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Phase evolution and the mechanism of J_c increase during post-annealing in Ag/(Bi, Pb)2223 tapes

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Abstract

We have investigated the influence of post-annealing on the current transport and phase/microstructure evolution in Ag/(Bi, Pb)2223 tapes. After post-annealing, the critical current density was about 1.4 times higher. Also the superconducting transition was greatly sharpened and T_c became much higher, even when a magnetic field was applied. The Rietveld analysis suggested that, before post-annealing, a liquid phase remained in the tape, whose mass fraction was estimated as 9.3% from the sum of the phases that formed during post-annealing. Transmission electron microscopy observations revealed that amorphous layers or lattice disorder areas existed at a large number of the (Bi, Pb)2223 grain boundaries. These insulating layers block the superconducting current paths or produce a Josephson-coupled weak link boundary. Upon post-annealing, phase and structure changes took place at the grain boundaries. The insulating amorphous layer converted into metallic 2212 layers. In addition, a faceted structure was formed at the boundary interface such that the tilt boundary became a superconducting through-path. It is strongly suggested that these two factors are critical for increase of the J_c upon post-annealing.

1. Introduction

(Bi, Pb)₂Sr₂Ca₂Cu₃O_{10-δ} ((Bi, Pb)2223) is a high temperature superconducting oxide material, and has been studied in various respects with a view to practical applications. In particular, the control of phase and structure evolution in the tapes is a key factor for realizing high critical current density (J_c). Therefore, in efforts to achieve a high J_c for a (Bi, Pb)2223 tape, the relationship between the phase evolution and heat treatment conditions have been carefully investigated in recent years [1–3]. Recently, the post-annealing process has had attention paid to it because of its remarkable effect of increasing J_c for the (Bi, Pb)2223 tape [4, 5]. In particular, it was reported that J_c became about two times higher upon the tape undergoing a combination of stepwise heat treatments [5].

During the (Bi, Pb)2223 phase transformation, Bi₂Sr₂CaCu₂O₈ (Bi2212) melts partially, with secondary phases present, and a liquid phase appears at the early stage of the

sintering process [6, 7]. After the partial melting, the reaction of (Bi, Pb)2212 with the liquid phase leads to (Bi, Pb)2223 formation.

Even after the (Bi, Pb)2223 phase formation is mostly completed, some liquid phase remains in the sample. The remaining liquid phase exists at boundaries of the (Bi, Pb)2223 grains and consequently limits J_c for the tape. It is suggested that the post-annealing makes this liquid phase convert into some crystallized phases with no adverse effect on J_c when the post-annealing conditions are appropriate. However, the phase evolution in the sample during post-annealing and the mechanism of increase of J_c are still unclear and clarification is desirable for a more detailed understanding, for investigating more effective heat treatment conditions.

Powder XRD Rietveld analysis is very powerful and useful for investigating the phase evolution inside the Ag sheath during the heat treatment process [7, 8]. We have succeeded in determining quantitatively the content of phases

in the superconducting filament through Rietveld analysis of powder XRD data in spite of the *ex situ* observation using the conventional powder XRD technique [8]. The combination of Rietveld analysis and microstructure observation gives us much more detailed and precise information, helping us to understand the post-annealing effects on J_c .

In this paper, we investigate the effect of post-annealing on the current transport, phase and microstructure evolution in the tape. The phase formation from the residual liquid phase was studied using powder XRD Rietveld analysis. And we demonstrate microstructure changes at the (Bi, Pb)2223 grain boundaries using transmission electron microscopy (TEM) observation. We also suggest the mechanisms of the increase of J_c and discuss the relationship between the current transport and the microstructure evolution.

2. Experimental details

The precursor powder from Merck Corporation was used after re-calcining at 1073 K in air for 72 ks. Its chemical composition is $\text{Pb}_{0.33}\text{Bi}_{1.80}\text{Sr}_{1.87}\text{Ca}_{2.00}\text{Cu}_{3.00}\text{O}_x$. The precursor consisted of Bi2212 , CaPbO_4 (CP), $(\text{Sr}, \text{Ca})_{14}\text{Cu}_{24}\text{O}_{41}$ (14-24), $(\text{Ca}, \text{Sr})_2\text{CuO}_8$ (2-1), $(\text{Sr}, \text{Ca})_{0.78}\text{CuO}_x$ (1-1) and $(\text{Pb}, \text{Bi})_3\text{Sr}_2\text{Ca}_2\text{CuO}_x$ (Pb3221). Ag-sheathed monofilamentary tapes were prepared by the PIT method.

