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Size dependence of the trapped magnetic field in large single-grained Gd-Ba-Cu-O bulk superconductors

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Abstract. Based on careful consideration to crystal growth mechanism, three different sizes of Gd-Ba-Cu-O cylindrical bulk superconductors have been fabricated. The trapped magnetic field proportionally increased with the sample size and the peak heights at 77 K were 0.91-0.95 T, 1.38-1.51 T and 1.91-2.03 T for samples 30 mm, 46 mm and 64.5 mm in diameter, respectively. In addition, macroscopic \(J_c\) of approximately 13,000 A/cm\(^2\) was estimated for all samples with different sizes. Such a high value of macroscopic \(J_c\) and the linear increase of trapped fields with size can be ascribed to excellent quality of large single-grained Gd-Ba-Cu-O bulk superconductors.

1. Introduction
High temperature superconducting (HTS) bulks of RE-Ba-Cu-O (RE: Y or rare earth elements) are highly attractive from a practical viewpoint because of their strong pinning force and a variety of interesting bulk applications have been proposed. In order to realize bulk applications, large single-grained HTS bulks with high critical current density, \(J_c\), are indispensable. Regarding enlargement of a single-grained HTS bulk crystal, a breakthrough was achieved by Morita et al. shortly after the discovery of Y-Ba-Cu-O: control of nucleation and crystal orientation by seeding [1, 2]. This seeding technique is very useful and has been widely utilized for fabricating single-grained HTS bulks in many laboratories around the world. The values of \(J_c\) are usually evaluated for small specimens 1-2 mm in size and these \(J_c\) values are defined as microscopic \(J_c\) in this paper. Microscopic \(J_c\) values higher than 10,000 A/cm\(^2\) at 77 K and 1 T are often reported and in most cases they are for small specimens cut from HTS bulks 20-30 mm in size. It is well-known that, according to the Bean model, the trapped field capability is proportional to the product of their single-domain size and \(J_c\). Assuming that \(J_c\) is constant, the peak height of the trapped field should be proportional to the sample size. There were, however, few experimental reports on the size dependence of the trapped field so far. This is probably because it is still difficult to produce large single-grained HTS bulks with high \(J_c\) kept constant, due to undesirable nucleation and deterioration, especially for HTS bulks larger than 30 mm.

In this paper, in order to experimentally demonstrate the relationship between the trapped field and the sample size, we have fabricated and investigated three different sizes of Gd-Ba-Cu-O cylindrical bulk superconductors: 30 mm, 46 mm and 64.5 mm in diameter.

2. Crystal growth mechanism of RE123 bulk crystals with much excess RE211
It is very important to understand the complicated crystal growth mechanism of a REBa\(_2\)Cu\(_3\)O\(_x\) (RE123) crystal with much excess RE\(_2\)BaCuO\(_5\) (RE211) inclusions finely dispersing in the
superconducting matrix, in order to fabricate HTS bulks of good quality, especially for large samples. It is known that the RE123 phase is produced by the following peritectic reaction:

\[
\text{RE211} + \text{L} \rightarrow \text{RE123} + \text{excess RE211}
\]

where L denotes a liquid phase consisting BaCuO\(_2\) and CuO. Crystal growth is generally limited by interface kinetics, mass transport and heat transport. Since HTS bulk crystals exhibit faceted growth, it is no doubt that the effect of interface kinetics is essential in these materials. Both mass and heat transports are also important for stable growth of the faceted interface.

