Internal friction and Young's modulus in the Bi(pb)-Sr-Ca-Cu-0 superconductor

Du Jiaju, Sun Yuping, Jiang Jianyi, Zeng Fanchun, and Yin Huaqing Institute of Solid State Physics, Academia Sinica, Hefei, People's Republic of China (Received 30 May 1989; revised manuscript received 27 October 1989)

Measurements of internal friction and resonant frequency in the kHz range have been conducted in Bi(Pb)-Sr-Ca-Cu-O superconductors between 80 and 300 K. The specimens are of pure low- T_c 2:2:1:2 phase, pure high- T_c 2:2:2:3 phase, or binary phase. The experimental results show that the internal-friction spectra of Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O, and Bi(Pb)-Sr-Ca-Cu-0 superconductors are obviously different and are affected by the preparation conditions of the specimens. This may be related to the difference of the oxygen deficiency and the ions with mixed valence in the specimens. There should not always be an internal-friction peak near T_c associated with the superconducting transition.

The measurement of internal friction is a nondestructive and sensitive tool for studying defects and microscopic processes in solids. Since the discovery of the La-Ba-Cu-0 and Y-Ba-Cu-0 superconductors, much work has been carried out with this technique;¹⁻¹⁰ however, as for the experimental results and theoretical explanations about the internal-friction peaks, there are different opinions. The most important differences are whether there is an internal-friction peak appearing near T_c in spite of the different measuring frequencies used, and whether this peak is associated with the change of the microstructure and directly related to the superconducti: transition. In our previous papers,^{1,2,6} three internal friction peaks were reported at about 250, 110, and 88 K in YBa₂Cu₃O₇₋₈ by acoustic frequency internal-friction measurements in the kHz range. The peak at about 250 K may be associated with the adjustment of oxygen deficiency and the other two peaks with the activated relaxation processes. The 88-K peak appeared in both the superconductor with orthorhombic structure and the semiconductor with tetragonal structure, while the 110-K peak was only observed in the superconductor with orthorhombic structure. The 88-K peak was interpreted as the jump of electrons in the Cu(2)-O plane, and the 110-K peak was attributed to that in the Cu(1)-O plane.⁶ The structure of the Bi(Pb)-Sr-Ca-Cu-0 superconductor is different from that of the Y-Ba-Cu-0 superconductor, and it will be interesting to find out whether there is a difference in the internal-friction spectra between these two systems. By studying the internal friction and its relation to the superconductivity of Bi(Pb)-Sr-Ca-Cu-0 superconductor, we can get some information for understanding the origin and mechanism of the internalfriction peaks and the superconductivity of these new superconductors. In this paper we report some results of this study.

INTRODUCTION EXPERIMENTAL TECHNIQUES

The specimens used in this study were prepared from the mixture of highly pure $Bi₂O₃$, PbO, CaO, and CuO powders by a standard ceramic sintering process. The dimension of which is about $80 \times 5 \times 4$ mm³.

The structure and the lattice parameters of the specimens were examined by a Phillips PW1700 automatic powder x-ray diffractometer. The resistance (R) was measured by the standard four-probe method. The internal friction Q^{-1} and the Young's modulus E were deter mined with the method of the free decay of a resonant bar in the kHz frequency f range

The internal friction Q^{-1} is given by

$$
Q^{-1} = (1/n)\ln(A_0/A_n) ,
$$

where n is the number of the vibration cycles, while its amplitude attenuates from A_0 to A_n . The Young's modulus (E) can be derived from

$$
E = (4\pi^2 spl^4/m^4I)f^2,
$$

where f is the resonant frequency, p is the density of the sample, l is the length of the sample, I is the moment of inertia, s is the cross-sectional area of the sample, and m is the constant 4.730 for the vibration in the fundamental mode.

EXPERIMENTAL RESULTS AND DISCUSSION

The temperature dependence of Q^{-1} , f^2 , and R in the Bi-Sr-Ca-Cu-0 specimens prepared at different sintering temperature is shown in Fig. 1. An internal-friction peak at about 170 K appears in all the specimens, although

FIG. 1. The temperature dependence of Q^{-1} , f^2 , and R in the BiSrCaCu₂O_y specimens sintered at (a) 865° C/20 h, (b) 870'C/46 h, and (c) 875'C/46 h.

there is a little difference among the peaks. However, the internal-friction spectrum of the Bi-Sr-Ca-Cu-0 specimens is obviously different from that of the $YBa₂Cu₃O_{7-\delta}$ specimens (see Fig. 2). In order to study the origin of the peak, we made the specimens with the same composition and synthesis temperature but different

FIG. 3. Effect of the cooling rate on the internal friction of the Bi₂Sr₂CaCu₂O_y specimens. \circ : quenched in LN₂, $f \approx 1400$ Hz, T_{c0} =89 K. \times : quenched in air, $f \approx 1500$ Hz, T_{c0} =83 K. • cooled in furance, $f \approx 1530$ Hz, $T_{c0} = 75$ K.

cooling rates. They are of a single low- T_c 2:2:1:2 phase with different T_{c0} . The height of the Q^{-1} peak is different as shown in Fig. 3. The faster the cooling rate of the specimens, the higher the T_{c0} (89, 83, and 75 K for the liquid N_2 quenched, the air cooled, and the furance cooled specimens, respectively), the higher the Q^{-1} peak and the lower the peak temperature. At the same time, we measured the lattice parameters and compared the oxygen content of the specimens of different cooling rates by thermogravimetric analysis (TGA). As reported in by thermogravimetric analysis (TGA) . As reported in our other paper,¹¹ the oxygen content and the lattice parameters increase with the increasing cooling rate of the specimens. This shows that the Q^{-1} peak is associated with the oxygen content.

