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Melt textured $YBa_2Cu_3O_y$ bulks with artificially patterned holes: a new way of processing *c*-axis fault current limiter meanders

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Abstract

$YBa_2Cu_3O_y$ (Y123) bulk materials were textured with artificially patterned holes, creating a 'perforated structure'. The goal is to facilitate sample oxygenation and decrease crack formation in order to address the problem of hot spot formation in fault current limiter (FCL) applications. As-processed samples contained mechanically patterned holes parallel to the mean *c*-axis interconnected to the textured domain. This makes samples easier to oxygenate and cool. The microstructure is not distorted in the vicinity of the holes. The single domain character of the sample is evidenced by XRD pole figure investigations. Meander tracks were prepared by drilling the upper and lower surfaces of the samples. Samples containing holes still trap high fields, comparable with samples without holes. Values of J_c are increased in samples with holes.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

A wide variety of materials are processed with a regular array of holes for various industrial applications, such as inkjet printer, fuel injection systems, flow and dosage regulators, etc. Superconductors until recently have been elaborated as dense structures, with a few exceptions. Until now, only one regular array of antidots in YBaCuO (Y123) thin films with a lattice parameter comparable to the vortex lattice parameter has been reported [1]. Concerning bulk materials, superconducting Y123 foams with a large and opened porosity have been successfully processed and characterized [2, 3], and some authors have reported the growth of Y123 single domain through complex porous geometries [4–6], some prepared from a 3D wax model [7].

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Using the conventional route, the resulting textured tetragonal bulk sample needs to be transformed into the orthorhombic phase by annealing in flowing oxygen, or under oxygen pressure, at a low temperature around 500 °C. But because of its size and the low oxygen diffusion rate, a full oxygenation of the sample cannot be achieved without macrocracks due to the tetragonal-to-orthorhombic phase transition [8]. To overcome these problems, an alternative route is proposed to prepare bulk Y123 material with a thin wall geometry, i.e. with regularly spaced artificial holes [4–6]. The perforated morphology is proposed to play a significant role in solving some problems which have remained up to now.

- Perforation could decrease the sample porosity of the superconducting volume, since the gas trapped in the sample's core can be easily evacuated during the melting stage along the holes.

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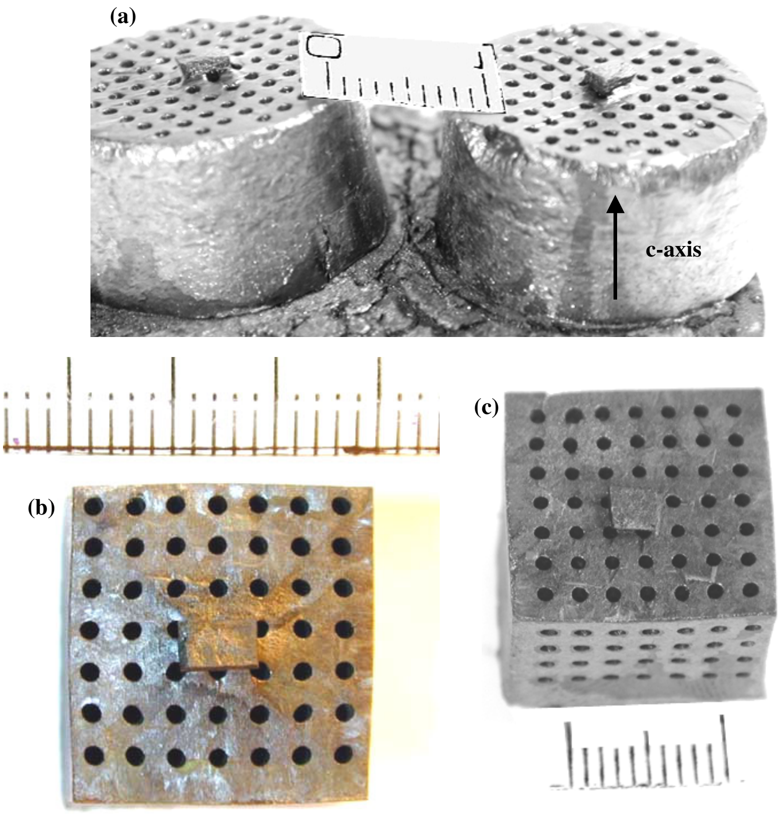


Figure 1. A batch of different perforated (a) pellets, (b) square form and (c) interconnected samples.

