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New multi-seeding method of RE–Ba–Cu–O superconductor

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Abstract

We have developed a new multi-seeding method, multi-seeded seamless bulk (MUSLE) technique, for improving a problem of the conventional multi-seeding method, i.e. the existence of the excluded non-supercondcting phases (liquid phases and segregated RE211 phases) at the grain boundaries in the bulk superconductor. It is the basic concept of this technique that the precursor is composed of two or more RE–Ba–Cu–O layers with different peritectic temperatures. Thereby, the directions of the crystal growth from the seed crystals can be controlled.

RE–Ba–Cu–O superconductors were fabricated by the MUSLE technique with four seeds. The trapped field distribution obtained at 77 K showed a single peak with a value of 0.9 T, indicating that the MUSLE technique is effective in eliminating the excluded phases formed at the grain boundaries in the multi-seeded bulk superconductor. We suggest that the elimination of the excluded phases can be explained by the seed crystal proximity effect.

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1. Introduction

Top-seeded melt-process [1–3] is known as a process to fabricate RE–Ba–Cu–O (RE: Y and rare earth elements) bulk superconductors. According to this method, it is possible to manufacture samples with their diameter up to 100 mm [4]. However this method require quite a long time for crystal growth.

Up to date, the multiple-seeding technique has been applied to fabricate the sample [5–13]. This multi-seeding technique has a advantage in shortening the processing time because a region for growing a crystal grain from each seed crystal becomes small. However, the bulk superconductor fabricated by this technique has several peaks for the trapped field distribution. This is caused by the existence of the excluded non-superconducting phases at grain boundaries.

In order to improve this problem, several studies have been performed on the seed arrangement of the (1 1 0)/(1 1 0) junction [6,8,10,12] and the proximal seed arrangement where there is a small distance between the seed crystals [6,7,11]. The seed arrangement of the (1 1 0)/(1 1 0) junction makes it difficult to form the excluded phases, however, the trapped field distribution does not have a single peak, and thus the excluded phases

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cannot be removed sufficiently. The proximal seed arrangement is effective in eliminating the excluded phases. However, it is not practical from the view of productivity because a lot of the seed crystals need to be embedded on the top surface of the precursor. Therefore, there has been a demand for a method of manufacturing bulk superconductors without the excluded phases at the grain boundaries.

We have developed a new multi-seeding method, multi-seeded seamless bulk (MUSLE) technique, for fabricating RE–Ba–Cu–O bulk superconductors that have a single peak in the trapped field distribution in order to improve the problem of the conventional multi-seeding method, i.e. the existence of the excluded phases at the grain boundaries in the bulk superconductor. It is the basic concept of the MUSLE technique that the precursor is composed of the two or more RE–Ba–Cu–O layers with different peritectic temperatures. In this study, RE–Ba–Cu–O bulk superconductors were fabricated by the MUSLE technique. To prove the validity of this technique, measurements of the trapped field, and observation of the microstructure around the grain junction were performed.

2. Experimental

Two bulk superconductors were fabricated by the top-seeded melt-growth technique [14]. One of the specimens was used for measuring the trapped field, and the other was used for observing the microstructure around the grain boundary. Fig. 1(a) shows a schematic of the precursor, which had two layers of different compositions (Layer-A and Layer-B). The Layer-A and Layer-B were $\mathrm{Dy_{0.75}}$-$\mathrm{Gd_{0.25}}$–Ba–Cu–O layer ($\mathrm{Dy_{0.75}Gd_{0.25}Ba_2Cu_3O_x}$: $\mathrm{Dy_{1.5}Gd_{0.5}BaCuO_5} = 3:1$ in molar ratio with 0.5 wt.% Pt) and Dy–Ba–Cu–O layer ($\mathrm{DyBa_2Cu_3O_x}$: $\mathrm{Dy_2BaCuO_5} = 3:1$ in molar ratio with 0.5 wt.% Pt), respectively. The (Sm,Nd)–Ba–Cu–O seed crystals were arranged to make the crystal grain with (1 0 0)/(1 0 0) grain junctions (Fig. 1(b)), and placed on the Layer-A of the precursors before the melt-growth process. The precursors were heated up to 1060 °C, kept for 4 h, and then cooled down to 1020 °C. For the crystal growth, the precursors were cooled to 990 °C at 0.1–2.0 °C/h rate, and then cooled to the room temperature. One of the specimens was cut into the Layer-A part and the Layer-B part (Fig. 1(c)), and then annealed at 450 °C for 100 h in flowing oxygen gas.

