UPCommons

Portal del coneixement obert de la UPC

<http://upcommons.upc.edu/e-prints>

Aquesta és una còpia de la versió *author's final draft* d'un article publicat a la revista [*IEEE transactions on applied superconductivity*].

URL d'aquest document a UPCommons E-prints:

<http://hdl.handle.net/2117/85591>

Article publicat¹ / Published paper:

Teixeira Dias, F., das Neves Vieira, Valdemar, Wolff-Fabris, E., kampert, M. H., Schaf, J., Farinela, G.F., De paiva Gouvea, C., Roa Rovira, J.J. IEEE transactions on applied superconductivity, 26, 3. 1-5. Doi: 10.1109/TASC.2016.2526005

Comparison Between the Magnetic Irreversibility and Zero Resistance of High-Quality Melt-Processed YBaCuO Superconductors

Fábio Teixeira Dias, Valdemar das Neves Vieira, Frederik Wolff-Fabris, Erik Kampert, Marlon Hneda, Jacob Schaf, Grazielle Fernanda Farinela, Cristol de Paiva Gouvêa, and Joan Josep Roa Rovira

Abstract—This paper portrays a detailed study of the magnetic irreversibility limit T_{irr} (H) and of the zero resistance point T_{c0} (H) of three different top-seeding melt-textured $YBa_2Cu_3O_{7-\delta}$ superconducting samples, with well-aligned c-axis and doped with a high density of nonsuperconducting $Y_2Ba_1Cu_1O_5$ (Y211) pinning centers. We have performed measurements for applied magnetic fields up to 140 kOe and for the whole set of the different field-current configurations. The magnetization measurements were performed using an MPMS-XL SQUID magnetometer and a vibrating sample magnetometer, both from Quantum Design. The electric transport measurements were made using a physical properties measurement system from Quantum Design. The goal of this exhaustive study is obtaining precise data about magnetic flux mobility along the various directions in the sample for the different field-current configurations, thereby defining the nature and effects, due to the strength and anisotropy of the pinning mechanisms and disclosing the various physical mechanisms dissipating electric transport in these systems below the superconducting transition temperature. We discuss our results in terms of the anisotropic flux pinning by the Y211 grains dispersed into the superconducting matrix.

Index Terms—Flux pinning, granular superconductors, high-temperature superconductors, type-II superconductors.

I. INTRODUCTION

THEORETICALLY, below the superconducting transition temperature of homogeneous superconductors a non-zero electric current can flow without any resistance. In zero applied magnetic field the electric resistance falls to zero closely underneath the T_c . However, if a magnetic field is present inside the

superconducting material, the electric current will interact with the quantized magnetic fluxons causing magnetic flux dynamics and dissipation of its energy. It can flow without resistance, only if the magnetic fluxons are sufficiently stabilized by pinning centers. In homogeneous type II superconductors, under an applied magnetic field, the magnetic irreversibility limit line T_{irr} (H) is a boundary, in the field-temperature (H-T) plane, below the superconducting transition at which the flux pinning just begins to stabilize the first magnetic fluxons. However, the pinned fluxons percolate through the sample only at some temperature below the T_{irr} line. Hence, a nonzero electric current can flow without resistance only somewhat below this T_{irr} (H) line.

Our high quality melt-processed $YBa_2Cu_3O_{7-\delta}$ samples are doped with up to 30 percent of non-superconducting $Y_2Ba_1Cu_1O_5$ (Y211) particles, which provides them with a high density of very effective flux pinning centers [1]. The goal of our present study is to disclose experimentally the correlation between the magnetic irreversibility limit lines, T_{irr} (H), and the zero resistance lines, T_{c0} (H), for all the different field-current configurations and thereby obtaining information about the magnetic flux mobility in the various directions and the respective flux pinning mechanisms. We have obtained precise profiles of T_{irr} (H), corrected for relaxation effects, due to field non-homogeneity of the magnetometers and of T_{c0} (H) as a function of applied field for three high quality melt-processed $YBa_2Cu_3O_{7-\delta}$ samples.

II. EXPERIMENTAL PROCEDURES AND TECHNIQUES

A. Sample Preparation

The three melt-processed $YBa_2Cu_3O_{7-\delta}$ samples investigated in this work were produced at the Institute of Materials Science of Barcelona, and at the Department of Materials Science and Metallurgical Engineering of the University of Barcelona, in Spain. The samples, labeled as TS17, TS30-I, and TS30-II, were prepared by the top-seeding method. Single crystalline seeds of the $Nd_1Ba_2Cu_3O_{7-\delta}$ superconductor were placed on top of ceramic $YBa_2Cu_3O_{7-\delta}$ pellets in order to induce the crystallographic texture. More information may be found in [2] and [3].

The top-seeding sample TS17 was produced starting from a ceramic pellet of $YBa_2Cu_3O_{7-\delta}$ containing 17 wt% of the

Manuscript received September 7, 2015; accepted January 22, 2016. This work was supported under the Brazilian Grant MCTI/CNPq Universal 14/2012 (Contract 477506/2012-7). The experimental work at the HLD was supported by EuroMagNET II (Contract 228043).

F. T. Dias and V. das Neves Vieira are with the Instituto de Física e Matemática, Universidade Federal de Pelotas, 96010-900 Pelotas-RS, Brazil (e-mail: fabio.dias@ufpel.edu.br).

