

# Growth of YBCO single domains through an array of holes for FCL *c*-axis superconducting elements

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## Abstract

Considering resistive YBCO fault current limiter, a more compact design can be achieved with the current flowing along the *c*-axis instead of the (*a*, *b*) planes. The higher resistivity along the *c*-axis allows reducing the required length of material.

The oxygen annealing step of the samples is the key factor for developing the necessary superconducting properties. Usually an intense cracking along the (*a*, *b*) plane is observed due to the mechanical constraints introduced during the oxygen uptake. An annealing treatment has been designed to reduce the cracking drastically. However, this treatment can only be applied to samples with thickness lower than 1.5 mm.

To simplify manufacturing and increase reproducibility, single domains (Ø 20 and 40 mm) were grown by the TSMG method through a triangular array of equidistant holes. Holes as small as 0.8 mm in diameter and distant of 2 mm were drilled parallel to the *c*-axis in YBaCuO sintered pellets in order to produce a geometry with walls having a thickness less than 1.5 mm. The growth proceeds similarly to a plain pellet. The holes remain open. The growth front is slightly distorted by the holes, but reaches the edges. The growth of a single domain is confirmed by flux mapping experiment as well as by microscopic observation under polarized light. An indirect advantage of this geometry is the disappearance of the porosity usually trapped within a plain single domain.

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## 1. Introduction

Superconducting fault current limiter (SFCL) is a promising power application with no competing equivalent in traditional technologies. SFCL is based on the self-triggering transition (quench) from a superconducting state to a dissipative normal state as soon as a threshold current is reached in the superconducting material. The high impedance so introduced in the circuit provides a fast and efficient protection against currents occurring during short circuits which can be 20–30 times higher than the rated current. SFCL are particularly attractive at high voltage where protection devices can open the circuit only when current alternations pass

through zero. They are used to diminish electrical constraints on classical electrical devices or to allow interconnections of feeding paths to increase the power quality of a grid without addition of the fault currents.<sup>1</sup> This paper deals with resistive SFCL using YBCO bulk textured materials. They present the advantages to be cheaper to produce than thin films and to provide a faster and more effective limitation than Bi-compounds.<sup>1</sup>

Limiting elements made of meanders cut out in (*a*, *b*) plane slices of YBaCuO single domains elaborated by a top seeding technique (TSMG)<sup>2</sup> were tested in a SFCL 1 kV/100 A.<sup>3</sup> However, because of their high critical current density ( $J_c$ ) at 77 K and their low thermal conductivity, the meanders are very sensitive to hot spots, a local transition caused by a material defect and usually leading to the sample destruction. This can be avoided by operating close to their critical temperature,  $T_c$ , where the local energy density is absorbed by the material specific heat itself. In those conditions, the  $J_c$  is

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limited as well as the power dissipation and the transition is self-protective.<sup>1,3</sup>

This necessary lowering of  $J_c$  for safe transition has been the triggering opportunity to investigate another original configuration for current limitation: the use of YBaCuO single domain sample with currents flowing along  $c$ -axis.<sup>4</sup> This configuration leads to a more compact design of the current limiter elements thanks to a resistivity higher along  $c$ -axis than in ( $a$ ,  $b$ ) planes by two orders of magnitude. Electric fields up to 4000 V/m have been obtained with  $c$ -axis elements as compared to an average of 100 V/m with meanders.<sup>3</sup>

However, cracks in the ( $a$ ,  $b$ ) planes appeared as a major drawback for the  $c$ -axis elements. The occurrence and propagation of cracks was found to be directly linked with the oxygenation of the material. The oxygen uptake results in a decrease of the cell unit in the  $c$ -direction. The low oxygen diffusion rate yields large oxygen gradient during a classical annealing treatment. The surface oxygenated layer shrinks as compared to the non-oxygenated bulk material and is finally the localization of a large tension stress resulting in the opening of cracks.<sup>5</sup> The cracks are then an easy diffusion path and the shrinkage of the unit cell along the  $c$ -axis at the crack tip provides the driving force for the cracks propagation.

A modified annealing treatment has been successfully proposed to drastically reduce the cracks occurrence. By progressively adjusting the oxygen partial pressure during annealing in accordance to the temperature and to the oxygen content of the material surface layer, the oxygen gradients can be maintained below the limit where they induce cracks. There is however a strong limit to this solution: about 10 days are needed to oxygenate samples with thickness of 1–1.5 mm.<sup>5</sup> A scale-up to larger sections is not realistic.

