Single phase $YBa_2Cu_3O_x$ high Tc ceramic superconductor (*)

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ABSTRACT

Single phase high temperature superconductor material in the Y-Ba-Cu-O chemical system was prepared and identified by powder X-ray methods, its superconductivity determined by magnetic and resistivity measurements. Samples prepared to directly obtain the stoichiometric $YBa_2Cu_3O_x$ phase showed zero resistance at 87K with the onset of the superconductive transition starting at 94K for $B = 0$.

INTRODUCTION

The electrical conductivity of materials has been the object of attention of researchers all over the world since it has a wide variation of at least 45 orders of magnitude when one goes from a good insulator to a superconductor. However, the search of high-temperature superconducting materials remained almost stagnant in recent years until the discovery of Bednorz and Muller (1) of superconductivity above 30K in a perovskite oxide composed of La, Ba and Cu. This event dramatically altered the perspetives of scientists that started a febrile investigation of other similar compounds. As a consequence another major breakthrough followed almost imediately. Wu (2), Chu (3), and coworkers reported a new material based on a composition of Y-Ba-Cu-O with Tc well above 90K. These results were also confirmed by other groups that identified the YBaCuO material as a mixture of several unidentified phases being only a small fraction of the sample responsible for the superconductivity. In this paper we report the sinthesis of single phase $Y_1Ba_2Cu_3O_x$ and the results of resistivity and magnetic measurements.

SAMPLE PREPARATION

Single phase $YBa_2Cu_3O_x$ compound was prepared by throughly mixing Y_2O_3, $BaCO_3$ and CuO in a 1:4:6 mass ratio. The mixture was then grinded and heated for 12 hours at 950°C under a constant flow of O_2 at atmospheric pressure. The mixture was then slowly cooled to room temperature at a ratio of 25°C per hour. After this procedure, a standard powder metalurgical method followed by grinding, pressing and sintering pellets of the compound at 950°C under O_2 for 12 hours and again slowly cooled to room T at a ratio of 25°C per hour. Powder X-ray diffraction pattern (figure 1) showed the presence of single phase $YBa_2Cu_3O_x$ compound identified according to published data (4). The amount of single phase $YBa_2Cu_3O_x$ present in the samples verified by X-ray diffraction pattern was found to critically depend on the preparation conditions and thermal cycling. Pellets directly pressed from the prepared mixture of oxides and then sintered at 950°C for 12 hours followed by a slow cooling procedure exibited approximately the same behaviour. Small deviations from the stoichiometric composition showed in the phase diagram Y-Ba line (figure 2) produced single phase $YBa_2Cu_3O_x$ in the presence of small amounts of precipitated Y_2O_3 or BaO identified by X-ray.

EXPERIMENTS AND RESULTS

Resistive measurements were carried out in a sample of dimensions 1.3 x 3 x 11 mm^3 by means of the standard four terminal method. The terminal leads were attached with silver paint. The current was set at 10mA. The sample was in contact with He gas in a temperature controlled cryostat. The temperature was measured with carbon and platinum thermometers. The data were taken at fixed field values up to 30 KOe and sweeping the temperature. Figure 3 shows several curves obtained at different applied fields. The data at $B = 0$ indicate the onset of the superconductive transition at $T = 94K$, the zero resistance state being attained at 87K. The results in the presence of an external magnetic field show clearly a departure of the behaviour observed for the $B = 0$ data. In this case the inicial rapid drop of the resistance R with decreasing temperature is followed by a much slower decrease in R. The zero resistance state being attained at temperatures considerably lower than the observed for $B = 0$. This behaviour is already observed in fields of few hundreds Gauss. In figure 4 the temperatures indicated in the insert of the figure 3 are plotted as a function of the applied field. Temperatures T_1 and T_2 correspond to the onset of the transition and to the temperature were the change in the resistive curve behaviour at $B \neq 0$ is located. The upper critical fields obtained in this way for the onset temperature a $\frac{dB}{dT} \sim 1$ to 1.5 T/K. This value is in the same order as a previously reported value for the $Y_{2-x}Ba_xCuO_{4-x}$ $(x = 4)$ system (5). The curve B_c vs T for the zero resistance temperatures rises with a positive curvature and at $B_c = 30$ kg, $\frac{dB_c}{dT}$ is smaller than the obtained for T_1.