F. Wolff-Fabris and E. Kampert are with Dresden High Magnetic Field Laboratory, Helmholtz-Zentrum Dresden-Rossendorf, 01314 Dresden, Germany.

M. Hneda, J. Schaf, and G. F. Farinela are with the Instituto de Física, Universidade Federal do Rio Grande do Sul, 91501-970 Porto Alegre-RS, Brazil.

C. de Pavia Gouvêa is with Inmetro, 25250-020 Duque de Caxias-RJ, Brazil. J. J. R. Rovira is with Universitat Politècnica de Catalunya, 08028 Barcelona, Spain.

Color versions of one or more of the figures in this paper are available online at <http://ieeexplore.ieee.org>.

Digital Object Identifier 10.1109/TASC.2016.2526005

Fig. 1. SEM image of the TS30-I sample showing profiles of the platelet structure stacked along the c-axis as a consequence of the texture process.

Fig. 2. Magnetoresistance data for TS30-II sample in the configuration $H // J // ab$ from 0 up to 140 kOe. The inset shows the zero resistance temperature for the magnetic fields of 0 and 140 kOe, denoted as T_{c0} (H).

76 non-superconducting Y211 phase. The two top-seeding melt-processed TS30-I and TS30-II samples were cut from two different top-seeding melt-processed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ buttons containing 30 wt% of the non-superconducting Y211 phase.

80 The samples were small parallelepipeds with 4 mm in length along the ab plane, 1.9 mm in thickness (ab plane) and 2.2 mm along the c-axis. We have characterized the samples by electron microscopy in order to verify the grain alignment. Fig. 1 is a representative image obtained for TS30-I sample using a scanning electron microscope Nova NanoLab 600 from Fei Company. Fig. 1 shows profiles of long platelets (superconducting grains) along the ab plane stacked along the c-axis. Similar structures could be observed in all of the investigated samples. The good quality of the samples also was characterized by measurements of the critical currents. In 5 K, all the samples supported critical currents of about 10^6 A/cm^2 up to about 20 kOe and more than 10^5 A/cm^2 up to 50 kOe.

B. Physical Measurements

94 The magnetoresistance measurements were made with a PPMS (Physical Properties Measurement System) from Quantum Design and under applied magnetic fields ranging from zero up to 140 kOe. Good four electrical contacts on the surface of the samples were achieved by heating with freshly painted silver paint stripes to 420°C in oxygen for 1.5 h. The measurements were made by applying a low measuring current density parallel to the ab plane and also along the c-axis in the five possible field-current configurations: $H // J // ab$, $H // ab - J // ab$ ($H \perp J$), $H // ab - J // c$, $H // c - J // c$, and $H // c - J // ab$. The same samples used in the magnetoresistance experiments were employed for the magnetic measurements.

Fig. 2 shows magnetoresistance results of the TS30-II sample for magnetic fields within the range of from 0 up to 140 kOe applied parallel to the electric current and both along the ab plane. We obtained the zero resistance temperature, denoted by T_{c0} , adopting a simple and practical criterion, where the zero resistance temperature is the beginning of the plateau where dR/dT definitely falls to zero value. The inset of Fig. 2 displays

details of this method to define the limit of zero resistance in our samples, and the arrows show the points where the electric resistance becomes zero for 0 and 140 kOe. The error in the determination of zero resistance is less than 0.5 K for all applied fields.

The magnetization measurements were always performed on the same samples used in the magnetoresistance measurements, with an MPMS-XL SQUID magnetometer and a vibrating sample magnetometer (VSM) both from Quantum Design in magnetic fields from 0.01 to 140 kOe applied along the c-axis or parallel to the ab plane. The magnetic irreversibility limit was obtained by using the standard method consisting in first cooling the sample down to a low temperature in zero applied field (ZFC), switching on a desired field and measuring the magnetic moment M (T) while warming the sample (rate of 0.3 K/min) in the constant (persistent) field up to a temperature above the superconducting transition temperature, and subsequently measuring M (T) while cooling the sample back in the same magnetic field (FC procedure).

The experimental results were always corrected for temperature gradient effects. The irreversibility limit, T_{irr} (H), for an applied magnetic field is the temperature point where the difference FC-ZFC deviates from the zero baseline, marked by the high-temperature experimental data, where the magnetic moment is always reversible. Fig. 3 is representative and shows our criterion adopted to define the magnetic irreversibility limit. It shows the difference between FC and ZFC magnetic moments for the TS30-I sample under a magnetic field of 1 kOe applied along the ab plane. The arrow shows the irreversibility limit T_{irr} (H) for the specific magnetic field, and the inset shows the original ZFC and FC curves. By this method we could determine $T_{irr}(H)$ within an experimental error lower than 0.5 K and in low fields lower than 0.2 K.

III. EXPERIMENTAL RESULTS

A. Magnetic Irreversibility

The magnetic irreversibility line T_{irr} (H) of the samples was determined for applied magnetic fields ranging from 0.01 kOe up to 140 kOe. Figs. 4–6 display the irreversibility lines of the three top-seeding melt-processed samples TS17, 152

Fig. 3. Difference between FC and ZFC magnetic moments for a magnetic field of 1 kOe applied along the ab plane of the TS-30-I sample. The arrow indicates the irreversibility limit and the inset shows the original ZFC and FC curves.

Fig. 4. Magnetic irreversibility lines $T_{irr}(H)$ for $H // ab$ (blue) and for $H // c$ (red), and the zero resistance lines $T_{c0}(H)$ for all the possible field-current configurations, according to listed in the inset, for the TS-17 sample. The coarse lines through the $T_{irr}(H)$ data are fittings with the gfc equation (see text), and the fine lines through the $T_{c0}(H)$ data are only guides to the eyes.

