

## Direct identification of extended defects as vortex pinning centers in melt textured $\text{YBa}_2\text{Cu}_3\text{O}_7 - \text{Y}_2\text{BaCuO}_5$ composites

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**Abstract**—Single domain  $\text{YBa}_2\text{Cu}_3\text{O}_7 - \text{Y}_2\text{BaCuO}_5$  melt textured ceramic composites have revealed a very rich microstructure, which has usually impeded, by using standard measurements, to evaluate the contribution of each defect to the enhancement of the critical current. We have measured the in-plane magnetoresistance anisotropy and the anisotropic in-plane inductive critical currents and we show that together with the microstructural TEM analysis, the contribution of the different extended pinning centers can be separated. These results have allowed us to infer the kind of microstructure modifications required to improve the critical current. In particular, we present an isostatic pressing deformation technique as a very promising post-processing treatment to strongly increase the critical currents of these composites.

### I. INTRODUCTION

Melt-textured  $\text{YBa}_2\text{Cu}_3\text{O}_7 / \text{Y}_2\text{BaCuO}_5$  superconducting composites have appeared as the promising bulk material for superconducting power applications, especially since preparation techniques based on solidification processes succeeded in growing single domain composites avoiding the high angle grain boundaries [1]. Y123/211 composites with critical current densities,  $J_c$ , of the order of  $50.000 \text{ A/cm}^2$  at 77 K have since then been achieved [2]. Further enhancement of  $J_c$  has required a detailed investigation of the microstructure and its correlation to flux pinning mechanisms. TEM analysis has revealed that these materials have a very rich and complex microstructure [3]: second phase precipitates, twin boundaries, dislocations, stacking faults, microcracks. Some of these defects are supposed to greatly influence the superconducting properties. However, the main difficulty is still to define the proper measurements to isolate the effect of each type of defect and thus be able to infer the kind of microstructure modifications required to improve the critical currents.

Recent studies [2,4] have shown that 211 second phase precipitates induce a new pinning mechanism at the interfaces with the Y123 matrix, which strongly increases the critical currents at low and intermediate magnetic fields.

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Measurements of samples with different contents of 123/211 interface area have confirmed that this pinning mechanism exists [2] and follows the correlated disorder pinning theories [5]. On the other hand, preliminary results on pinning by twin boundaries in Y123/211 have been reported through angle dependent magnetoresistance and critical current measurements for  $H // ab$  [6,7]. It has been shown that these planar defects can be considered linear correlated pinning centers in the liquid and also solid vortex state. Instead, the role of the linear defects like dislocations and partial dislocations associated to the stacking faults has been less investigated [8]. The difficulties in controlling its concentration through well established processing techniques have limited their relevance. However TEM observations have shown that these linear defects tend to align in specific directions in the  $ab$  plane [9] and thus could initially act as point defects for  $H // c$  whereas they might act as linear defects for the orientation  $H // ab$ .

In this paper, we report on the flux pinning effect induced by partial dislocations associated to the stacking faults generated by a isostatic pressure post-processing treatment. A 100 % enhancement of  $J_c$  at 77 K has been achieved. It has been recently shown that these partial dislocations tend to align with the twin boundaries. Thus, a study of the twin boundary pinning effect on the same samples was previously performed and is here also presented. We show that the combination of anisotropic magnetoresistance measurements and inductive critical current measurements for  $H // ab$  and  $H // c$  can help to elucidate the effect of both microstructural extended defects in the liquid and solid vortex state [10].

### II. EXPERIMENTAL

Single domain  $\text{YBa}_2\text{Cu}_3\text{O}_{7-d} / \text{Y}_2\text{BaCuO}_5$  (Y123/211) melt-textured composites were grown by a modified Bridgman directional solidification technique in air [11]. This preparation technique provides single domain bars of 100 mm in length and 7 mm in diameter with a maximum mosaic spread of  $3-5^\circ$  and allows us to vary the content of Y211 additives distributed in the Y123 matrix between 4 % and 38 % in weight. The results here reported belong to a sample with 28 % wt of Y211. Upon the oxygenation treatment, polarized light microscopy analysis revealed a random distribution of Y211 particles with mean sizes of  $1 \mu\text{m}$  and a

high density of twin boundaries showing the two orthogonal families. A post-processing deformation technique under isostatic pressing conditions (2 Kbar in Ar atmosphere at room temperature), CIP treatment, was afterwards applied to the same samples. TEM analysis revealed one remarkable new feature [12]: the generation of a high density of nanometric stacking fault loops in the  $\text{CuO}_2$  plane expanding following the twin boundary structure. This mechanism results in an increase of the total length of the associated  $1/6\langle 031 \rangle$  partial dislocations which have also been proposed as efficient pinning centers for this material [13]. The superconducting transition temperature was 92 K with  $\Delta T_c \sim 0.5$  K. The angular dependence of the anisotropic magnetoresistance was measured using the standard four point technique up to 9 T with a resolution of  $0.1^\circ$ . The inductive critical currents were determined with a SQUID magnetometer provided with a 5.5 T superconducting solenoid.

