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Letter

Facile preparation of large Mg-NdBCO crystal for seeding growth of high-performance YBCO single-grain superconductor

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ABSTRACT

Multi-grain Mg-doped Nd–Ba–Cu–O (Mg-NdBCO) bulk was facilely prepared by employing just one precursor pellet of composition $Nd_{1.6}Ba_{2.3}Cu_{3.3}O_x + 1$ wt% MgO. The sample exhibits typical large grains up to 15 mm in dimension, and a series of large Mg-NdBCO crystals of size 5×5 mm^2 can be cleaved from the multi-grain sample. Single-grain Y–Ba–Cu–O (YBCO) superconductor of 16 mm in diameter has been successfully grown on the seeding of the Mg-NdBCO large crystal by the infiltration growth (IG) technique, which presents superior performances of levitation force (32.17 N) and trapped field (0.3962 T) compared with the sample grown with a small seed. As a result, by combining the large Mg-NdBCO seed with the novel IG technique we proposed, a practical method for fabricating high-performance YBCO bulk superconductors has been developed.

1. Introduction

Bulk high temperature superconductors RE–Ba–Cu–O (REBCO, where RE is a rare-earth element such as Y, Nd, Sm, Eu, Gd, etc.) have been developed over the past decades for practical applications, such as magnetic levitation, flywheel energy storage systems, motors and high field magnets [1–4]. As is proved, these materials are required in the form of large, single grains without weak links for higher performances [5]. Thus the top seeding technique is employed for inducing the heterogeneous nucleation and directional growth of REBCO individual grain to a large scale [6]. And typically, the so-called top-seeded melt growth (MG) technique [7] and the top-seeded infiltration growth (IG) technique [8] are the two of the most popular processes.

In practice, the top-seeding method consists of two processing routes, i.e. the hot-seeding [9] and the cold-seeding [10]. For hot-seeding route, the seed must be put on the top of the pellet just after the high-temperature melting stage and before the slow-cooling growth stage. The major advantage of the hot-seeding is that the seed crystal has not to suffer the high-temperature melting stage, so its stability and seeding ability are not affected. But because the operation is carried out when the furnace is at high temperatures above 1000 °C, it is dangerous for the operators and is also very difficult to put the seed just at the centre of the pellet surface. While for the cold-seeding, the seed is put on the top of the preform at room temperature initially, so it is safer and can be easily performed. But subsequently, the seed will undergo the same high-temperature melting process with the preform, so its thermostability and chemical stability are challenged. As can be seen, the cold-seeding requires the seeds to have higher qualities.

Since the seed initiates the nucleation and directional growth of the REBCO grain, the quality of the seed directly determines the growth quality and hence the superconducting performance of the final REBCO bulks. Even though each seed is selected carefully, the growth failure of single-grain sample due to an inferior seed is still inevitable in practice, which directly leads to the failure of the whole experiment. Consequently, development of new, superior seeds or novel seeding techniques is crucial during the progress of bulk REBCO materials. Yao et al. have reported that the NdBCO thin films grown on MgO substrate can be used as cold seeds for growing REBCO grains [11], and recently they reported the successful seeding growth of YBCO grains employing different-sized NdBCO films (e.g. 2×2, 5×5, and 9×9 mm^2) [12]. As is indicated, the large-sized seed results in a larger *c*-growth sector (*c*-GS) in the final bulk sample, which tends to be beneficial for achieving a higher performance (such as levitation force and trapped field). But on the other hand, the large-sized seed covers a larger area on the sample surface, which makes the releasing of gas from the pellet more difficult and results in a high pore density in the microstructure. As a result, the bulk performance presents an ascending and subsequently descending tendency with the increasing seed size, and the sample with a seed of size 5×5 mm^2 exhibits highest properties. The relatively high pore density has also been observed in the bulk sample covered with a large buffer layer [13], so the negative effect of a large seed/covering on the microstructure of the final bulk indeed exists and should be considered by the researchers during designing and optimizing the experiments.