Fig. 5. Same as Fig. 4, for TS30-I sample.

Fig. 6. Same as Fig. 4 for TS30-II sample, for a maximum magnetic field of 140 kOe.

TS30-I and TS30-II, for magnetic fields H applied along the 153 c-axis ($H // c$) and along the ab-plane ($H // ab$). In fact 154 these T_{irr} data were corrected for the effects of relaxation, 155 induced by motion of the sample in the non-homogeneous field 156 within the sample space of the magnetometers. In 50 kOe of 157 applied field, we observed displacements of T_{irr} of 7.5 K to 158 lower temperature. In order to correct for these distortions, we 159 have measured the magnetic irreversibility as a function of the 160 amplitude of the sample transport and extrapolated the obtained 161 T_{irr} to zero amplitude, which gave a plausible and visibly the 162 correct irreversibility limit [4]. The continuous lines through 163 the $T_{irr}(H)$ data are fittings with the usual equation describing 164 the profile of magnetic irreversibility as a function of applied 165 magnetic field, related with the giant flux creep (gfc) [5], 166 $H_{irr}(T) = H_0(1 - t)^\alpha$, where $\alpha = 3/2$. In the equation, $t = 167 T_{irr}(H)/T_{irr}(0)$ is the reduced temperature, H_0 is the value 168 of the irreversibility field extrapolated to zero temperature, 169 and $T_{irr}(0)$ is the irreversibility temperature at zero applied 170 magnetic field. All the magnetic irreversibility data are well 171 fitted with this equation. 172

The anisotropy for $H // ab$ and $H // c$ is large, similar 173 to observations in $YBa_2Cu_3O_{7-\delta}$ single crystals, and shows 174 the good alignment of the crystallite c-axis. The irreversibility 175 lines of all our melt-processed samples do not exhibit the de 176 Almeida-Thouless [6] and the Gabay-Toulouse [7] regimes in 177 the low field region, typical of granular systems [8]. Usually 178 good melt-processed materials do not display these character- 179 istics of superconducting granularity, which attests to the good 180 crystallite alignment as well as very close crystallite coupling. 181

B. Magnetoresistivity

182

Figs. 4–6 also show the zero resistance data as a function of 183 applied field for each one of our three melt-processed samples, 184 and for all the five possible field-current configurations. The 185 continuous lines are a guide to the eyes. 186

Our zero resistance lines $T_{c0}(H)$ in Figs. 4–6 show the 187 same c-axis-ab-plane anisotropy as the magnetic irreversibility. 188 Moreover, the T_{c0} lines fall all closely underneath the T_{irr} 189 lines for the respective directions of the applied field. These 190

results show that the electric resistance below the transition temperature is due to excitation of magnetic flux dynamics. In this case the magnetic flux pinning avoids the electric current from dissipating its energy. In relation to applied fields along the c-axis, the two zero resistance lines fall closely together and practically are coincident. On the other hand, for magnetic fields along the ab plane, we observe differences larger than the error bars and for some field-current configurations the T_{c0} lines even lie significantly below the T_{irr} line.

IV. DISCUSSION

The usual idea that the magnetic irreversibility line T_{irr} (H) is a boundary in the $H \times T$ plane, below which a non-zero electric current may flow without resistance, stems from the conventional homogeneous isotropic metallic alloy superconductors with high density of pinning centers. In general this is not true for the high-temperature superconductors (HTSC). The coherence length in the HTSC is very short (less than 10 Å). They are planar and intrinsically granular superconductors [8]. However, the present work shows that good melt-processed polycrystalline $YBa_2Cu_3O_{7-\delta}$ materials exhibit large intrinsic anisotropy, and do not show the characteristics of superconducting granularity. In the melt-processed materials, doped with a high density of pinning centers, the electric resistivity falls to zero closely underneath the magnetic T_{irr} (H) line for $H //$ ab-planes, as well as for $H //$ c-axis as shown by Figs. 4–6. However, in order to get these T_{irr} (H) lines to the right place, it was necessary to measure the magnetic irreversibility for each applied field as a function of the amplitude of the sample transport and to extrapolate the irreversibility limits to zero amplitude. Otherwise, the magnetic irreversibility line appears below the zero resistance line, as seen in the work by Doyle *et al.* [9], which flagrantly contradicts established knowledge of superconductivity. In Figs. 4–6, it may be observed that in all of our samples, when the magnetic field is increasing, the zero resistance data (T_{c0}) deviate from the magnetic T_{irr} (H) lines toward lower temperatures, both for $H //$ ab and $H //$ c. This is a well known feature of superconductors that is due to under-pinning of the pinning centers by the low density of fluxons and hence still large mobility of the fluxons. Another well known feature of the melt-processed materials is the anisotropic pinning due to the strongly anisotropic (elongated) form and the geometrical arrangement of the Y211 grains [10], especially in the materials processed by the top-seeding method [11]. The growth of the Y211 grains and their geometrical configuration during the crystallization process has a complicated dependence on the temperature gradient and on the crystallization fronts. This anisotropic pinning affects differently the flux mobility along the several directions in the sample. Hence, the Lorentz force of the applied measuring current and the intrinsic pinning are not the unique features determining the magnetic flux mobility for the different field-current configurations. Theoretically the Lorentz force of the measuring current on the magnetic fluxons is strongest when the electric current flows perpendicularly to the direction of the applied magnetic field and is weakest when it is parallel. Magnetoresistivity measurements in very good $YBa_2Cu_3O_{7-\delta}$ single crystals agree with this expectation [8].

