

# Perforated monodomain $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ bulk superconductors prepared by infiltration-growth process

S. Meslin<sup>a</sup>, C. Harnois<sup>a</sup>, D. Chateigner<sup>a</sup>, B. Ouladdiaf<sup>b</sup>,  
X. Chaud<sup>c</sup>, J.G. Noudem<sup>a,\*</sup>

<sup>a</sup> CRISMAT-ENSICAEN, CNRS-UMR 6508, 6 Boulevard du Maréchal Juin, 14050 Caen Cedex, France

<sup>b</sup> ILL, 6, rue J. Horowitz, BP 156, 38042 Grenoble Cedex 09, France

<sup>c</sup> CNRS/CRETA, BP 166, 38042 Grenoble Cedex 09, France

Available online 22 April 2005

## Abstract

For various applications such as FCL, motor flywheel or bearing, . . . the core of bulk superconductors need to be fully oxygenated and some defects like cracks, pores and voids suppressed, in order that the material can carry high current densities. In order to study and minimise the above defects, we have developed a new elaboration technique.  $\text{YBa}_2\text{Cu}_3\text{O}_y$  (Y123) bulks have been prepared by combining liquid infiltration and top seed growth (ITSG) process. This process involves negligible shrinkage and an uniform distribution of Y211 inclusions. In addition, we prepare a regular perforation of the Y123 sample in view to magnify the specific surface and by then increase oxygen diffusion into the core of the material. Neutron texture analysis demonstrates the non-perturbative effect of the holes in the bulk from the orientation point of view. The advantages of the ITSG-process and of the novel perforated Y123 bulk are discussed.

© 2005 Elsevier Ltd. All rights reserved.

**Keywords:** Shaping; Grain growth; X-ray method; Composites; Superconductivity; Infiltration

## 1. Introduction

Nowadays the batch of textured Y123 materials can be processed reproducibly. But, shaping the materials after processing without introducing cracks or defects is one of the objective to solve to be able to use the bulk YBCO ceramics in some devices for practical applications. It is well known that in the conventional melt processing,<sup>1,2</sup> the Y123 phase is decomposed above its peritectic temperature, where it incongruently melts into solid  $\text{Y}_2\text{BaCuO}_5$  (Y211) and liquid phases. The melting is accompanied by the liquid phase losses and a change in the sample dimensions such as shrinkage up to 25%<sup>3</sup> and cracks.

In the infiltration-growth (IG) technique,<sup>4</sup> the molten liquid phase is allowed to fill or percolate into a starting Y211

preform. We can list many advantages to use these processing techniques such as:

- Near-net-shaped produced which are free of macrodefects, shrinkage, voids and porosities.
- Uniform distribution of the Y211 particles into the textured Y123 matrix.

In this paper, we report the preparation of Y123 bulks using the IG-process with a network of artificial holes. This new morphology seems to be an alternative to solve the mechanical properties/handling for the 2D Y123 fabric<sup>5</sup> or 3D Y123 foam<sup>6</sup> materials. On the other hand, Y123 with holes can be a good candidate for increasing interfacial flux pinning if the pores can be made sufficiently small. Many other prospects are related to the hole structure like, e.g. more efficient heat transfer, faster oxygenation and less microcracking, possibility of reinforcement and of interlocking connections, etc. . . . The development of processing methods of the perforated and textured Y123 with a high performance can open new pathways towards practical applications.

\* Corresponding author. Tel.: +33 231 45 29 15;  
fax: +33 231 95 16 00.

E-mail address: [noudem@ensicaen.fr](mailto:noudem@ensicaen.fr) (J.G. Noudem).  
URL: <http://www-crismat.ismra.fr>.

