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PHASE DISCRIMINATION OF HIGH TEMPERATURE SUPERCONDUCTING PHASES IN THE BiSrCaCuO SYSTEM BY MICROWAVE ABSORPTION

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Abstract - The formation of the so-called 80 K and 120 K superconducting phases in the BiSrCaCuO system has been investigated by microwave absorption. This material is characterized by an intense low field microwave absorption (LFMA) below the superconducting transition temperature. LFMA is shown to discriminate between the formation of the two high temperature superconducting phases in this system. Various heating stages in the sample preparation are studied.

INTRODUCTION

Several studies of the newly discovered high temperature superconductors by microwave absorption with an electron spin resonance spectrometer have been reported.[1-6] (Space limits us to only representative references throughout). For ceramic oxide superconductors, an intense low-field microwave absorption (LFMA) has been observed. It is now established that this low field microwave absorption is diagnostic of the superconducting phase.[4] This signal is associated with magnetic flux trapping and may allow an estimation of the amount of superconducting phase present . In this study we show that LFMA may be used to detect and discriminate superconducting phases with different transition temperatures(T_C).

Recently, several groups have published interesting studies on the BiSrCaCu system.[7-12] Stoichiometric variations, and different sintering times and temperatures influence the ratio of the two superconducting phases that are found with T_C's of 80 K and 120 K. The structure responsible for the transition close to 80 K has been

described as an orthorhombic unit cell with a = 5.4 Å, b = 5.4 Å and c = 30.6 Å[13-15] or as a tetragonal unit cell with a = 3.8 Å and c = 30.6 Å.[16-18] The composition of this phase has been reported to be close to $Bi_2(Sr,Ca)_3Cu_2O_8$.[18] It has been established that the composition of the 120 K phase is probably close to $Bi_2Sr_2Ca_2Cu_3O_{10}$.[19-20] But this phase has not been isolated and has been only observed together with the 80 K phase.

Here, our focus is to investigate and characterize the BiSrCaCu system at different stages of sample preparation, primarily by low-field microwave absorption.

Experimental

The Bi-Sr-Ca-Cu samples, with a nominal composition $BiSrCaCu_2O_y$ were prepared by mixing appropriate amounts of Bi_2O_3, $CaCO_3$, $SrCO_3$ and CuO. These powders were ground with an agate mortar and pestle for 35 minutes. The precursor powder was heated at 1073 K for 12 h (sample 1) followed by heat treatment at 1093 K for 6 h in a porcelain crucible (sample 2). A compact black material was obtained which was reground and pressed into pellets prior to a reheat treatment at 1143 K for 12 h (sample 3) and then at 1153 K for 8 h (sample 4). The final step in the synthesis involved the heating of sample 4 at 1158 K for 7 h (sample 5), for 20 h (sample 6), for 27 h (sample 7) and for 42 h (sample 8). All samples were heated and cooled at 80 K/h in air.

Microwave absorption measurements were performed on a Bruker ESP 300 ESR spectrometer using a modulation amplitude of 10 G and a microwave power of 2 mW. The derivative of the imaginary part of the magnetic susceptibility with respect to magnetic field ($d\chi''/dH$) was recorded as a function of the applied

magnetic field. The temperature in the cryostat was controlled with an Oxford Instruments ESR 900 helium flow system. In addition to the internal thermocouple mounted in the Oxford cryostat, an external thermocouple was used to monitor the temperature at the sample position. This thermocouple was taped in contact with the sample tube. Approximately 5 mg of each reground pellet were placed into 2 mm i.d. by 3 mm o.d. Suprasil quartz tubes, evacuated and sealed for measurements.

Results and Discussion

The X-ray patterns obtained for 2θ = 15 to 60^{o} for samples 4 to 8 are characteristic of the $Bi_2Sr_2CaCu_2O_y$ low T_c phase. Nevertheless, the presence of an additional diffraction peak at $2\theta = 32^{o}$ suggests the multiphase character of the samples.

An intense low field microwave absorption is observed for all samples. After cooling the sample in a 3 kG field, the magnetic field is switched to 0 G and the microwave absorption is recorded as a function of the magnetic field for temperatures from 4 K to 125 K.

Below the second transition temperature (T_c ~ 120 K), the microwave absorption shows a maximum (Figure 1) which has been described as the critical field where flux slippage occurs.[1] The position of this maximum is slightly shifted towards lower magnetic fields as the temperature is raised from 4 K to 120 K. Also as the temperature increases the signal maximum becomes narrower and higher.

Figure 1 shows the intensity of $(d\chi''/dH)_{max}$ plotted versus temperature. Two maxima are observed which correspond to the 80 K and 120 K high T_c phases. Figure 1 shows the growth of the

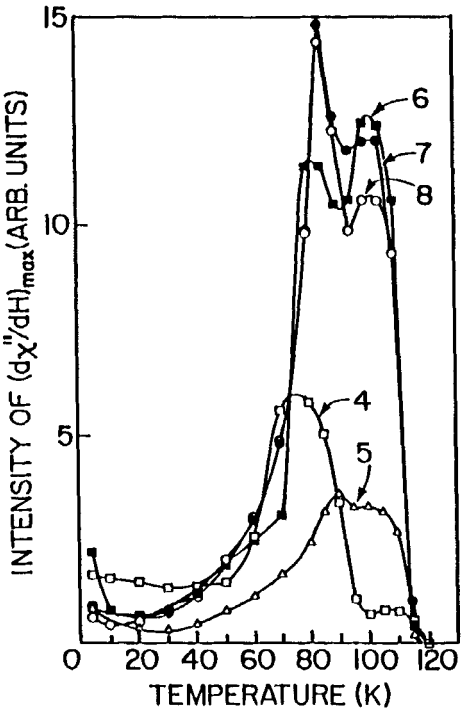

FIGURE 1 Intensity of the maxima of the low field microwave absorption versus temperature for 5 mg of samples 4 to 8.

high T_c phase from sample 4 to 8. A prolonged heat treatment at 1158 K seems to increase the amount of the high T_c phase as well as the amount of the low T_c phase. However, the high T_c phase seems optimized for sample 6.

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