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PREPARATION AND RESISTANCE MEASUREMENTS OF **BSCCO** SUPERCONDUCTORS

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Abstract

Three Bismuth based superconducting samples with the composition $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ *were prepared using different solid state reaction techniques. The* $Bi_2O_3, PbO, SrCO_3$ *and* CuO *powders of* 99% *purity were used as starting raw materials. The powders were mixed with the nominal cation ratio* $Bi : Pb : Sr : Ca : Cu = 0.7 : 0.3 : 1 : 1 : 1.5$ *and then mixed well using dry grinding, wet grinding and wet ball milling methods. The powders were calcined at* 820^0C *and pallets of diameter* 13 *mm and thickness* $1-2$ *mm were prepared from the calcined powder under* 300 *kg* cm^{-2} *pressure. The samples were then sintered at different appropriate temperatures.*

The resistance measurements at low temperatures were performed on three samples using a resistivity probe in the temperature range from $55K$ *to* $200K$. *The samples prepared by dry grinding, wet grinding and wet ball milling methods have shown the superconducting transition Temperatures* T_C *at* $112K, 115K$ *and* $110K$ *respectively.* T_{Co} *of the three samples were found to be* $70K, 80K$ *and* $68K$ *respectively. In this work we developed a technique and successfully achieved a temperature as low as* $55K$ *while the resistivity probe was in liquid nitrogen.*

keywords: :Superconductor, BSCCO

1 Introduction

When some metals, alloys and certain compounds are cooled to extremely low temperature, they transform to a new state called the superconducting state exhibiting remarkable changes in electric, magnetic and thermal properties. The most important change being the complete disappearance of d.c electrical resistance. This transition phenomenon is called superconductivity, and these materials are known as superconductors. The temperature at which the transition occurs is called the superconducting transition temperature. Superconductivity was first discovered in mercury by Kamarling Ones in 1911. The breakthrough of higher transition temperature resulted from Bednortz and Muller's discovery[1] in the copper oxide system. In 1986 they found evidence for superconducting transition occurring between $30K$ and $40K$ in the $LaBaCuO$ system. After this discovery research groups have intensified their research in superconductivity to find superconductors with higher and higher transition temperatures. In early 1987 it was shown that superconductivity occurs in $YBaCuO$ system at $90K$. Further, in early 1988 $BiSrCaCuO$ superconductor was discovered with transition temperature at $110K$. In the same year $TlBaCaCuO$ superconductor was discovered with the transition temperature at $120K$. The superconducting transition temperatures and the transition widths of the superconductors depend on the preparation techniques employed. The objective of this work is to characterise the BSCCO superconductors prepared by employing different preparation techniques. Series of BSCCO superconductors were prepared using dry grinding, wet grinding and wet ball milling methods. In order to characterise the samples the resistance measurements were performed on these samples in the temperature range from 55 K to 200 K.

2 Sample preparation

2.1 Powder preparation

The series of $BSCCO$ samples with composition $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ were prepared using solid state reaction techniques. The $Bi_2O_3, PbO, SrCO_3$ and CuO powders of 99% purity were used as starting raw materials. The powders were mixed with the nominal cation ratio $Bi : Pb : Sr : Ca : Cu = 0.7 : 0.3 : 1 : 1 : 1.5$ and then mixed well. Three different techniques were used for mixing these materials. They were dry grinding, in which a pestle and a mortar were used to grind, wet grinding, in which acetone was used and wet ball milling in which the starting materials were ball milled with ethyl alcohol and the milling media was alumina balls. Finally mixed starting materials were calcined at 820^0C in air for 12 $hours$[2]. A locally made stainless steel die and a piston of diameter 13 mm were used to pelletize the sample. A hydraulic jack attached to a pressure gauge was used to press the samples and the samples were pelletized under the pressure of 300 $Kgcm^{-2}$.

2.2 Sintering

2.2.1 Sample 1 (dry grinding method)

The sample 1 prepared by dry grinding method was first subjected to a preliminary sintering at 830^0C in air for 55 *hours* and air quenched to room temperature. The sample was subjected to a second sintering at 860^0C for 70 *hours*. In order to improve the quality of the sample the pellet was reground and palletised again and resintered at 860^0C for 70 *hours*.