The tapes were sintered at 1095 K for 432 ks with two intermediate pressings. Some of tapes were air-quenched after sintering. The rest of them were continuously cooled for 7.2 ks to 1053 K and post-annealed for 48.6 or 165.6 ks at 1053 K. Post-annealed samples were also air-quenched after each annealing process.

The J_c measurement was carried out at 77 K in the self-field by means of the four-probe method with the criterion of $1 \mu\text{V cm}^{-1}$. Superconducting properties were measured by a Quantum Design Physical Property Measurement System (PPMS). Cooling from 115 to 80 K, the resistivities of the air-quenched sample and the sample post-annealed for 165.6 ks were measured in the self-field and applying a magnetic field perpendicular to the broad tape surface from 25 to 100 mT. A current of 0.2 A was applied to the tapes in the resistivity measurements.

Then the Ag sheath was removed from the tape by chemical etching with $\text{NH}_3:\text{H}_2\text{O}_2 = 2:1$ solution. After removing the Ag sheath, superconducting filaments from the tapes were wet-ground with acetone to a powder-like form. The ground powder was mixed with acetone-diluted cellulose glue so as to decrease the effect of the preferred orientation of the plate-like grains. Then the powder mixed with cellulose glue was vacuum-dried and ground again to make samples for the powder XRD measurements.

The powder XRD measurement was performed with a RIGAKU RAD diffractometer in Bragg-Brentano geometry using $\text{Cu K}\alpha$ radiation. Diffraction data were collected from θ - 2θ scanning. The collected data were analysed using the Rietveld method in order to identify the phases present and refine their crystal parameters, such as the lattice parameter. The Rietveld analysis was carried out with RIETAN-2000 software [9].

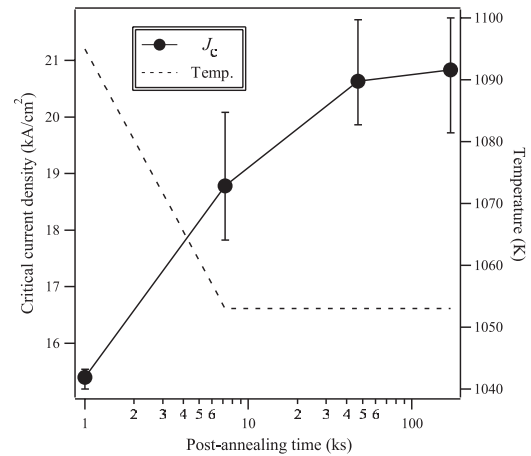


Figure 1. Temperature profile of the post-annealing and J_c (77 K, 0 T) for samples quenched at different stages.

Specimens for TEM observation were prepared by the following procedures. Firstly, two or three (Bi, Pb)2223 tapes were stacked and sandwiched with 0.5 mm thick Si substrates. The sandwiched tapes were made to adhere with Vishay M-BOND 610 adhesive. The adhered samples were thinned to ~0.5 mm thick in the direction of the cross section by wet-grinding. Then they were adhered to $\varnothing$ 1 mm holed Cu mesh and thinned to ~100 μm thick; then they were dimpled with a Fishione Model 150 dimple grinder. Finally, they were ion-polished with a Gatan 691 Precision Ion Polishing System (PIPS). TEM observations were performed with a Philips CM-200FEG high resolution transmission electron microscope (HRTEM).

3. Results and discussion

J_c increased during post-annealing as shown in figure 1, where the temperature was reduced to the post-annealing temperature, and then kept constant. As shown in this figure, J_c increased monotonically during the post-annealing, and saturated at almost the end of the post-annealing. And J_c became 1.4 times higher upon post-annealing.

