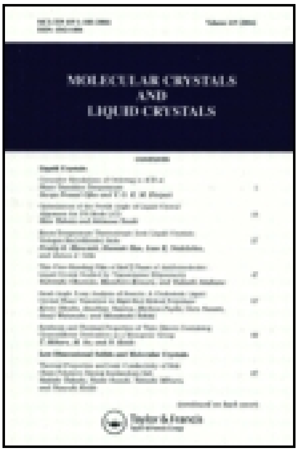

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Grain Boundaries in Oxide Superconductors Examined by Transmission Electron Microscopy

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GRAIN BOUNDARIES IN OXIDE SUPERCONDUCTORS EXAMINED BY TRANSMISSION ELECTRON MICROSCOPY

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Abstract Grain boundaries in $YBA_2Cu_3O_{7-x}$ and $Bi_2(Sr,Ca)_3Cu_2O_x$ were investigated by high resolution electron microscopy. A flat boundary parallel to a basal plane for one of neighboring grains was frequently observed in sintered specimens. In a bismuth superconductor, a Bi_2O_2 layer tended to connect both the grains. A relationship between a [001] twist grain boundary and a low J_c property in a bicrystal specimen is discussed.

Introduction:

There are three important parameters in superconductors; critical temperature T_c, upper critical field H_{c2} and critical current J_c. The T_c is an intrinsic value of materials and it is very advantageous for practical applications that the T_c is above liquid nitrogen temperature in both $YBA_2Cu_3O_{7-x}$(YBCO) and Bi-Sr-Ca-Cu-O(BSCCO). The H_{c2} is also large enough at 77K. However, the third parameter J_c changes extensively by the synthesis process. Although $J_c > 10^6 A/cm^2$ is achieved in single crystal and epitaxial thin film, it is less than $10^4 A/cm^2$ in a sintered specimen. Some defects might be related to the large discrepancy. It is difficult to decide whether or not they act favorably as a pinning center of fluxoid, because the materials belong to type-II superconductors but coherence length is extremely short. Recently, H. Kumakura et al[1] and D. C. Larbalestier et al[2] reported a small J_c value measured by the direct resistive method as compared to that evaluated by a hysteresis of magnetization curve. They indicated a weak coupling of grains in sintered specimens.

In this report, we show a structural analysis of grain boundaries(G.B.) in YBCO and BSCCO by high resolution TEM observation. The obtained results suggest a barrier effect of G.B., which are consistent with their macroscopic measurements.

Expeimentals

The YBCO smaple was prepared by a solid state reaction method using Y_2O_3, $BaCo_3$ and CuO in a stoichiometric composition. A transition temperature measured by four terminal method was about 90K. The BSCCO sample was prepared similarly. After mixing powders in $Bi_2Sr_2Ca_2Cu_3O_x$ ratio, a final sintering was performed at 1123K for 14hr. The specimen is composed of some phases. A resistivity curve showed that most of them is 70K phase, but it contained a few 110K phase.

Thin foils for TEM observation were prepared by argon ion thinning method. High resolution electron microscopy was performed by JEM-2000EX operated at 200KeV.

High Resolution Electron Microscopy of the Grain Boundary

Figure 1 shows high resolution image of G.B. in YBCO. In most cases, G.B. is intimately connected to each other grains as shown in Fig.1[3]. A flat boundary observed in a sintered specimen is often parallel to a basal plane of a grain (c-plane boundary). The boundary is sometimes decorated by an amorphous layer (10nm), which is mainly caused by a G.B. fracture owing to an anisotropy of thermal expansion coefficients between a-(or b-)axis and c-axis.

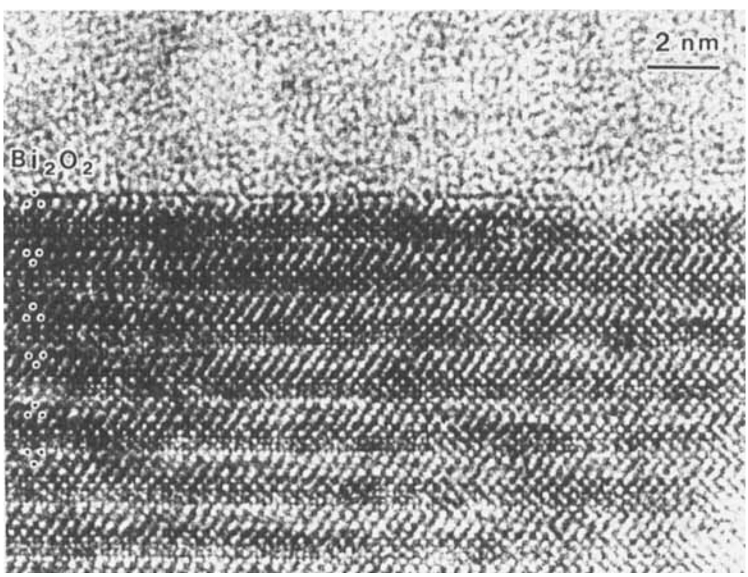

FIGURE 1. High resolution image of c-plane boundary in BSCCO.