The design of  $c$ -axis elements has to deal with this constraint. In prototypes tested by the CRETA, the desired section (dictated by the nominal current to flow in the conductor) was obtained by assembling thin  $c$ -axis slabs of 1–1.5 mm in parallel using silver paste.<sup>4</sup> Such individual elements can fit preliminary test, but this assembly is delicate and time consuming. Furthermore, reproducibility is a major concern. So the concept of bulk Y123 single domains with a thin wall geometry was introduced, which consists in growing single domains on sintered pellets being already machined with an array of holes. This material stands in one piece but benefits from reduced diffusion paths and thin walls.

## 2. Experimental

The starting material is a mixture of commercial powders with a ratio of 70 wt.% of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub>, 30 wt.% of Y<sub>2</sub>BaCuO<sub>5</sub> and 0.15 wt.% in excess of PtO<sub>2</sub>. Cylindrical pellets, typically 25 mm in diameter and 20 mm in height, are pressed under an 80 MPa uniaxial load and sintered at 910 °C during 6 h. Machining the ceramic pellet is the most convenient at this stage. The pellet will not crumble as just compacted powder, and has not reached yet the hardness of a

single domain. Some preliminary tests have been made using 1.5 mm diameter holes and a square pattern of 5 mm distant holes. Typically, a triangular array of 1 mm diameter holes distant of 2.4 mm is drilled parallel to the pellets axis through its whole height (20 mm). The wall thickness of the obtained geometry is less than 1.5 mm.

The single domain is then grown on the machined pellet from a SmBa<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub> seed. This material is very similar to the YBa<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub> compound, but with a 45 °C higher melting point. The seed is placed on top of the pellet. The pellet is melted, but not the seed. In the solidification temperature range, the single domain nucleates below the seed with the crystallographic orientation of this latter, usually chosen to grow the single domain with its  $c$ -axis parallel to the pellet axis. The pellet is maintained in a very narrow temperature range to grow the single domain without further nucleation from the melt. The growth is controlled thanks to a video monitoring of the sample surface, the contrast between the growing crystal and the melted pellet being revealed by a halogen lightning [see<sup>2</sup> for details]. Once the growth is finished, the sample is cooled down to room temperature under flowing nitrogen in order to freeze the tetragonal phase. Oxygenation is performed afterwards.

## 3. Results

Fig. 1 illustrates the growth at an intermediate stage. It proceeds as for a plain pellet. Starting from the seed, the single domain grows following a square pattern. This pattern is due to the growth of the YBa<sub>2</sub>Cu<sub>3</sub>O<sub>*x*</sub> compound along the  $a$ - and  $b$ -directions in tetragonal phase. The growth speed is about 0.3 mm/h. The samples are kept about 60 h in the solidification temperature range, which is far enough for the single domains to reach the edges of the pellets. To get a single domain over the entire volume of a drilled pellet seems even easier and more reproducible than for plain pellets. This can be ascribed to a better gas exchange associated with reduced diffusion paths.

Some distortions need however to be pointed out when the growth front crosses a hole. Two cases can be distinguished: steps and streaks. In the diagonal region of the pattern, the hole forces the growth front to dissociate into two perpendicular  $a$ - and  $b$ -directions. Both growth fronts join again behind the hole, but create a step since the  $a$ -growth has been favoured for one front and the  $b$ -growth for the other (see for instance at the bottom of the left pellet Fig. 1). In the middle region of the growth fronts, i.e. where the growth proceeds roughly along only one direction, a streak appears behind the holes like the one left by the recombination of a flow behind a cylindrical obstacle. An increasing number of holes leads to an increasing distortion of the growth front square pattern as can be seen in Fig. 1.

However, once the surface is polished, it seems impossible to evidence sign of defects around the holes. The holes remain open, but their diameter reduces during the process from 1

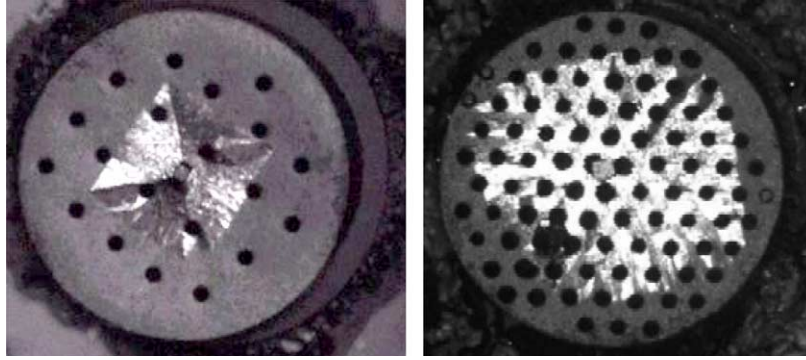


Fig. 1. Pictures of the surface of drilled pellets with different hole spacings (every 5 mm on left and every 2.4 mm on right) taken at an intermediate stage during the growth process. The bright shape is the growing domain with its seed in the centre. Steps and streaks result from the interaction of the holes with the growth front. The distortion of the regular square growth pattern increases with the number of holes.