First, as for mass transport, the supply of RE elements into the growing interface is crucial for steady crystal growth. A model that RE ions dissolving in the liquid phase move toward the growing interface can be considered and this model may be true for crystal growth with no or little excess RE211 phase, but the flux of dissolving RE ions alone is not sufficient for the growth of large HTS bulks since the solubility of RE ions in the melt is very small, a few % or less. In the growth of HTS bulks containing much excess RE211 phase of a few tens %, another model should be proposed that RE211 particles trapped on the growing interface play a much more important role on the supply of RE elements into the growing interface. In this model, RE211 particles trapped on the growing front are decomposed at the three phase interface between the liquid phase, RE211 and RE123 and, as shown in Figure 1, the resultant RE ions move along the interface plane for a certain time determined by interface kinetics. Some of them are incorporated into the growing interface at kinks and others are dissolved into the liquid. According to this model, it is possible to supply a sufficient amount of RE element to the growing interface even for large HTS bulks. This model also predicts that stable faceted interface is difficult to grow in the case that the distribution of excess RE211 particles is not uniform, leading to instability in crystal growth. It is thus particularly important to enhance the uniformity of excess RE211 phase distribution inside the precursor for producing large HTS bulks of good quality.

Next, as for heat transport, the internal temperature gradient at solid/liquid interface is important for stable faceted growth. Compared with the positive temperature gradient where the liquid phase temperature is higher than the solid phase one, it is more difficult to keep the solid/liquid interface flat due to the Mullins-Sekerka instability in the case of negative temperature gradient. Although positive temperature gradient models based on directional solidification are often proposed for HTS bulks, the internal temperature gradient is much different. In the HTS bulk melt-process, the internal temperature gradient is negative rather than positive, because the entire precursor including both the crystallized and liquid parts is slowly cooled during the crystal growth step and the crystallized phase temperature rises due to the latent heat generated at the growing interface. The HTS bulk melt-process therefore requires more careful heat treatments for stable faceted growth, especially for larger samples.

![Figure 1](image.png)

**Figure 1.** (a) Schematic diagram, (b) RE concentration and (c) temperature near the interface.

### 3. Sample preparation

Large single-grained Gd-Ba-Cu-O bulk superconductors were prepared by a melt-growth process called the modified QMG method. QMG originally stood for “Quench and Melt-Growth”, but in the
modified QMG process the quench step is eliminated by employing a small amount of Pt or CeO₂ addition into the starting materials [2] and thus “Q” means “Quality” at present instead of “Quench.

In order to enhance the uniformity of excess RE211 phase distribution within the precursor, very careful attention was paid to the precursor preparation. First, appropriate amounts of high purity Gd₂O₃, BaO₂ and CuO powders were ground and mixed well with 0.5 wt% of Pt. These mixed powders were calcined at about 1173 K and then pulverized. This calcination process was repeated twice to ensure complete reaction. Next, separately-calcedined powders of Gd123 and Gd211 were ground and blended well in a molar ratio of Gd123:Gd211=3:1 with 10 wt% Ag₂O. This mixture process was also repeated three times to ensure complete mixing. Pt was added for the refinement of Gd211 particles and Ag was for improving the fracture strength properties. Finally, the well-mixed powders were pressed into three different sizes of cylindrical precursors.

Special care was also taken of the heat treatment for crystal growth. First, the precursor was heated to a relatively high temperature, approximately 1443 K, for a certain length of time in order to transform the whole precursor into the partially-melted state completely. If part of the precursor is incompletely melted, undesirable nucleation occurs and the sample becomes poly-grained, not single-grained. Next, the melted precursor was rapidly cooled to about 1310 K and the seed crystal of (Nd, Sm)-Ba-Cu-O was placed on the top of the precursor. The precursor was then cooled to the point just above the peritectic temperature, followed by slow cooling to 1245 K for crystal growth in air. The as-grown samples were machined into three different sizes of Gd-Ba-Cu-O cylindrical bulk superconductors: 30 mm, 46 mm and 64.5 mm in diameter, as shown in Table 1. Finally, the samples were annealed for 100 h at 673 K in flowing oxygen gas.

