

# AC MAGNETIC SUSCEPTIBILITY MEASUREMENTS OF Bi(Pb)-Sr-Ca-Cu-O SUPERCONDUCTOR IN DC MAGNETIC FIELDS

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## Abstract

*In this work a Bi based superconductor with the composition  $Bi_{1.6} Pb_{0.4} Sr_2 Ca_2 Cu_3 O_y$  was prepared using sol-gel method and AC Magnetic Susceptibility measurements  $\chi'(T)$  and  $\chi''(T)$  were carried out on the sample in the temperature range from 4.2 K to 120 K in both zero and applied dc magnetic fields. The ac Hartshorn bridge was used to measure the ac susceptibility of the sample. From  $\chi'(T)$  data in zero field the onset superconducting transition temperature of the sample was found to be 107.5 K. A single drop in the  $\chi'(T)$  data suggests that the sample has only one superconducting phase which is identified as Bi-2223. A small kink was observed in the  $\chi'(T)$  at a temperature of 105.6 K due to the weak link coupling between the superconducting grains. The  $\chi''(T)$  data in zero dc field shows an intergrain peak at a temperature of 100 K indicating that the maximum energy dissipation and the penetration of the ac field and the shielding current into the center of the sample take place at this temperature. In the dc applied magnetic fields the  $\chi'(T)$  and the  $\chi''(T)$  data show broad transitions and broad peaks respectively indicating that the magnetic fields suppressed superconductivity of the sample. The intergrain peaks in  $\chi''(T)$  were found to shift to low temperatures with increasing dc magnetic fields.*

*In low dc magnetic fields two drops were observed in  $\chi'(T)$  data which are attributed to the superconducting grains and the weak links in the sample. In high magnetic field  $\chi'(T)$  data shows only one single drop suggesting that the magnetic field simultaneously penetrated into the superconducting grains and the weak links. The high magnetic fields also shifted the superconducting transition temperature of the sample to lower temperatures.*

**keywords:** Superconductor, BSCCO

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## 1 Introduction

It is apparent that the electrical resistance of a conductor decreases when it is cooled. It was experimentally found that when some conductors are cooled to very low temperatures their resistances fall abruptly to zero. The conductors with zero resistance are called superconductors and the temperature at which a superconductor loses its resistance is called its superconducting transition temperature. The breakthrough of higher superconducting transition temperature resulted from Bednortz and Muller's discovery in the La-Ba-Cu-O system[1] in 1986. In early 1988 Maeda et.al[2] discovered a superconductor with composition Bi-Sr-Ca-Cu-O which showed superconducting transition temperature at 110 K. The ac magnetic susceptibility measurements are most useful for characterizing superconducting samples and these measurements can be conveniently carried out using Hartshorn's mutual inductance technique[3]. The ac susceptibility can be written as a complex quantity such that

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

where  $\chi'$  and  $\chi''$  are the in-phase and the out-of-phase components of the complex susceptibility. In general temperature dependence of the in-phase component  $\chi'(T)$  of the superconducting sample shows the perfect diamagnetism of the sample below its superconducting transition temperature which indicates that sample exhibits Meissner effect. The temperature dependence of the out-of-phase component  $\chi''(T)$  reflects the energy dissipation in the superconductor during the superconducting transition. Usually each drop in  $\chi'(T)$  is accompanied by a dissipative peak in  $\chi''(T)$ . In high  $T_c$  superconductors the complex susceptibility is the most useful parameter for studying the nature of the intergranular and the intragranular superconductivity. In a very small ac field a sharp drop is observed in  $\chi'(T)$  showing diamagnetism of the sample. However if the ac field is relatively large then typically there will be two superconductive components in  $\chi'(T)$ . The component with the higher critical temperature can be regarded as being due to the bulk superconductivity appearing in the inside of the superconducting grains. On the other hand the component with the lower critical temperature and the corresponding peak in  $\chi''(T)$  are attributed to the presence of weak coupling between superconducting grains. This two stage behaviour in  $\chi'(T)$  is found even more clearly in the presence of an applied dc magnetic field.

$\chi'(T)$  also determines the onset temperature of the superconducting transition, superconducting transition width, and the number of superconducting phases existing in the sample. The objective of this work was to characterize a Pb doped  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  superconductor prepared by employing sol-gel method. In order to characterize and to study the granular superconductivity of the sample ac susceptibility measurements were performed on the sample both in zero and applied dc magnetic fields.