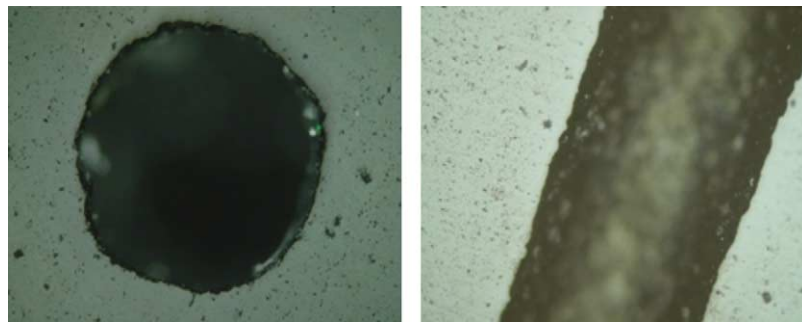


Fig. 2. Micrographs under polarized light of the vicinity of a hole ( $\varnothing$  0.8 mm) showing a classical distribution of  $Y_2BaCuO_5$  particles in the  $YBa_2Cu_3O_x$  matrix. No indication of non recombined liquid phase or segregation is found around the hole.

to 0.8 mm. Fig. 2 shows the microstructure in the vicinity of a hole. The growth proceeds clearly up to the edge. No segregation or non-recombined liquid phase is detected. Further investigations are underway to characterize the distortions introduced by the holes.

No detrimental effects are evidenced by macroscopic characterizations. On the contrary, they confirm that the growth through an array of holes yield a single domain with similar, if not better, properties than a single domain grown in a plain pellet. Crystallographic orientation has been checked by X-ray and neutron diffraction analyses. Superconducting properties in the  $(a, b)$  planes have been investigated using flux mapping. Fig. 3 shows a 2D representation of the flux trapped by a drilled sample. The sample is magnetized by a permanent magnet (NdFeB with a surface induction of 0.5 T) lying on its surface while cooling down to 77 K. Once the permanent magnet removed, the sample surface is scanned with a Hall probe at 0.2 mm of the surface using a 0.5-mm step. The current induced by the magnetization cannot flow through defects such as grain boundaries, planar segregation or cracks. The contour profile of Fig. 3 corresponds to a single current loop flowing at the sample scale: the sample is a single domain. The array of holes has no significant effect on the current loops at the macroscopic scale.

Although the trapped flux peak (454 mT) does not correspond to a saturated value (the magnetization is limited to the

0.5 T of the permanent magnet), it is already 37% better than the one measured on plain pellets (330 mT). The levitation force measured on this drilled sample (42 N) is also larger than the best value obtained so far in comparable plain pellet (35 N). These results are obtained with just a classical oxygenation process, whereas the material volume is decreased

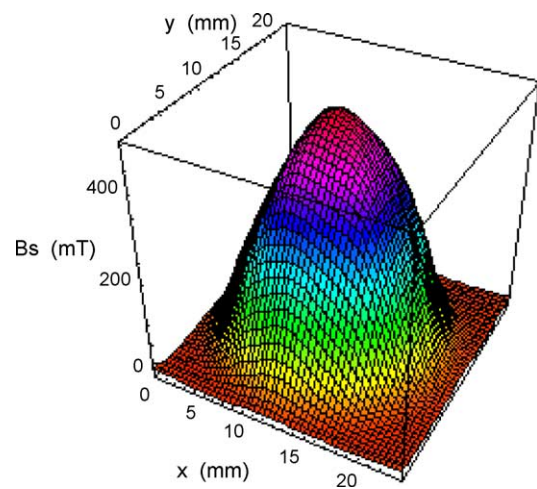


Fig. 3. Flux trapped measurement by a Hall probe scanning on a drilled pellet showing a single current loop typical of a single domain. The sample was magnetized by a permanent magnet ( $B_s = 0.5$  T).