Magnetic measurements were taken with a mutual inductance bridge in the same temperature controlled cryostat. In figure 5 we show the magnetic susceptibility data for the same sample. A diamagnetic signal is ob-

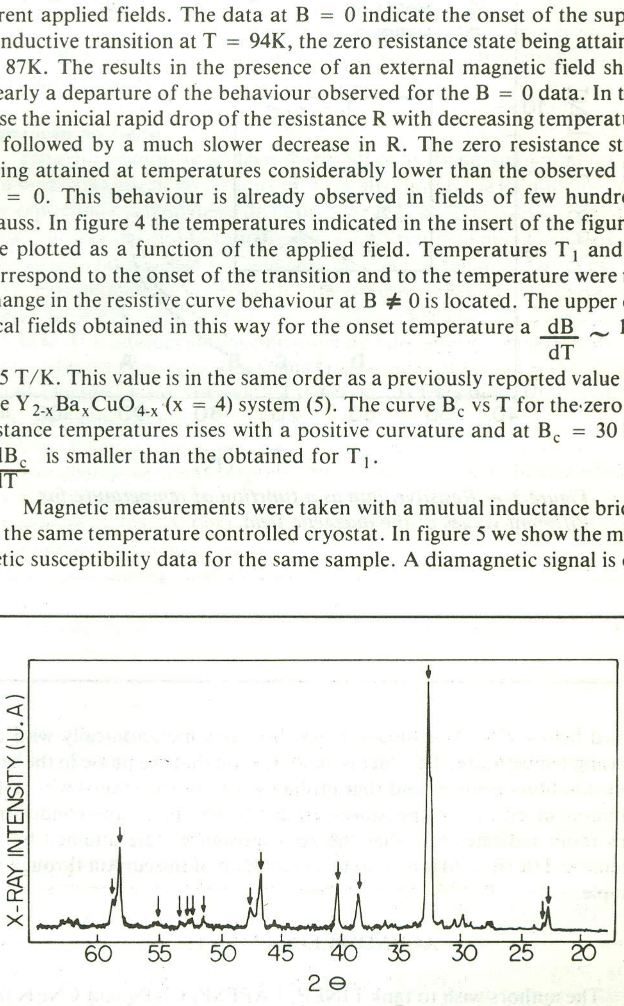

Figure 1 — Powder X-ray pattern showing single phase $YBa_2Cu_3O_x$.

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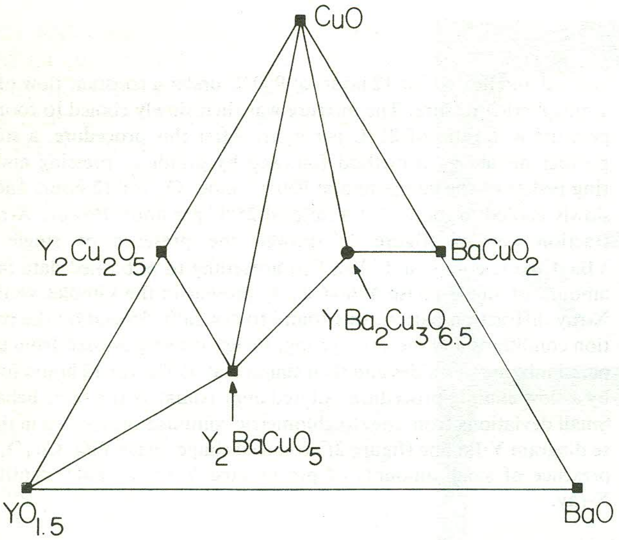

Figure 2 — Projection into the zero oxygen plane of the quaternary chemical system Y-Ba-Cu-O phase diagram.

