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Simple and inexpensive technique for measuring the transition temperature of superconducting thick films from 60 to 300 K using liquid nitrogen and subcooled liquid oxygen

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ABSTRACT

This paper describes a simple and low-cost experimental setup intended for AC susceptibility measurements between 60 and 300 K. A mutual inductance bridge for measuring the transition temperature of high-T_c superconductor thick films was constructed. The device is based on using liquid nitrogen and subcooled liquid oxygen; it allows temperatures down to 60 K to be achieved. Experimental details are described and illustrative measurements on high-T_c superconductor thick films are included.

1. Introduction

AC susceptibility is a powerful technique for studying high temperature superconducting materials [1–5]. It is can be used to determine transition critical temperatures (T_c) and critical currents for a wide range of different specimen types including bulk slabs, cylinders, discs, films and powder specimens. In addition, it can be used to investigate inside the crystalline grains (intragranular) and outside the grains (intergranular), magnetic flux penetration, loss mechanisms, vortex dynamics and pinning mechanisms [6]. The mutual inductance bridge is commonly used in AC susceptibility measurements [1,7–11]. The bridge consists of a driving primary coil generates the AC magnetic field and two secondary or sense coils detect the change in flux passing through the sample generating a secondary voltage according to Faraday's law. Since the two secondary pick up coils wound opposite to each others, introducing the sample to one secondary coil produces a differential output signal which should be primarily due to only the sample properties. However, some errors such as non-ideal coils characteristics, series resistance and the change in the inductance with temperature affect the technique accuracy. This can be largely overcome by subtracting the background signal. The magnitude and phase of the detected signal can then be measured using phase sensitive detector or lock in amplifier and real and complex susceptibilities calculated.

One of the key techniques to realize measurements and applications of high temperature superconductors (HTS) in practice is the cryogenic design such as cooling system size, weight and power consumption. Liquid nitrogen is very common as a cryogenic cooling liquid and it is easy to obtain. In most cases, HTS systems are cooled by saturated liquid nitrogen which has natural boiling temperature (NBT) 77.344 K [12]. Subcooled liquid nitrogen is considered as an important cooling technique for obtaining lower temperature than NBT which is suitable for obtaining lower operating temperature. The important benefit of working with subcooling liquid nitrogen is the lower operating temperature 65 K instead of 77 K and the suppression of bubbles that may be generated from an internal or external heat load. Since bubbles play a critical role in the degradation of electrical insulation performance of liquid nitrogen, subcooling is essential for high-voltage devices such as fault current limiters (FCL) or transformers [13].

It is well known that materials exist in three phases, solid, liquid and vapour. Subcooled liquid nitrogen can be explained by the low temperature nitrogen phase diagram shown in Fig. 1 [14]. Subcooled liquid refers to liquid that is below its saturation temperature for a given pressure or above its saturation pressure for a given temperature. In other words, subcooled liquid is a liquid that is not in equilibrium with its saturated vapour. Subcooling moves the thermodynamic state of the saturation line into the pure liquid region. The common method to make subcooled liquid nitrogen is evacuation on the liquid nitrogen surface by a vacuum

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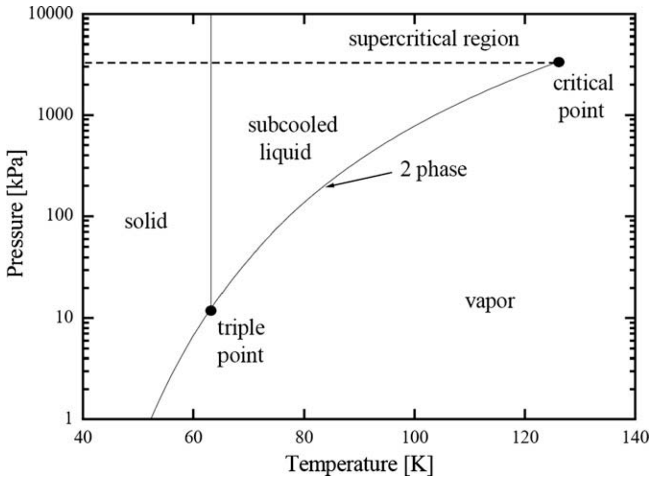

Fig. 1. Phase diagram for low temperature nitrogen.

pump. The lowest temperature of subcooled liquid is at its triple point which is at 63.149 K for liquid nitrogen and at 58.361 K for liquid oxygen [14,15].