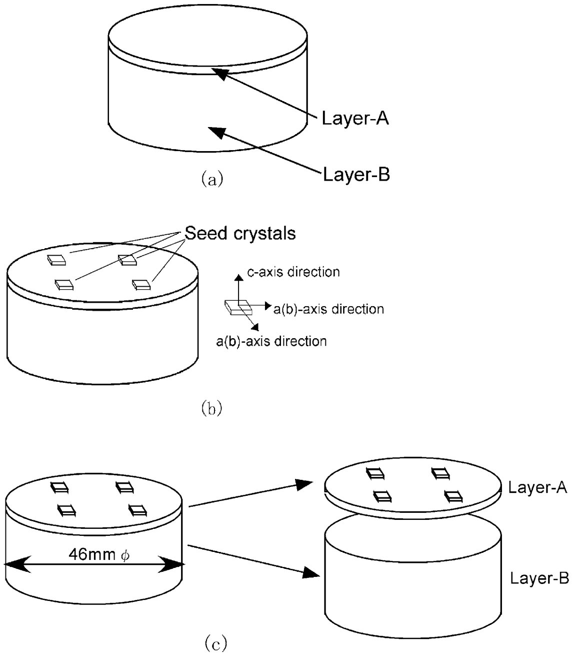


Fig. 1. Schematic of the sample in the study: (a) schematic of the precursors, (b) the (Sm,Nd)–Ba–Cu–O seed crystals arrangement on the precursor, (c) the specimen cut into the Layer-A part and the Layer-B part.

The trapped field distributions were measured by scanning a Hall sensor at a height of 1 mm above each specimen surface after the specimens were field-cooled to liquid nitrogen temperature, at 77 K.

3. Results and discussion

Fig. 2 shows a photograph for the top surface of the bulk superconductor fabricated by the MUSLE technique. The four grains grown from the seed crystals almost covered the top surface of the precursor, and the (1 0 0)/(1 0 0) grain junctions were formed between seed crystals. The distance between seed crystals was long, i.e. 15 mm.

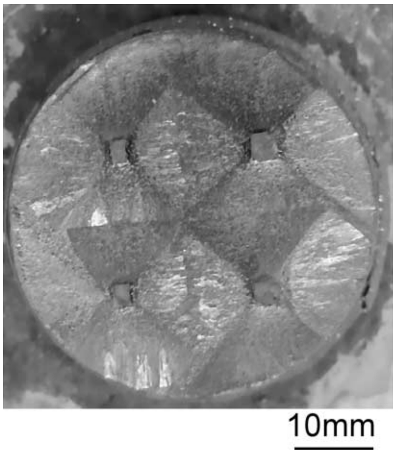


Fig. 2. Photograph for the top surface of the bulk superconductor fabricated by the MUSLE technique.

