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Growth of Bi-2212 single crystals and oxygen diffusion studies

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Bi-2212 superconducting single crystals were grown by the travelling solvent floating zone method at different oxygen ambient pressure. The weight change of Bi-2212 single crystalline samples as a function of temperature, time and surrounding atmosphere was investigated by a classical thermogravimetric technique. From the size dependence it was found that oxygen out-diffusion takes place in the a-b plane in a quite anisotropic way, i.e. mainly along the a-direction. At 500°C $D_a/D_b \geq 5-6$, $D_{ab} \approx 6.12 \times 10^{-8} (\text{cm}^2/\text{s})$.

1. SINGLE CRYSTAL GROWTH

For some physical measurements probing the local field distribution of the vortex lattice phase, there is an increasing need for high-quality and large-size single crystals of Bi-2212. Therefore we have grown several large boules of the Bi-2212 superconducting phase at different oxygen ambient pressures up to 300kPa by the TSFZ method in the NEC SC-N35HD image furnace. During the growth process the feed and seed shafts were counter rotated at a rate of 30rpm. The growth rate was approximately 0.2-0.26mm/h in the static ambient atmosphere of air, 200 and 300kPa oxygen pressure.

From all growth experiments, single-crystalline boules of typically 6-8mm in diameter and 60-100mm in length were obtained. Single-crystalline samples were prepared by first cutting and then cleaving the boules. A "large-volume" and a "large-surface" sample were made for neutron scattering and muon-spin rotation experiments, respectively [1]. The "large volume" sample was a big crystal of approximately $12 \times 5 \times 1.5 \text{mm}^3$ which weighted 690mg. Neutron diffraction results showed that this

sample actually consisted of one large and one small single crystal which were closely aligned with their c-axis coinciding within 2° and with respective rocking curve widths of 0.4° , and 0.6° [2]. The "large-surface" sample for muon-spin rotation experiments was composed from 5 thin single-crystalline pieces of typical size of $5 \times 5 \times 0.5 \text{mm}^3$ with well-defined a- and b-directions, and the c-direction perpendicular to the surface [3].

2. OXYGEN DIFFUSION STUDIES

We now turn to study of the anisotropic oxygen diffusion. Compared with other methods, a classical thermogravimetric technique can be more accurate and direct in determining the amount of oxygen change and in studying the anisotropy of oxygen diffusion. But, for the calculation of the diffusion coefficients, the determination of the effective diffusion lengths is still a question to be solved. Therefore the use of single-crystalline sample is extremely desirable.

Bi-2212 single crystalline samples were prepared by the Traveling Solvent Floating Zone (TSFZ)

method [1]. In order to directly investigate the mechanism and structural anisotropy of the oxygen diffusion, two samples were prepared: sample A consisting of crystals of typical size $1 \times 1 \times 0.01(\text{mm}^3)$, total weight 178.00(mg), and sample B consisting of crystals of typical size $3.5 \times 1.2 \times 0.1(\text{mm}^3)$, total weight 203.80(mg). The dependence between crystal size and timescale for oxygen out-diffusion in nitrogen atmosphere at 500°C was studied.

Thermogravimetical analysis (TGA) was performed using a thermogravimetric microbalance (Setakam TGA 24 thermoanalyzer) connected with a gas mixture system. This thermobalance is able to detect weight change within $\pm 2.5(\mu\text{g})$. For samples of the Bi-2212 phase of about 200(mg), it is possible to measure the change of Δx with an accuracy of about 0.001. We choose the oxygen content at 500°C in air as a reference point.

The result of size dependent oxygen out-diffusion in nitrogen at 500°C , as can be seen in Fig. 1.

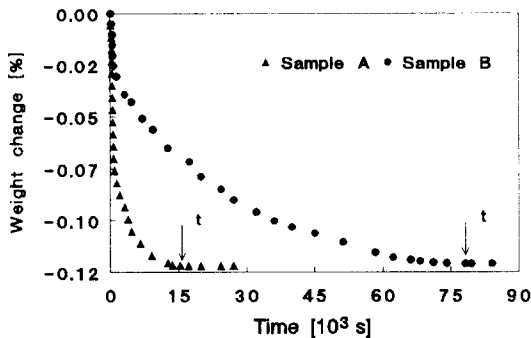


Fig.1. The size dependence of the timescale for oxygen out-diffusion at 500°C in nitrogen. The arrows indicate the relaxation time t .

If the oxygen diffusion would be in the c -direction, the relaxation time for sample B had to be 100 times larger than that of sample A. This is in contradiction with the experimental result, the relaxation time for sample B being only 6 times larger than that of

Sample A. It means that the oxygen out-diffusion takes place in the a - b planes. This is in agreement with the oxygen tracer diffusion result for Bi-2212, namely $D_{ab}/D_c \approx 10^4$ - 10^6 [5]. For a crystal of dimensions $a \times b \times c$, the in-plane diffusivity D_{ab} is given by:

$$D_{ab} = [1/a^2 + 1/\alpha b^2 + 1/\beta c^2]^{-1} / \pi^2 t \quad (1)$$

where α is the anisotropy ratio between D_a and D_b , and β is the anisotropy ratio between D_{ab} and D_c . Since β is about 10^4 - 10^6 , the third term in Eq.(1) may be neglected, then

$$D_{ab} = [1/a^2 + 1/\alpha b^2]^{-1} / \pi^2 t \quad (2)$$

After substitution of the values for a , b , and t (the relaxation time is defined in Fig.4), for sample A and B, we get $\alpha \geq 5$ -6 and $D_{ab} \approx 6.12 \times 10^{-8}(\text{cm}^2/\text{s})$ at 500°C . It follows that the oxygen out-diffusion in the a - b plane is also quite anisotropic and it is mainly along the a -direction.

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