In this article, a mutual inductance bridge susceptometer was built for measuring T_c of HTS thick films in the temperature range 60–300 K using liquid nitrogen and subcooled liquid oxygen.

2. Susceptometer construction

The measurement of AC magnetic susceptibility by mutual inductance techniques is well documented [7–11]. The principle of

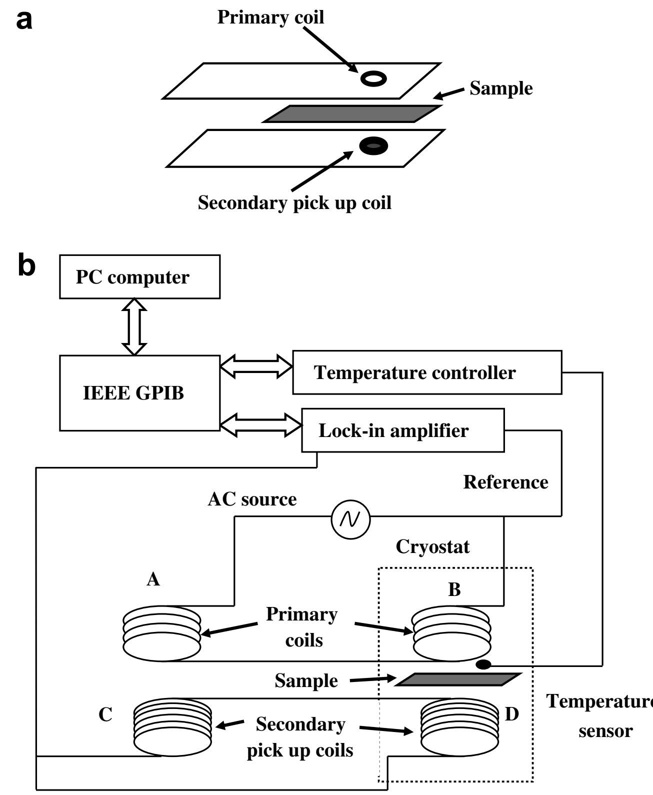

Fig. 2. Schematic diagram illustrating the principle of AC magnetic measurements (a) a sample holder, primary and single sensing coils and (b) circuit diagram.

