SUPERCONDUCTIVITY IN Bi-Pb-Ca-Sr-Cu-O SYSTEM

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In this work, we will report the results on preparing the Bi-Pb-Ca-Sr-Cu-O superconductors. A phase diagram around the compositional ratio 2:2:2:3(=Bi:Ca:Sr:Cu) will be studied systematically. It is found that the superconducting properties of the samples are very sensitive to the annealing conditions. The results of annealing conditions, resistivity, magnetic susceptibility, and TGA data were applied to characterized the physical properties of samples in this system.

Since the discovery of a new high T_c system Bi-Ca-Sr-Cu-O¹, it is known that the high T_c phase (2223) usually appears with other low T_c phases and many efforts were focused on synthesizing the high T_c monophasic (2223). Several new processes have been successfully applied to produce a large fraction of the high T_c phase.²⁻⁴ One of these processes is the Pb-addition to Bi-Ca-Sr-Cu-O samples. In this paper, we will study systematically the correlations of Pb addition, composition change, and the annealing condition in the synthesis of the 2223 phase.

Samples with a nominal composition were prepared from an appropriate powder mixture of Bi₂O₃, CaCO₃, SrCO₃, PbO and CuO. The powder was preheated at 820°C for 1 day, then pressed into pellet and annealed at 850°C for 3 days either in air or in oxygen atmosphere. All samples were furnace cooled. Sample A' was prepared in a different way which was preheated at 880°C for 20 min and then annealed at 860°C for 10 hrs in air.

Figure 1 shows the temperature dependence of resistivity for samples reported here. The electrical resistivity was measured by a four-probe method with Ag-paint as electrical contacts. The compositions, annealing environment and T_c 's are summarized in Table I. From the T_c results for samples A, B, and D', we

TABLE I

Compositions, annealing environments, and T_c 's for samples A, B, C, D, A', B', C', D', E', and F'.

Sample	Composition (Bi:Pb:Ca:Sr:Cu)		Annealing Environment	T_c (50%) (K)
A	2: 0.3:2.2: 2:3.2		air	111
B	2: 0.3:2.2: 2:3.2		oxygen	60
C	1.84:0.34: 2:2.1:3.1		oxygen	83
D	1.84:0.34: 2:2.1:3.1		air	Semicond.
A'	2: 0: 2: 2: 3		air	75
B'	2: 0: 2: 2: 3		oxygen	63
C'	2: 0: 2: 2: 3		air	68
D'	2: 0.3: 2: 2: 3		air	108
E'	1.8: 0.4: 2: 2: 3		air	3 phases
F'	1.8: 0.4: 2: 2: 3		oxygen	100

observed that 0.3 Pb addition to a nominal composition around 2223 and an air annealing environment have the highest T_c (~110K). As one increases the Pb doping to 0.34(samples C) and decreases the Bi-content, the superconducting properties degrade. It is worth noting that the

FIGURE 1
Temperature dependence of resistivity for samples A, B, C, D, A', B', C', D', E', and F'. The nominal compositions, annealing environments and T_c 's are shown in Table I.

air annealed sample (sample D) with 0.34 Pb doping behaved as a semiconductor rather than a superconductor. If we continue to increase the Pb-content up to 0.4, the oxygen annealing turns out a much better way to obtain a higher T_c superconductor than the air annealing. In our experiments, it seems to us that the more Pb added, the more oxygen needed in order to achieve superconductivity. From sample A', B', and C', no 2223 phase was found from pure 2223 nominal composition.

Sample A ($T_c=111K$) was chosen to do the magnetic susceptibility measurement by means of a SQUID magnetometer (Quantum Design). The results were shown in Figure 2. As can be seen, although the powder X-ray diffraction pattern and resistivity data show almost a single phase for sample A, the high T_c Meissner signal is still much smaller than it has to be.

Due to the fascinating electrical and thermal properties for both Bi-Ca-Sr-Cu-O system and their thin films⁵⁻⁷, samples A and B were taken to do TGA measurements which were carried out on Du Pont 9900 thermal system with heating and cooling rates of about 5°C/min and the results were shown in Figure 3. Sample A shows a sharp drop in weight loss at $T=265^\circ C$ in the heating while sample B reveals almost

FIGURE 2
Magnetization as a functional of temperature for sample A in a field of 20 G.

FIGURE 3
TGA data run in O_2 atmosphere for samples A and B.

unchanged temperature dependence curve, which is very different from that of Bi-Ca-Sr-Cu-O samples.⁵⁻⁷ The TGA data for sample A is a typical one and the sharp weight loss at $T=265^\circ C$ has been attributed to a thermodynamic decomposition. The TGA data of sample B suggest that sample B is a more stable 60K phase.

In summary, we found that superconductivity of Bi-Pb-Ca-Sr-Cu-O sample is determined by both annealing environments and the Pb content. O_2 annealing tends to degrade the superconductivity as Pb-0.3, while it improves the superconducting properties of samples with Pb-0.4. Although oxygen content is less essential in the superconductivity of Bi-Pb-Ca-Sr-Cu-O samples compared with that of YBaCuO system, it may not be true for Pb doping case.

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