Superconductivity near 90 K in the Lu-Ba-Cu-O System

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Superconducting onsets near 90 K are observed in multiphase samples with nominal composition $Lu_{1.8}Ba_{0.2}CuO_4$. Results of electrical-resistance, mutual-inductance, and dc-magnetization measurements are reported. The magnitude of the superconducting transitions indicates that about 2% of the best sample is superconducting.

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Superconductivity in the metallic-oxide La-Ba-Cu-O system[1,2] was observed at temperatures of up to 30 K by Bednorz and Müller.[3] Subsequently the superconducting behavior was convincingly attributed to $La_{2-x}Ba_xCuO_4$, which has the K_2NiF_4-type structure.[4,5] It was shown soon thereafter that a variety of closely related materials become superconducting with transition temperatures up to 40 K.[6,7] Recently, Wu *et al.*[8] reported superconductivity in an unidentified phase in the Y-Ba-Cu-O system at 93 K. We report superconductivity near 90 K in samples with nominal composition $Lu_{1.8}Ba_{0.2}CuO_4$. Samples are multiphase with no observable K_2NiF_4-type structure. Superconductivity occurs in up to 2% of the sample volume.

Samples were prepared from dried Lu_2O_3, high-purity $BaCP_3$, and fully oxidized CuO. Powders were ground together in an agate mortar. The resulting powders were fired in air overnight at 1000 °C in Pt crucibles. This result was ground again, pelletized, and then fired at 1100 °C in air for 4–12 h in Pt crucibles. Additional samples fired solely at 1000 °C, and those fired at 1200 °C, show no signs of superconductivity. Samples with compositions differing by 10% to 50% in Ba content (from $Lu_{1.8}Ba_{0.2}CuO_4$) were not superconducting. Two of three samples at the $Lu_{1.8}Ba_{0.2}CuO_4$ composition prepared at Brookhaven National Laboratory show superconductivity above 70 K; one sample prepared at Ames also shows the high-temperature superconductivity. Similar samples containing Sr and Ca in place of Ba were also prepared, but showed no sign of superconductivity between 5 and 100 K as measured by ac mutual inductance.

Superconducting transitions were measured in three ways: low-frequency (200 Hz) mutual-inductance measurements, dc-magnetization measurements, and electrical-resistance measurements. Electrical-resistance

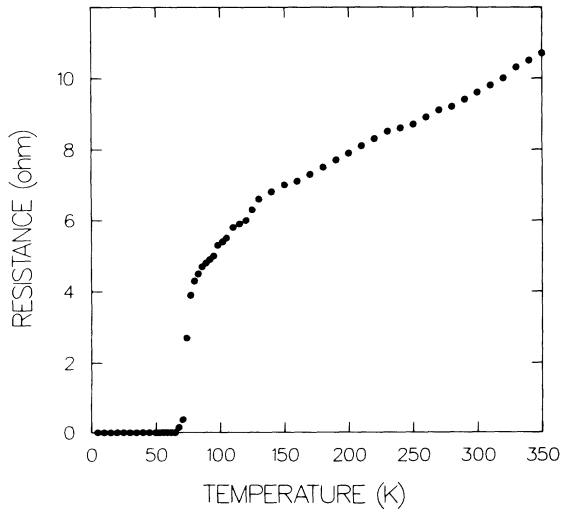

FIG. 1. Temperature dependence of electrical resistance of Lu-Ba-Cu-O.

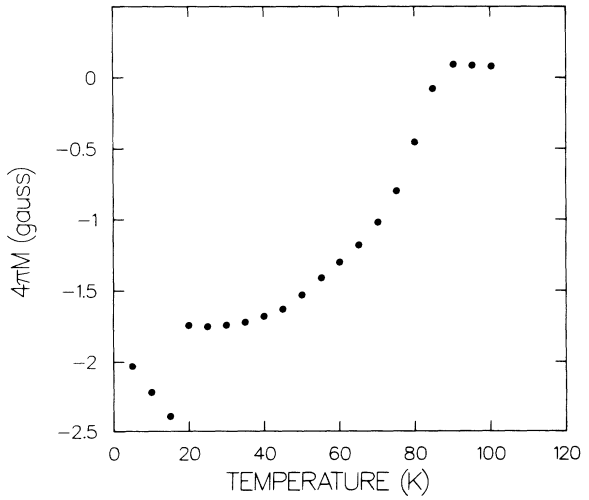

FIG. 2. Temperature dependence of dc magnetization of $Lu_{1.8}Ba_{0.02}CuO_4$ (taken at 100 Oe), on warming after cooling in zero applied field.