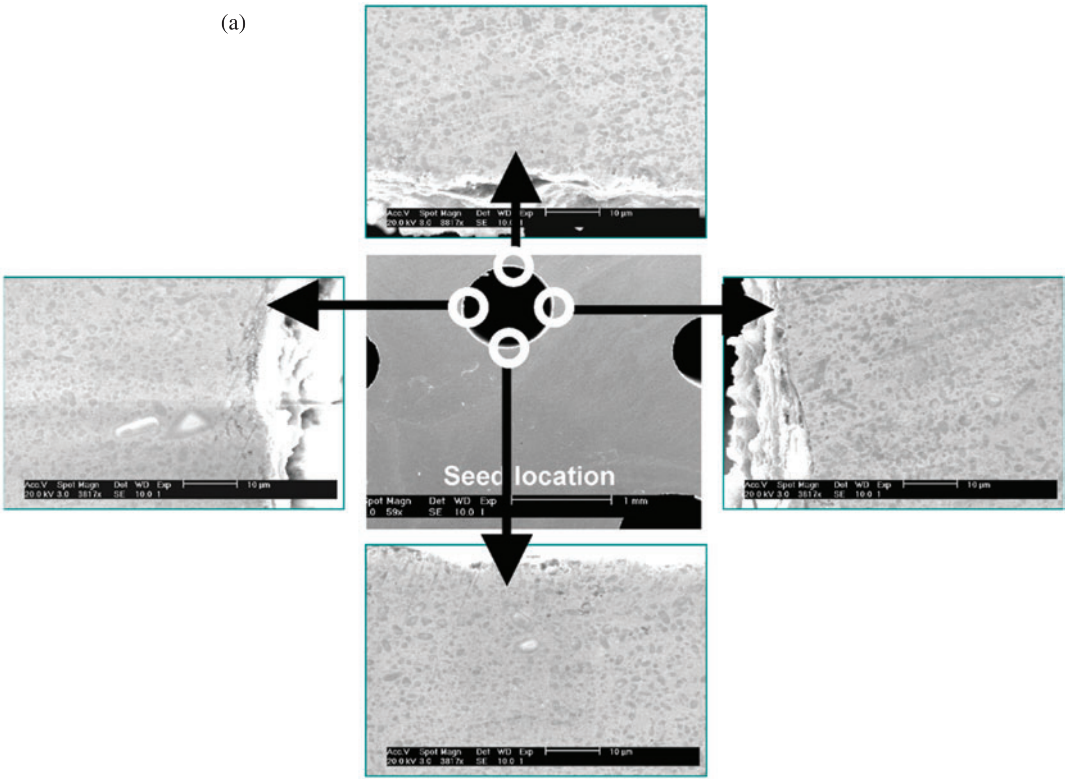
- Holes should facilitate the oxygenation of the samples, thereby minimizing crack development during the oxygenation step [8].
- Such a structure could advantageously help during sample use and characterization, e.g. by overcoming hot spot formation frequently observed during the critical current measurements of the bulk form samples, and favour heat transfers between the bulk material and the cryogenic coolant due to a larger exchanging surface.

One of the central questions is whether this novel superconductor structure is similar to the parent YBCO bulk material in terms of microstructure, superconducting properties and performances. The present investigation not only shows that the newly prepared structures have potential applications in electrotechnique devices, but also opens up a new route for processing alternative morphologies of superconducting ceramic materials.

