Investigation of artificially patterned YBCO single domain bulks

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Abstract. Mechanical stress is introduced into the bulk of large YBCO single domains during oxygenation, mainly because of the tetragonal to orthorhombic phase transition. Due to the slow oxygen diffusion rate, full oxygenation is nearly impossible in reasonably large samples without the occurrence of macro-cracks. An alternative morphology of the bulk, i.e. a periodic array of holes drilled parallel to the \textit{c}-axis prior to oxygenation, could lead to a solution of this problem, because the perforation limits the wall thickness (diffusion distance) to the distance between the holes and provides additional paths for oxygen flow during processing. Conventional flux density mapping of the fully magnetised bulks showed, that the artificial holes were beneficial for the material properties, resulting in an enhancement of the trapped flux density compared to reference samples without these holes. However, variations of the flux density profile with the periodicity of the perforation are observable at higher resolution. This indicates that the array of holes influences the local current distribution. The bulks were also investigated more locally, by the magnetoscan technique. These measurements clearly display the array of holes and provide additional information about the cracks.

1. Introduction
The occurrence of cracks during the oxygenation procedure and their reduction is a key problem in the fabrication of bulk superconductors. A possible approach to solve this problem is a modification of the morphology of the bulk, for instance by drilling an array of holes parallel to the \textit{c}-axis, thus providing additional diffusion paths and an increase in the surface to volume ratio. Moreover the perforation is beneficial for the material quality itself because of the reduction of pores trapped in the material [1].

On the other hand the perforation implies a loss of superconducting volume and, therefore this improvement due to better oxygenation could be counterbalanced by the material loss. Secondly, the influence of the holes on the supercurrent flow and, therefore, on the flux density profile is of interest.

2. Experimental
2.1. Samples
Two samples grown at CRETA from a mixture of commercial powders (70 wt.% YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x}, 30 wt.% Y\textsubscript{2}BaCuO\textsubscript{5} and an excess of 0.15 wt.% PtO\textsubscript{2} [1]) were analysed.

The samples had a diameter of 21 mm and a thickness of 7.5 mm and 7.2 mm, respectively. Holes of 1 mm in diameter were drilled parallel to the \textit{c}-axis into one of the two samples prior
to the melt texture growth and after a sintering step. They are separated by 2.4 mm and form a triangular array. A total of 81 holes yield a volume loss of about 20%. The second sample is taken as a reference sample. Both samples were subjected to the same oxygenation treatment, which consisted of two stages, the first at 420 °C (144 h) and the second at 380 °C. Optical investigations of the reference sample revealed an extended grain on the top surface with different reflectivity.

2.2. Measurement Techniques

The samples were analysed by both the conventional flux density mapping method and the magnetoscan technique at liquid nitrogen temperature. The trapped flux density profiles were mapped 15 min after activation in an 8 Tesla magnet. The gap between the active area of the Hall probe and the sample surface was approximately 0.2 mm.

In all measurements the field perpendicular to the sample surface, \( B_z \), was assessed using Hall probes from AREPOC. The size of the active area of the Hall probes is \( 25 \times 25 \mu m^2 \) for the flux density maps and \( 50 \times 50 \mu m^2 \) for the magnetoscans, respectively. The voltage of the Hall probes was measured by a KEITHLEY DMM. The scan mesh was \( 0.25 \times 0.25 \) mm\(^2\), the data were taken by a computer controlled \( xy \)-positioning system.

The magnetoscan technique offers the possibility of obtaining information of the bulk superconducting properties on a more local scale. In contrast to conventional flux density maps, the currents are induced in a thin layer (\( \approx 1 \) mm) of the top surface during the scan by a small permanent magnet. The magnet is moved together with a Hall-probe which simultaneously scans the sample surface. Details of this setup can be found in [2]. A magnet with a height of 18 mm and a diameter of 6 mm, reaching an induction of about \( \mu_0 H=100 \) mT on top, was used for these experiments.

3. Results

3.1. Flux density maps

Both samples were activated with a magnetic field of 1.5 T (\( zfc \)). The trapped flux density map of the reference sample exhibits a smooth profile only disturbed by a larger crack penetrating the sample from its edge (fig. 2.4). No influence of the extended grain on the top surface of the reference sample could be observed. The maximum trapped field was 353 mT.

A higher trapped field was obtained in the perforated sample (478 mT). A periodic perturbation of the trapped flux density was found close to the sample edge. The periodicity is associated with the underlying array of holes.

Additionally, the profile was perturbed by a crack and a larger defect. The latter may result from a perturbation of the growth front.

Furthermore magnetic fields below \( 2 H^* \) were applied. Increased activation (0.2, 0.4 and 0.6 T) pushes the remanent profile towards the center of the bulk. Especially at an applied
field of 0.4 T nearly all holes close to the edge are detected (fig. 3). Additionally both defects mentioned above are evident in all scans.

3.2. Magnetoscan
The grain with different reflectivity on the top surface of the reference sample displayed a Bean-like magnetisation, indicating an area of bad superconducting properties with a smaller diamagnetic response (fig. 5). Around this region numerous smaller cracks penetrating the sample from the boundary are evident in the scans.

The structure of the perforation was entirely reproduced with this method. Additionally all defects discussed above are visible in the magnetoscan, even though they are partly masked by the strong variation of the signal induced by the holes. In general one would expect the diamagnetic response of the bulk to decrease above the holes because of a lack of shielding currents in this region. However, maxima with a diamagnetic response beyond that of the bulk were also obtained and found to be aligned along the scan direction. They can be understood by numerical simulations and are related to geometrical effects of the relative arrangement between the holes, the magnet and the Hall probe [3].

4. Summary
Perforated samples showed a trapped field enhancement by about 35 % compared to reference samples. The enhancement in the trapped field is similar to that reported elsewhere for similar geometries and shapes [1]. Although the reference sample exhibited an area of bad superconducting properties on the top surface, its influence on $B_z$ is negligible. The results of the flux density maps together with the magnetoscan suggest that this area is only a thin layer of insufficiently oxygenated tetragonal phase.

A periodic pattern of small signals in the trapped flux density profile was found in the perforated sample, indicating that the current flow is slightly perturbed by the holes. The perforation is even more evident at activation fields below $2H^*$. Regarding the trapped fields we showed that under the present oxygenation conditions the benefit resulting from the modified geometry (reduced porosity, shorter processing) overcomes the loss in superconducting sample volume. It is likely that an increase of the oxygenation time will lead to similar $J_c$ values in both samples and, hence, the volume loss will become dominant. However, the perforated samples provide us with a method of obtaining higher trapped fields at a reduced oxygenation time.

References
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[2] Eisterer M, Haindl S, Wojcik T, Weber HW 2003 Supercond. Sci. Technol. 16 1282–85
[3] Zehetmayer M private communication
Figure 2. Flux profile of the perforated (left) and the reference sample (right).

Figure 3. Activation fields of 0.2, 0.4 and 0.6 mT. The holes are indicated by white circles.

Figure 4. Contour lines of the flux density profile of the perforated (left) and the reference sample (right). The circle indicates the large defect, the arrows indicate cracks.
Figure 5. Magnetoscan of the perforated (left) and the reference sample (right): The arrow indicates the scanning direction, the arrangement of the magnet and the probe is indicated by the circle and the cross. The grain on the top surface of the reference sample is indicated by the white circle. Since the magnetoscan is a qualitative method, all data are normalised.