Fabrication and Characterization of Melt-Processed YBCO

RECEIVED

OCT 30 1996

OSTI

CONF-960850--16

ANL/MSD/CP--91355

S. Sengupta, J. Corpus, J.R. Gaines, Jr.
Superconductive Components, Columbus, Ohio

V.R. Todt, X.F. Zhang, D.J. Miller
*Materials Science Division and the Science and Technology Center for Superconductivity
Argonne National Laboratory, Argonne, IL 60439*

and

C. Varanasi, and P.J. McGinn
Department of Chemical Engineering, University of Notre Dame, Indiana

The submitted manuscript has been created by the University of Chicago as Operator of Argonne National Laboratory ("Argonne") under Contract No. W-31-109-ENG-38 with the U.S. Department of Energy. The U.S. Government retains for itself, and others acting on its behalf, a paid-up, non exclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, by or on behalf of the Government.

**1996 Applied Superconductivity Conference, August 25-30, 1996, Pittsburgh, PA, to appear in
IEEE Transactions on Superconductivity, Spring 1997.**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Work supported by U.S. Department of Energy, Division of Basic Energy Sciences-Materials Sciences, Conservation and Renewable Energy - Advanced Utility Concepts - Superconducting Technology Program, under contract #W-31-109-ENG-38; by the National Science Foundation Office of Science and Technology Centers for Superconductivity under contract DMR #91-20000, and DOE Grant DE-FG02-ER45427 through Midwest Superconductivity Consortium at University of Notre Dame.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Fabrication and Characterization of Melt-Processed YBCO

S. Sengupta, J. Corpus, and J. R. Gaines, Jr.
Superconductive Components, Columbus, Ohio

V. R. Todt, X. F. Zhang and D. J. Miller

Materials Science Division and Science and Technology Center for Superconductivity, Argonne National Laboratory, Argonne, Illinois

C. Varanasi and P. J. McGinn

Department of Chemical Engineering, University of Notre Dame, Indiana

Abstract — Large domain YBCO are fabricated by using a melt processing technique for magnetic levitation applications. A $\text{Nd}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_y$ seed is used to initiate grain growth and to control the orientation of YBCO grains. Samples as large as 2 inch have been fabricated by utilizing this method. Microstructural studies reveals two distinct regions in these levitators due to different growth mechanism along a/b and c axis. Some initial result on the mass production of these levitators are also reported.

I. INTRODUCTION

The stable levitation or suspension of a magnet is a fascinating property of a superconductor. A magnet can be levitated above or suspended below a superconductor and vice versa. A large number of applications are envisioned using this unique property. These applications include rotary motion bearings, cryo-coolers, cryo-flowmeters, energy storage devices, contact less transportation, and vibration isolators [1].

For a given magnet and for superconductors with similar dimensions, the levitation force depends on the critical current density, J_c , and the length scale of the induced current loop, d in the superconductor. The melt processing technique offers an attractive way to fabricate $\text{YBa}_2\text{Cu}_3\text{O}_x$ (YBCO) that can strongly levitate a magnet [2]-[7]. In a typical melt processing method, YBCO is heated above its peritectic point where it melts incongruently into Y_2BaCuO_5 and a Ba- and Cu-rich liquid. The semi-solid melt is cooled slowly to obtain aligned grains of YBCO or domains. In order to achieve high levitation forces, large domain size which determines the d , and high J_c is desirable. One way to increase the domain size is by initiating grain growth by using a seed crystal. In presence of a favorable temperature gradient the seed not only ensures a single nucleation site but also permits controlled orientation of the grains. Using the seeding technique along with a controlled temperature gradient YBCO domains as large as that of the sample size (single domain) can be fabricated.

Manuscript received August 27, 1996.

The work at Argonne National Laboratory is partially supported by the US Department of Energy, Division of Basic Energy Science-Materials Sciences, Conservation and Renewable Energy Utility Concepts-Superconductivity Technology program, under Contract # W-31-109-ENG-38 and by NSF(DMR91-20000) through Science and Technology Center for Superconductivity. The work at University of Notre Dame is supported by DOE Grant DE-FG02-90-ER45427 through Midwest Superconductivity Consortium.

Fig. 1. Photograph of a large domain YBCO levitator with a diameter of 2 inch. The edge of the sample was coated with a thin layer of Yb_2O_3 in order to minimize nucleation from the edges.

Seeding with a single crystal of MgO , Al_2O_3 , $\text{SmBa}_2\text{Cu}_3\text{O}_x$ and $\text{Nd}_{1-x}\text{Ba}_2\text{Cu}_3\text{O}_y$ have been reported by various groups [2]-[7]. The seed can be added to the coldest point of the sample prior to heating or after an extra melting step. The addition of the seed prior to heating is preferable as it reduces the difficulty of mass production. The critical current density, J_c , is generally controlled by introducing defects like Y_2BaCuO_5 inclusions and structural defects like dislocations, stacking faults etc. during melt processing.

