Unconventional superconducting granularity of the $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ compound

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We report on fluctuation magnetoconductivity and magnetic irreversibility of $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ single crystals and a polycrystalline sample. Although our samples are all single phase (orthorhombic) and the single crystals show no sign of structural inhomogeneity, all the samples exhibit two close and sharp genuine superconducting transitions. On the other hand, while the resistive transition of the polycrystalline sample exhibits in addition a coherence transition characteristic of a granular superconductor and the magnetic irreversibility displays the signature of the intergrain flux dynamics, the single crystals show no sign of these features. In view of these facts, we conclude that the well characterized split superconducting transition must result from a peculiar phase separation related with oxygen doping.

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I. INTRODUCTION

Chemical substitutions in the $YBa_2Cu_3O_{7-\delta}$ compound have been an important tool to disclose the planar superconducting morphology of the high-T_c superconducting cuprates (HTSC). The experimental observations, however, have not always been so clear-cut. Contrasting with the rather mild effect on the superconducting properties by most rare-earth substitutions at the Y site,[1–5] doping with Pr, Ce, or Nd has been observed to severely depress the superconducting transition temperature.[6–11] In the particular case of Pr doping, the location of Pr in the crystal lattice by usual neutron and x-ray diffraction is especially difficult. However, polarized neutron scattering[12] and magic-angle spinning nuclear magnetic resonance[13] have evidenced that some Pr goes to the Ba site. On this ground people have related the T_c depression to the misplaced Pr ions.[14–16] Careful analysis of resistivity data of $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$, $Y_{1-x}Pr_xCa_xBa_2Cu_3O_{7-\delta}$, and $YBa_{2-x}Pr_xCu_3O_{7-\delta}$ samples, in which Pr expressly replaces Y atoms and/or Ba atoms, indicated that Pr at Ba sites causes weak pair breaking (linear in x) and a largely dominant hole filling effect (quadratic in x).[17,18]

Scarcity of information specifically on the exact nature of the superconducting transition and on the morphology of the superconducting state was the early motivation of the present work. Initially the occurrence of two close inflections in the magnetization curves and two very sharp resistive transitions in the polycrystalline sample was seen as an indication of structural problems of our samples. Nevertheless, the observation of quite analogous split transitions in the single crystals with no trace of structural inhomogeneity strongly indicated a more unusual inhomogeneity. Split resistive transitions have often been observed in HTSC samples,[7,8,10,19–22] but in general could be attributed to conventional sample inhomogeneities. This, however, is not possible anymore in the case of our single crystals.

II. SAMPLE PREPARATION AND MEASURING TECHNIQUES

We have prepared a pellet of polycrystalline $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ by the usual solid-state reaction technique. From the pellet we have cut a sample for the magnetic measurements and a parallelepiped of $8\times1\times1$ mm^3 for the magnetoresistivity measurements. Single crystals with the same nominal stoichiometry $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ were grown in a tilted ZrO_2 tray by the CuO self-flux method. For our study we chose two single crystals from the same batch with a very clean appearance and an elongated rectangular shape with about 1 mm^2 in area and nearly 0.1 mm in thickness. Energy dispersive x-ray analysis indicated the presence of Pr in all samples to be distributed homogeneously over each single crystal. The Pr concentration, however, changed considerably in different crystals of the same batch. This is confirmed by the rather different T_c values. X-ray diffraction showed the pure orthorhombic structure of all samples. Detailed inspection of several $K\alpha_1$ diffraction peaks down to angular resolutions of 10^{-2} degrees showed no conventional structural inhomogeneities. Examination by electron micros-

copy showed that the naked single crystals are of a very homogeneous texture and that the polycrystalline sample is a porous grain aggregate. Moreover, examination of the single crystals by polarized light microscopy gave account of twinning fields throughout.

In our magnetoresistivity measurements we used a four contact, low current-low frequency ac null technique. The experimental setup was a system in which a lock-in amplifier is used as a null detector. The resistivity measurements were performed under applied fields ($H \| ab$) within the range $0 \leqslant H \leqslant 1$ kOe for $H \| J$ while sweeping the temperature very slowly (0.05 K/min). The magnetic irreversibility was measured by a superconducting quantum interference device MPMS-XL magnetometer from Quantum Design. The method consisted of measuring the dc-magnetization while warming the sample after cooling in zero field (M_{ZFC}) and then while cooling back in the same field (M_{FC}).

III. EXPERIMENTAL RESULTS

A. Magnetoresistive transition

Usually, the electric resistivity of a superconductor falls steeply to zero below the transition temperature. In granular superconductors, however, this occurs only after a coherence transition, when the Josephson coupling energy between the grains overcomes the phase entropy.[23,24] This coupling is known to depend strongly on the applied field. The upper-left panel in Fig. 1 displays $\rho(H,T)$ of the polycrystalline sample under the indicated applied fields and a measuring current density $J=55$ mA/cm^2. The upper-right panel displays the resistive transition of a single crystal measured for a current density J of 500 mA/cm^2 along the ab plane $[H \| J]$ under the indicated applied fields. The profile of the resistive transition is well-known to be practically the same for field applied along the c-axis or the ab-planes. The lower panels in Fig. 1 display the respective temperature derivatives $d\rho/dT$. Note that $\rho(H,T)$ falls in two equally abrupt steps of about the same magnitude at $T_{c_1}=92.3$ K and $T_{c_2}=91.2$ K in the case of the polycrystalline sample and $T_{c_1}=70.4$ K and $T_{c_2}=69.2$ K in the case of the single crystal. From the T_c values of the polycrystalline sample it is seen that the Pr concentration is close to the nominal concentration.[8] Scaling the T_c value of our single crystal onto the curve of Ref. 8 would lead to 24 at. % Pr. The concentration of Pr in the single crystals is hardly controllable. The considerably lower T_c values of the single crystals, used in the resistivity measurements, can be explained by a higher Pr concentration or oxygen underdoping. The relatively narrow superconducting transitions, however, indicate that oxygen deficiency can hardly be the cause of this T_c depression. To know the exact cause of T_c depression of our samples is obviously important. However, our main emphasis will be put on specifically the split superconducting transition itself.