## 2 Sample preparation

In this study a Pb doped Bi-Sr-Ca-Cu-O ceramic superconductor with the composition  $\text{Bi}_{1.6} \text{Pb}_{0.4} \text{Sr}_2 \text{Ca}_2 \text{Cu}_3 \text{O}_y$  was prepared using sol-gel method. The appropriate amount of the powders of  $\text{Bi}(\text{NO}_3)_3$ ,  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{Ca}(\text{NO}_3)_2$ ,  $\text{Sr}(\text{NO}_3)_2$  and  $\text{Cu}(\text{NO}_3)_2$  of purity around 99% were mixed in a solution of citric acid, ethylene glycol and distill water and the solution was heated at  $180^\circ\text{C}$  for one day. The resultant powder was first pelletised and furnace at  $660^\circ\text{C}$  for 24 hours. Subsequently it was ground, pelletised and furnace at  $825^\circ\text{C}$  for 24 hours, and finally the powder was reground, pelletised and furnace once again at  $844^\circ\text{C}$  for 10 days. All these processes were carried out in air.

## 3 Experimental techniques

The major part of the ac susceptibility apparatus is the ac Hartshorn bridge[3] consisting of a primary coil, two oppositely wound secondaries, mutual inductance box, potentiometer, ac power supply and a two phase Lock-in-amplifier. The sample chamber is a long glass tube sealed to the upper plate of the cryostat. The primary and the secondary coils are wound on a tufnel rod which is sealed onto the lower end of the glass tube. The superconducting magnet is connected to the lower part of the cryostat. The sample holder is a thin non-magnetic aluminium plate which was attached to one end of a beryllium-copper rod. The heater wire is non-inductively wound around this rod and the other end of the rod is soldered to a long stainless steel tube. A sensitive Gold/Iron-chromel thermocouple which was placed using G.E. varnish on to the one side of the aluminium plate was used to measure the temperature of the sample.

## 4 Experimental procedure

The sample was mounted on one side of the aluminium plate and the stainless steel tube gently inserted into the sample chamber and sealed with o-ring to the upper plate of the cryostat. The sample chamber was pumped out to a good vacuum. A small ac current was applied to the primary coil. The ac magnetic field produced by the primary coil and the frequency of the ac signal were 0.25 G(r.m.s) and 370 Hz respectively. Initially the bridge was balanced by adjusting the mutual inductance box and the potentiometer so that  $\chi'(T)$  and  $\chi''(T)$  were zero. Subsequently the sample was cooled down to 4.2 K using liquid nitrogen and liquid helium.

### 4.1 The measurements of $\chi'(T)$ and $\chi''(T)$ in zero applied dc magnetic field

The sample had rectangular shape with the dimensions of 11.1mm x3.6mm x1.3mm. The increasing current was applied to the heater coil by a current source and

the measurements of  $\chi'(T)$  and  $\chi''(T)$  were recorded by the two phase lock-in-amplifier while the sample was warming. The corresponding temperatures of the sample in the range from 4.2 K to 120 K were also recorded by a voltmeter. In this experiment the current source, the voltmeter and the lock-in-amplifier were all interfaced to a computer and the data were recorded by the computer.

#### 4.2 The Measurements of $\chi'(T)$ and $\chi''(T)$ in applied dc magnetic fields

The sample was cooled to 4.2 K in zero magnetic field. A dc magnetic field of 5 G was applied to the sample by the superconducting magnet. Then the sample was slowly warmed by passing a current through the heater, and the measurements of  $\chi'(T)$  and  $\chi''(T)$  and the corresponding temperatures were recorded in the presence of the magnetic field. These measurements were repeated with the magnetic fields of 15 G, 25 G, 50 G, 100 G and 500 G being applied to the sample. The measurements of  $\chi'(T)$  and  $\chi''(T)$  carried out in this way are referred to as zero field cooled (ZFC) measurements.

### 5 Results and discussion

#### 5.1 The $\chi'(T)$ and the $\chi''(T)$ in zero applied dc magnetic field

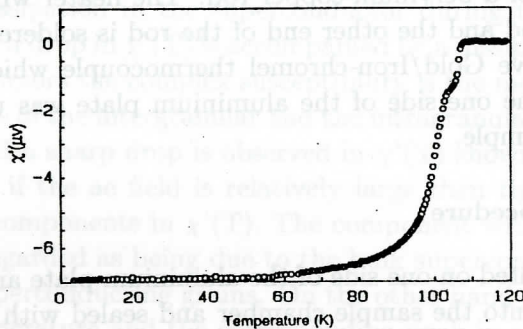


Figure 1: Measurement of  $\chi'(T)$  on Bi-Pb-Sr-Ca-Cu-O superconductor in zero dc magnetic field

The  $\chi'(T)$  data on the Bi-Pb-Sr-Ca-Cu-O superconducting sample in zero d.c magnetic field is presented in the Figure 1. It shows that the onset temperature of the superconducting transition of this sample is 107.5 K. As can be seen in the  $\chi'(T)$  data a small kink is observed at the temperature of around 105.6 K and this kink may be due to the weak link coupling between the superconducting grains. This kink for Pb doped Bi-Sr-Ca-Cu-O sample was also reported by other research groups [3]-[6].