### III. RESULTS AND DISCUSSION

#### A. Twin boundary pinning

Fig 1 shows the magnetoresistance of a Y123 / 28%wt 211 single domain sample as a function of the angle,  $\theta$ , between the magnetic field and the crystallographic c-axis, for a magnetic field of 1 T and 9 T at the indicated temperatures. The transport current, 100  $\mu\text{A}$ , was applied in the ab plane keeping the orthogonality with the magnetic field when this was rotated  $90^\circ$ . The expected  $\sin^2(\theta)$  curve showing the anisotropic character of the layered superconductors is clearly visible. Shown around the angle of  $0^\circ$  ( $H \parallel c$ ) is a small dip manifesting a decrease of dissipation around this magnetic field orientation and which should be related to an anisotropic pinning mechanism active at  $H \parallel c$ . We have ascribed this effect to the dense twin boundary structure that this melt-textured material possess along the  $\langle 110 \rangle$  directions.

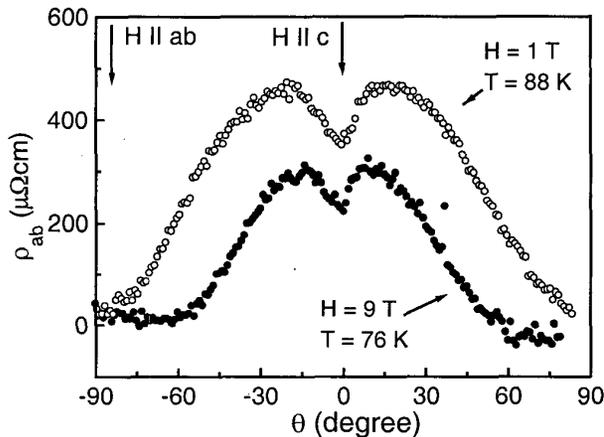


Fig. 1 Angular dependence of the magnetoresistance at 1 and 9 T for the indicated temperatures. The angle  $\theta = 0$  corresponds to the orientation  $H \parallel c$ .

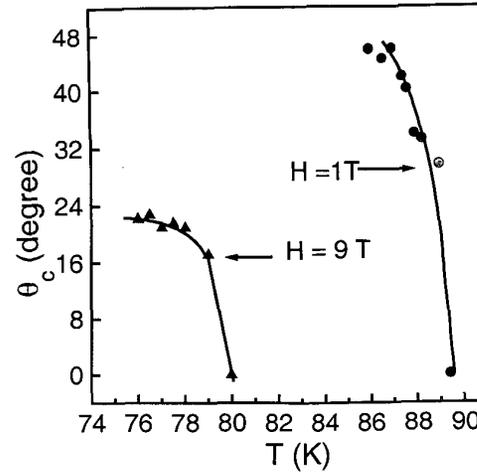


Fig.2 Temperature dependence of the critical angle,  $\theta_c$ , for 1 T and 9 T.

A critical angle,  $\theta_c$ , may be then defined from the results of Fig 1, by taking the angle between the two maxima surrounding the dip. This angle reflects the actual region where twin boundary pinning is active in the liquid vortex state, i.e. the angular range where a kinked vortex structure persists [14]. In Fig 2 the temperature dependence of the critical angle is presented. At high temperatures, a fast decrease of the pinning efficiency is observed probably due to thermal activation processes. Instead, at relatively low temperatures, the effect of twin boundary pinning tend to a saturation. This is in agreement with the results reported on single crystals [15]. Taking into account that a 1 T magnetic field is already above a kind of matching field for the twin boundary spacing of Y123/211 composites ( $d \approx 1000 \text{ \AA}$ ), the above results suggest that the dense twin boundary structure may pin the full vortex lattice even in the liquid vortex state close to the irreversibility line.