Considering that high quality NdBCO thin films can not be home made by the bulk superconductor groups and need to be supplied at a high expense by the researchers working on thin-film

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fabrication, we still expect that such large seeds can be self made on the basis of the conventional NdBCO crystal. In this study, we demonstrate a practical approach for facile preparation of large Mg-doped NdBCO (Mg-NdBCO) seed crystals, and then high-performance YBCO single-grain bulk superconductor was successfully grown by the large seed. The addition of Mg can increase the melting point and chemical stability of the NdBCO seed [14,15].

2. Facile preparation of large Mg-NdBCO crystal

The powders of Nd_2O_3, BaO and CuO were mixed thoroughly in a molar ratio Nd_2O_3:BaO:CuO = 0.8:2.3:3.3, then sintered at 920 °C for 30 h to prepare the compound of nominal composition $Nd_{1.6}Ba_{2.3}Cu_{3.3}O_x$ (named as Nd-1.6 powder for short, corresponding to a equivalent composition: $NdBa_2Cu_3O_{7-\delta}$ (Nd-123) + 0.15 $Nd_4Ba_2Cu_2O_{10}$ (Nd-422)). Powders of Nd-1.6 + 1 wt% MgO were well mixed using a ball milling machine, and then pressed into pellet of diameter 26 mm under a pressure of 200 MPa. The pellet was then mounted on five MgO single crystals, which themselves were placed on an alumina plate. As is indicated, only a precursor pellet is employed here, because no substrate is required to prevent nucleations caused by MgO crystals since we aim to obtain a multi-grain NdBCO bulk.

The sample was heated to 920 °C at a rate of 240 °C/h and held for 10 h, then further heated to 1140 °C at a rate of 60 °C/h and held for 1 h, then cooled to 1090 °C at a rate of 60 °C/h, then cooled slowly at a rate of 0.5 °C/h to 1040 °C, and finally furnace-cooled to room temperature, as shown in Fig. 1(a).

Fig. 1(b) shows the top surface morphology of the melt grown Mg-NdBCO bulk by employing just one pellet of Nd-1.6 + 1 wt% MgO. It is clear that the sample exhibits typical multi-grain morphology, because no seed was used for the Mg-NdBCO bulk growth and the grown grains were indeed induced by the random, spontaneous nucleations. It is noticed that the diameter of the bulk shrinks to about 21 mm, i.e. about 20% shrinkage ratio compared with the initial preform (26 mm), which is commonly observed in the MG process. Different from the former studies on fabricating Mg-NdBCO bulks [14–16], in which only small grains up to a few mm sizes were obtained, the Mg-NdBCO bulk in this work presents two absolutely large grains with a size up to 15 mm. This may be result from the different processing details we used, such as employment of Nd-1.6 powder, a larger preform and a prolonged slow-cooling growth process (100 h). The large grains were cut from the bulk sample, then roughly crushed, and finally a series of large Mg-NdBCO crystals up to 5 × 5 mm^2 in size were successfully cleaved.