However, the results in the Figs. 4–6 show some anomalous scatter. Although for $H //$ c-axis these deviations are low, falling within the experimental error bar, for $H //$ ab-plane they are more significant. In fact, because of the large c-axis-ab-plane anisotropy in the magnetization and in the resistivity of the Y123 superconductors, the local field-current configuration may be much more complicated than theoretically expected. While the magnetic field locally has a strong tendency to go along the c-axis, the current flows more easily along the ab-planes. Besides this, we must not forget the small misalignment of the pretended sample axis with the applied magnetic field, especially for $H //$ ab-plane. Misalignment favors disarray of the field. In our view, the results displayed in Figs. 4–6 show that the anisotropic pinning of the Y211 grains introduces considerable anisotropy in the flux mobility that in most of our melt-processed Y123 samples supersedes the Lorentz forces. We appoint the anisotropic flux pinning by the Y211 grains as the main reason of the subtle anomalous dispersion in the T_{c0} (H) lines for the different field-current configurations for $H //$ ab-plane.

V. CONCLUSION

We have investigated three different melt-textured $YBa_2Cu_3O_{7-\delta}$ samples in order to obtain a precise and complete profile of the correlation between the magnetic T_{irr} (H) lines and the T_{c0} (H) data, and consequently the flux mobility and the pinning mechanisms in the different field-current configurations. The results show the usual large anisotropy of the magnetic T_{irr} (H) lines and of the electric resistance due to the intrinsic pinning by the planar superconducting structure. This large anisotropy evidences the well aligned c-axes of the crystallites and hence the good quality of our samples. The data also show that, although their polycrystalline character, the melt-textured $YBa_2Cu_3O_{7-\delta}$ samples do not show the features of superconducting granularity. The resistivity of these samples falls to zero closely underneath the magnetic T_{irr} (H) lines for $H //$ c-axis and for $H //$ ab-plane as expected from the well established science of superconductivity. However, in order to achieve this, it was necessary to refine the standard method for measuring magnetic irreversibility.

REFERENCES

- [1] S. Sanfilippo *et al.*, “Experimental evidence of twin-planes pinning in the ab plane studied in large c-axis $YBa_2Cu_3O_7$ samples,” *Phys. Rev. B*, vol. 58, no. 22, pp. 15 189–15 197, Dec. 1998.
- [2] P. Diko, K. Zmorayova, X. Granados, F. Sandiumenge, and X. Obradors, “Growth related Y_2BaCuO_5 particle concentration micro-inhomogeneity in the growth sectors of TSMG $YBa_2Cu_3O_7/Y_2BaCuO_5$ bulk superconductor,” *Phys. C, Supercond.*, vol. 384, no. 1/2, pp. 125–129, Jan. 2003.
- [3] K. Zmorayova, P. Diko, A. E. Carrillo, F. Sandiumenge, and X. Obradors, “Quantitative analysis of Y_2BaCuO_5 particle distribution in a melt-grown $YBa_2Cu_3O_7/Y_2BaCuO_5$ bulk superconductor,” *Supercond. Sci. Technol.*, vol. 18, no. 7, pp. 948–952, Jul. 2005.
- [4] F. T. Dias *et al.*, “Magnetic irreversibility: An important amendment in the zero-field-cooling and field-cooling method,” *Jpn. J. Appl. Phys.*, vol. 55, no. 2, Feb. 2016, Art. ID 023101.
- [5] Y. Yeshurun and A. P. Malozemoff, “Giant flux creep and irreversibility in an Y-Ba-Cu-O crystal: An alternative to the superconducting-glass model,” *Phys. Rev. Lett.*, vol. 60, no. 21, pp. 2202–2205, May 1988.

- [6] J. R. L. de Almeida and D. J. Thouless, "Stability of the Sherrington-Kirkpatrick solution of a spin glass model," *J. Phys. A, Math. Gen.*, vol. 11, no. 8, pp. 983–990, May 1978.
- [7] M. Gabay and G. Toulouse, "Coexistence of spin-glass and ferromagnetic orderings," *Phys. Rev. Lett.*, vol. 47, no. 3, pp. 201–204, Jul. 1981.
- [8] F. T. Dias *et al.*, "Correlation between the magnetic irreversibility limit and the zero resistance point in different granular $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductors," *Phys. Rev. B, Condens. Matter Mater. Phys.*, vol. 77, no. 13, Apr. 2008, Art. ID 134503.
- [9] R. A. Doyle *et al.*, "High field behavior of artificially engineered boundaries in melt-processed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$," *Appl. Phys. Lett.*, vol. 73, no. 1, pp. 117–119, Jul. 1998.
- [10] C.-J. Kim and G.-W. Hong, "Defect formation, distribution and size reduction of Y_2BaCuO_5 in melt-processed YBCO superconductors," *Supercond. Sci. Technol.*, vol. 12, no. 3, pp. R27–R41, Mar. 1999.
- [11] J. Figueras, T. Puig, and X. Obradors, "Influence of twin boundaries and randomly oriented correlated disorder on the liquid vortex plasticity of $\text{YBa}_2\text{Cu}_3\text{O}_7$," *Phys. Rev. B, Condens. Matter Mater. Phys.*, vol. 67, no. 1, Jan. 2003, Art. ID 014503.