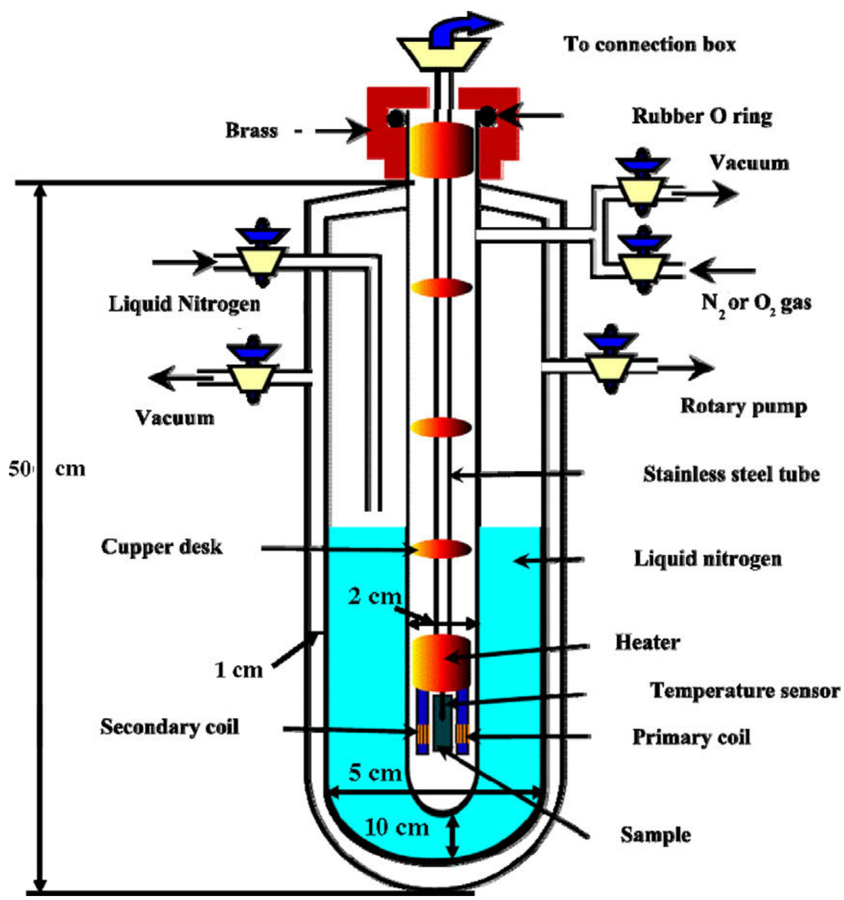

Fig. 3. Schematic diagram for the cryostat.

the operation is simple. As shown in Fig. 2, an alternating current in the series connecting primary coils A and B produces an alternating magnetic field, which induces a voltage in the pick up coils. The secondary coils are wound in opposition to each other and connected in series. The internal diameter of primary and secondary coils was 2 mm while their numbers of turns were 300 and 1000, respectively. With no sample between the coils, the mutual inductance of the combined coils is ideally zero. However, with a superconducting thick film (or paramagnetic or diamagnetic materials) between coils B and D the produced signal depends upon the magnetization, hence upon the AC susceptibility of the sample. The output and reference signals were connected to SR530 lock in amplifier and the real and imaginary parts of the output signal which were proportional to the in-phase χ' (the real part) component and the out-of-phase χ'' (the imaginary part), respectively, were recorded as a function of temperature. Then T_c can be defined in terms of the onset, midpoint or end of the diamagnetic transition.

In the real case, the two secondary coils were not identical and hence there was very small output signal voltage. This could be overcome by recording the background signal without the sample as a function of temperature and then correcting the sample reading.

3. Cryostat design and operation

A detailed diagram of the cryogenic system is shown in Fig. 3. The cryostat was made of three concentric Pyrex glass tubes. The external diameters of the two outer tubes were 7 and 5 cm, respectively. The internal diameter of the sample chamber tube was 2 cm. The height between the bottom of the sample chamber and the bottom of the cryostat is 10 cm. The total cryostat height is 50 cm as can be seen in the figure. The sample holder was 1 cm diameter stainless steel tube in which electrical wires connection were passed.

The system operation can be divided into two cases as follows. The temperature was decreased down to 60 and 65 K using subcooled liquid oxygen and subcooled liquid nitrogen, respectively. Firstly, the temperature lowering down to 60 K using subcooled liquid oxygen. An Edward rotary pump was switched on to evacuate the space between the two outer tubes for two hours before starting the cooling process. The sample is fixed in the sample holder and another rotary pump was used to evacuate the sample chamber. After that the vacuum valve of the sample chamber was closed and oxygen gas valve was released. This process was performed three times. The oxygen gas pressure in the sample chamber was kept at 1 atm before starting the cooling process. The same rotary pump was used to evacuate the liquid nitrogen chamber. Opening the liquid nitrogen valve applies indirect pressure on the surface of liquid nitrogen in the dewar and causes liquid nitrogen flow from the dewar to liquid nitrogen chamber. This flow process is safe since liquid nitrogen is at atmospheric pressure in the dewar. The liquid nitrogen flow was continued during all the experiment. As shown in Fig. 4a, the temperature of the sample decreases slowly till it reaches about 110 K. After that, the pressure inside the sample chamber started to decrease as a result of decreasing temperature. As a result the temperature in the sample chamber rabidly decreased to 92 K. The temperature was constant at 92 K for some time and this was ascribed to the conversion of oxygen gas into liquid oxygen. Then temperature started to decrease slowly to 78 K. After that pressure suddenly reduced and temperature decreased below 78 K. With reducing pressure more using the rotary pump in the sample chamber, the temperature was decreased to about 60 K.

The second case in which the temperature can be reduced to 65 K is similar to the previous case. This is can be achieved by replacing the oxygen gas in the sample chamber by nitrogen gas.

