic of the experimental set–up used for obtaining the resonance frequency of the tank circuit.

The inductance of the tank circuit consists of two coil connected in series. One coil is of sixty turns (1mm diameter) and the other coil of ten turns. The latter was kept in close contact with the superconducting sample. The tank circuit was designed in such a way that its resonance frequency was around 18MHz at room temperature. An rf-oscillator (Hewlett Packard 3325B) was used to inject rf- signal (f=17-32 MHz) in the tank circuit. The reflected rf-signal was amplified by a low noise pre-amplifier (gain 60dB) and detected by a diode detector. For determining the resonance frequency of the tank circuit, the detector output was monitored while the oscillator frequency was swept from f_0-δf to f_0+δf where f_0 was the expected resonance frequency and 2δf was the range in which rf frequency was swept. At resonance frequency, the detector output will have a maximum value. For precise determination of the resonance frequency, a voltage signal corresponding to the frequency sweep from f_0-δf to f_0+δf was fed to X-channel of a CRO and the detector output was connected to the Y-channel of the CRO.

The YBCO thin film was deposited on 1cm × 0.5 cm $SrTiO_3$ (100) substrate by dc magnetron sputtering technique [3] and YBCO bulk was prepared using conventional solid state reaction method[4].

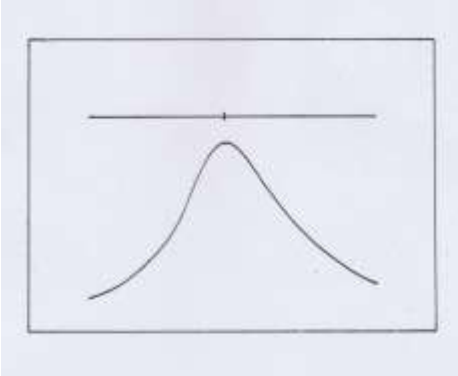

Figure 6. Variation of the detector output with frequency of the tank circuit at 77K. The marker indicates the resonance frequency

Figure 6 shows a typical curve for the variation of the detector output with an injected rf frequency. The maximum value in this curve corresponds to resonance frequency and its value was obtained using a pointer as shown at the top of the figure.

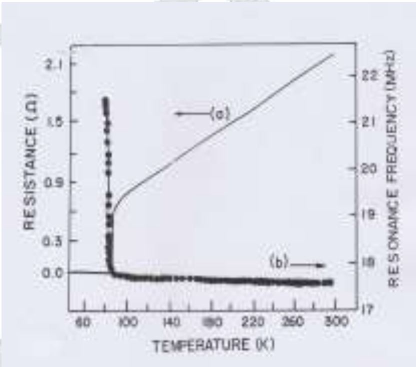

Fig. 7 (a): Resistance-verses temperature curves for YBCO thin film sample and (b) Variation of resonance frequency with temperature when the film is inductively coupled to a tank circuit.

In the present experimental set-up, a change of 0.01 MHz in the resonance frequency can be easily measured. This technique has been used to determine the transition temperature of a YBCO thin film and a small piece of YBCO bulk and the results have been compared with resistance verses temperature (R-T) measurements.

For the measurement of transition temperature of YBCO thin film, a flat spiral coil of ten turn was glued on the top of the film and this coil was connected to the other sixty turn coil of the tank circuit. The value of f_0 and Q of the tank circuit at the room temperature for thin film sample was 17.6 MHz and 20 respectively. Figure 7 shows the variation of resistance verses temperature (curve a) for a YBCO thin film and the variation of the resonance frequency (f_0) with temperature (curve b) for the same film. For YBCO bulk sample, a coil of ten turns wound around a small piece of bulk pellet (3mm long, 1mm diameter) was connected to the sixty turn coil. The values of f_0 and Q of the tank circuit for bulk sample at the room temperature were 18.11 MHz and 19 respectively. Variation of resonance frequency, f_0 of the tank circuit with temperature was measured for both the cases.

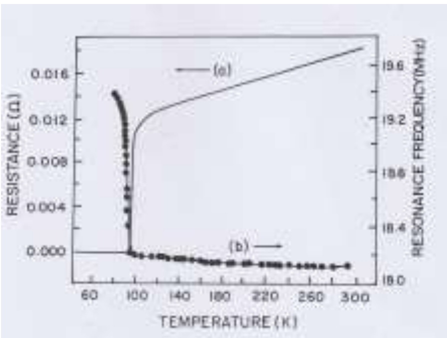

Fig. 8 (a): Resistance-verses temperature curve for YBCO bulk sample and (b) Variation of resonance frequency with temperature when the sample is inductively coupled to a tank circuit.

It is evident that there is a sharp change in the value of f_0 just at the temperature where the film becomes superconducting at T_c =84K. Figure 8 shows the plots of R-T (curve a) and f_0-T (curve b) measurements for the YBCO bulk sample. Here also f_0 changes drastically at a temperature corresponding to T_c =91K.

Comparison of f_0-T and R-T plots for YBCO film and bulk YBCO clearly indicate that the transition temperature of the superconducting samples can be determined accurately from the sharp change in the resonance frequency f_0 of the tank circuit at the transition temperature T_c. This technique is useful for samples available in small quantities. It can also be used for samples of any shape and dimension.

A non-contact superconducting transition temperature (T_c) measurement system using a Hall probe array was devised to measure T_c of high-T_c superconductor coated conductors without physical contact to the surface, thereby avoiding contacts with electrodes which can cause damage to the conductor surface [5].

VI CONCLUSION

A cheaper noncontact method for T_c measurements based on a modification of resonant frequency of an LC circuit has been described. This is based on measurement of resonance frequency of a tank circuit at different temperatures. The applicability of this method in obtaining T_c of a YBCO thin film and a small piece of YBCO bulk sample is demonstrated. This technique can be effectively used for characterizing the samples of irregular shape and small dimensions where it is difficult to make proper contacts required for resistivity measurements.

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