AUTHOR QUERY

NO QUERY.

IEEE Proof

Comparison Between the Magnetic Irreversibility and Zero Resistance of High-Quality Melt-Processed YBaCuO Superconductors

Fábio Teixeira Dias, Valdemar das Neves Vieira, Frederik Wolff-Fabris, Erik Kampert, Marlon Hneda, Jacob Schaf, Grazielle Fernanda Farinela, Cristol de Paiva Gouvêa, and Joan Josep Roa Rovira

Abstract—This paper portrays a detailed study of the magnetic irreversibility limit T_{irr} (H) and of the zero resistance point T_{c0} (H) of three different top-seeding melt-textured $YBa_2Cu_3O_{7-\delta}$ superconducting samples, with well-aligned c-axis and doped with a high density of nonsuperconducting $Y_2Ba_1Cu_1O_5$ (Y211) pinning centers. We have performed measurements for applied magnetic fields up to 140 kOe and for the whole set of the different field-current configurations. The magnetization measurements were performed using an MPMS-XL SQUID magnetometer and a vibrating sample magnetometer, both from Quantum Design. The electric transport measurements were made using a physical properties measurement system from Quantum Design. The goal of this exhaustive study is obtaining precise data about magnetic flux mobility along the various directions in the sample for the different field-current configurations, thereby defining the nature and effects, due to the strength and anisotropy of the pinning mechanisms and disclosing the various physical mechanisms dissipating electric transport in these systems below the superconducting transition temperature. We discuss our results in terms of the anisotropic flux pinning by the Y211 grains dispersed into the superconducting matrix.

Index Terms—Flux pinning, granular superconductors, high-temperature superconductors, type-II superconductors.

I. INTRODUCTION

THEORETICALLY, below the superconducting transition temperature of homogeneous superconductors a non-zero electric current can flow without any resistance. In zero applied magnetic field the electric resistance falls to zero closely underneath the T_c . However, if a magnetic field is present inside the

superconducting material, the electric current will interact with the quantized magnetic fluxons causing magnetic flux dynamics and dissipation of its energy. It can flow without resistance, only if the magnetic fluxons are sufficiently stabilized by pinning centers. In homogeneous type II superconductors, under an applied magnetic field, the magnetic irreversibility limit line T_{irr} (H) is a boundary, in the field-temperature (H-T) plane, below the superconducting transition at which the flux pinning just begins to stabilize the first magnetic fluxons. However, the pinned fluxons percolate through the sample only at some temperature below the T_{irr} line. Hence, a nonzero electric current can flow without resistance only somewhat below this T_{irr} (H) line.

Our high quality melt-processed $YBa_2Cu_3O_{7-\delta}$ samples are doped with up to 30 percent of non-superconducting $Y_2Ba_1Cu_1O_5$ (Y211) particles, which provides them with a high density of very effective flux pinning centers [1]. The goal of our present study is to disclose experimentally the correlation between the magnetic irreversibility limit lines, T_{irr} (H), and the zero resistance lines, T_{c0} (H), for all the different field-current configurations and thereby obtaining information about the magnetic flux mobility in the various directions and the respective flux pinning mechanisms. We have obtained precise profiles of T_{irr} (H), corrected for relaxation effects, due to field non-homogeneity of the magnetometers and of T_{c0} (H) as a function of applied field for three high quality melt-processed $YBa_2Cu_3O_{7-\delta}$ samples.

II. EXPERIMENTAL PROCEDURES AND TECHNIQUES

A. Sample Preparation

The three melt-processed $YBa_2Cu_3O_{7-\delta}$ samples investigated in this work were produced at the Institute of Materials Science of Barcelona, and at the Department of Materials Science and Metallurgical Engineering of the University of Barcelona, in Spain. The samples, labeled as TS17, TS30-I, and TS30-II, were prepared by the top-seeding method. Single crystalline seeds of the $Nd_1Ba_2Cu_3O_{7-\delta}$ superconductor were placed on top of ceramic $YBa_2Cu_3O_{7-\delta}$ pellets in order to induce the crystallographic texture. More information may be found in [2] and [3].

The top-seeding sample TS17 was produced starting from a ceramic pellet of $YBa_2Cu_3O_{7-\delta}$ containing 17 wt% of the

Manuscript received September 7, 2015; accepted January 22, 2016. This work was supported under the Brazilian Grant MCTI/CNPq Universal 14/2012 (Contract 477506/2012-7). The experimental work at the HLD was supported by EuroMagNET II (Contract 228043).

F. T. Dias and V. das Neves Vieira are with the Instituto de Física e Matemática, Universidade Federal de Pelotas, 96010-900 Pelotas-RS, Brazil (e-mail: fabio.dias@ufpel.edu.br).

F. Wolff-Fabris and E. Kampert are with Dresden High Magnetic Field Laboratory, Helmholtz-Zentrum Dresden-Rossendorf, 01314 Dresden, Germany.

M. Hneda, J. Schaf, and G. F. Farinela are with the Instituto de Física, Universidade Federal do Rio Grande do Sul, 91501-970 Porto Alegre-RS, Brazil.

C. de Pavia Gouvêa is with Inmetro, 25250-020 Duque de Caxias-RJ, Brazil. J. J. R. Rovira is with Universitat Politècnica de Catalunya, 08028 Barcelona, Spain.