Figure 2 demonstrates the superconducting transitions in the self-field and various magnetic fields as the resistivity changes as a function of temperature. The transitions of the air-quenched and 165.6 ks post-annealed sample are shown in figures 2(a) and (b), respectively. The resistivity was normalized to that at 120 K. As shown in figure 2, the critical transition temperature (T_c) in the self-field was increased by about 5 K upon post-annealing. It is suggested that the increase of the superconducting temperature can be attributed to the accommodation of the oxygen concentration during the post-annealing [10].

As shown in figures 2(a) and (b), for both samples, the superconducting transitions became wider and T_c was shifted to lower temperature as the higher magnetic field was applied. However, the transition became greatly sharper and T_c was much higher for the post-annealed sample, even when a high magnetic field was applied (figure 2(b)). It should be noted that the T_c difference between the air-quenched and post-annealed

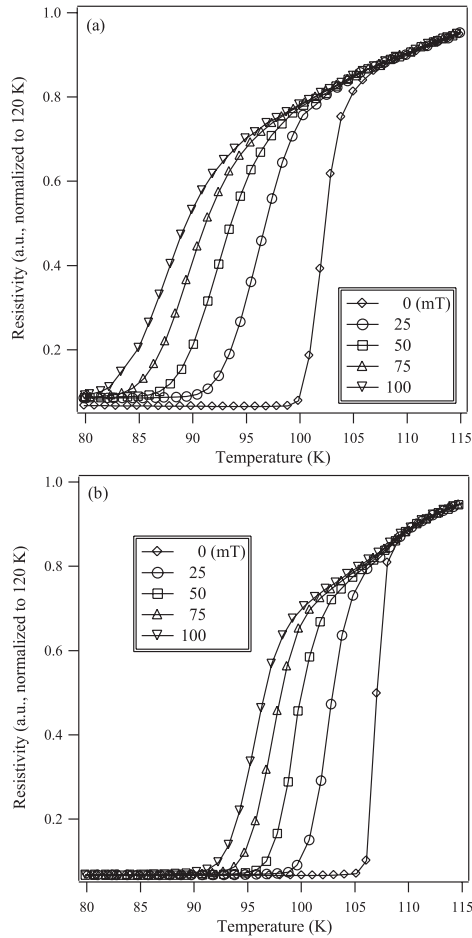


Figure 2. (a) Sample air-quenched from 1095 K; (b) sample post-annealed at 1053 K for 165.6 ks and air-quenched.

samples in the same magnetic field became nearly 10 K, which was relatively great compared with the difference in the self-field.

In a previous report, an increase of T_c upon post-annealing was also observed as the change of the DC magnetization was measured by a SQUID [5]. Basically, the superconducting behaviour differs in two areas in polycrystalline superconductors—in and between grains. In cuprate superconductors, especially in (Bi, Pb)2223, grain boundaries (weak links) are the critical factor in the superconducting behaviour due to the short coherence length. As demonstrated in figure 2, the measurements were performed in conditions in which a current of 0.2 A and a magnetic field of 25–100 mT were applied. This is similar to the conditions in which a high density of superconducting current passes through the tapes in the self-field. Figure 2 indicates that the air-quenched sample had more weak link boundaries and so found it easy to transit from the superconducting to the metallic or insulating state. This means that the superconducting current transport property itself was drastically improved at grain boundaries by post-annealing.

Figure 3 shows the results of Rietveld analysis of the air-quenched sample (a) and that post-annealed for 165.6 ks (b). In the air-quenched sample, only (Bi, Pb)2223 and (Bi, Pb)2212 were detected, and it had a J_c of 15.4 kA cm⁻², whereas in