2. Experimental procedure

$YBa_2Cu_3O_y$ (Y123) ceramic samples were prepared from commercial powders. A Y123, 25 mol% Y211 and 0.5 wt% $CeO_2 + 0.25$ wt% SnO_2 powder mixture was pelletized uniaxially and then sintered at 920 °C for 24 h. The formation of holes by a mechanical process was made. Regularly spaced, 0.5–2 mm diameter holes were drilled into the sintered bulk sample prior to melt processing. For the fault current limiter (FCL) elements, the holes did not emerge on both faces of the $\sim 15 \times 15 \times 15$ mm^3 sample, but stopped at half-thickness, using a shifted mesh on opposite faces. Details of the top seed melt-texture growth process (TSMTG) are given elsewhere [9]. The as-processed perforated samples were subsequently annealed in flowing oxygen at 450 °C for 100 h. The texture characterization was determined from XRD diffraction analysis using an experimental set-up and methodology detailed in a previous paper [10]. The sample was oscillating during the acquisition in order to increase the irradiated area up to several mm^2. The orientation distribution (OD) refinement was calculated using the Beartex program [11] and the {005/014/104}, {006/020/200} and {113} pole figures. Optical and scanning electron microscopies (SEM) were used to investigate the surface morphology. A DC Squid magnetometer (Quantum Design MPMS 5) was used to measure the superconducting transition and the hysteresis loop $M(B)$ at 77 K of the samples with the magnetic field applied along the c-axis. The critical current density (J_c) was calculated from the magnetization measurement using the modified Bean model [12]. Additionally, trapped field experiments were performed on polished samples with a Hall probe system in order to verify the homogeneity of the samples and to investigate the field ability. The Hall probe measures the magnetic field in a 0.4 mm radius area approximately. Scanning steps of 0.2 mm in two directions (x and y) were chosen. The gap between the polished top surfaces of the sample and the Hall sensor was adjusted to 1 mm. The sample was field cooled in liquid nitrogen with a $Nd_2Fe_{14}B$ magnet of 15 mm diameter and 0.4 T surface field.

(a)



(b)

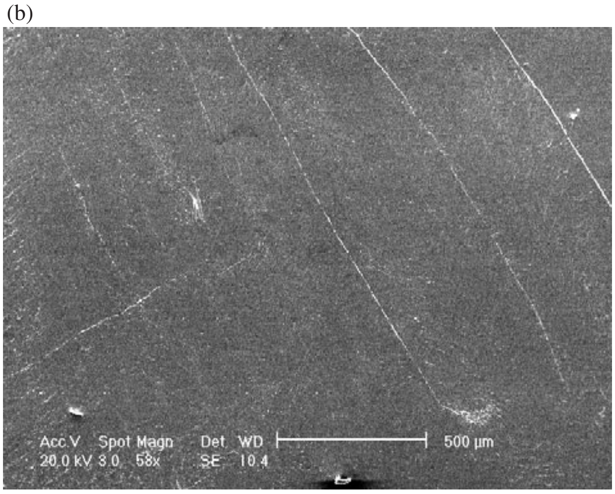


Figure 2. (a) Microstructures around a hole. (b) The microstructure of a non-patterned sample.

3. Results and discussion

Optical micrographs of as-grown samples with 1 mm holes are shown in figure 1. All the pictures show the top surface of the formed single domains, grown from Sm123 seeds. Seed-induced growth lines (figure 1(b)) are visible up to the edge of the samples. The single domain growth occurs completely throughout the total height of the samples (figure 1(a)) as was demonstrated previously using bulk neutron texture analysis [13], and without large hole deformation thanks to the homogeneous deformation of the melt. Figure 1(c) proves the feasibility of preparing Y123 materials with interconnected holes. This structure can help heat extraction from the sample due to the increased surface exchange. Alternatively, a Y123 domain with open holes could be reinforced, for example by infiltration with a low melting metal alloy, in order to improve the mechanical properties useful for levitation applications and trapped field magnets.

The growth lines of faceted growth on the surface of the perforated single domain are clearly analogous to the ones observed on top-seeded melt-textured bulks processed in the same conditions but with no artificial holes. This shows that the pre-formed holes do not seem to disturb the growth of the single domain, which is confirmed by the video[4] of the melt growth process of other perforated samples prepared by Chaud *et al* [5].