In this paper, some of our recent progress in fabricating large domain levitators is reported. We also report the development of microstructure and some of our initial results on the feasibility of large scale manufacturing of these samples.

II. EXPERIMENTAL METHOD

Phase pure $\text{YBa}_2\text{Cu}_3\text{O}_x$ and Y_2BaCuO_5 powders were prepared by using a low pressure calcination method. Typically, in a low pressure calcination process, the required oxides and carbonates are mixed and ball milled in a proper molar ratio. The resulting powders are then calcined in a low pressure furnace to produce the desired phase. An oxygen atmosphere with pressure of 2-5 torr is used during calcination. A partial vacuum is used to increase the efficiency of removal of CO_2 . Once the reaction is completed, the vacuum is discontinued and the powders are cooled in an oxygen atmosphere at ambient pressure to fully oxygenate the superconducting powders. The low pressure calcination method is

beneficial in reducing the carbon content and processing time while maintaining small particle size [8].

The $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_y$ single crystals which were used as seeds are grown using standard flux growth method. The details have been reported earlier [6].

$\text{YBa}_2\text{Cu}_3\text{O}_x$, Y_2BaCuO_5 (~20-25 wt %) and PtO_2 (0-0.5 wt %) were mixed homogeneously by ball milling overnight (~8-10 hr.). The mixed powder were then pressed in to a desired shape and seeded with a $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_y$ single crystal and melt-processed in a temperature gradient as reported earlier [6]. In brief, the samples are heated to about 1050 °C and then slowly cooled through the peritectic temperature to about 950 °C and then finally cooled to the room temperature at a rate of 100 °C/hr.. The samples (levitators) are then oxygenated between 450 -700 °C for about 8-10 days in a flowing oxygen atmosphere.

Microstructural development was investigated by using a scanning electron microscopy (SEM), with a energy dispersive x-ray spectroscopy (EDS) and optical microscopy.

III RESULTS AND DISCUSSIONS

Although, considerable progress in increasing the size of the domain in melt-processed YBCO has been achieved through seeding methods, the diameter of the domain is typically limited to roughly about 1.5 inches. Only with sizable temperatures gradient and/or compositional gradient larger samples can be produced. This size limitation is believed to be in part due to the high supercooling along the direction of growth, which often leads to disorderly production of nuclei at the later stage of crystal growth resulting in multi-dominated YBCO and partly due to the lower temperature at the edges of the sample where grain nucleation can occur resulting in multiple impinging domains. The nucleation at the edges of the sample can be minimized by coating the edges with Yb_2O_3 . Because of the considerably low melting temperature of $\text{YbBa}_2\text{Cu}_3\text{O}_x$ (~920 °C), the edge nucleation can be reduced. Figure 1 shows a photograph of a 2 inch diameter YBCO levitator fabricated by coating the edges of the sample with Yb_2O_3 .

Due to the differences in the growth mechanism in the a/b axis and the c-axis two distinct microstructures results in seeded YBCO levitators. The nucleation of the $\text{YBa}_2\text{Cu}_3\text{O}_x$ phase at the surface of the $\text{Nd}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_y$ single crystal occurs after the slow cooling process has started. The crystal begins growing epitaxially in form of a thin, square platelet, with well developed (100), (010) and (001) planes. The pedestal that develops below the seed is formed by well defined growth steps. These steps are relatively high close to the seed but much less pronounced at the edge of pellet. The growth stops when the growth front reaches the reaction layer that forms at the interface between the crucible and the sample (Fig. 2). The reaction layer is usually 1-3 mm thick and consists of a large amount of Y_2BaCuO_5 embedded in Ba-Cu-O phases. Small grains of $\text{YBa}_2\text{Cu}_3\text{O}_x$ and some complex Y-Ba-Cu-Al-Pt-O phases are also observed in these regions.

As mentioned earlier, two distinct regions due to the growth in a/b axis and c axis are observed in these levitators. Just under the seed the region grows mainly due to the growth along the c-axis. In this region, generally a pyramid shaped volume (Fig. 3) develops with low Y_2BaCuO_5 concentration (Fig 4) and a blocky sub-domain structure (Fig. 5). The sub-domains are mostly square-like shape with sizes up to 1 mm². The c-axis misalignment between adjacent sub-domains is usually well below five degree. The volume fraction

Fig. 2. Vertical cross-section of a YBCO levitator. A thin reaction zone can be seen at the bottom of the sample.

Fig. 3. Vertical cross section of a YBCO levitator under a polarized optical microscope at a magnification of 50 X.

Fig. 4. Microstructure of the c-axis growth region showing the distribution of Y_2BaCuO_5 inclusions.

of the Y_2BaCuO_5 phase in this area directly below the seed is usually around 10 vol. % and increases with decreasing height.