A sharp drop in  $\chi'(T)$  could have been observed if smaller ac field had been employed. In any case it is gratifying to observe that there is one single drop in  $\chi'(T)$  indicating that this sample has only one superconducting phase which is identified as the Bi-2223 superconducting phase.

The  $\chi''(T)$  data for the sample in zero dc magnetic field is presented in the Figure 2. The  $\chi''(T)$  data in zero applied magnetic field shows an intergrain peak indicating that energy dissipation takes place in the intergranular region of the sample. The intergrain peak in  $\chi''(T)$  is found to be at a temperature of 100 K at which the energy dissipation in the intergranular region of the sample takes its maximum value and also at this temperature the ac field and the shielding current penetrated into the centre of the sample.

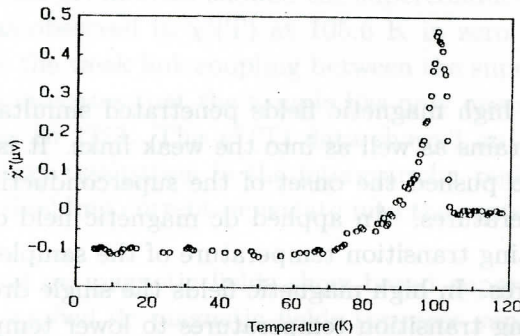


Figure 2: Measurement of  $\chi''(T)$  on Bi-Pb-Sr-Ca-Cu-O superconductor in zero dc magnetic field

## 5.2 The $\chi'(T)$ and $\chi''(T)$ in applied dc magnetic fields

The  $\chi'(T)$  data in several applied dc magnetic fields are presented in Figure 3. From this data it is clear that when the applied magnetic field is increased, the superconducting transition of the sample become broadened since the applied magnetic fields suppressed superconductivity of the sample. The suppression of superconducting transition of Bi based superconductors in the presence of dc magnetic fields was also reported by others [6]-[9]. The  $\chi'(T)$  in low applied dc magnetic fields showed two diamagnetic contributions. However, in high magnetic fields the  $\chi'(T)$  showed only one single stage transition. The two drops in  $\chi'(T)$  in low magnetic fields are usually attributed to the superconducting grains and the weak link couplings between the superconducting grains of the sample. These two drops in  $\chi'(T)$  in the presence of magnetic fields for the Bi based sample were also observed by W. Lang et.al [6].

In high magnetic fields these two drops in  $\chi'(T)$  disappeared and a single drop

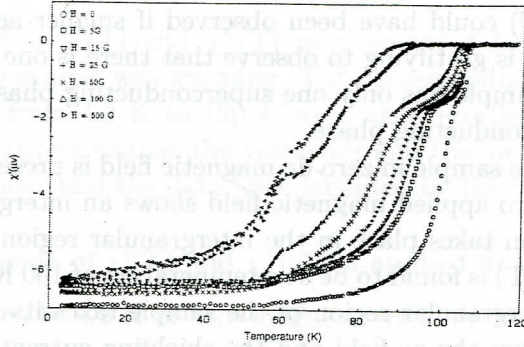
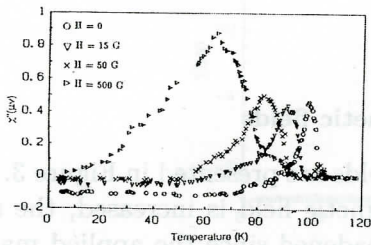
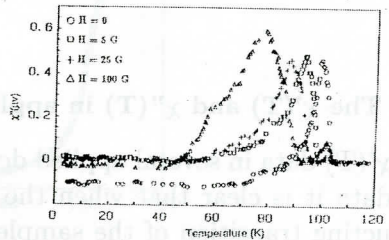


Figure 3: Measurement of  $\chi'(T)$  on Bi-Pb-Sr-Ca-Cu-O superconductor in several applied dc magnetic fields

was observed because high magnetic fields penetrated simultaneously deeply into the superconducting grains as well as into the weak links. It is clear that the high dc magnetic fields also pushed the onset of the superconducting transition of the sample to lower temperatures. An applied dc magnetic field of 500G was able to shift the superconducting transition temperature of the sample by as much as 14.2 K to lower temperatures. In high magnetic fields the single drop in  $\chi'(T)$  and the shift of superconducting transition temperatures to lower temperatures for the Bi based superconductors were also reported by others[6],[8],[9].



