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Study of the superconductivity of the 132 K Sb-doped Bi-system oxide superconductor

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Abstract. According to powder x-ray diffraction analyses and electron diffraction for the 132 K Sb-doped Bi-system oxide superconductor, it can be found that a new phase exists in this system; the new phase belongs to the monoclinic system with cell dimensions $a = 22.10 \text{ \AA}$, $b = 5.90 \text{ \AA}$, $c = 19.91 \text{ \AA}$ and $\beta = 99.6^\circ$ and is not superconducting itself. Furthermore, there exists intergrowth between the monoclinic phase and 2:2:2:3 (= Bi: Sr: Ca: Cu) phase, and for this reason crystals of the 2:2:2:3 phase are distorted. The superconductivity at 132 K may be caused by the distorted 2:2:2:3 phase. After thermal cycles, this kind of intergrowth phenomenon disappears so that only superconductivity at 110 K occurs as usual. All this can explain why the 132 K phase is not stable. In addition, how and why the measurement current influences T_c will also be discussed in this paper.

1. Introduction

After Maeda *et al* [1] discovered that there are two superconducting transitions in the Bi–Sr–Ca–Cu–O system, it was reported by Mizunyw *et al* [2] that the higher- T_c phase which undergoes transition at 110 K has the ideal chemical formula $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$, and that this phase can be made stable by doping with Pb. Recently we found the zero-resistance phenomenon at 132 K for a $\text{Bi}_{1.9-x}\text{Pb}_x\text{Sb}_{0.1}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ sample ($x = 0.3, 0.4$) [3], but this sample was not stable and its transition temperature (T_c) decreased apparently after several thermal cycles [4]. Luo *et al* [5], Sastry *et al* [6] and Chandrchood *et al* [7], from their experimental results, also indicated that this higher- T_c phase is not stable. Meanwhile, we also discovered that T_c has a relation to the measurement current. The values of the zero-resistance temperature T_{c0} equal 130 K and 110 K, respectively, when the measurement currents are $10 \mu\text{A}$ and $100 \mu\text{A}$. This suggests that the crystal which undergoes transition at 132 K occurs only on quite a small region of the system. In this paper, the x-ray powder diffraction patterns, the electron diffraction patterns and the AC susceptibility are studied for the 132 K superconductor before and after thermal cycles. All the results obtained from these experiments can explain the mechanism responsible for the superconductivity at 132 K and why this system is not stable during thermal cycles and is stable at 110 K subsequently.