| Sample No. | Diameter (radius) | Thickness | Measured peak height | Corrected for r/t =1.5 |
|------------|-------------------|-----------|----------------------|-----------------------|
| A-1        | 30 mm (15 mm)     | 10 mm     | 0.91 T               | 0.91 T                |
| A-2        | 46 mm (23 mm)     | 15 mm     | 0.95 T               | 0.95 T                |
| B-1        | 64.5 mm (32.25 mm)| 20 mm     | 1.38 T               | 1.38 T                |
| B-2        |                   |           | 1.51 T               | 1.51 T                |
| C-1        |                   |           | 1.94 T               | 1.94 T                |
| C-2        |                   |           | 2.03 T               | 2.03 T                |

4. Results and discussion

Figure 2 shows the appearance and the trapped field distribution at 77 K for three different sizes of Gd-Ba-Cu-O. The samples were field-cooled under an applied field of 3.5 T and the trapped fields were measured by scanning a Hall sensor two-dimensionally at a gap of 0.6 mm above the sample surface. As shown in Figure 2(b), each sample exhibited a symmetric trapped field distribution with a single peak, indicating that the sample was a single crystal with no serious weak-links. In other words, stable crystal growth continued to the sample edge even in large HTS bulks 64.5 mm in diameter. The peak values of the trapped field at 77 K were 0.91-0.95 T, 1.38-1.51 T and 1.91-2.03 T for samples 30 mm, 46 mm and 64.5 mm in diameter, respectively. Figure 3(a) displays the relationship between the measured values of the peak height and the sample diameter, and the peak height increases linearly with the diameter. This result experimentally demonstrates that the trapped field ability proportionally increases with the sample size up to at least 64.5 mm.

![Figure 2](image-url)  
**Figure 2.** (a) Appearance of Gd-Ba-Cu-O bulks and (b) the trapped field distributions at 77 K.
The Bean model simply predicts that the peak height of the trapped magnetic field is proportional to the sample size in a one-dimensional model, but it is more appropriate to use a two-dimensional model for real HTS bulks. For a cylindrical HTS bulks with a finite thickness assuming a field-independent $J_c$, the peak height of the trapped field, $B_p$, is described as follows [3]:

$$B_p = \frac{\mu_0 J_c t}{2} \ln \left( \frac{r}{t} + \sqrt{1 + \left(\frac{r}{t}\right)^2} \right)$$

where $\mu_0$ is the magnetic permeability in air, $r$ the radius and $t$ the thickness. According to the above equation, the peak height is strictly proportional to the sample size only in the situation where the aspect ratio, $r/t$, is constant. The aspect ratio of the samples used in this study was not constant as shown in Table 1 and thus we should correct the peak height. Open circles in Figure 3(b) are the corrected values for $r/t = 1.5$ and it seems that the linearity is slightly improved.

In addition, $J_c$ can be estimated from the trapped magnetic field by using the above equation. This $J_c$ value is defined as macroscopic $J_c$ in this paper because it is calculated on the assumption that the superconducting current flows throughout the whole sample. The estimated vales of macroscopic $J_c$ are approximately 13,000 A/cm$^2$ at 77 K for all samples with different sizes. It is amazing that there are no large differences in macroscopic $J_c$ between different-sized HTS bulks and that these macroscopic $J_c$ value are as high as the microscopic $J_c$ ones reported so far. Such a high value of macroscopic $J_c$ and the liner increase of trapped fields with size can be attributed to good quality of large single-grained Gd-Ba-Cu-O bulk superconductors.

![Figure 3](image)

Figure 3. Relationship between trapped field and sample diameter of Gd-Ba-Cu-O bulks: (a) before correction (measured values) and (b) after correction (aspect ratio $r/t = 1.5$).

5. Summary
We have fabricated three different sizes of Gd-Ba-Cu-O cylindrical bulk superconductors, based on careful consideration to crystal growth mechanism. The trapped field ability proportionally increased with the sample size up to at least 64.5 mm and macroscopic $J_c$ of approximately 13,000 A/cm$^2$ at 77 K was estimated for all samples with different sizes. High quality of large single-grained Gd-Ba-Cu-O bulk superconductors is responsible for such a high value of macroscopic $J_c$ and the liner increase of trapped fields with size.

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