(a)



(b)

Figure 4: (a) Measurement of  $\chi''(T)$  on Bi-Pb-Sr-Ca-Cu-O superconductor in several applied fields (0 G, 5 G, 25 G, 100 G) (b) Measurement of  $\chi''(T)$  on Bi-Pb-Sr-Ca-Cu-O superconductor in several applied fields (0 G, 15 G, 50 G, 500 G)

The  $\chi''(T)$  data in several dc magnetic fields are shown in Figures 4 (a) and 4 (b) where it can be seen the broad intergrain peaks associated with the energy dissipation in the intergranular region of the sample. These intergrain peaks in  $\chi''(T)$

became broadened and shifted to lower temperatures as the applied dc magnetic field was increased. The intragrain peak shift to lower temperatures in the presence of dc magnetic fields for Bi based superconductor was also previously reported [6],[7]. From this data it is clear that the penetration of ac field and the shielding currents to the center of the sample take place well below the superconducting transition temperature of the sample in the presence of dc magnetic fields.

## 6 Conclusions

The Pb doped BSCCO superconductor with the composition  $\text{Bi}_{1.6} \text{Pb}_{0.4} \text{Sr}_2 \text{Ca}_2 \text{Cu}_3 \text{O}_y$  prepared by sol-gel method showed the superconducting transition at 107.5 K. A small kink was observed in  $\chi'(T)$  at 105.6 K in zero applied magnetic field which may be due to the weak link coupling between the superconducting grains. A single drop in  $\chi'(T)$  indicates that the sample has only one superconducting phase which is identified as Bi-2223. The  $\chi''(T)$  data showed an intergrain peak at 100 K indicating the energy dissipation in the intergranular regions of the sample and the ac field and the shielding current penetrate into the center of the sample at this temperature

The  $\chi'(T)$  in applied dc magnetic fields show broad superconducting transitions indicating that the applied dc magnetic fields suppress superconductivity.  $\chi''(T)$  also shows broad intergrain peaks in accordance with  $\chi'(T)$  and these peaks shifted to a lower temperature with increasing dc magnetic fields. In low dc magnetic fields  $\chi'(T)$  shows two drops attributed to the superconducting grains and the weak links of the sample. However, in high magnetic fields these two drops disappear and a single drop in  $\chi'(T)$  was observed since the high magnetic field simultaneously penetrated into the superconducting grains and the weak links. High magnetic fields also pushed the superconducting transition temperatures of the sample to lower temperatures.

## References

- [1] J.C. Bednortz and K.A. Muller,(1987) *Z.Phys*, **B64**, 189.
- [2] H. Maeda, Y. Tanaka, M. Fukutomi and T. Asano(1988) *Japanese Journal Applied Physics Lett.*, **4**, L.209.
- [3] L. Hartshorn(1925). *Journal of Scientific Instruments*, **5**, 145.
- [4] N. Knauf, J. Harnischmacher, R. Muller, R. Borowski, B. Rader and D. Wohlleben (1991). Preparation and characterization of single-phase Bi-Pb-Sr-Ca-Cu-O high temperature superconductors, *Physica C*, **173**, 414-424.

- [5] N. Savvides, A. Katsaros and S.X. Dou(1991). Critical current density and flux pinning in silver superconductor composites and tapes, *Physica C* , **179**, 361-368.
- [6] W. Lang, H. Jodlbauer, W. Markowitsch, P. Schaffarich and H. Kusmany(1989). Systematic investigation of complex susceptibility and transport properties in (Bi,Pb)Ca SrCuO, *Physica C* , **162-164**, 1639-1640.
- [7] G. J. Russel, K.N.R. Tayler, D.N. Matthews, A. Bailey and R.A.Vaile(1989). Effect of intergranular flux pinning of the magnetic properties of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  and  $(\text{Bi}_{0.9}\text{Pb}_{0.1})\text{Sr}_1\text{Ca}_1\text{Cu}_{1.8}\text{O}_x$ , *Physica C* , **162-164**, 327-328.
- [8] C.J. Van der Beek, M. Essers, P.H. Kes, M. J. V. Menken and A.A. Menovsky(1992). Vortex response to ac-fields in  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ , *Superconductor Science and Technology* , **5**, S260-263.
- [9] C. Duran, J. Yazyi, A. Arribere, F. de la Cruz(1992). Superconducting interlayer decoupling and softening of the flux line structure in BSCCO single crystals, *Superconductor Science and Technology* **5**, S272-275.