Color versions of one or more of the figures in this paper are available online at <http://ieeexplore.ieee.org>.

Digital Object Identifier 10.1109/TASC.2016.2526005

Fig. 1. SEM image of the TS30-I sample showing profiles of the platelet structure stacked along the c-axis as a consequence of the texture process.

Fig. 2. Magnetoresistance data for TS30-II sample in the configuration $H // J // ab$ from 0 up to 140 kOe. The inset shows the zero resistance temperature for the magnetic fields of 0 and 140 kOe, denoted as T_{c0} (H).

76 non-superconducting Y211 phase. The two top-seeding melt-
77 processed TS30-I and TS30-II samples were cut from two
78 different top-seeding melt-processed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ buttons
79 containing 30 wt% of the non-superconducting Y211 phase.

80 The samples were small parallelepipeds with 4 mm in length
81 along the ab plane, 1.9 mm in thickness (ab plane) and 2.2 mm
82 along the c-axis. We have characterized the samples by electron
83 microscopy in order to verify the grain alignment. Fig. 1 is
84 a representative image obtained for TS30-I sample using a
85 scanning electron microscope Nova NanoLab 600 from Fei
86 Company. Fig. 1 shows profiles of long platelets (superconduct-
87 ing grains) along the ab plane stacked along the c-axis. Similar
88 structures could be observed in all of the investigated samples.
89 The good quality of the samples also was characterized by
90 measurements of the critical currents. In 5 K, all the samples
91 supported critical currents of about 10^6 A/cm^2 up to about
92 20 kOe and more than 10^5 A/cm^2 up to 50 kOe.

93 B. Physical Measurements

94 The magnetoresistance measurements were made with
95 a PPMS (Physical Properties Measurement System) from
96 Quantum Design and under applied magnetic fields ranging
97 from zero up to 140 kOe. Good four electrical contacts on the
98 surface of the samples were achieved by heating with freshly
99 painted silver paint stripes to 420°C in oxygen for 1.5 h. The
100 measurements were made by applying a low measuring current
101 density parallel to the ab plane and also along the c-axis in
102 the five possible field-current configurations: $H // J // ab$,
103 $H // ab - J // ab$ ($H \perp J$), $H // ab - J // c$, $H // c -$
104 $J // c$, and $H // c - J // ab$. The same samples used
105 in the magnetoresistance experiments were employed for the
106 magnetic measurements.

107 Fig. 2 shows magnetoresistance results of the TS30-II sample
108 for magnetic fields within the range of from 0 up to 140 kOe
109 applied parallel to the electric current and both along the ab
110 plane. We obtained the zero resistance temperature, denoted
111 by T_{c0} , adopting a simple and practical criterion, where the zero
112 resistance temperature is the beginning of the plateau where
113 dR/dT definitely falls to zero value. The inset of Fig. 2 displays

details of this method to define the limit of zero resistance in
our samples, and the arrows show the points where the electric
resistance becomes zero for 0 and 140 kOe. The error in the
determination of zero resistance is less than 0.5 K for all applied
fields.

The magnetization measurements were always performed on
the same samples used in the magnetoresistance measurements,
with an MPMS-XL SQUID magnetometer and a vibrating
sample magnetometer (VSM) both from Quantum Design in
magnetic fields from 0.01 to 140 kOe applied along the c-axis
or parallel to the ab plane. The magnetic irreversibility limit
was obtained by using the standard method consisting in first
cooling the sample down to a low temperature in zero applied
field (ZFC), switching on a desired field and measuring the
magnetic moment M (T) while warming the sample (rate of
0.3 K/min) in the constant (persistent) field up to a temperature
above the superconducting transition temperature, and subse-
quently measuring M (T) while cooling the sample back in the
same magnetic field (FC procedure).

The experimental results were always corrected for temper-
ature gradient effects. The irreversibility limit, T_{irr} (H), for
an applied magnetic field is the temperature point where the
difference FC-ZFC deviates from the zero baseline, marked by
the high-temperature experimental data, where the magnetic
moment is always reversible. Fig. 3 is representative and shows
our criterion adopted to define the magnetic irreversibility
limit. It shows the difference between FC and ZFC magnetic
moments for the TS30-I sample under a magnetic field of 1 kOe
applied along the ab plane. The arrow shows the irreversibility
limit T_{irr} (H) for the specific magnetic field, and the inset shows
the original ZFC and FC curves. By this method we could
determine $T_{irr}(H)$ within an experimental error lower than
0.5 K and in low fields lower than 0.2 K.

III. EXPERIMENTAL RESULTS

A. Magnetic Irreversibility

The magnetic irreversibility line T_{irr} (H) of the samples
was determined for applied magnetic fields ranging from
0.01 kOe up to 140 kOe. Figs. 4–6 display the irreversibility
lines of the three top-seeding melt-processed samples TS17, TS21,

Fig. 3. Difference between FC and ZFC magnetic moments for a magnetic field of 1 kOe applied along the ab plane of the TS-30-I sample. The arrow indicates the irreversibility limit and the inset shows the original ZFC and FC curves.

Fig. 6. Same as Fig. 4 for TS30-II sample, for a maximum magnetic field of 140 kOe.

Fig. 4. Magnetic irreversibility lines $T_{irr}(H)$ for $H // ab$ (blue) and for $H // c$ (red), and the zero resistance lines $T_{c0}(H)$ for all the possible field-current configurations, according to listed in the inset, for the TS-17 sample. The coarse lines through the $T_{irr}(H)$ data are fittings with the gfc equation (see text), and the fine lines through the $T_{c0}(H)$ data are only guides to the eyes.

