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### Effect of Annealing on Superconductivity in Bi-Pb-Sr-Ca-Cu-O System

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## EFFECT OF ANNEALING ON SUPERCONDUCTIVITY IN Bi-Pb-Sr-Ca-Cu-O SYSTEM

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**Abstract** The effect of annealing on the superconductivity in the Bi-Pb-Sr-Ca-Cu-O system was investigated. The samples annealed at temperatures ranging from 600°C to 825°C exhibited lower zero-resistance temperatures than that of the as-sintered sample and the tailing phenomenon in the R-T curves. This phenomenon was pronounced particularly for the sample annealed at 700°C. From the ac susceptibility measurement, the weak-coupling was confirmed to exist between the high- $T_c$  grains in the annealed samples. Therefore, it was elucidated that the tailing phenomenon was caused by the weak-coupling generated between the high- $T_c$  grains during annealing.

## INTRODUCTION

Since the Ba-Y-Cu-O system was found to exhibit the superconducting transition above liquid nitrogen temperature,<sup>1</sup> several superconducting oxides with critical temperature above 100K were discovered.<sup>2,3</sup> The properties of ceramic superconductors are closely related to various preparatory conditions.<sup>4-7</sup> In our previous paper, it was reported that the  $T_{c,zero}$  values of Bi-Pb-Sr-Ca-Cu-O superconductors were extremely dependent on the cooling rate.<sup>8</sup> Especially, the sample obtained at a very slow cooling rate ( $<10^\circ\text{C/h}$ ) showed  $T_{c,zero}$  lower than 85K. This result suggested the existence of a certain temperature range in the cooling process where  $T_{c,zero}$  was decreased. In the present work, we investigated the effect of annealing on the superconductivity in a Bi-Pb-Sr-Ca-Cu-O system by the ac susceptibility measurement.

## EXPERIMENTAL

The samples were prepared by a solid-state reaction method. Commercial powders of  $\text{Bi}_2\text{O}_3$  (purity 99.99%),  $\text{PbO}$  (purity 99.99%),  $\text{SrCO}_3$  (purity 99.9%),  $\text{CaCO}_3$  (purity 99.9%) and  $\text{CuO}$  (purity 99.9%) were mixed in the molar ratio of  $\text{Bi:Pb:Sr:Ca:Cu}=1.6:0.4:1.6:2.0:4.0$ . The sintering was carried out at  $845^\circ\text{C}$  in air and a cooling rate of  $100^\circ\text{C/h}$  was adopted. Then, some of the samples were annealed at  $300^\circ\text{C} \sim 845^\circ\text{C}$  for 24h in air.

The resistivity of the sample was measured in a closed-cycle helium refrigerator by a conventional four-probe resistive method, changing the current direction alternatively. The electrodes were prepared by evaporating gold films and the lead-in wires were attached to the gold electrodes using silver paste. The temperature was measured by a Pt-Co resistive thermometer. The ac susceptibility measurement was performed using an impedance analyzer (YHP-4192A) at 1kHz.

## RESULTS AND DISCUSSION

Figure 1 shows the zero-resistance temperature ( $T_{c,\text{zero}}$ ) as a function of the annealing temperature. The  $T_{c,\text{zero}}$  values of the samples annealed at  $600^\circ\text{C}$  to  $825^\circ\text{C}$  were lower than that of the as-sintered sample. Moreover, the  $T_{c,\text{zero}}$  values of all samples were reduced when the measuring current density was increased. The maximum decrease in  $T_{c,\text{zero}}$  was observed in the sample annealed at  $700^\circ\text{C}$ . From the above results, it could be considered that the decrease in  $T_{c,\text{zero}}$  of the sample obtained at a very slow cooling rate, as shown in the previous paper,<sup>8</sup> was caused by holding the samples at temperatures ranging from  $600^\circ\text{C}$  to  $825^\circ\text{C}$  for a long time.

Figure 2 shows the temperature dependences of the resistivity for the as-sintered sample and the sample annealed at  $700^\circ\text{C}$ . The normal-state resistivity of the sample annealed at  $700^\circ\text{C}$  was much higher than that of the as-sintered sample. In addition, the tailing phenomenon<sup>9</sup> was observed in the R-T curve for the sample annealed at  $700^\circ\text{C}$  though the as-sintered sample showed a sharp superconducting transition above 100K. This phenomenon was also observed for all samples annealed at temperatures ranging from  $600^\circ\text{C}$  to  $825^\circ\text{C}$ . As the

cause of the tailing phenomenon observed in Fig.2(b), two possibilities can be thought as follows.

(1): A low  $T_c$  phase generated between the high- $T_c$  grains during annealing.

(2): A weak-coupling generated between the high- $T_c$  grains.

To elucidate which case occurred, the ac susceptibility measurement was performed. Figure 3 shows the temperature dependence of the ac susceptibility for the powder sample obtained by crushing after annealing at 700°C. Only one step was observed around 112K. If the low- $T_c$  phase had been contained in the annealed samples, two steps

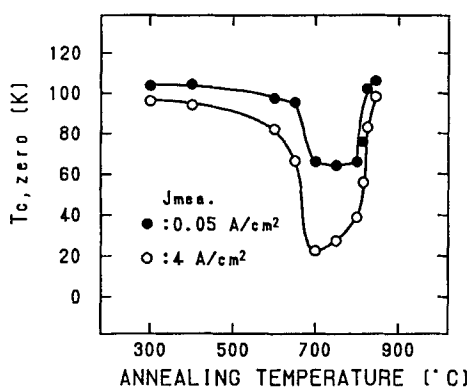


FIGURE 1 Zero-resistance temperature ( $T_{c,zero}$ ) as a function of the annealing temperature. The measuring current densities are  $0.05 \text{ A/cm}^2$  (●) and  $4 \text{ A/cm}^2$  (○).