The effect of the twin boundaries in the solid vortex state was investigated by measuring the component,  $J_c^c$  of the critical currents for  $H \parallel ab$  [2].  $J_c^c$  was determined from the hysteresis loops using the generalized anisotropic Bean model. Fig 3(a) shows a comparison of the temperature dependence of the critical current for the sample Y123 / 28%wt 211 when  $H \parallel \langle 110 \rangle$  and when  $H \parallel \langle 100 \rangle$ . Fig 3(b) shows the corresponding magnetic field dependence at 3 different temperatures. Notice that in both figures, an increase of the critical current density by 30-40 % is obtained when  $H \parallel \langle 110 \rangle$ . Thus, confirming that pinning by twin boundaries is effective when the vortex lattice is well below the irreversibility line, deep in the solid vortex state.

Finally, the influence of the twin boundaries for  $H \parallel ab$  near the irreversibility line was also investigated. Fig 4(a) shows the magnetoresistance measurements for the Y123/28%wt211 when the magnetic field was rotated within the ab plane and the transport current was directed along the c-axis. In this case, a minimum in dissipation is expected every  $90^\circ$ , indicating that the two twin boundary families may pin the vortices. The expected minima are certainly seen in Fig 4(a)

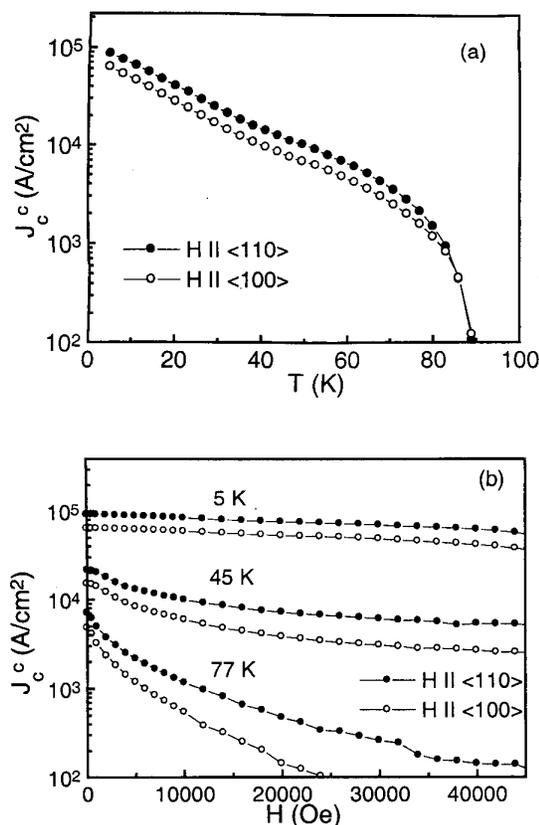


Fig 3 (a) Temperature dependence of the critical current density,  $J_c^c$ , for  $H \parallel \langle 110 \rangle$  and  $H \parallel \langle 100 \rangle$  orientation. (b) Magnetic field dependence of the critical current,  $J_c^c$ , for the three indicated temperatures for  $H \parallel \langle 110 \rangle$  and  $H \parallel \langle 100 \rangle$  orientation.

at  $\theta = 90^\circ$  and  $180^\circ$ . However, we notice that one is much more important than the other, which probably just indicates that the volume percentage of the two twin families is different in this sample.

### B. $1/6\langle 031 \rangle$ Partial dislocation pinning

Upon studying flux pinning by twin boundaries, we induced a CIP process [12] to the same samples. TEM analysis have remarkably shown [12] the generation of short stacking fault loops lying parallel to the  $\text{CuO}_2$  planes and growing following the twin boundary structure.

Flux pinning by the  $1/6\langle 031 \rangle$  partial dislocations which surround the stacking faults, has been theoretically proposed [13]. However, the difficulty in controlling their growth have usually impeded its study. We have found that after a CIP process,  $1/6\langle 031 \rangle$  partial dislocations are generated and aligned following the  $\langle 110 \rangle$  directions and therefore their

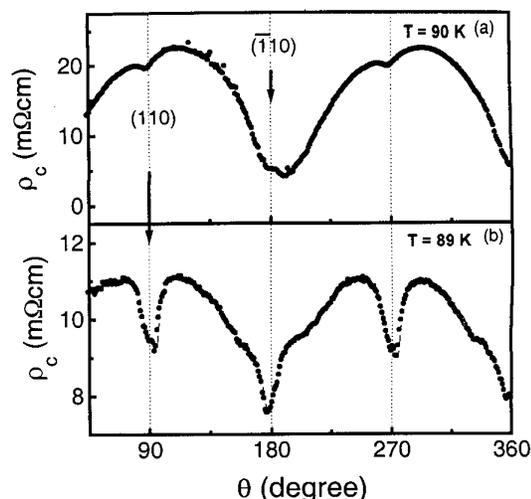


Fig. 4 A representative data of the angular dependence of the magnetoresistance within the ab plane at 3 T for the Y123/211 composite (a) previous to the CIP treatment and (b) after CIP treatment.

pinning efficiency may be now investigated. A thorough study both by TEM and magnetization has been published elsewhere [12]. The CIP process is proposed as a good post-processing technique for the generation and control of  $1/6\langle 031 \rangle$  partial dislocations.