2.2.2 Sample 2 (wet grinding method) and Sample 3 (wet ball milling method)

The samples prepared by wet grinding and wet ball milling method were first subjected to a preliminary sintering at 830^0C in air for 55 *hours* and quenched to room temperature. The pellets were subjected to a second sintering at 860^0C for 70 *hours*[3].

3 Experimental Techniques

3.1 Resistivity Probe

A locally made resistivity probe[4] was used to measure the resistivity of the samples in the temperature range from $55K$ to $125K$.

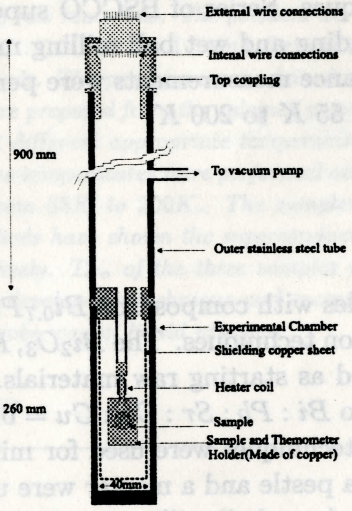

Figure 1: Schematic diagram of the resistivity probe

The diagram of the probe is shown in figure 1. To measure the resistance of the sample, the four probe method was employed. The temperature of the sample was

measured by a $Rh - Fe$ resistance thermometer. The temperature of the sample was controlled by electrical heating.

3.2 Experimental Procedure

The resistivity probe was pumped out to a good vacuum and resistance measurements were carried out on the samples while the samples were heated and cooled. The samples were gradually warmed by electrical heating and the resistances and corresponding temperatures of the samples were recorded. The measurements of resistance were repeated while the samples were cooled.
When the stainless steel probe was evacuated using the vacuum pump and left immersed inside the liquid nitrogen dewer for sufficient time, it reached equilibrium at $77K$. Then a small amount of liquid nitrogen was admitted into the probe and then the probe was evacuated once again. During this process the temperature inside the probe was observed to be dropped suddenly to about $55K$. After that it was found that the temperature increased slowly and came to equilibrium at $77K$. This temperature drop may be due to the Joule Thompson effect.

4 Results

4.1 Resistance of $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ superconductor (sample 1) prepared by dry grinding method

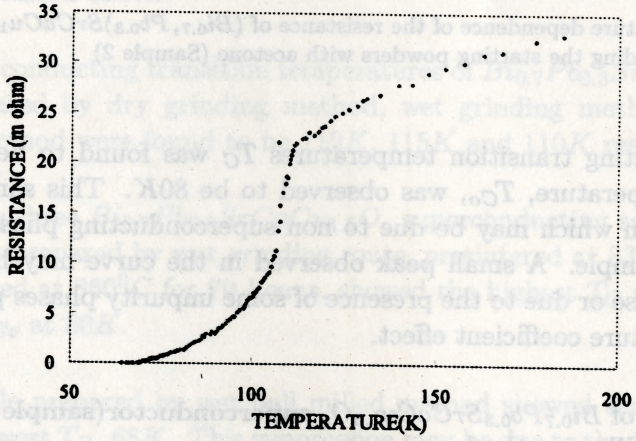

Figure 2: The temperature dependence of the electrical resistance of $(Bi_{0.7}, Pb_{0.3})SrCaCu_{1.5}O_x$ superconductor prepared by dry grinding method (Sample 1)

The variation of resistance of sample 1 in the temperature range from $55K$ to $180K$ is shown in Figure 2. A considerably broad transition was observed. This may be due

to the presence of other non superconducting phases in the sample or due to the presence of impurities in the sample. The superconducting transition temperature,T_C and the zero critical temperature, T_{Co}, of the sample were observed at $112K$ and, at $70K$ respectively.