Fig. 3 shows an optical micrograph of a vertical section of the specimen. The boundary between the Layer-A and the Layer-B was determined by measuring Gd element along the *c*-axis direction by the electron probe microscopic analyzer (EPMA). In the Layer-A, a line-shaped segregation was found along the grain boundary. The width of the segregation was 10–120 μm, which values are similar to those in the previous reports [6,7,9]. The main compositions of the segregations were $Dy_{1.5}Gd_{0.5}BaCuO_5$ phase, CuO phase and Ba–Cu–O phase, which were determined by the EPMA. On the other hand, the line-shaped segregations were not found around the grain boundary in the Layer-B. It means that the (1 0 0)/(1 0 0) grain junctions without the segregations at the grain boundary were realized in the Layer-B. This indicates that the MUSLE technique is effective in eliminating the segregations at the grain boundary and realizing a clean grain boundary.

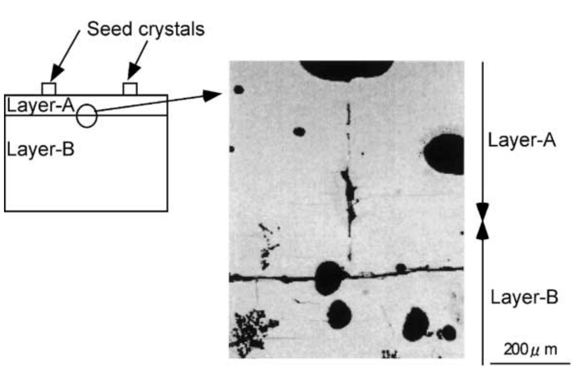
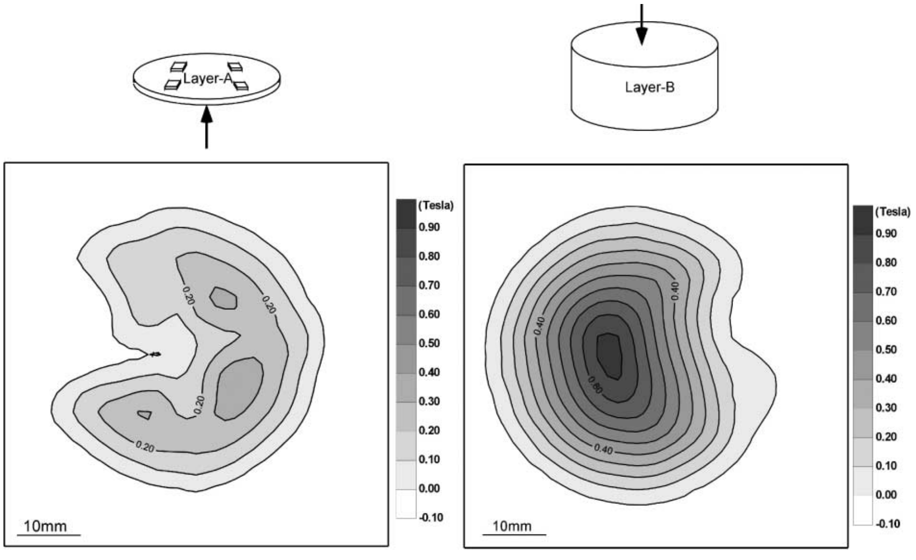


Fig. 3. Optical micrograph of a vertical cross-section of the specimen.

Fig. 4 shows the trapped field distributions for the Layer-A and the Layer-B superconductors. In the case of the Layer-A, the distribution had several separated peaks that correspond to the grains grown from the seed crystals. It is typical distribution with the multi-peaks for the conventional multi-seeded bulk superconductor [5–8,11, 12]. However, in the case of the Layer-B, the trapped field distribution had a single peak with a value of 0.9 T, which is a characteristic of the bulk superconductor without weak-links and segregations. It is an important fact that the seams of the grains grown from the seed crystals cannot be distinguished on the trapped fields distribution even if we use the four seed crystals separated by a long distance, i.e. 15 mm, for the crystal growth of the bulk superconductor.