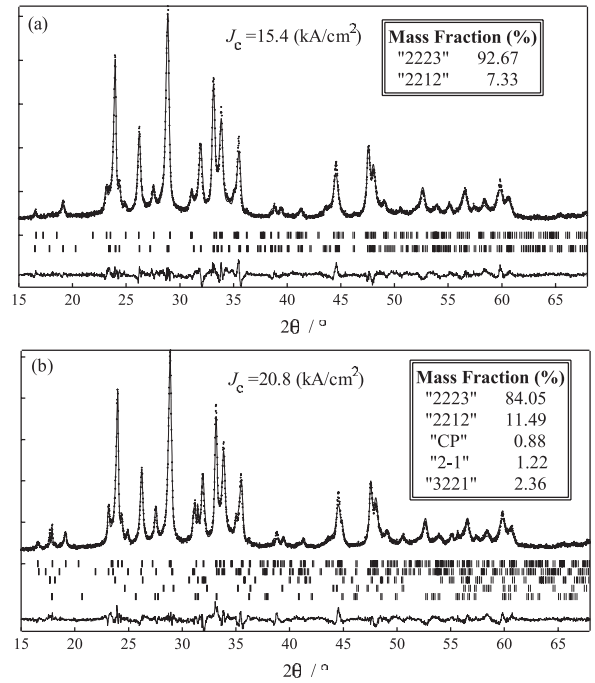


Figure 3. The Rietveld analysis of the samples (a) air-quenched from 1095 K and (b) post-annealed at 1053 K for 165.6 ks and air-quenched. J_c is 15.4 and 20.8 kA cm⁻² for the air-quenched and post-annealed samples, respectively. In the graphs, the dots and solid line indicate the experimental data and the calculated pattern, respectively. The bands at the bottom are the reflections from the detected phases. The solid line at the bottom is the error between the experimental and calculated patterns.

the sample after post-annealing, 2-1, CP and Pb3221 were detected in addition to (Bi, Pb)2223 and (Bi, Pb)2212, and it had a J_c of 20.8 kA cm⁻². As is clearly shown in figure 3, the mass fraction of the (Bi, Pb)2223 phase decreased, although J_c increased by nearly 1.4 times upon post-annealing at 1053 K for 165.6 ks. However, it is hardly acceptable to suggest that (Bi, Pb)2223 decomposes and induces other phases such as Bi2212 or Pb rich phases during post-annealing because of the increase of J_c . Furthermore, a small amount of amorphous phase (liquid phase) was *not* detected in the XRD measurement even if it exists in the sample. Thus, it is suggested that these newly formed phases indicated in figure 3(b) have been converted from the liquid phase remaining in the sample just before the post-annealing process.

In the present study, we assumed that the mass fraction of (Bi, Pb)2223 was kept constant, and all of the newly formed phases were converted from the liquid phase during post-annealing. And the mass fraction of the liquid phase w_L could be estimated from the sum of the newly formed phases:

$$w_L = (w_{2212}^{PA} - w_{2212}^{AQ}) + \sum w_i^{PA} \quad (1)$$

where w_{2212}^{PA} and w_{2212}^{AQ} are the mass fractions of (Bi, Pb)2212 in the sample after post-annealing and air-quenching, respectively; w_i^{PA} is the mass fraction of the newly formed phases except for (Bi, Pb)2212. With this assumption, the mass fraction of the residual liquid phase in the air-quenched sample was estimated as 9.30%. And the mass fractions in

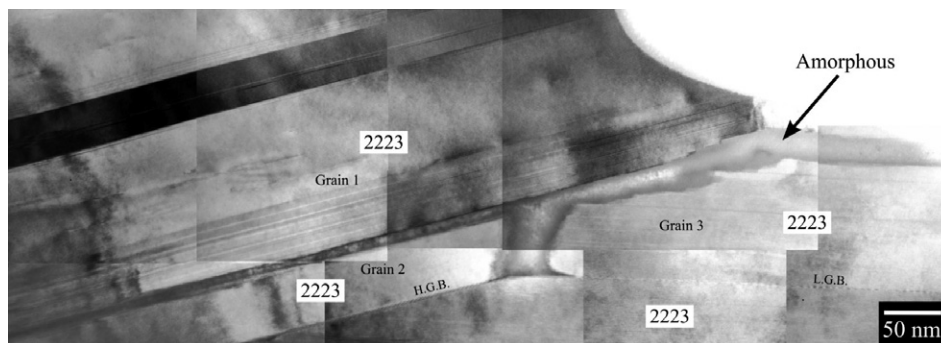


Figure 4. TEM image of the sample air-quenched from 1095 K. Grains 1, 2 and 3 are isolated by the amorphous layer.

figure 3(a) were re-evaluated as follows:

(Bi, Pb)2223: 84.05 (mass%),

(Bi, Pb)2212: 6.65,

Liquid phase: 9.30.