Fig 4(b) shows representative results of the angular dependence of magnetoresistance within the ab plane for a Y123/28%wt211 sample upon a CIP process was performed. The transport current, also 100  $\mu\text{A}$ , were applied in the  $c$ -axis direction ensuring a non-zero Lorentz force onto the vortex lattice. The partial dislocations may here act as linear pinning defects for  $H \parallel ab$  whereas they would as point defect pinning centers for  $H \parallel c$ .

Notice comparing the results of Fig 4(b) with those of Fig 4 (a), that the minima expected every  $90^\circ$  are not only still present but their effect seems to be more pronounced and well defined after the CIP process. This may indicate that the new microstructural defect identified by TEM behaves more efficiency as linear pinning center overwhelming any effect associated to an unbalanced ratio of the volume of the two twinned families.

Finally, Fig 5(a) and (b) show the temperature and magnetic field dependence of the critical current density before and after a CIP treatment for  $H \parallel c$ ,  $J_c^{ab}$ . The critical current density has strongly increased after this post processing treatment for all temperatures and magnetic fields and it has particularly reached a 100 % increase at 77 K and zero field. This impressive result not only certifies that the new microstructural defect represents an important pinning contribution also in the solid vortex state as point defect, but it also paves the way to a new processing methodology of melt-textured material for artificial defect engineering.

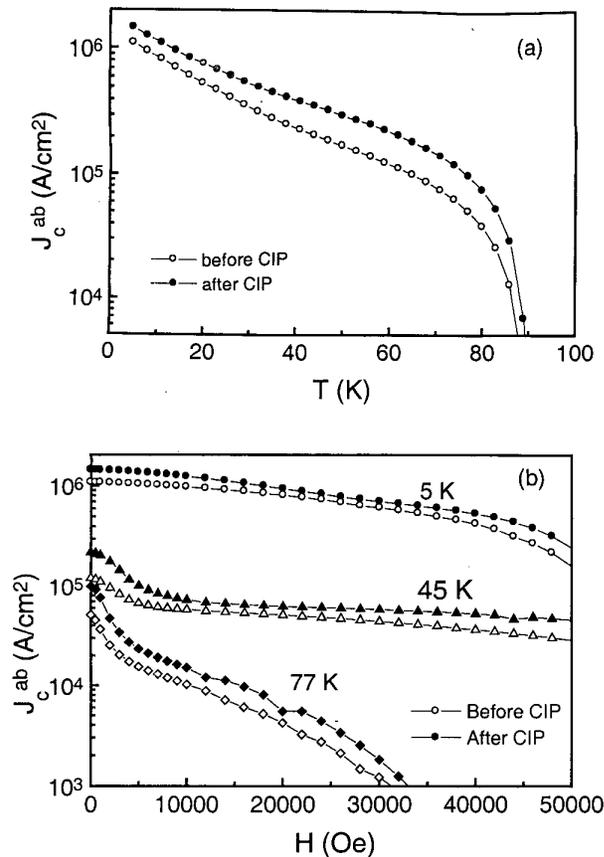


Fig 5 (a) Temperature dependence of the critical current density,  $J_c^{ab}$ , for (open symbols) sample before CIP treatment, (closed symbols) after CIP treatment. (b) Magnetic field dependence of the critical current density,  $J_c^{ab}$ , for the three indicated temperatures for (open symbols) sample before CIP treatment, (closed symbols) after CIP treatment.

#### IV. CONCLUSIONS

Flux pinning results of the extended defects of twin boundaries and  $1/6\langle 031 \rangle$  partial dislocations have been briefly reported. Both, the behavior in the liquid and solid state have been studied by measuring the angular dependence of the magnetoresistance and the inductive critical current density respectively. We have shown that by properly measuring these samples, the pinning effect of the main microstructural defects can be determined. In particular, we have shown that a isostatic pressing treatment at room temperature may be taken as a post processing technique to enhance pinning by  $1/6\langle 031 \rangle$  partial dislocations which grow aligned with the  $\langle 110 \rangle$  direction. The potential of this technique to enhance the critical currents of Y123/211 composites has been demonstrated.