To study the characters of the Q^{-1} peak, we have mea sured the Q^{-1} using different frequencies. The change of the Q^{-1} with the measuring frequency is shown in Fig. 4. The peak shifts to higher temperature by raising the measuring frequency. It shows that the peak is of the relaxation characteristic. The activation energy obtained is about 0.27 eV. Viewing the fact that the activation energy is very small and comparing the preceding experimental results with those obtained by us in the $YBa₂Cu₃O₂$ superconductor, 1,2,6 we can suggest that the relaxatio

FIG. 2. The temperature dependence of Q^{-1} , f^2 , and R in the YBa₂Cu₃O₇₋₈ specimens (Ref. 2).

FIG. 4. The change of internal friction with measuring frequency in the same specimen. 1: $f \approx 0.9$ Hz and 2: $f \approx 2.5$ Hz.

FIG. 5. The x-ray diffraction patterns of specimen (c).

process causing this peak is electronic relaxation. In a crystal containing ions in two valence states or holes and electrons trapped at the impurity or vacancy, the Q^{-1} peak caused by the jump of the electrons among two or more equivalent sites can be observed.¹² The high- T_c superconductors are such crystals. There are ions in two valence states and oxygen deficiency, so the \mathcal{Q}^{-1} peak associated with the jump of the electrons is observed. Because of the complexity of the structure of the compounds, the details of the relaxation process of this Q^{-1} peak are not clear yet.

However, from the preceding experimental results, it can be concluded that the Q^{-1} peak is not caused by the change of the microscopic structure in the specimens and is not directly related to superconducting transition. The arguments are as follows. First, the peak is a relaxation peak. Second, for the specimens with different temperature dependence of resistance, there always exists a peak

FIG. 6. The internal friction of the specimens with different phase compositions. (a): single low- T_c 2:2:1:2 phase ($f \approx 1.7$ Hz). (b): both 2:2:1:2 phase and 2:2:2:3 phase ($f \approx 1.7$ Hz). (c): single high- T_c 2:2:2:3 phase ($f \approx 1.2$ Hz).

FIG. 7. The internal-friction spectra of the specimens with binary-phase sintered under low oxygen pressure $(\frac{1}{17}$ atm) $f \approx 1340 \text{ Hz}, T_c = 107 \text{ K}.$

at about 170 K (see Fig. 1) for both superconducting and semiconducting specimens in spite of some difference. Third, by raising the cooling rate of the specimens, its T_{c0} rises; the peak temperature, on the contrary, declines at lower temperature.

The preceding viewpoint can be further proved by experimental results obtained in lead-doped Bi-Sr-Ca-Cu-0 specimens. We have prepared three specimens with the same chemical composition but with different phase composition. The first consists of a single low- T_c 2:2:1:2 phase. The second contains not only the low- T_c 2:2:1:2 phase, but also the high- T_c 2:2:2:3 phase. The third consists of a single high- T_c 2:2:2:3 phase. The x-ray diffraction patterns of the third specimen, shown in Fig. 5, can all be indexed assuming that $2:2:2:3$ phase had an orthorhombic cell with lattice parameters $a=5.403$ Å, $b=5.412$ Å, and $c=37.062$ Å. It coincides with the results reported by Endo et al .¹³ The temperature dependence of the Q^{-1} and R of these specimens is shown in Fig. 6. It is clear that there is not an appreciable Q^{-1} peak and an abrupt change of modulus about T_{c0} for these specimens. Figure 7 shows the curves of Q^{-1} $f²$ of another specimen sintered in low oxygen pressure conditions, which is of binary phase containing both the low- T_c and the high- T_c phase, and 107 K T_{c0} . In this figure, the Q^{-1} peak and abrupt change of modulu about T_{c0} is also not obvious, but a small Q^{-1} peak can be observed at about 270 K. Why the addition of lead can effectively repress the appearance of 170-K peak in the Bi-Sr-Ca-Cu-0 superconductor is not clear. It needs further study.

In conclusion, our experimental results show that there is an obvious difference of internal-friction spectra among the Y-Ba-Cu-0, the Bi-Sr-Ca-Cu-O, and the Bi-Pb-Sr-Ca-Cu-0 superconductors. The preparation condition also affects the Q^{-1} of the specimens. This is due to the difference of the microstructure, especially the oxygen deficiency or the ions in two valence states. Although details of this are not very clear at present, the further study of the internal-friction spectra should provide useful information about the microstructure defects and mechanisms of the superconductivity of the new high- T_c ceramic superconductors. Because of our study one thing ceramic superconductors. Because of our study one thin
is clear—there should not be a Q^{-1} peak at T_c tempera ture associated with the superconducting transition.

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