[4] Video at webpage: http://www-crismat.ismra.fr or http://www.grenoble.cnrs.fr/CRETA/creta.html

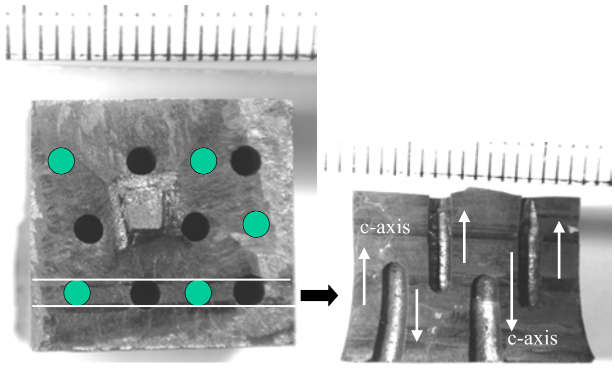


Figure 3. The feasibility of *c*-axis meander shape for a fault current limiter taken from a single domain monolith sample with 2 mm holes.

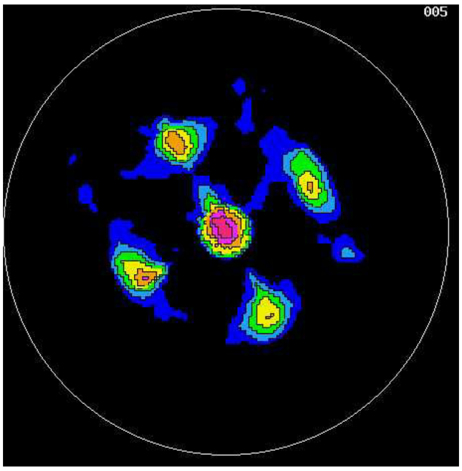


Figure 4. {005/104/014} normalized multipole figure.

The elaboration of the perforated samples can give rise to problems like microcrack induction during drilling of the pellets, but after melt processing no micropore-like defects appear in the microstructures. Indeed, the presence of holes in the sample seems to limit the porosity between the holes in the bulk material. Figure 2(a) shows the typical microstructure of a single domain of Y123 around the holes. One can clearly observe (i) the compact, crack-free microstructure with respect to a non-patterned sample (figure 2(b)) and (ii) a uniform distribution of fine Y211 particles in the Y123 matrix.

Figure 3 shows the feasibility of elaborating *c*-axis resistive fault current limiter (FCL) elements from the perforated bulks. It is well known that the material characteristics required for a FCL are: (i) a negligible resistance during normal operation but (ii) the development of a sufficient resistance to limit the current to the desired value during a fault current. Many efforts have been devoted to develop a meander FCL from a bulk textured material [14–18]. Generally elements are cut into meander-like shapes using a diamond saw, the current path being oriented along the *ab*-planes or *c*-axes. According to the relative brittleness of the Y123 material, the successive cutting operations can induce cracks or microcracks to form in the sample, sometimes breaking it. In our case, from a $\sim 15 \times 15 \times 15$ mm^3 cube, we are able to prepare *in situ* a *c*-axis meander shape before melt texturing, which greatly simplifies the element shaping for FCL application. In addition, the obtained meander is dense and shows an easy injection of the current along the *c*-axis. This new concept for the elaboration of superconducting ceramic materials in designed shapes can be an efficient alternative to build compact engineering devices such as FCLs, motors, etc.

The {005/014/104} multipole figure, recalculated from the refined orientation distribution of the crystallites, is presented in figure 4. This figure shows a very strong 005 pole at the centre of the pole figure (the maximum of the orientation distribution is of 968 multiples of a random distribution), indicating that the mean *c*-axis of the crystallite distribution is aligned with the cylinder axis of the samples, and that mechanical drilling of the initial pellet does not much perturb the crystalline alignment. Interestingly, the 104/014 poles exhibit a single domain-like character, showing the strong alignments of the **a** and **b** axes of the Y123 phase in the plane of the sample. These latter poles are however slightly elongated in azimuth, illustrating a small misalignment of **a** and **b** in the plane. A contribution which comes from a small domain can be seen at larger tilt angles. However, this component is lower than 1% in volume from the observed densities.