#### REFERENCES

- [1] P. McGuinn, "High Temperature Superconducting Materials Science and Engineering", ed. Bonglu Shi, Elsevier Science Ltd. 1995, Ch. 8, pp345-382
- [2] B. Martínez, X. Obradors, A. Gou, V. Gomis, S. Piñol, J. Fontcuberta, H. Van Tol, "Critical currents and pinning mechanisms in directionally solidified  $YBa_2Cu_3O_{7-x}Y_2BaCuO_5$  composites", Phys. Rev. B **53**, 2797-2810 (1996)
- [3] Y. Zhu, "High Temperature Superconducting Materials Science and Engineering", ed. Bonglu Shi, Elsevier Science Ltd. 1995, Ch.5, pp199-258; F. Sandiumenge, S. Piñol, X. Obradors, E. Snoek, and Ch. Roncau, Phys. Rev. B **50**, 7032 (1994)
- [4] M. Murakami, "Proceedings and Properties of High Tc Superconductors", vol. 1, Ed. S. Jin, Singapore World Scientific 1993, Ch 6, pp 215-268
- [5] D. R. Nelson, V. M. Vinokur, "Boson localization and correlated pinning of superconducting vortex arrays", Phys. Rev. B **48**, 13060-13097 (1993)
- [6] S. Sanfilippo, D. Bourgault, C. Villard, R. Tournier, P. Gautier Picard, E. Beaunon, A. Sulpice, Th. Fournier and P. Germi, "Twin planes influence on critical transport current along the c-axis in  $YBaCuO$ ", Europhys. Lett. **39**, 657 (197)
- [7] B. Martínez, T. Puig, A. Gou, V. Gomis, S. Piñol, J. Fontcuberta and X. Obradors, "Ub-plane flux pinning in melt textured  $YBa_2Cu_3O_{7-x}Y_2BaCuO_5$  composites", submitt. to Phys. Rev. B
- [8] P. X. Zhang, L. Zhou, P. Ji, W. M. Bian, X.Z. Wu and Z. H. Lai, "The effect of annealing on stacking faults and  $J_c$  values of PMP processed YBCO" Supercond. Sci. Technol. **8**, 15-18 (1995)
- [9] F. Sandiumenge, S. Piñol, X. Obradors, E. Snoek and C. Roncar, "Microstructure of directionally solidified high critical current  $YBaCu_3O_{7-x}Y_2BaCuO_5$  composites", Phys. Rev. B **50**, 7032 (1994)
- [10] G. Blatter, M.V. Feigel'man, V.B. Geshkenbein, A.I. Larkin and V.M. Vinokur, "Vortices in high temperature superconductors", Rev. Mod. Phys. **66**, 1125 (1994)
- [11] S. Piñol, V. Gomis, B. Martínez, A. Labarta, J. Fontcuberta and X. Obradors, "Bridgman growth and enhanced critical currents in textured  $YBaCu_3O_{7-x}Y_2BaCuO_5$  composites", J. Alloys Compounds. **195**, 11 (1993)
- [12] B. Martínez, L. Richard, F. Sandiumenge, T. Puig, J. Rabier, X. Obradors, "Enhanced critical currents in melt-textured  $YBa_2Cu_3O_7$  by cold isostatic pressing", submitt. Appl. Phys. Lett.
- [13] A.M. Campbell and J.E. Evetts, "Flux vortices and transport currents in type II superconductors", Adv. Phys. **21**, 199-428 (1972); D. Dew-Hughes and J.M. Witcomb, Phil. Mag. **2673** (1972)
- [14] A.A. Zhukov, G.K. Perkins, J.V. Thomas, A.D. Caplin, H. Küpfer, T. Wolf, "Direct observation of tilted vortex structures induced by twin boundaries in  $YBa_2Cu_3O_7$  single crystals", Phys. Rev. B **56**, 3481-3487 (1997)
- [15] L.M. Paulius, J.A. Fendrich, W.K. Kwok, A.E. Koshelev, V.M. Vinokur, G.W. Crabtree, B.G. Glagola, "Effect of 1-GeV uranium ion irradiation on vortex pinning in single crystals of high-temperature superconductor  $YBa_2Cu_3O_{7-d}$ ", Phys. Rev. B **56**, 913-924 (1997)