Basically, when the (Bi, Pb)2223 phase nucleates from the liquid phase, its chemical composition must be stoichiometrically suitable for (Bi, Pb)2223. As sintering proceeds, the mass fraction of the liquid phase decreases corresponding to 2212 to 2223 phase conversion, so the area of liquid phase is gradually isolated, and exchange of cations between the areas scarcely occurs, to retain the stoichiometry. In addition, as indicated in previous reports [11, 12], the stable range of 2212 in phase equilibrium is fairly wide compared with that of 2223. Therefore, the 2212 phase preferentially precipitates from the off-stoichiometric liquid phase, when the heat treatment temperature is beyond the range of the partial melting state that is needed for (Bi, Pb)2223 formation.

The existence of amorphous phase was proved by TEM observation. Figure 4 shows amorphous phase existing at (Bi, Pb)2223 grain boundaries in the air-quenched sample. The amorphous layers are 10–30 nm thick. It is clearly seen that the (Bi, Pb)2223 grains 1, 2 and 3 are isolated by amorphous phase, which was the residual liquid solidified during air-quenching after sintering. Such amorphous layers are likely to be insulating, and the superconducting current never passes through. Because the amorphous phase remained at almost 10% of the mass fraction, the superconducting paths were seriously limited, and thus the critical current is suppressed in the air-quenched sample.

As shown in figure 3(b), some 2212 was newly formed during post-annealing. Corresponding to the 2212 phase formation, a microstructure change was taking place at the grain boundary including an amorphous layer, as demonstrated in figure 5. The amorphous layer is seen at the tilt boundary in the air-quenched sample (figure 5(a)). After post-annealing, newly formed 2212 layers occupy the tilt boundary (figure 5(b)). A high magnification image near the phase-to-phase interface is also shown in the small window in figure 5(b). It is clearly seen that the c -axis lengths were different, and the shorter c -axis proved that the arrowed area was 2212 phase.

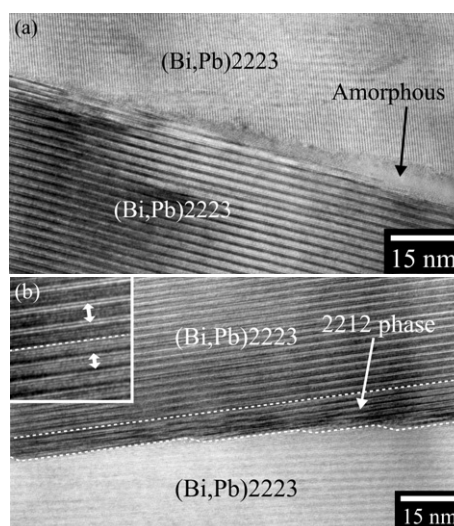


Figure 5. Structural changes at the grain boundaries sandwiching an amorphous layer. (a) The amorphous layer is seen at the tilt boundary in the air-quenched sample. (b) After post-annealing, 2212 layers formed at the grain boundary.

In figure 6, we suggest a mechanism of increase of J_c by 2212 formation at the grain boundaries. Before post-annealing, the residual liquid phase existed as an insulating amorphous phase at a number of grain boundaries (figure 6(a)). Superconducting current cannot pass through such an insulating layer to an adjacent grain. Therefore, the number of current paths is fairly limited. Meanwhile, upon post-annealing, the residual liquid converted into 2212 phase, which is in the metallic state at 77 K, especially when a superconducting current is applied (figure 6(b)). At this time, the supercurrent can pass through the metallic layer to an adjacent grain. In this way, the newly formed 2212 phase works as a metallic layer instead of a completely insulating amorphous one, so the number of current paths is increased by post-annealing.