## 2. Experimental details

The samples for infiltration process have been prepared using commercial powders  $\text{Y}_2\text{BaCuO}_5$  (Y211) (SSC Neyco 3N, purity 99.9%),  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  (Y123) (SR 30 Solvay, purity 99.9%) and the  $\text{Ba}_3\text{Cu}_5\text{O}_8$  (Y035) prepared in our laboratory. A pellet of Y211 was formed isostatically to obtain a relatively dense preform free of cracks. The liquid source used for infiltration is a pellet of a mixture of Y035 and 50 wt% Y123 powders, uniaxially pressed. Sm123 was used as a seed to initiate single grain 123 nucleation. Details concerning the top seed melt-texture growth process (TSMTG) and combination of infiltration and top seed growth method (ITSG) are given elsewhere.<sup>7</sup>

## 3. Results and discussion

Fig. 1a shows an optical macrograph of the top view of a 15 mm diameter pellet. We can observe the Sm123 seed position at the center and see that, the domain grew from the seed crystal until the edges, which indicates that a single domain perforated specimen could successfully be processed

using infiltration-growth method. Fig. 1b shows the SEM pictures of the polished surface of the sample after oxygenation. The higher (left) and lower (right) magnifications have been taken in the core of the sample. One can readily see the holes of 0.8 mm and the relative dense microstructure. At high magnification, the average size of the Y211 inclusions is determined to be 2  $\mu\text{m}$  and homogeneously distributed into the Y123 matrix. According to the microstructure, we can notice that, the bulk sample seems to be free of macrocracks in contrast to the conventional melt processed samples<sup>7</sup> with particles in the range of 2–10  $\mu\text{m}$ . The small size of Y211 particles obtained without any dopant addition is clearly one of the advantages of infiltration and top seed growth method. This refinement of Y211 inclusions is considered for inducing a good flux pinning and high critical current density ( $J_c$ ). The magnetic  $J_c$  value at 77 K is estimated from the hysteresis cycle on the basis of the Bean model taking  $J_c = 20 \Delta M / [a(1 - (a/3b))]$ , where  $\Delta M$  is magnetic hysteresis measured in  $\text{emu}/\text{cm}^3$ ,  $a$  and  $b$  the sample dimensions (with  $a < b$ ) in cm. A critical current density of  $J_c \approx 23 \text{ kA}/\text{cm}^2$  at 0 T is calculated. This value is similar to those of single domain bulks with a non-optimized Y211 content and size distribution. There is a lot of scope