A c-plane boundary is observed more frequently in BSCCO than in YBCO. The former is composed of a Bi_2O_2 layer. Dots in Fig. 1 indicate a position of bismuth atom, where Bi double layers are found to be flat in atomic scale at G.B. Another example is shown in Fig. 2. A crystal orientation of a bottom grain is [100]. An ordered structure is seen at G.B. and the periodicity coincides to lattice parameters of BSCCO(5b X c/2 = 2.7 X 1.5nm). In addition, a Bi_2O_2 layer connects a neighboring grain in a facet of G.B. These preferential selectivity of Bi_2O_2 layer is explained by the interlayer bonding; two bismuth planes is widely separated by oxygen plane, then, the bonding of the layer is weak and it can accommodate an incommensurate modulation. The Bi_2O_2 layer makes G.B. energy lower relaxing the G.B. strain. However, a Bi_2O_2 layer at G.B. is considered not beneficial from a view of

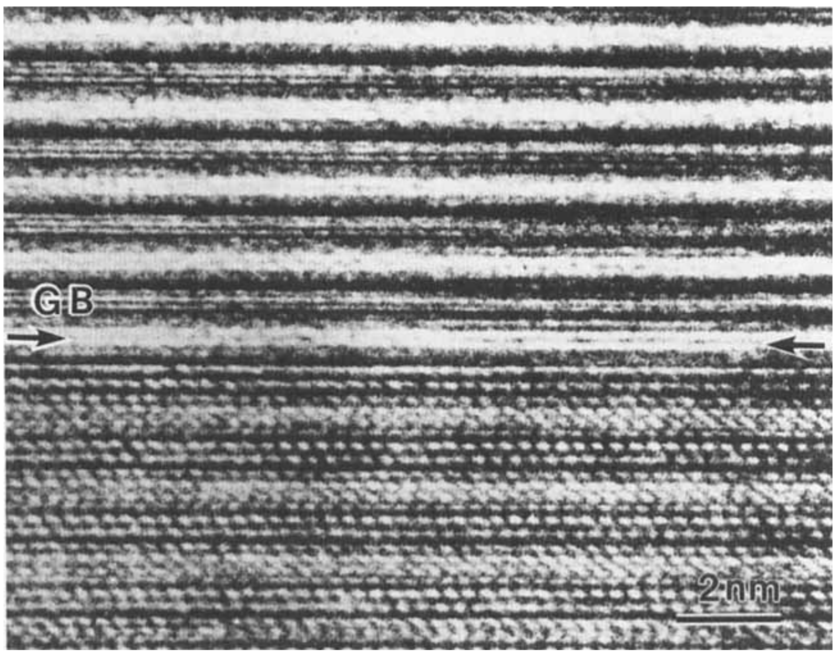

FIGURE 2. A [001] twist boundary of BSCCO.

J_c. It is believed that a Cu-O plane in a perovskite unit plays a main role of conductivity and that a Bi_2O_2 layer only separates these sheets. Thus a at G.B. may be referred as a S-I-S junction, where the coupling is very weak.

Grain Boundary Structure and J_c

HREM observation showed that there is no thick layer at G.B., but it still acts as a barrier. Recently, D. Dimos et al[4] postulated an orientation dependence of J_c across G.B.(J_c^{gb}) using bicrystal thin films in YBCO. J_c^{gb} decreased by two orders of magnitude across [001] tilt boundary. Takahashi et al analyzed their results and estimated the G.B. width as a barrier to be result predicted a weak coupling across the G.B. The coherent length is so small and anisotropic in the layered structure (ξ=3.1nm is a basal plane and ξ=0.51nm parallel to c-axis) that the reduction in the width of G.B. (w/ξ) varies from 0.58 to 3.5. It indicates J_c^{gb} depends heavily on the crystal orientation; The [001] tilt boundary should be most advantageous since the current direction is parallel to c-planes for both grains. The [001] twist boundary should be least advantageous since the boundary plane is c-plane and the reduced thickness is

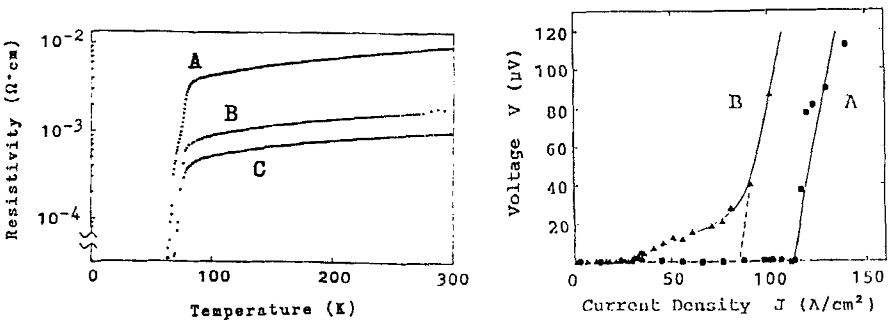

FIGURE 3. The T_c and J_c values in an [001] twist boundary in BSCCO.

the largest. The latter type of boundary is most abundant in a sintered specimen and may explain the low J_c values in polycrystalline specimens.

Fig. 3(a) shows T_c values of BSCCO, (A) sintered polycrystalline sample, (B) an [001] twist bicrystal and (C) single crystalline sample. They differ to each other only by small amount especially with respect to T_c. The J_c values, however, differ extensively (Fig.3(b)) when a large shoulder is evident in bicrystalline specimen.

Conclusions

In summary, G.B.s in YBCO and BSCCO were examined by high resolution electron microscopy. Grains are tightly connected in atomic scale, however it is electrically weak coupling due probably to the short coherence length. The poor J_c at G.B. may be enhanced when a boundary is parallel to c-plane, which is abundant in a sintered specimen.

Improvement in the connection between grains should enhance the J_c property in a polycrystalline material.

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