Fig. 5. Same as Fig. 4, for TS30-I sample.

TS30-I and TS30-II, for magnetic fields H applied along the 153 c-axis ($H // c$) and along the ab-plane ($H // ab$). In fact 154 these T_{irr} data were corrected for the effects of relaxation, 155 induced by motion of the sample in the non-homogeneous field 156 within the sample space of the magnetometers. In 50 kOe of 157 applied field, we observed displacements of T_{irr} of 7.5 K to 158 lower temperature. In order to correct for these distortions, we 159 have measured the magnetic irreversibility as a function of the 160 amplitude of the sample transport and extrapolated the obtained 161 T_{irr} to zero amplitude, which gave a plausible and visibly the 162 correct irreversibility limit [4]. The continuous lines through 163 the $T_{irr}(H)$ data are fittings with the usual equation describing 164 the profile of magnetic irreversibility as a function of applied 165 magnetic field, related with the giant flux creep (gfc) [5], 166 $H_{irr}(T) = H_0(1 - t)^\alpha$, where $\alpha = 3/2$. In the equation, $t = 167 T_{irr}(H)/T_{irr}(0)$ is the reduced temperature, H_0 is the value 168 of the irreversibility field extrapolated to zero temperature, 169 and $T_{irr}(0)$ is the irreversibility temperature at zero applied 170 magnetic field. All the magnetic irreversibility data are well 171 fitted with this equation. 172

The anisotropy for $H // ab$ and $H // c$ is large, similar 173 to observations in $YBa_2Cu_3O_{7-\delta}$ single crystals, and shows 174 the good alignment of the crystallite c-axis. The irreversibility 175 lines of all our melt-processed samples do not exhibit the de 176 Almeida-Thouless [6] and the Gabay-Toulouse [7] regimes in 177 the low field region, typical of granular systems [8]. Usually 178 good melt-processed materials do not display these character- 179 istics of superconducting granularity, which attests to the good 180 crystallite alignment as well as very close crystallite coupling. 181

B. Magnetoresistivity

182

Figs. 4–6 also show the zero resistance data as a function of 183 applied field for each one of our three melt-processed samples, 184 and for all the five possible field-current configurations. The 185 continuous lines are a guide to the eyes. 186

Our zero resistance lines $T_{c0}(H)$ in Figs. 4–6 show the 187 same c-axis-ab-plane anisotropy as the magnetic irreversibility. 188 Moreover, the T_{c0} lines fall all closely underneath the T_{irr} 189 lines for the respective directions of the applied field. These 190

results show that the electric resistance below the transition temperature is due to excitation of magnetic flux dynamics. In this case the magnetic flux pinning avoids the electric current from dissipating its energy. In relation to applied fields along the c-axis, the two zero resistance lines fall closely together and practically are coincident. On the other hand, for magnetic fields along the ab plane, we observe differences larger than the error bars and for some field-current configurations the T_{c0} lines even lie significantly below the T_{irr} line.

IV. DISCUSSION

The usual idea that the magnetic irreversibility line T_{irr} (H) is a boundary in the $H \times T$ plane, below which a non-zero electric current may flow without resistance, stems from the conventional homogeneous isotropic metallic alloy superconductors with high density of pinning centers. In general this is not true for the high-temperature superconductors (HTSC). The coherence length in the HTSC is very short (less than 10 Å). They are planar and intrinsically granular superconductors [8]. However, the present work shows that good melt-processed polycrystalline $YBa_2Cu_3O_{7-\delta}$ materials exhibit large intrinsic anisotropy, and do not show the characteristics of superconducting granularity. In the melt-processed materials, doped with a high density of pinning centers, the electric resistivity falls to zero closely underneath the magnetic T_{irr} (H) line for H // ab-planes, as well as for H // c-axis as shown by Figs. 4–6. However, in order to get these T_{irr} (H) lines to the right place, it was necessary to measure the magnetic irreversibility for each applied field as a function of the amplitude of the sample transport and to extrapolate the irreversibility limits to zero amplitude. Otherwise, the magnetic irreversibility line appears below the zero resistance line, as seen in the work by Doyle *et al.* [9], which flagrantly contradicts established knowledge of superconductivity. In Figs. 4–6, it may be observed that in all of our samples, when the magnetic field is increasing, the zero resistance data (T_{c0}) deviate from the magnetic T_{irr} (H) lines toward lower temperatures, both for H // ab and H // c. This is a well known feature of superconductors that is due to under-pinning of the pinning centers by the low density of fluxons and hence still large mobility of the fluxons. Another well known feature of the melt-processed materials is the anisotropic pinning due to the strongly anisotropic (elongated) form and the geometrical arrangement of the Y211 grains [10], especially in the materials processed by the top-seeding method [11]. The growth of the Y211 grains and their geometrical configuration during the crystallization process has a complicated dependence on the temperature gradient and on the crystallization fronts. This anisotropic pinning affects differently the flux mobility along the several directions in the sample. Hence, the Lorentz force of the applied measuring current and the intrinsic pinning are not the unique features determining the magnetic flux mobility for the different field-current configurations. Theoretically the Lorentz force of the measuring current on the magnetic fluxons is strongest when the electric current flows perpendicularly to the direction of the applied magnetic field and is weakest when it is parallel. Magnetoresistivity measurements in very good $YBa_2Cu_3O_{7-\delta}$ single crystals agree with this expectation [8].