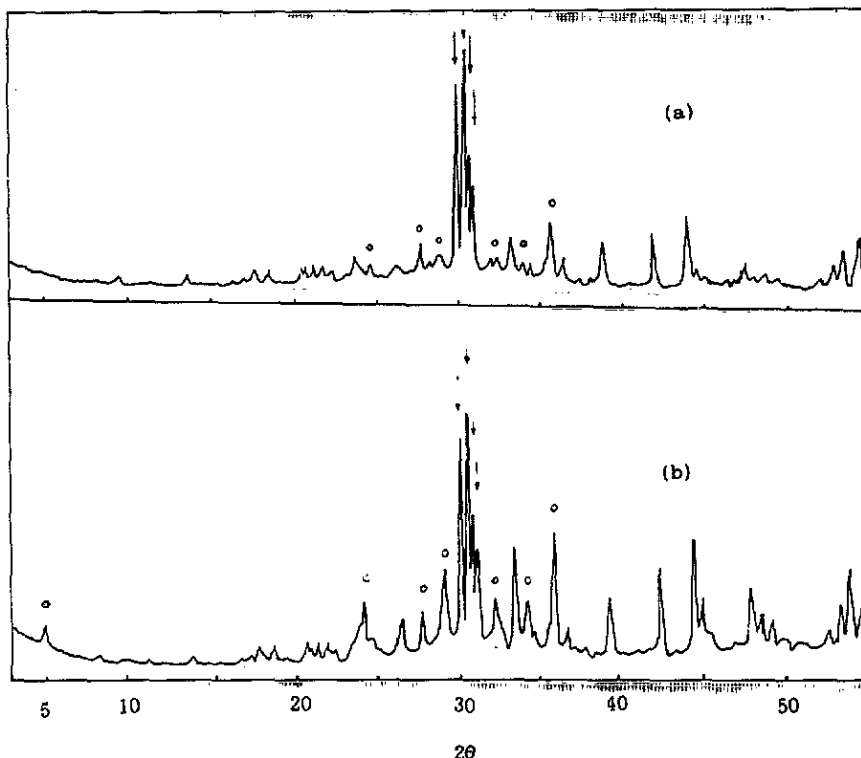


Figure 1. X-ray diffraction patterns of $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ for (a) an as-prepared sample and (b) a sample which had experienced a room temperature thermal cycle: O, diffraction peaks of the 2:2:2:3 phase; ↓, diffraction peaks of the monoclinic phase.

2. Experiment

2.1. Sample preparation

A sample with the nominal composition $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ was prepared by solid state reaction. Powders of Bi_2O_3 , PbO , Sb_2O_5 , SrCO_3 , CaCO_3 and CuO were mixed, ground and then pre-heated at 820°C for 12 h in air. After the pellets had been reground and pressed into disc-shaped pellets, they were heated at 865°C for 60 h and finally quenched in air.

2.2. Behaviour of the initial sample

Figure 1(a) is the x-ray powder diffraction pattern for the $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ sample. It is very obvious that there is no typical peak of 2:2:2:3 (= Bi: Sr: Ca: Cu) phase at $2\theta = 5^\circ$ and the other peaks of the 2:2:2:3 phase are weaker. The main diffraction peaks in this figure suggests that a new phase exists in this system. The TEM analyses indicate that the new phase belongs to the monoclinic system. Figure 2 gives the fractured-section SEM image for this sample; it shows that two series of crystal exists in this system, namely larger thick grains (labelled 1) belonging to the monoclinic crystal and the smaller flaky grains (labelled 2) belonging to the 2:2:2:3 phase crystal. Figure

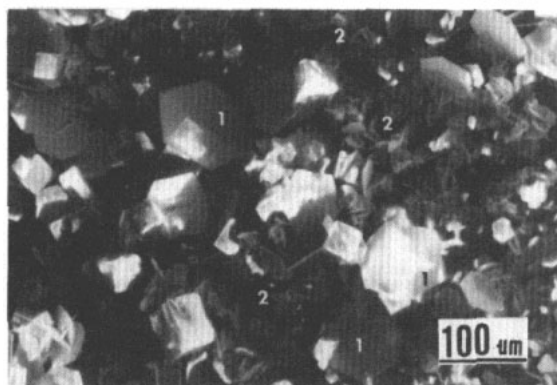


Figure 2. SEM photograph of the $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ sample: 1, grains of the monoclinic phase; 2, grains of the 2:2:2:3.