Figure 5 corresponds to the trapped field mappings at 77 K of one of the perforated pellet samples (figure 5(a)) and of a reference sample (figure 5(b)), which has the same composition and same thermal treatment, but no holes. All the perforated samples exhibit similar trapped field maps, indicating that there is no significant difference induced by the two hole sizes (0.5 and 1 mm) in these measurement conditions. No clear effect of the holes on the trapped field can be evidenced from the mappings illustrated in figure 5: both have the same shape, with no fall of the trapped field around the holes. Note that mappings with smaller scanning steps (<0.2 mm) have been performed around the holes and have given the same result. The trapped field value is similar in the three samples. Since the samples were cooled with a magnetic field of only 0.4 T, no optimal trapped field value can be given here. Field cooled samples under high fields must be performed to really compare the samples' capacity in term of continuity and value of the trapped fields. However, these measurements confirm that the perforated samples are homogeneous and that the presence of holes does not affect the domain growth.

The temperature dependence of the resistance is shown (figure 6) for magnetic fields up to 3 T applied parallel to the mean *ab*-planes and *c*-axes of the perforated textured material. The measurements show a transition temperature $T_c \approx 90$ K, and a narrow transition width, indicating the good homogeneity of the sample. The magnetoresistance curves show a high T_c when the magnetic field is applied in *ab*-planes, corresponding to the strong pinning force in this case. We can deduce a magnetic anisotropy ratio of 3.5 (at 88 K) coherent with the strong texturization of the perforated sample and with the SEM microstructure and trapped field observations.

The zero field cooled magnetization curve (inset of figure 6) shows a very narrow transition with an onset at 92 K, comparable to values taken from the resistivity $R(T)$ curves mentioned above. The steep transition characterizes an essentially pure Y123 phase and indicates no substantial contamination originating from the drilling of the samples.

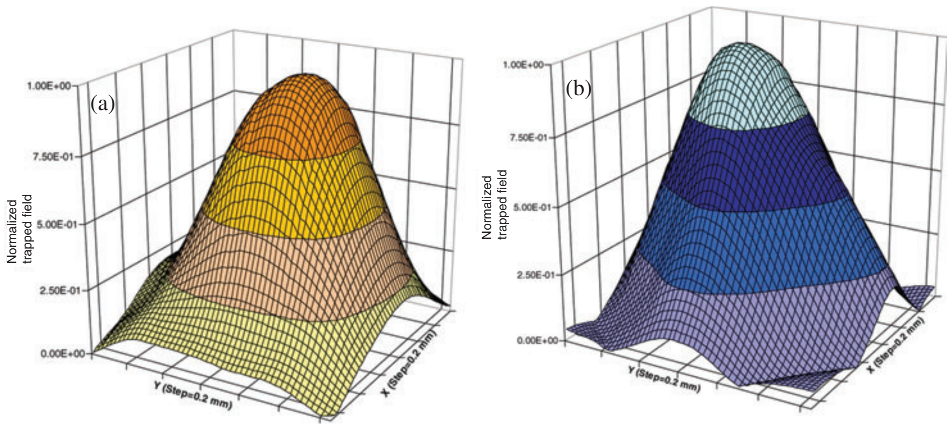


Figure 5. (a) Normalized trapped field (77 K) of the perforated sample. (b) Normalized trapped field (77 K) of the hole-free sample.

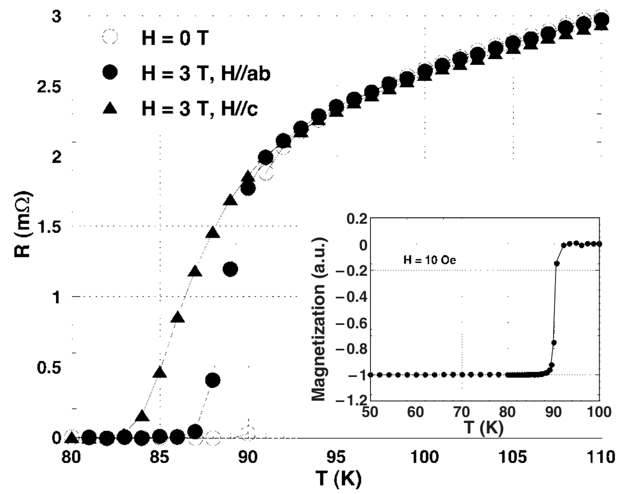


Figure 6. The temperature dependence of resistivity under magnetic field. Inset: magnetic transition versus temperature.