We have discussed the microstructure change of (Bi, Pb)2223 grain boundaries, indicating the phase conversion from the residual liquid to the 2212 phase. Although the newly formed 2212 layers were metallic in the supercurrent flow at 77 K, the superconducting transition became greatly sharper upon post-annealing, as demonstrated in figure 2(b). This is not fully explained just with the phase conversion at grain

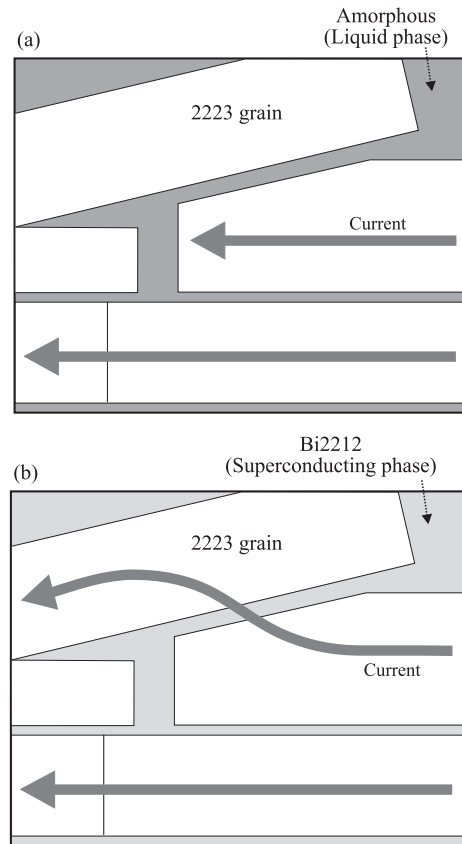


Figure 6. Illustration explaining the mechanism of increase of J_c via 2212 formation at the grain boundaries. (a) Before post-annealing, the residual liquid phase exists as an amorphous phase at a number of grain boundaries. (b) After post-annealing, the residual liquid converts into the 2212 phase.

boundaries. Thus, we must consider another factor improving the current transport property at (Bi, Pb)2223 grain boundaries, or eliminating weak link connections.

Now we investigate nanoscaled structure changes of the grain boundary interface. Figure 7 shows TEM images of a typical (Bi, Pb)2223 tilt grain boundary in the air-quenched sample. Atomic columns containing Bi and Pb atoms exhibited dark contrast and the (Bi, Pb)O layers could be easily discerned [13]. The 2223 c -plane periodicity was seen in the upper grain. As shown in figure 7(a), the grain boundary had a smooth interface. And the grain boundary seemed to be solidly connected.

However, HRTEM observations revealed lattice distortion and disorder at such boundary interfaces. A HRTEM image of the tilt boundary is shown in figure 7(b). The [001] direction is arrowed both in the upper and lower grains. The lattice contrast of the upper grain shows that the beam direction is parallel to the [100] axis. Lattice periodicity was clearly seen in the upper grain. However, the lattice contrast suddenly became diffuse in the 1.8 nm thick area. Basically, when the lattice periodicity becomes weak, the lattice contrast in HRTEM images tends to become diffuse. These areas were not considered as amorphous areas that had completely lost their lattice periodicity. However, when an amorphous layer ultimately became thinner, it was more affected by the

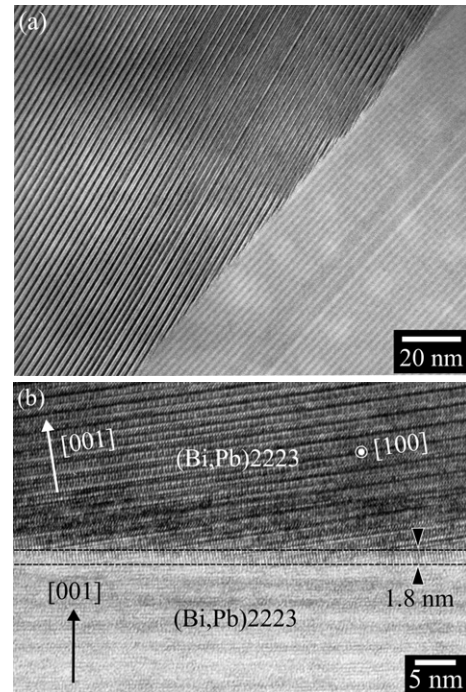


Figure 7. TEM micrographs showing a typical grain boundary in the sample air-quenched from 1095 K. (a) The boundary had a smooth interface. (b) The lattice contrast is clearly seen in the 2223 grain, but the lattice contrast suddenly becomes diffuse in the 1.8 nm thick area.

periodicity of an adjacent grain. Therefore, the diffuse contrast indicated that a lattice disorder area existed at the boundary interface in the air-quenched sample. And the present disorder area results in decrease of T_c and J_c at the grain boundary.