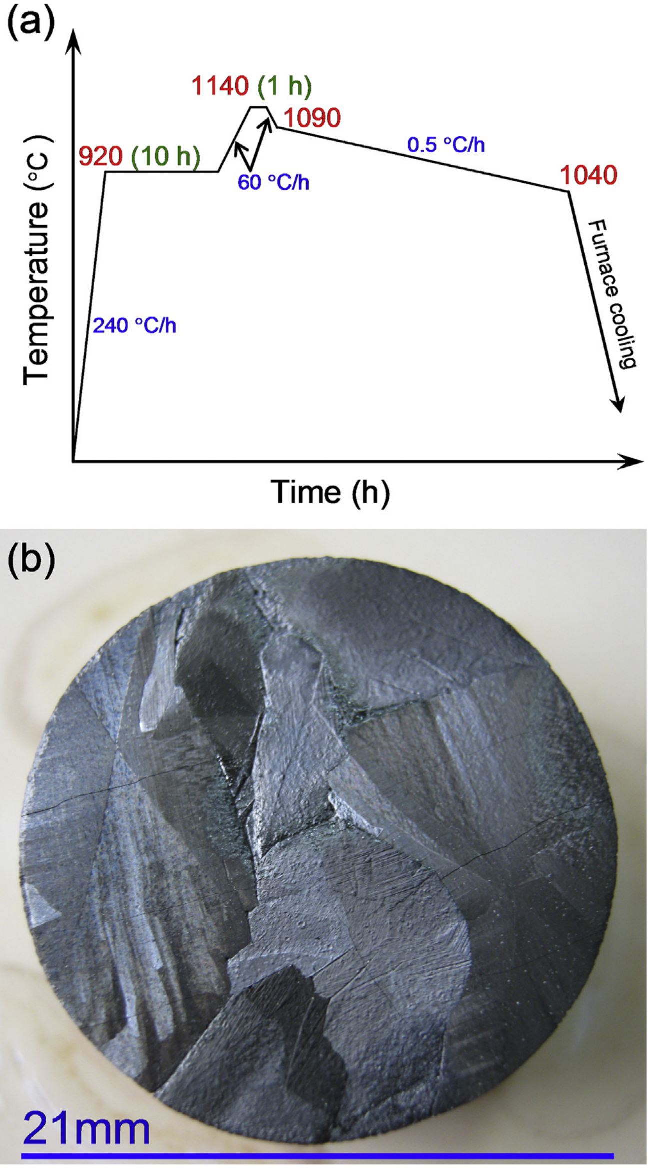

Fig. 1. (a) Heat treatment profile used for the melt growth of Mg-NdBCO bulk. (b) Top surface morphology of the as-grown Mg-NdBCO multi-grain sample.

3. Growth and characterization of single-grain YBCO superconductor using Mg-NdBCO large seed

The fabrication process of single-grain YBCO superconductor was performed by employing a novel IG technique we reported earlier [17], which can accomplish the introduction of nano-sized Y_2BaCuO_5 (Y-211) inclusions in $YBa_2Cu_3O_{7-\delta}$ (Y-123) superconducting matrix. The difference only exists in the size of the seed, i.e. large seed of 5 × 5 mm^2 is used here, while in that study a small seed of size about 2 × 2 mm^2 was used.

Fig. 2 shows the top surface morphology of the as-grown YBCO bulk of diameter 16 mm and height about 8.2 mm seeded by the large Mg-NdBCO crystal. As can be seen, the sample has been grown in the form of a single grain, which proves the reliable seeding ability of the large Mg-NdBCO crystal, and also indicates that our facile method for preparing Mg-NdBCO seeds is successful. In addition, as is known, it is the bottom surface of the seed that promotes the nucleation and epitaxial growth of the YBCO grain, so a large seed can effectively reduce the growth distance from the edge of the seed to the edge of sample (i.e. growth process in the *a*-axis direction), so the required growth time could also be reduced and the YBCO grain orientation within the sample (especially at the edge position) can be controlled increasingly [18,19]. Moreover, the large seed has better thermostability than the small and thin seeds, so we can expect that this kind of Mg-NdBCO large seed will play an important role on the fabrication process of large YBCO samples with diameters above 50 mm, in which a prolonged high-temperature melting stage is required.

The superconducting properties of the whole sample including levitation force and trapped field were measured at the liquid nitrogen temperature by a self-designed 3D magnetic force & field measuring device [20]. Fig. 3 presents the levitation force as a function of the distance between the sample and a permanent magnet ($\phi = 18$ mm, $B = 0.5$ T) for the large-seed YBCO bulk superconductor. As can be seen, the sample achieves a maximum levitation force $F_{L,max}$ of 32.17 N at the smallest separation of about 0.5 mm with the