Surrounding the inner region (the pyramid shaped region) the solidification mostly occurs by ab plane growth. The transition from the c-axis growth to the ab plane growth region is well defined, as can be revealed by the abrupt change in Y_2BaCuO_5 density. This region shows a high Y_2BaCuO_5 concentration and a strip - sub-domain structure (Fig. 5). The strip like sub-domain structure consists mostly of rectangular sub domains of very small sizes (<0.1 mm²). The volume fraction of Y_2BaCuO_5 in this area

Fig. 5. Sub-domain structure of c-axis and a/b axis growth region as observed by a polarized optical microscope at magnification of 200X.

Fig. 6. Microstructure of the a/b axis growth region showing the distribution of Y_2BaCuO_5 inclusions.

is usually close to the starting volume fraction with high volume fractions near the edge of the sample (Fig. 6).

Large domain YBCO levitators with various shapes and dimensions are now routinely fabricated. These levitators can be machined to desired shapes without significantly deteriorating its properties. In order to demonstrate, the feasibility of mass production about 90 samples has been recently fabricated in one furnace. These samples are hexagonal in shape and about 0.8 inch wide from side to side and 0.45 inch thick. Only on two samples the seed diffused during the melt during processing. Figure 7 shows the distribution of the zero-distance levitation force for these samples. Although, levitation forces are sufficient for various practical purposes, further optimization is necessary to obtain results similar to that of smaller batch sizes.

IV CONCLUSION

Large domain YBCO levitators are produced by melt processing techniques using a $Nd_{1-x}Ba_xCu_3O_y$ seed to initiate the grain growth. Two distinct regions resulting from the two different growth mechanism are observed in the microstructure. Industrial scale manufacturing of these levitators with levitation forces sufficient for various practical purposes seems feasible.

Fig. 7. Distribution of the zero distance levitation forces in a single furnace run.

ACKNOWLEDGMENTS

We would like to thank Dr. John Hull at Argonne National Laboratory for measuring the levitation forces.

REFERENCES

- [1] F. C. Moon, *Superconducting Levitation, Applications to Bearings and Magnetic Transportation*, 1st ed., pp 198-212, March 1994.
- [2] M. Morita, L. Trouilleux, S. Takebayashi, K. Kimura, M. Tanaka, K. Miyamoto and M. Hashimoto, "Growth of QMG Crystals," Proc. Int. Workshop on Superconductivity, Honolulu, pp. 119-122, June 1992.
- [3] K. Y. Blohowiak, D. F. Garrigus, T. S. Luhman, K. E. McCrary, M. Strasik, I. A. Aksay, F. Dogan, W. B. Hicks, J. Liu, and M. Sarihaya, "Evaluation of $YBa_2Cu_3O_{7-x}$ Bulk Superconductors For High Field Magnet Applications," IEEE Trans. Appl. Supercond. 3, pp. 1049-1052, 1993.
- [4] M. Murakami, T. Oyama, H. Fujimoto, T. Taguchi, S. Gotoh, Y. Shiohara, N. Koshizuka, and S. Tanaka, "Large Levitation Force due to Flux Pinning in YBCO Superconductors Fabricated by Melt-Powder-Melt-Growth Process," Jpn. J. Appl. Phys. vol. 29, Nov. 1990, pp. L1991-L1994.
- [5] R. Weinstein, I.-G. Chen, J. Liu, D. Parks, V. Selvamanikam, and K. Salama, "Persistent Magnetic Fields Trapped in High T_c Superconductor," Appl. Phys. Lett. vol. 56, pp. 1475-1477, April 1990.
- [6] V. R. Todt, S. Sengupta, D. Shi, J. R. Hull, P. R. Sahm, P. J. McGinn, R. B. Poeppel, "Processing of Large $YBa_2Cu_3O_x$ - Domains for Levitation Applications by $NdBa_2Cu_3O_x$ -Seeded Melt-Growth Technique," J. of Electronic Materials, vol. 23, pp. 1127-1130, Feb. 1994.
- [7] U. Balachandran, W. Zhong, C. A. Youngdahl and R. B. Poeppel, "Microstructure and Associated Properties of YBCO Superconductors Prepared by Melt-Processing Techniques," J. of Electronic Materials, vol. 22, pp. 1285-1288, Oct. 1993.
- [8] U. Balachandran, R. B. Poeppel, J. E. Emerson, S. A. Johnson, M. T. Lanagan, C. A. Youngdahl, D. Shi, K. C. Goretta, and N. G. Error, "Synthesis of Phase-Pure Orthorhombic $YBa_2Cu_3O_x$ under Low Oxygen Pressure," Materials Lett., vol 8, pp. 454-456, Nov. 1989.