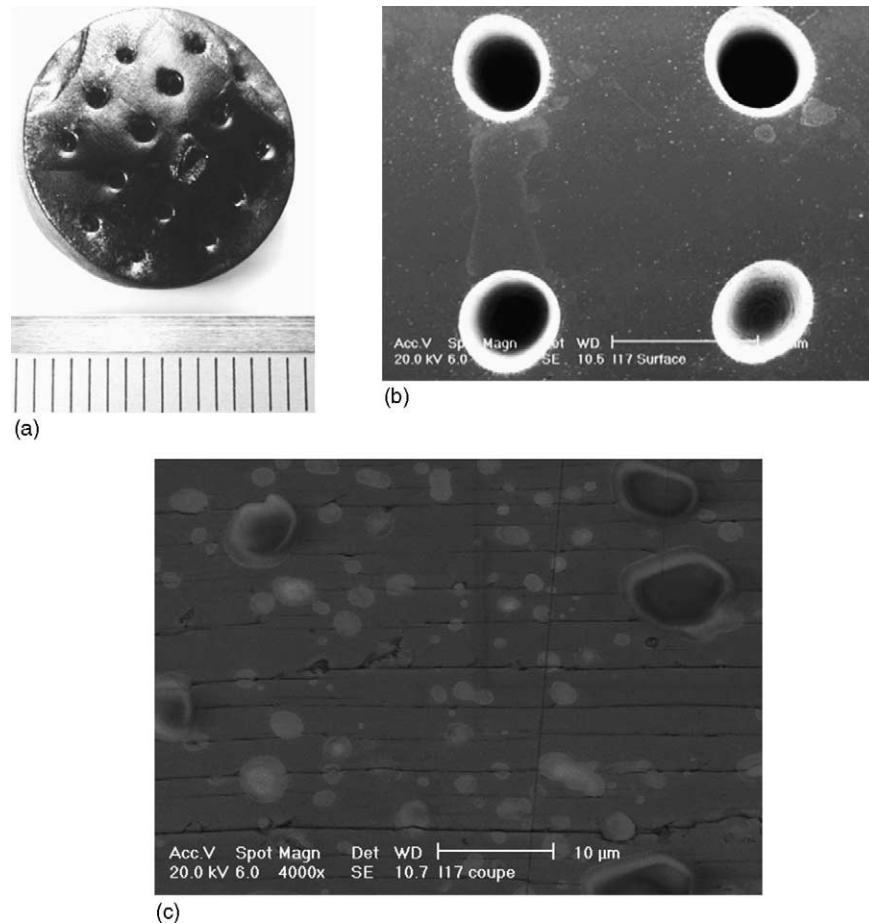


Fig. 1. (a) Textured Y123 as-processed, (b) and (c) microstructures showing the holes and Y211 particles into Y123 matrix.

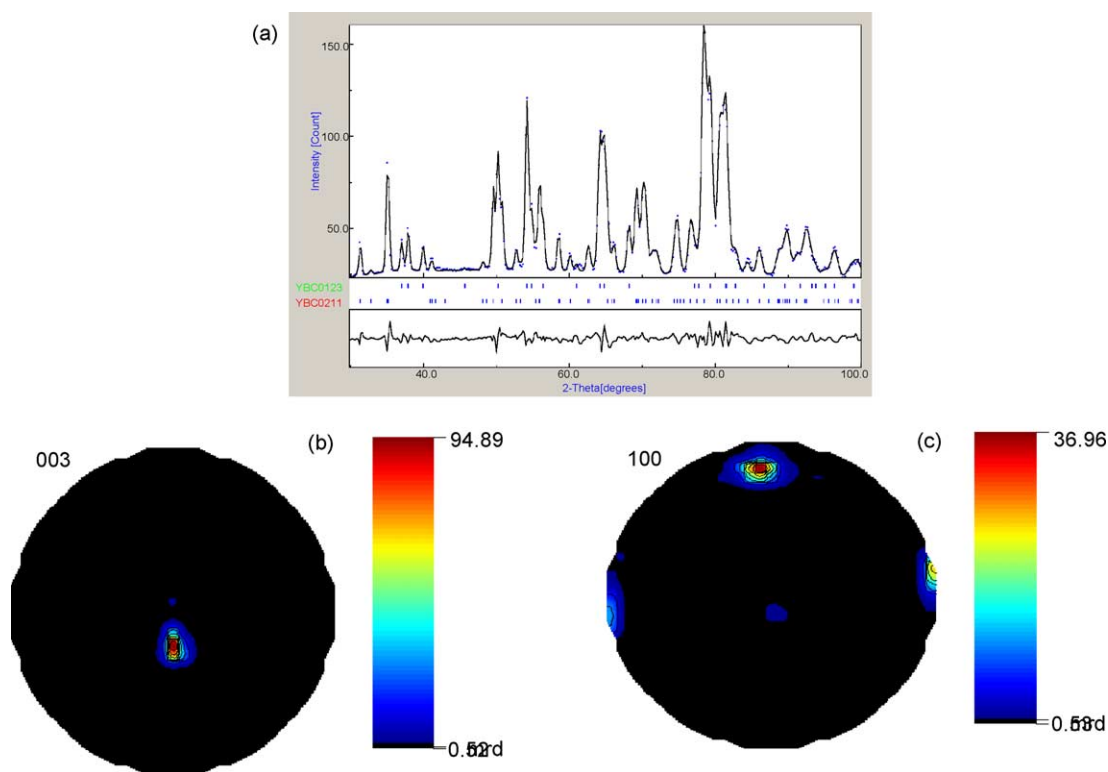


Fig. 2. (a) Rietveld refinement of the sum of the 1368 measured diagrams, allowing phase, cell parameters quantitative determinations. (b)  $\{003\}$  and (c)  $\{100\}$  pole figure showing the strong orientation of the Y123 phase with one single domain.

for further improvement of  $J_c$  in the IG-Y123-perforated by refining the Y211 distribution in their microstructure using, e.g. doping methods known from bulk materials processing.<sup>8</sup>