4.2 Resistance of $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ superconductor(sample 2)-prepared by wet grinding method

The result of the resistance measurements of sample 2 prepared by wet grinding method in the temperature range from $55K$ to $300K$ is given in Figure 3.

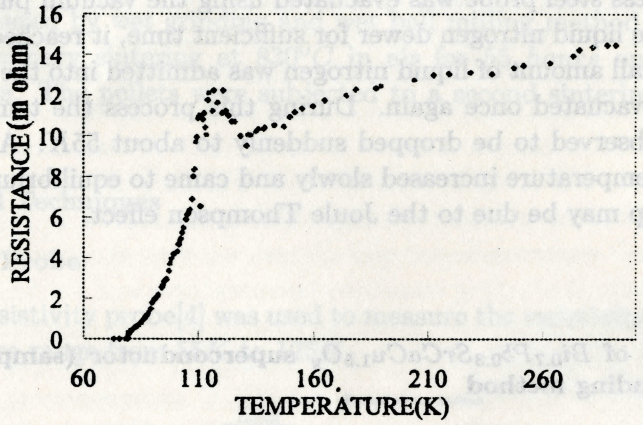

Figure 3: Temperature dependence of the resistance of $(Bi_{0.7}, Pb_{0.3})SrCaCu_{1.5}O_x$ superconductor prepared by grinding the starting powders with acetone (Sample 2)

The superconducting transition temperatures T_C was found to be $115K$ and the zero critical temperature, T_{Co}, was observed to be $80K$. This sample also shows a broad transition which may be due to non superconducting phases or impurities present in the sample. A small peak observed in the curve may be either due to the electronic noise or due to the presence of some impurity phases probably having positive temperature coefficient effect.

4.3 Resistance of $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ superconductor(sample 3)-prepared by wet ball milling method

The resistance measurements on sample 3 in the temperature range from $55K$ to $200K$ prepared by wet ball milling method is presented in Figure 4. The superconducting transition Temperatures T_C of the sample was observed at $110K$ and zero critical temperature was observed at $68K$. This sample also shows a broad transition.

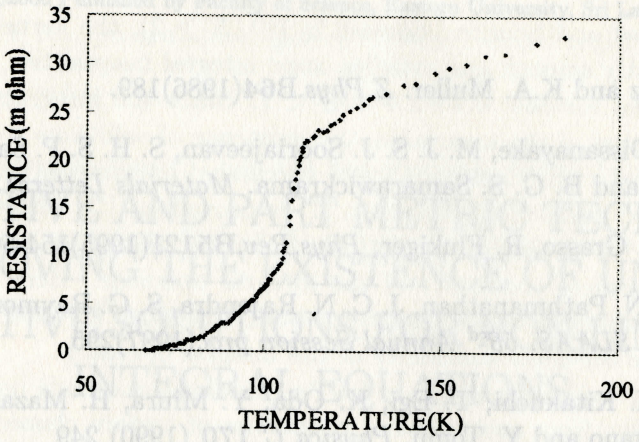

Figure 4: . Temperature dependence of the resistance of $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_x$ superconductor prepared by Ball Milling the starting powders using alumina balls and ethyl alcohol (Sample 3)

5 Conclusion

1. When a small amount of liquid nitrogen was admitted into the probe and the probe was evacuated once again the temperature of the sample dropped suddenly to about $55K$. This sudden temperature drop may be due to the Joule- Thomson effect.

2. The superconducting transition temperatures of $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ samples prepared by dry grinding method, wet grinding method and wet ball milling method were found to be $112K$, $115K$ and $110K$ respectively.

3. Out of the three $Bi_{0.7}Pb_{0.3}SrCaCu_{1.5}O_y$ superconducting samples, sample 2, which was prepared by wet grinding route, presintered at 830^0C for 55 *hours* and sintered at 860^0C for 70 *hours*, showed the highest T_C at 115 K and the highest T_{Co} at $80K$.

4. The sample prepared by wet ball milled method showed the lowest T_c $110K$ and the lowest T_{Co} $68K$. This suppression may be due to the impurity addition from the milling media - alumina (Al_2O_3)balls[5].

Acknowledgement

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