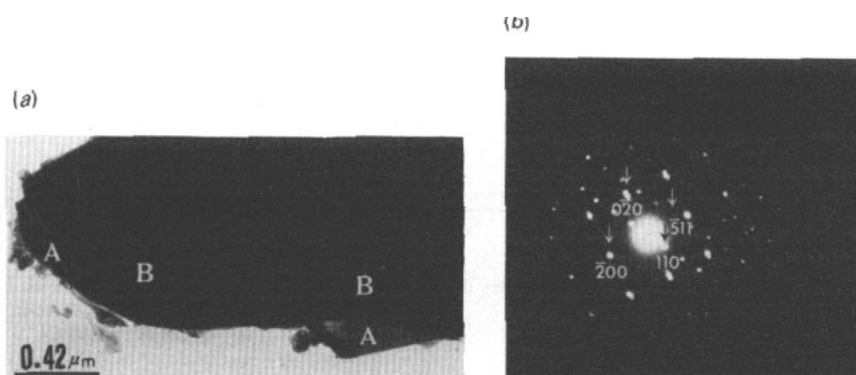


Figure 3. TEM pictures of the $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ sample. (a) Thick monoclinic grains with the 2:2:2:3 phase sheet growing on it: A, crystal of the 2:2:2:3 phase; B, crystal of the monoclinic phase. (b) Electron diffraction pattern of the sample. The diffraction spots indexed by $[200]$ and $[020]$ show the tetragonal lattice of the 2:2:2:3 phase. The diffraction spots indexed by $[110]$ and $[\bar{5}11]$ show the monoclinic lattice.

3 is the TEM picture of this sample. Figure 3(a) shows thick monoclinic grains (labelled B) with a 2:2:2:3 sheet (labelled A) growing on it. Figure 3(b) shows the electron diffraction pattern of this sample. It is clear that two kinds of diffraction spot appear in this pattern: one (indexed by $[200]$ and $[020]$) corresponding to tetragonal lattice of 2:2:2:3 phase in the Bi system with $a = 5.4 \text{ \AA}$ and $b = 5.4 \text{ \AA}$ and a distorted c which is different in the various intergrowth regions and larger than 36 \AA , and the other (indexed by $[110]$ and $[\bar{5}11]$) corresponding to the monoclinic system with $a = 22.10 \text{ \AA}$, $b = 5.90 \text{ \AA}$, $c = 19.90 \text{ \AA}$ and $\beta = 99.6^\circ$.

Figure 4 shows the AC susceptibility data. Curves (1), (2) and (3) are the experimental results for low-temperature thermal cycles (77–200 K) in vacuum; from these curves, the superconductivity can be observed at 141 K for this sample. However, for the higher-temperature thermal cycle (77 K–room temperature), the transition at 141 K disappears, as shown in curve (4), and the higher transition temperature decreases to 110 K. Further AC susceptibility measurements show that the sample is stable at 110 K after the room temperature cycle.

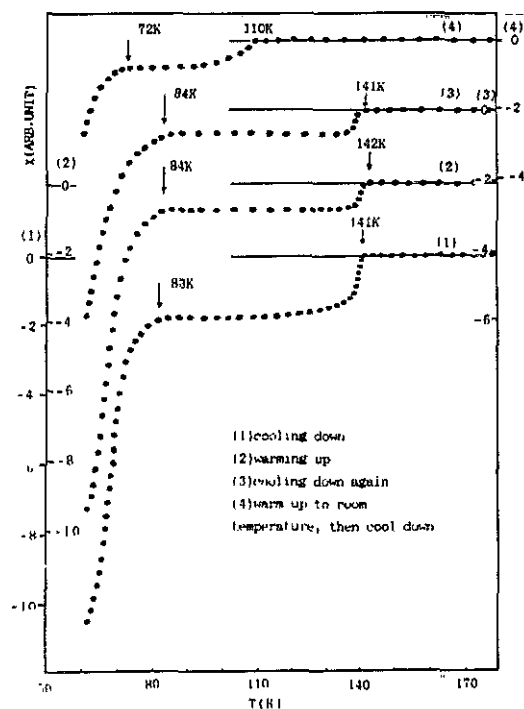


Figure 4. AC susceptibility of the Bi-Sr-Ca-Cu-O system doped with Pb and Sb.