With continuous flow, the temperature slowly increased due to heat leakage till 77 K with a rate of about 1 K/min as shown in Fig. 4b. After that a Lackshore 330 low temperature controller with upgraded ram was used to ramp temperature with a rate of 0.3 K/min [16]. Both of lock in amplifier and temperature controller was

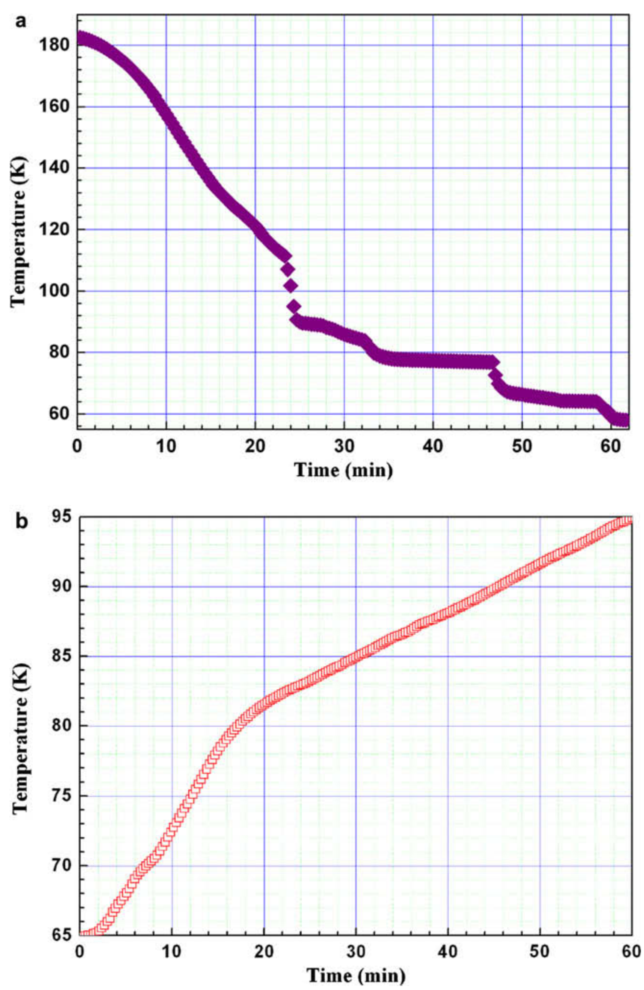

Fig. 4. The change of temperature as a function of time (a) cooling and (b) heating.

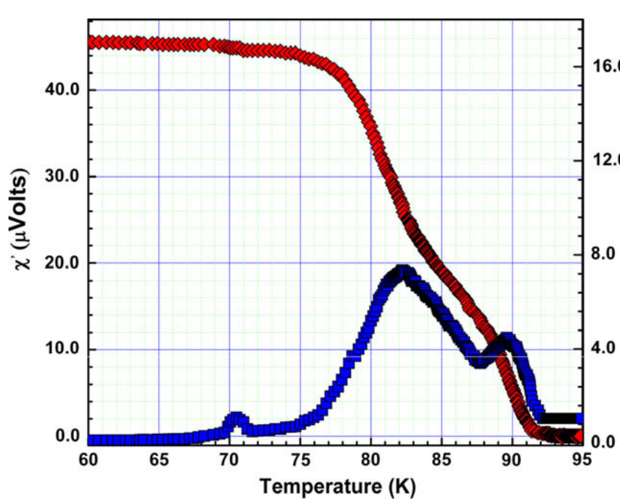

Fig. 5. The temperature dependence of the real and imaginary parts of AC susceptibility for an $YBa_2Cu_3O_z$ oxygen annealed superconducting thick film.