Comparing the trapped field distributions of the Layer-A and the Layer-B superconductors, we found that the existence of the excluded phases at the grain boundary reflects the existence of the depression of the trapped field. According to Bean's law [15], the depression of the trapped field at the grain boundary reflects the reduced current transport capability across the grain boundary. An obstacle to the transport current across the boundary can be caused by the excluded phases at the grain boundary. Hence, the depression of the trapped filed is attributed to the segregations and the excluded phases at the grain boundary. In other words, the multi-seeded bulk superconductor without the excluded phases at the grain boundaries has the trapped field distribution with a single peak.

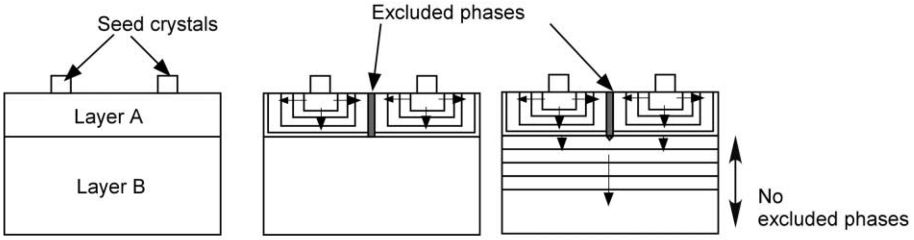
Fig. 5 shows a schematic of the precursor for the MUSLE technique. It is the basic concept of this technique that the precursor is composed of two or more RE–Ba–Cu–O layers with different peritectic temperatures. Thereby, the directions of the crystal growth from the seed crystals can be controlled. In the Layer-A, the crystal growth directions are partly opposite to each other as shown



(a)

(b)

Fig. 4. Trapped field distributions for the Layer-A on the back surface (a), and the Layer-B superconductors on the top surface (b). The arrows show the directions of view.



(a)

(b)

(c)

Fig. 5. Schematic of the specimen for the MUSLE technique: (a) one example of a structure of the MUSLE technique, (b) the schematic of the crystal growth of the Layer-A and (c) the schematic of the crystal growth of the Layer-B.

in Fig. 5(b) according to the ordinary multi-seeding method. However, in the Layer-B, these crystal growth directions can be changed to be in the same direction along the *c*-axis direction and be well combined as shown in Fig. 5(c). This phenomenon can be explained the proximity effect of the seed crystals reported by Jee et al. [6]. The proximity effect means that there are little excluded phases, i.e. liquid phases and segregated RE211 phases, at the boundary between the grains grown from the proximal seed crystals.

This proximity effect can be applied to the phenomenon in the Layer-B. When the crystal growth of the Layer-B starts, the Layer-A consists of the grains grown from the seed crystals and the excluded phases at the grain boundary. Therefore, from the viewpoint of the crystal growth in the Layer-B, the grains and the excluded phases in the Layer-A act as the proximal seeds and the gaps between the proximal seeds, respectively. Hence, in the Layer-B, the excluded phases were made to disappear at the grain boundary by the proximity effect.

As mentioned above, the MUSLE technique opens the way to fabricate the multi-seeded bulk superconductor with a single peak on the trapped field distribution. Furthermore, since this technique is a simple method of using the layers with the different peritectic temperatures in the precursor, it can be widely applied to the fabrication of large-sized and various-shaped bulk superconductors in a short time.

4. Conclusion

In order to improve the problem of the conventional multi-seeding method, i.e. the existence of the excluded phases at the grain boundaries in the bulk superconductor, we have developed a new multi-seeding method, the MUSLE technique for fabricating RE–Ba–Cu–O bulk superconductors. It is the basic concept of this technique that the precursor is composed of two or more RE–Ba–Cu–O layers with different peritectic temperatures. Thereby, the directions of the crystal growth from the seed crystals can be controlled. We found that the MUSLE technique is effective in eliminating the excluded phases at the grain boundaries for the multi-seeded bulk superconductor. We suggest that the elimination of the excluded phases can be explained by the seed crystal proximity effect, i.e. there are little liquid phases and segregations at the boundary between the grains grown from the proximal seed crystals.

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