However, the results in the Figs. 4–6 show some anomalous scatter. Although for H // c-axis these deviations are low, falling within the experimental error bar, for H // ab-plane they are more significant. In fact, because of the large c-axis-ab-plane anisotropy in the magnetization and in the resistivity of the Y123 superconductors, the local field-current configuration may be much more complicated than theoretically expected. While the magnetic field locally has a strong tendency to go along the c-axis, the current flows more easily along the ab-planes. Besides this, we must not forget the small misalignment of the pretended sample axis with the applied magnetic field, especially for H // ab-plane. Misalignment favors disarray of the field. In our view, the results displayed in Figs. 4–6 show that the anisotropic pinning of the Y211 grains introduces considerable anisotropy in the flux mobility that in most of our melt-processed Y123 samples supersedes the Lorentz forces. We appoint the anisotropic flux pinning by the Y211 grains as the main reason of the subtle anomalous dispersion in the T_{c0} (H) lines for the different field-current configurations for H // ab-plane.

V. CONCLUSION

We have investigated three different melt-textured $YBa_2Cu_3O_{7-\delta}$ samples in order to obtain a precise and complete profile of the correlation between the magnetic T_{irr} (H) lines and the T_{c0} (H) data, and consequently the flux mobility and the pinning mechanisms in the different field-current configurations. The results show the usual large anisotropy of the magnetic T_{irr} (H) lines and of the electric resistance due to the intrinsic pinning by the planar superconducting structure. This large anisotropy evidences the well aligned c-axes of the crystallites and hence the good quality of our samples. The data also show that, although their polycrystalline character, the melt-textured $YBa_2Cu_3O_{7-\delta}$ samples do not show the features of superconducting granularity. The resistivity of these samples falls to zero closely underneath the magnetic T_{irr} (H) lines for H // c-axis and for H // ab-plane as expected from the well established science of superconductivity. However, in order to achieve this, it was necessary to refine the standard method for measuring magnetic irreversibility.

REFERENCES

- [1] S. Sanfilippo *et al.*, “Experimental evidence of twin-planes pinning in the ab plane studied in large c-axis $YBa_2Cu_3O_7$ samples,” *Phys. Rev. B*, **288** *Condens. Matter Mater. Phys.*, vol. 58, no. 22, pp. 15 189–15 197, Dec. 1998.
- [2] P. Diko, K. Zmorayova, X. Granados, F. Sandiumenge, and X. Obradors, “Growth related Y_2BaCuO_5 particle concentration micro-inhomogeneity in the growth sectors of TSMG $YBa_2Cu_3O_7/Y_2BaCuO_5$ bulk superconductor,” *Phys. C, Supercond.*, vol. 384, no. 1/2, pp. 125–129, Jan. 2003.
- [3] K. Zmorayova, P. Diko, A. E. Carrillo, F. Sandiumenge, and X. Obradors, “Quantitative analysis of Y_2BaCuO_5 particle distribution in a melt-grown $YBa_2Cu_3O_7/Y_2BaCuO_5$ bulk superconductor,” *Supercond. Sci. Technol.*, vol. 18, no. 7, pp. 948–952, Jul. 2005.
- [4] F. T. Dias *et al.*, “Magnetic irreversibility: An important amendment in the zero-field-cooling and field-cooling method,” *Jpn. J. Appl. Phys.*, vol. 55, no. 2, Feb. 2016, Art. ID 023101.
- [5] Y. Yeshurun and A. P. Malozemoff, “Giant flux creep and irreversibility in an Y-Ba-Cu-O crystal: An alternative to the superconducting-glass model,” *Phys. Rev. Lett.*, vol. 60, no. 21, pp. 2202–2205, May 1988.

- [6] J. R. L. de Almeida and D. J. Thouless, "Stability of the Sherrington-Kirkpatrick solution of a spin glass model," *J. Phys. A, Math. Gen.*, vol. 11, no. 8, pp. 983–990, May 1978.
- [7] M. Gabay and G. Toulouse, "Coexistence of spin-glass and ferromagnetic orderings," *Phys. Rev. Lett.*, vol. 47, no. 3, pp. 201–204, Jul. 1981.
- [8] F. T. Dias *et al.*, "Correlation between the magnetic irreversibility limit and the zero resistance point in different granular $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ superconductors," *Phys. Rev. B, Condens. Matter Mater. Phys.*, vol. 77, no. 13, Apr. 2008, Art. ID 134503.
- [9] R. A. Doyle *et al.*, "High field behavior of artificially engineered boundaries in melt-processed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$," *Appl. Phys. Lett.*, vol. 73, no. 1, pp. 117–119, Jul. 1998.
- [10] C.-J. Kim and G.-W. Hong, "Defect formation, distribution and size reduction of Y_2BaCuO_5 in melt-processed YBCO superconductors," *Supercond. Sci. Technol.*, vol. 12, no. 3, pp. R27–R41, Mar. 1999.
- [11] J. Figueras, T. Puig, and X. Obradors, "Influence of twin boundaries and randomly oriented correlated disorder on the liquid vortex plasticity of $\text{YBa}_2\text{Cu}_3\text{O}_7$," *Phys. Rev. B, Condens. Matter Mater. Phys.*, vol. 67, no. 1, Jan. 2003, Art. ID 014503.

AUTHOR QUERY

NO QUERY.

IEEE Proof