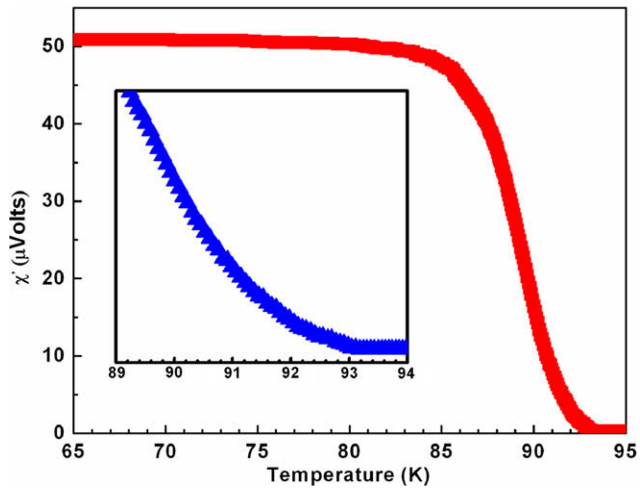

Fig. 6. The real part of AC susceptibility for an $YBa_2Cu_3O_z$ oxygen annealed superconducting thick film as a function of temperature.

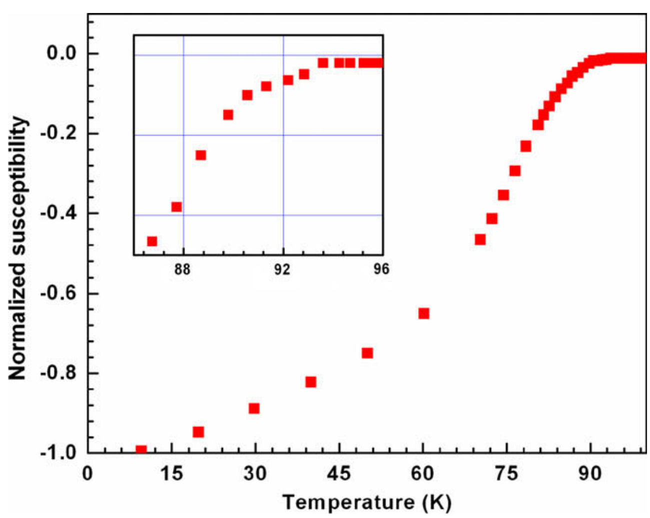

Fig. 7. The temperature dependence of zero field cooled DC normalized magnetic susceptibility measured with $H \| c$ axis of the film in Fig. 6.

connected to a PC through IEEE GPIB interface card. Time, real and imaginary parts of the output signal and temperature were recorded with a rate of 10 reading/min.

4. Results and discussions

Fig. 4b shows the change of temperature with time. Between 60 and 78 K the controller heater was off and the temperature increased by heat leakage. The line in Fig. 4b can be divided into two regions; below and above 78 K. Below 78 K, the slope of the line was about 1 K/min that means the heating rate of temperature was about 1 K/min. Although this heating rate is considered relatively slow, it can be more slowed by increasing thermal insulation using higher vacuum between the external double wall tubes. Above 78 K (the heater of the controller was switched on), the slope line was about 0.3 K/min and this means temperature was increased according to the adjusted heating rate. It is important to mention that liquid nitrogen flow is continuous during the whole experiment.

The temperature dependence of real and imaginary parts of an $YBa_2Cu_3O_z$ oxygen annealed superconducting thick film is shown in Fig. 5. Three transitions can be clearly seen in the real and imaginary components. Another $YBa_2Cu_3O_z$ thick film was measured on this technique and its transition temperature was 93 K as shown in Fig. 6. To confirm this unusual high-T_c, this sample was measured using a quantum design SQUID magnetometer (Quantum Design, Model MPMS7) at ISTEC Laboratory, Faculty of Marine Science and Engineering, Tokyo University. The details of this setup are described in elsewhere [17]. As shown in Fig. 7, the normalized susceptibility curve confirms this high value for T_c. This indicates that the experimental setup described in this work provides accurate and reliable AC characterization of superconducting thick films between 60 and 300 K.

5. Conclusions

In conclusion, a simple and inexpensive experimental setup suitable for measuring AC susceptibility of superconducting thick films has been constructed. It has been shown that cooling the system down to 60 K using subcooled liquid oxygen can be easily obtained. With controlling thermal leakage the AC susceptibility can be measured between 60 and 77 K. Subcooled liquid oxygen can be produced easily using liquid nitrogen. Therefore, it can be concluded that subcooled liquid oxygen is an alternative and interesting cryogenic cooler and can be successfully used in building cryogenic systems.

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