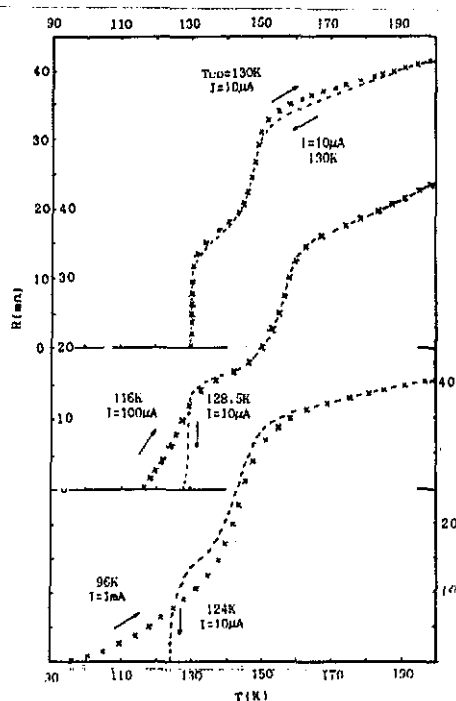


Figure 5. The different values of T_{c0} after low-temperature cycling at different currents in Bi-Pb-Sb-Sr-Ca-Cu-O samples.

2.3. Behaviour of the sample after thermal cycles

An x-ray powder diffraction pattern of a $\text{Bi}_{1.6}\text{Pb}_{0.1}\text{Sb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ sample which has experienced the room temperature thermal cycle is shown in figure 1(b). The typical diffraction peak of the 2:2:2:3 phase at $2\theta = 5^\circ$ appears clearly, and the intensity of other peaks of 2:2:2:3 phase increase, while hardly any change in the intensity and position of the monoclinic phase is observed. Further TEM observations for this sample show that the dimensions of the monoclinic crystal become small and the number of lamellae of the 2:2:2:3 phase obviously increase.

3. Discussion

3.1. The formation and stability of 132 K phase

On the basis of above experimental results and analyses, we can see that intergrowth exists between the crystal of the monoclinic phase and that of the 2:2:2:3 phase. This kind of intergrowth results in distortion for the 2:2:2:3 phase crystal, and the distorted 2:2:2:3 phase is essentially responsible for the superconductivity at 132 K. Since the intergrowth part is quite thin, its T_c is sensitive to the measurement current, as shown in figure 5, and its diamagnetism only appears in the AC susceptibility but cannot be observed in the DC Meissner effect. After the room temperature thermal cycle, the

monoclinic crystal breaks and becomes small grains and the 2:2:2:3 crystal separates from the monoclinic crystal; therefore the distortion of the 2:2:2:3 phase disappears and the superconductivity at 110 K occurs again. All this explains why the system is not stable.

3.2. Monoclinic phase

In order to determine whether the monoclinic crystal is superconducting, we prepared a pure phase specimen with the composition of the monoclinic phase, and the measurement of superconductivity for this sample indicates that the monoclinic crystal is not superconducting.

References

- [1] Maeda M, Tanaka Y, Fukutami M and Asano T 1988 *Japan. J. Appl. Phys. Lett.* **27** L209
- [2] Mizunoy M, Endo H, Tsuchiya J, Kijima N, Smiyama A and Dguri Y 1988 *Japan. Appl. Phys.* **27** L1225
- [3] Liu Hongbao, Cao Liezhao, Zhou Ling, Mao Zhiqiang, Li Xiaoxian, Yu Zhidong, Xue Bai, Mao Xianglei, Zhou Guien, Ruan Yaozhong, Chen Zhaojia and Zhang Yuheng 1989 *Solid State Commun.* **69** 867
- [4] Yang Li, Fan Chenggao, Li Fanghua, Cheng Tingzhu, Zhou Guien, Cheng Zhaojia, Mao Zhiqiang and Zhang Yuheng 1990 *Mod. Phys. Lett. B* **4** 363-67
- [5] Luo J S, Michel D and Chevalier J P 1989 *Appl. Phys. Lett.* **55** 1448
- [6] Sastry P V P S S, Yakhoni J and Iyer R M 1989 *Solid State Commun.* **71** 1935
- [7] Chandrachood M R, Mulla I S and Sinha A P B 1989 *Appl. Phys. Lett.* **55** 1742