Hysteresis cycles with magnetic field applied parallel to the direction of the single domain surface have been performed at 77 K on a square sample. The magnetic J_c values are estimated for $B \parallel c$-axes from M–B cycles on the basis of the Bean model taking $J_c = 20\Delta M/[a(1-(a/3b))]$, where $a(1-(a/3b))$ is a geometric factor related to the sample dimensions (in cm) with $a < b$ and ΔM (in emu cm^{-3}) is the hysteresis of the magnetization. Figure 7 presents the $J_c(B)$ determined from the width of the hysteresis loops for several pieces cut from the perforated and free of holes samples. A critical current density of $J_c \approx 40$ kA cm^{-2} at 0 T can be deduced for current parallel to the ab-planes compared to 25 kA cm^{-2} obtained on samples without holes. There is a lot of scope for further investigation/improvement of J_c in such perforated Y123 material such as performed on the cleaved samples (where the crystallographic ab-planes are well defined), because the J_c measurements were made on cut-out cubic samples where the misalignment of c-axes can reduce the J_c.

4. Conclusion

The association of sintering/drilling and top seeding melt texture was successfully applied to the preparation of regularly perforated Y123 bulk materials. SEM studies have shown that the hole presence does not hinder the domain growth and that the typical microstructure is conserved. Porosity is reduced

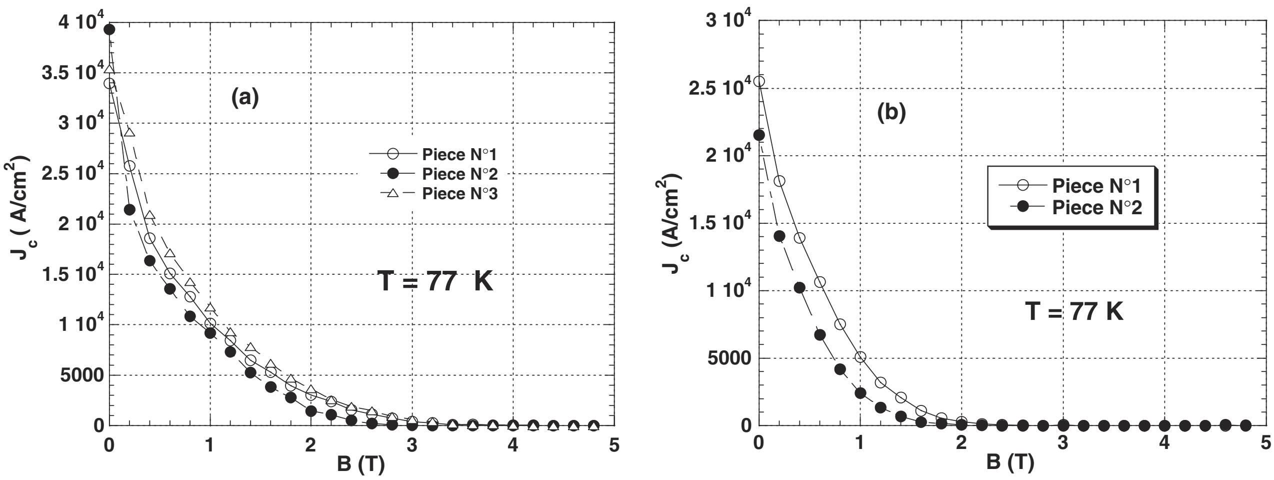


Figure 7. The magnetic field, B, dependence of the critical current density at 77 K for various pieces taken from the same sample: (a) perforated material, (b) sample without holes.

in the bulk. The perforated samples exhibit a c-axis grain orientation, confirmed by pole figures, and the single domain character is evidenced by trapped-field distribution. Magnetic J_cs are increased in samples with holes compared to the hole-free parent sample having the same thermal history.

This new structure has a great potential for many applications with improved performances in place of Y123 hole-free bulks, since it should be easier to oxygenate, and to maintain at liquid nitrogen temperature during application, avoiding hot spot occurrence. For meandering FCL elements, cutting is a crucial step as cracks appear during this stage. This can be solved by *in situ* zigzag shape processing of holes, as demonstrated by this work. Further investigations concerning the oxygenation effect, transport-J_c measurements, maximum trapped field capacity and hole density with respect to the properties are in progress.

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