In earlier TEM works [14, 15], it has been reported that 2201, 2212 and amorphous phase existed at the boundaries due to the compositional modulation in the sintered tapes. In the case of figure 7, low T_c phases such as 2201 or 2212 were not present near the grain boundary; only nanoscaled thick disordered areas existed. In the common sense, since disorder areas make the Gibbs free energy increase in the system, they are considered to be unstable. However, as mentioned previously, the residual liquid phase could exist as amorphous phases at grain boundaries due to the partial melting state present during sintering, especially when out of (Bi, Pb)2223 stoichiometry. Therefore the lattice disorder area—ultimately a thin amorphous phase—could exist at the grain boundary interface in the air-quenched sample.

On the other hand, after post-annealing, a significant structure difference was observed at (Bi, Pb)2223 grain boundaries. Figure 8 shows a typical grain boundary in the post-annealed sample. Before post-annealing, the grain boundaries had smooth interfaces as shown in figure 7(a). However, after post-annealing, it is clearly seen that a serrated interface was formed at the grain boundary (figure 8(a)).

This structure difference is more clearly demonstrated in a HRTEM image of the serrated interface as demonstrated in figure 8(b). The Bragg condition was satisfied with [110] parallel to the incident beam in the upper grain. The arrowed lines of the black lattice contrast indicate a single (Bi, Pb)O plane. The [001] direction is also arrowed in the figures. As

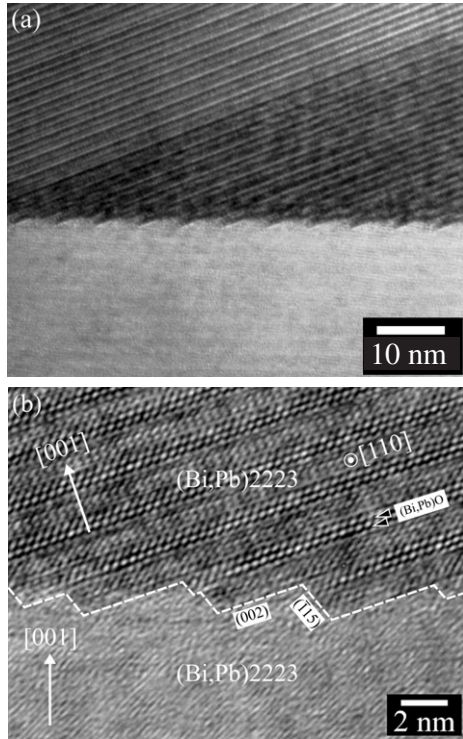


Figure 8. TEM images showing a typical grain boundary in the post-annealed sample. (a) The boundary had a serrated interface. (b) The HRTEM image of the serrated boundary. It is clear that the cations realigned and rearranged to form the faceted structure.

clearly shown in figure 8(b), cations realigned and rearranged to form the faceted structure at the boundary interface. The facet consisted of (002) and (115) planes. One of features of the terraced facet is that (Bi, Pb)O planes never contacted an adjacent grain. It should be noted that the lattice contrast never became diffuse and remained clear to the boundary interface. It is strongly suggested that the lattice disorder area at the interface was drastically decreased by post-annealing.

When the faceted structure was formed, the phase conversion during post-annealing helped its formation. As previously discussed, before post-annealing, the (Bi, Pb)2223 lattice was disordered in the area a nanometre thick at the boundary interface, because of the partial melting state that arose during sintering. The condition of post-annealing was beyond the range of the partial melting state, so the residual liquid started to crystallize to form several phases. Corresponding to the phase conversion during post-annealing, cations were frequently transported through the grain boundaries. Therefore, it is considered that fixing of the stoichiometry at the boundary interface has taken place, and this helped in the transformation from the disordered to the faceted structure. And, with the formation of the faceted structure, the interface became as coherent as possible, decreasing the Gibbs free energy at the interface.