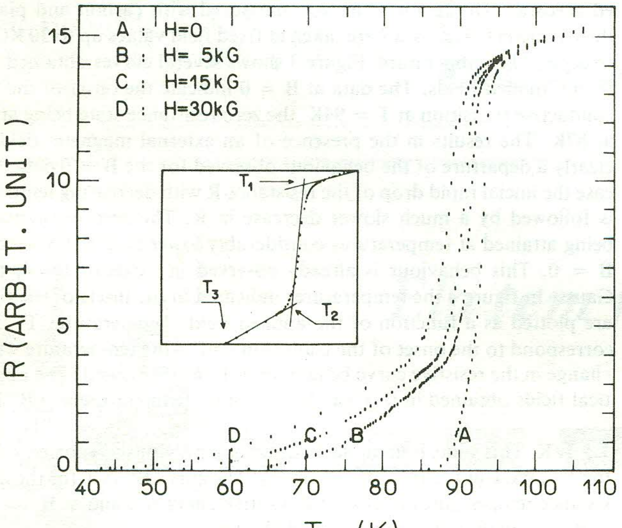

Figure 3 — Resistive data as a function of temperature for different values of the magnetic field.

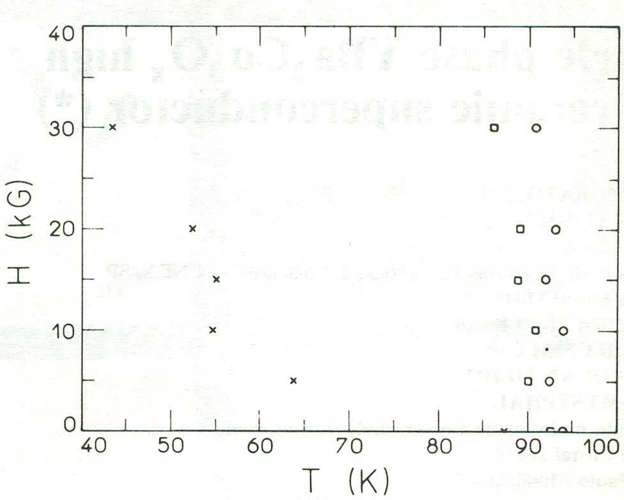

Figure 4 — Critical upper field as a function of temperature O-Ti; □-T_2; ⊡-T_3.

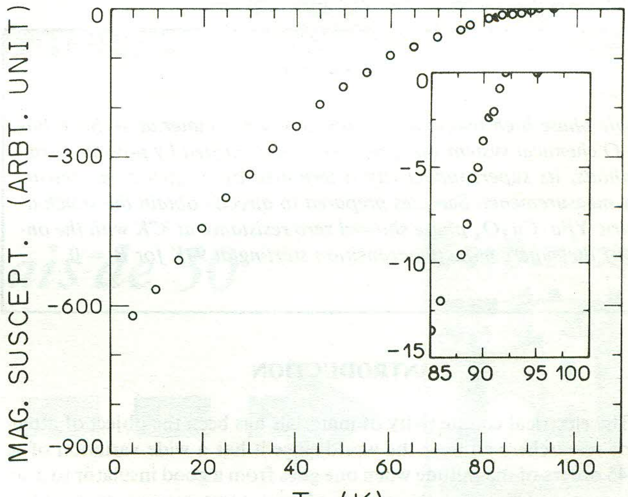

Figure 5 — Magnetic susceptibility as a function of temperature.

served below 92K. The susceptibility decreases monotonically with decreasing temperature. This fact is an indication that the phase in the sample is not homogeneous and that probably regions (or grains) with a distribution in critical temperatures gradually becomes superconducting. This result indicates also that the zero resistance state attained by the sample at 87K (B = 0) is due to the percolation of the current through the sample.

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