Quantitative texture analysis was performed on the perforated sample in order to check for eventual orientation perturbation. The whole sample volume was probed using neutron diffraction and the high flux reactor of the Institut Laue-Langevin, France, at the D1B beamline. Complete pole figures were measured using a procedure described elsewhere.<sup>9</sup> We used the combined approach to extract pole figures from diffraction spectra and calculate the orientation distribution function, this latter enabling calculation of the  $\{003\}$  and  $\{100\}$  pole figures shown in Fig. 2.<sup>10,11</sup> These pole figures clearly illustrate the presence of only one domain present in the sample, with  $c$ -axes inclined by approximately  $20^\circ$  from the normal to the plane of the figures (plane perpendicular to the perforated holes), and  $a$ -axes aligned with the main axes of the figure. The texture is single-crystal like, with pole dispersions within  $5^\circ$  to  $10^\circ$  of dispersion, at the limit of the refinability of the orientation distribution using our scanning resolution ( $5^\circ \times 5^\circ$  grid). Since no other domains are present, we can conclude that no significant orientational perturbation is introduced using our perforation process. The cell parameters of the two phases were refined from the diagram summed over the 1368 spectra corresponding to as many measured sample orientations. We obtained after refinement  $a = 3.8532(7) \text{ \AA}$ ,  $b = 3.8554(3) \text{ \AA}$  and  $c = 11.8230(9) \text{ \AA}$  for Y123,  $a = 12.169(1) \text{ \AA}$ ,  $b = 5.6526(7) \text{ \AA}$

and  $c = 7.1237(8) \text{ \AA}$  for Y211, with a reliability factor of  $R_w = 42\%$  corresponding to the refinement illustrated on Fig. 2. The Y123 phase is then poorly oxygenated, as expected for an as synthesised sample.

#### 4. Conclusion

Mono-domain composite of Y123/Y211 and perforated has been processed by combining infiltration and top seeded growth methods. The microstructure revealed the refinement of the Y211 particles and their uniform distribution in the Y123 matrix. It is interesting to note a single grain with high degree of texture in spite of the perforated structure showing by the neutron diffraction measurements.  $J_c$  at 77 K are comparable to that obtained by conventional method.

#### Acknowledgments

S. Meslin is grateful to the ‘‘French Ministère de la Recherche et de la Technologie’’ for his Ph.D. fellowship.

#### References

- Jin, S., Tiefel, T. H., Sherwood, R. C., Davis, M. E., Van Dover, R. B., Kamlot, G. W. et al., *Appl. Phys. Lett.*, 1988, **52**, 2352.
- Morita, M., Takebayashi, S., Tanaka, M., Kimura, K., Miyamoto, K. and Sawano K., *Adv. Supercond.*, 1991, **3**, 733.

3. Reddy, E. S. and Rajasekharan, T., *J. Mater. Res.*, 1998, **13**, 3389.
4. Hari Babu, N., Kambara, M., Smith, P. J., Cardwell, D. A. and Shi, Y., *J. Mater. Res.*, 2000, **15**, 1235.
5. Sudhakar Reddy, E., Noudem, J. G., Tarka, M. and Schmitz, G. J., *J. Mater. Res.*, 2001, **16**.
6. Sudhakar Reddy, E. and Schmitz, G. J., *Am. Ceram. Soc. Bull.*, 2002, **81**, 35.
7. Meslin, S. and Noudem, J. G., *Supercond. Sci. Technol.*, 2004, **17**, 1324.
8. Harnois, C., Desgardin, G., Laffez, I., Chaud, X. and Bourgault, D., *Physica C*, 2002, **383**, 269.
9. Chateigner, D., Wenk, H.-R. and Pernet, M., Orientation analysis of bulk YBCO from incomplete neutron diffraction data. *J. Appl. Crystallogr.*, 1997, **30**, 43–48.
10. Chateigner, D., Lutterotti, L. and Hansen, T., Quantitative phase and texture analysis on ceramics-matrix composites using Rietveld texture analysis. *ILL Report 97 "Highlights"*, 1998, pp. 28–29.
11. Guilmeau, E., Lambert, S., Chateigner, D., Noudem, J. G. and Ouladiaz, B., Quantitative Texture analysis of polyphased oxides by diffraction: example of Bi2223 sinter-forged ceramic and Y123 foam superconductors. *Mater. Sci. Eng. B*, 2003, **104**, 107–112.