In figure 9, we suggest a mechanism of increase of J_c by faceted structure formation. Before post-annealing, the residual liquid phase exists as an amorphous layer at the tilt grain boundary. And when the amorphous layer is sufficiently thin (1–2 nm), it is affected by the lattice periodicity of (Bi, Pb)2223 grains, becoming a lattice disorder area (figure 9(a)).

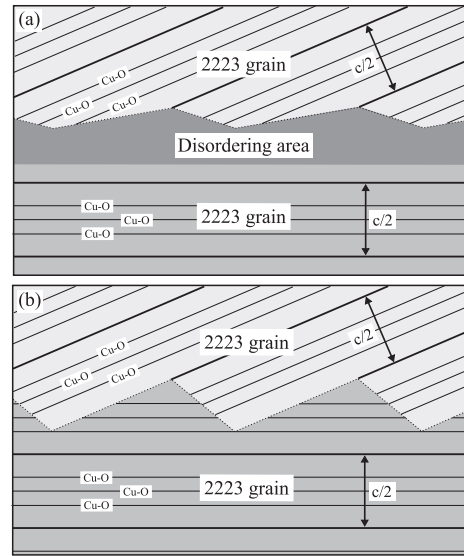


Figure 9. Illustration explaining the mechanism of increase of J_c by faceted structure formation. (a) Before post-annealing, a lattice disorder area exists at the 2223 boundary. Such boundaries are described as thin insulator sandwiching structures. (b) After post-annealing, the formation of a faceted structure takes place at the boundary interface. At this time, a nanoscaled rail switch structure is applicable for describing such boundaries.

Although this area is not completely amorphous, it works as a thin insulating (sometimes metallic) layer so as to form a Josephson-coupled weak link boundary. In this case, only a low density of superconducting current passes through such boundaries.

On the other hand, during post-annealing, the structure evolution takes place at the boundary interface (figure 9(b)). Cations in the disorder area realign and rearrange to make the faceted structure. And corresponding to the phase formation from the residual liquid, diffusion of cations frequently takes place to fix the stoichiometry at the grain boundary. It is considered that the boundary diffusion helps with the rearrangement of cations in making the faceted structure.

For describing the current transport, a ‘brick wall’ or ‘rail switch’ model has been used for polycrystalline BSCCO tapes [16, 17]. In the case of figure 9(b), the faceted grain boundary barely has lattice disorder areas, so a nanoscaled rail switch model is applicable for describing such boundaries. The electrical behaviour at such a boundary no longer shows a sign of a weak link regime. And the nanoscaled rail switch boundaries are also electrically well connected, thus being close to strong links like in-plane connected grain boundaries. In consequence, a much higher density of the superconducting current passes through such a strong link grain boundary. Therefore, due to the faceted structure formation, T_c and J_c at the grain boundary are drastically increased upon post-annealing.

4. Conclusion

In this paper, we studied the relationship between J_c and the phase evolution during post-annealing, by means of current transport measurements, TEM observation and powder XRD Rietveld analysis. We summarize our conclusions as follows.

J_c became about 1.4 times higher upon post-annealing at 1063 K. In the self-field, the critical transition temperature (T_c) increased by about 5 K upon post-annealing. When a magnetic field was applied, the superconducting transitions became wider and T_c was shifted to lower temperature. However, the transition was sharper and T_c was much higher in the post-annealed sample, even when a high magnetic field was applied.

During post-annealing, crystallized phases were formed from the residual liquid. The mass fraction of the residual liquid was estimated from the sum of the phases that were newly formed during post-annealing. It was suggested that 9.30 mass% of the liquid phase remained after sintering for 432 ks.

TEM observations revealed that an amorphous layer or a 1–2 nm thick lattice disorder area existed at a number of (Bi, Pb)2223 grain boundaries. These insulating layers limit the number of superconducting current paths or make a Josephson-coupled weak link. Upon post-annealing, phase and structure changes take place at the grain boundaries. The insulating amorphous layer converts into a 2212 layer which is in the metallic state at 77 K when the supercurrent passes through the tape. In addition, a faceted structure is formed at the boundary interface, so the tilt boundary becomes a strong link. Therefore, it is clearly suggested that these two factors are the key to understanding the mechanism of J_c increase during post-annealing.

Acknowledgment

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