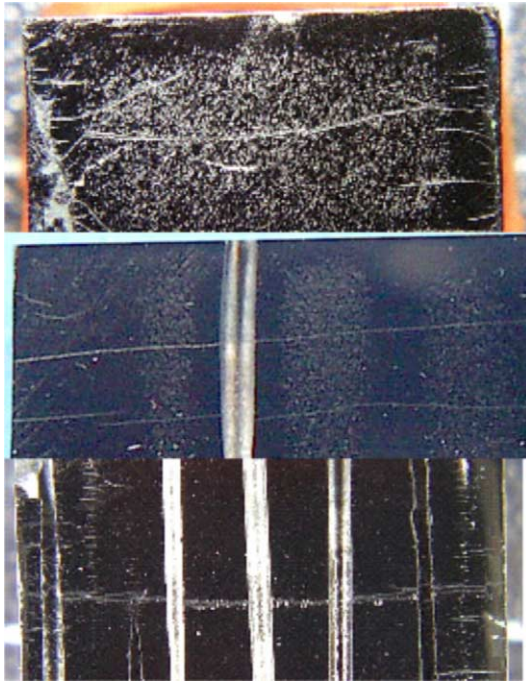


Fig. 4. Vertical section of a plain pellet and two drilled pellets with different hole spacings. A large porosity is noticeable inside the plain pellet. This porosity disappears in a 3–4 mm layer close to the surface of the sample as well as close to the holes. This porosity can be nearly eliminated for a suitable hole spacing (bottom pellet) yielding a high quality material.

by 20%: they underline a significant increase of the material quality.

One reason for this increase may be the elimination of porosity. Cross sections of a plain pellet and two drilled pellets, each with a different spacing between holes, are compared in Fig. 4. A large porosity exists in the bulk material, except for a 2-mm layer close to surface. This porosity is already reduced in the drilled pellet with a 5-mm spacing (middle) and a dense layer is found around the holes. For a suitable wall thickness (bottom), the material is exempt from pores. The porosity is able to diffuse outside of the material from the first millimetres close to the surface, but seems trapped inside the material once the dense surface layer has formed. Thin wall geometry helps avoid this phenomenon and so increase advantageously the material quality. Enhancement could also be related to the reduction of cracking, but characterizations

of the material with that regards are still underway, in particular implemented in a *c*-axis FCL element.

#### 4. Conclusion

The growth of Y123 single domains on pellet already machined with a regular array of holes is successfully demonstrated. Despite the distortion of the growth front introduced by the holes array, the enhancement of quality makes definitely this thin wall geometry material a good candidate for applications such as levitation and flux trapping. The microstructure is homogeneous and nearly free of porosity as a consequence of the short diffusion paths associated with thin wall geometry. These preliminary results are very encouraging to elaborate complex geometry single domains suitable for specific applications such as *c*-axis FCL, and to investigate the opportunities offered by thin wall geometries with regard to processing, doping, improved thermal exchange and mechanical reinforcement.<sup>6</sup>

#### References

1. Tixador, P., Obradors, X., Tournier, R., Puig, T., Bourgault, D., Grana-dos, X. *et al.*, Quench in bulk HTS materials - application to the fault current limiter. *Supercond. Sci. Technol.*, 2000, **13**, 493–497.
2. Isfort, D., Chaud, X., Beaugnon, E. and Tournier, R., Growth of large YBaCuO monodomains for current limiting applications. *Inst. Conf. Ser.*, 2000, **167**, 47–50; Chaud, X., Isfort, D., Beaugnon, E. and Tournier, R., Isothermal growth of large YBaCuO single domains up to 93 mm. *Physica C*, 2000, **341–348**, 2413–2416.
3. Floch, E., Buzon, D., Isfort, D., Porcar, L., Chaud, X., Tixador, P. *et al.*, Current limitation based in bulk YBaCuO meanders. *Cryogenics*, 2000, **41**, 531–538.
4. Tournier, R. F., Isfort, D., Bourgault, D., Chaud, X., Buzon, D., Floch, E. *et al.*, *c*-Axis YBCO domains for current limiting applications. *Physica C*, 2003, **386**, 467–473.
5. Isfort, D., Chaud, X., Tournier, R. and Kapelsky, G., Cracking and oxygenation of YBaCuO bulk superconductors: application to *c*-axis elements for current limitation. *Physica C*, 2003, **390**, 341–355; Diko, P. and Krabbes, G., Formation of *c*-macrocracks during oxygenation of TSMG YBaCuO single-grain superconductors. *Physica C*, 2003, **399**, 151–157.
6. Tomita, M. and Murakami, M., High-temperature superconductor bulk magnets that can trap magnetic fields of over 17 T at 29 K. *Nature*, 2003, **421**, 517–520.