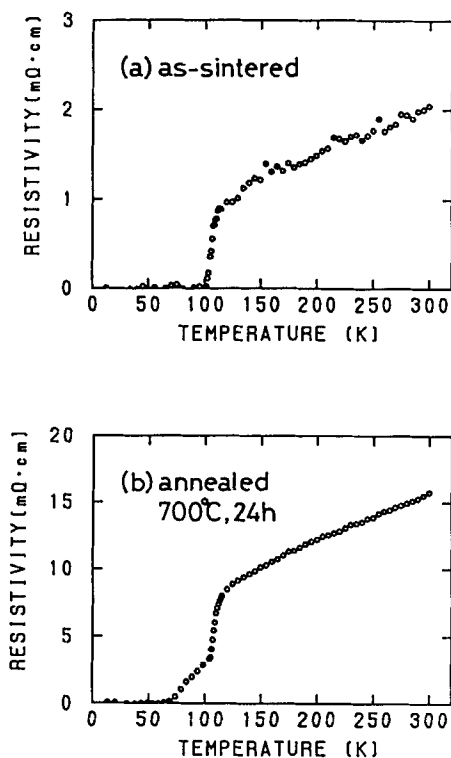


FIGURE 2 Temperature dependences of the resistivity for the as-sintered sample(a) and the sample annealed at 700°C(b). The measuring current density is  $0.05 \text{ A/cm}^2$ .

would have been confirmed in the temperature dependence of the ac susceptibility. This result indicates that the annealed sample does not contain the low- $T_C$  phase. In general, if the sample contains a weak-coupling, the temperature dependence of the ac susceptibility is strongly dependent on the ac field strength.<sup>10</sup> Accordingly, in order to verify the existence of the weak-coupling, the temperature dependences of the ac susceptibility for the bulk samples were investigated as a function of the ac field strength. The result is shown in Fig.4. The behavior of the ac susceptibility for the as-sintered sample rarely depended on the ac field strength. On the

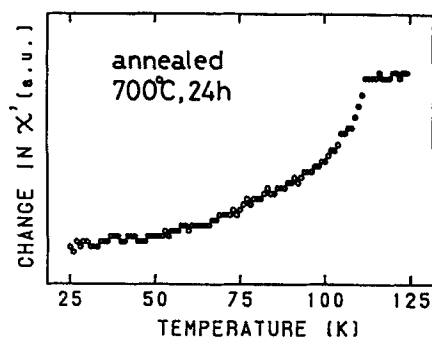


FIGURE 3 Temperature dependence of the ac susceptibility for the powder sample obtained by crushing after annealing at 700°C.

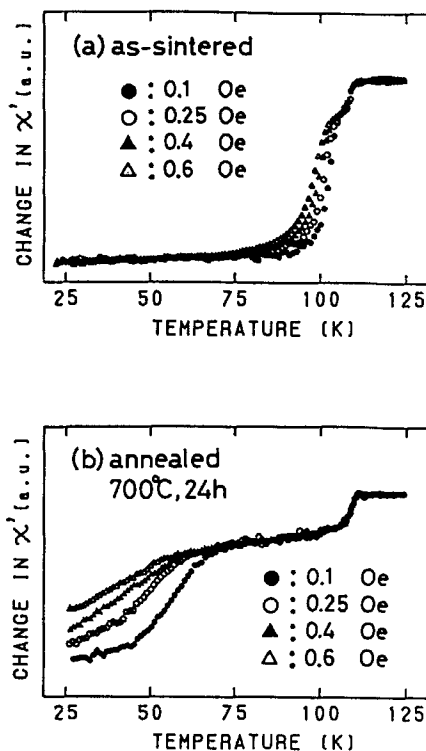


FIGURE 4 Temperature dependences of the ac susceptibility for the bulk samples as a function of the ac field strength. (a): The as-sintered sample. (b): The sample annealed at 700°C.

other hand, the aspect of the change in the ac susceptibility for the sample annealed at 700°C extremely differed from that for the as-sintered sample. The strong dependence on the ac field strength was observed below 80K, which is the evidence of the existence of the weak-coupling structure. These results indicate that the weak-coupling generates between the high- $T_c$  grains during annealing. Therefore, it was ascertained that the tailing phenomenon in the R-T curves was caused by the weak-coupling generated during annealing.

### CONCLUSION

We have investigated the effect of annealing on the superconductivity in the Bi-Pb-Sr-Ca-Cu-O system. The  $T_{c,zero}$  of the samples annealed at temperatures ranging from 600°C to 825°C were lower than that of the as-sintered sample and the tailing phenomenon was observed in the R-T curves. From the ac susceptibility measurement, the weak-coupling was confirmed to exist between the high- $T_c$  grains in the annealed samples. Consequently, it became clear that the tailing phenomenon was attributed to the existence of the weak-coupling.

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