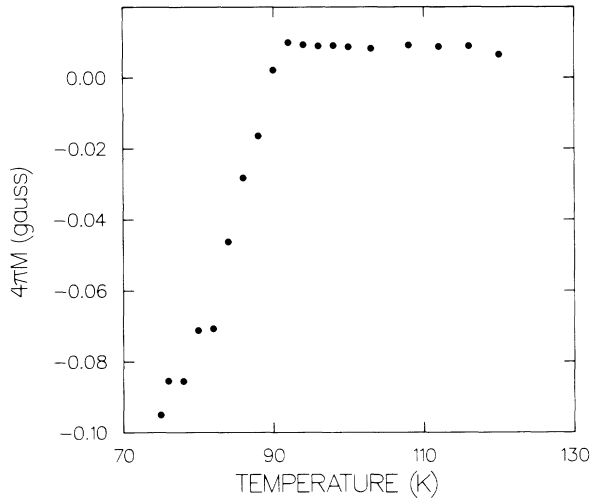

FIG. 3. dc magnetization of $La_{1.8}Ba_{0.2}CuO_4$ cooled in field of 10 Oe near the upper superconducting transition, illustrating the Meissner effect.

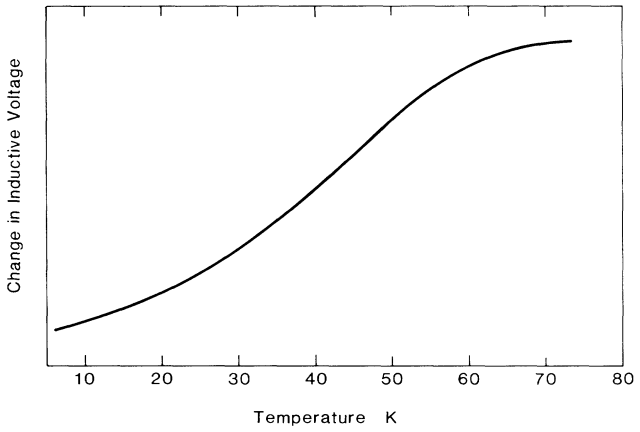

FIG. 4. Temperature dependence of mutual-inductance signal of $La_{1.8}Ba_{0.2}CuO_4$.

measurements were performed on an irregularly shaped sample about 5 mm long having a cross section of 2×2 mm^2. Pt wires were attached to the sintered piece with silver epoxy. Figure 1 shows the electrical resistance of a multiphase sample with nominal composition $Lu_{1.8}Ba_{0.2}CuO_4$. The superconducting transition is centered near 75 K (see Fig. 1). A deviation from the straight-line characteristic of a metal is visible at much higher temperatures. The drop in resistance from above to below T_c is at least five orders of magnitude—the limit imposed by the sensitivity of the technique.

Magnetization measurements provide additional evidence for superconductivity in this material. dc-magnetization data were taken on a different portion of the same sample with a commercial SQUID magnetometer in which the sample is moved slowly through the pickup coils. Temperature was measured with carbon-glass thermometers calibrated to an accuracy of 0.010 K. Figure 2 shows the results of the measurements taken on increasing temperature in a field of 100 Oe after first cooling in zero applied field. The superconducting transitions are superimposed on a paramagnetic signal, possibly caused by copper in one of the constituent phases. There appear to be two distinct superconducting transitions, one centered near 75 K and another at about 15 K. The onset of superconductivity is near 90 K. Figure 3 shows an additional magnetization measurement near the onset. Data taken during cooling in a field of 10 Oe show the expulsion of flux from the sample during the transition. A superconducting onset near 90 K is apparent.

Mutual-inductance measurements were made on samples having a mass of 0.01 to 0.02 g with use of a low-frequency ac field. The Ge resistance thermometer is calibrated below 100 K. Results on a different portion of the sample described above are shown in Fig. 4. The onset of superconductivity is above 80 K. Various pieces of two samples showed superconducting behavior, to a greater or lesser extent, but all with onsets at or above 80 K.

Preliminary attempts to identify the superconducting phase have been unsuccessful. X-ray diffraction reveals a significant amount of unreacted Lu_2O_3. Compositional analysis for metal-element content with energy-dispersive spectroscopy in a scanning electron microscope also shows the existence of large amounts of a phase containing Ba and Cu as well as a minor phase containing Lu, Ba, and Cu. In addition, photomicrographs taken from a polished surface of the specimen also indicated the presence of at least three phases.

The results of the three types of superconductivity measurements described above, taken together, demonstrate the existence of superconductivity in the Lu-Ba-Cu-O system. Much work remains to be done to identify the superconducting compound and optimize the superconducting properties.

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