The two prominent $d\rho/dT$ peaks of the single crystal and the polycrystalline sample are only weakly affected by the applied magnetic field. However, while the resistivity of the single crystal vanishes at once below T_{c_2}, that of the polycrystalline sample displays a hump below the lowest transition T_{c_2}. The drastic dependence of this hump on the applied magnetic field lets no doubt that this sample is an aggregate of superconducting grains.[23] These granularity symptoms will be retrieved again in the magnetic irreversibility of this sample. The clear split resistive transition of our samples raises an important question about the origin of the two close superconducting transitions.

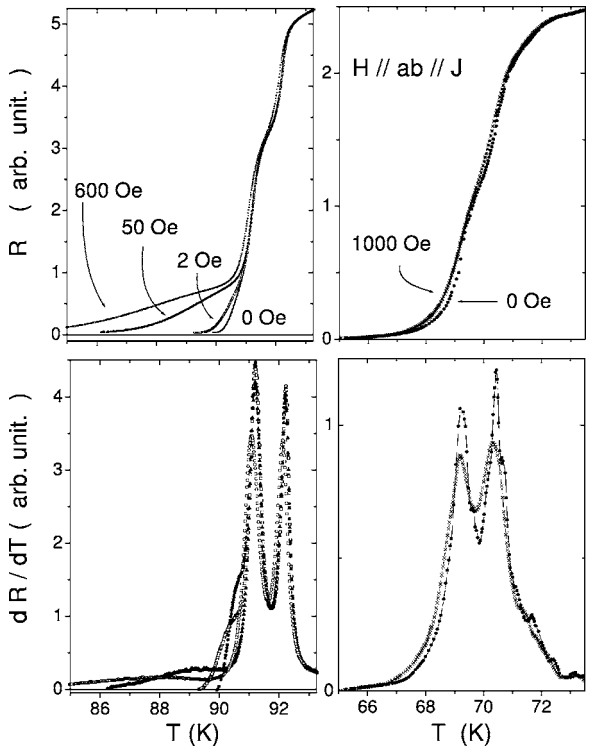

FIG. 1. Upper panels, the magnetoresistive transitions of the $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ polycrystalline sample (left) and the $Y_{0.76}Pr_{0.24}Ba_2Cu_3O_{7-\delta}$ single crystal (right) for the indicated applied fields. The lower panels display the respective temperature derivatives. Note the drastic effect of the applied field on the grain coupling of the polycrystalline sample at the approximation to zero resistivity and its complete absence in the single crystal.

B. Fluctuation magnetoconductivity

The superconducting fluctuations in the HTSC induce significant contributions to the electric conductivity even several degrees above T_c. Their contribution can be estimated by subtracting from the extrapolated linear resistivity, defined by the data well above T_c, the effectively measured resistivity. Assuming that the excess electric conductivity $\Delta\sigma(T)$ diverges when the temperature approaches T_c from above according to the power law[21,25]

$$\Delta\sigma(T) = A(T - T_c)^{-\lambda}, \tag{1}$$

where A is a critical amplitude and λ is a critical exponent. Then we calculate the temperature derivative of $\ln[\Delta\sigma(T)]$ and plot $[\chi_\sigma(T)]^{-1}$, obtaining[21,25]

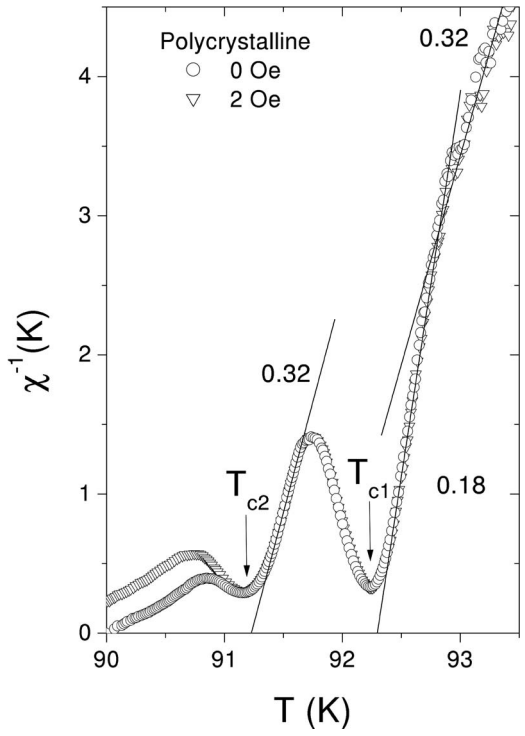

FIG. 2. The inverse of the logarithmic temperature derivative of $\Delta\sigma(T)$ of the polycrystalline sample for the indicated applied fields. The straight-line segments are fittings to Eq. (2) labeled by the respective genuinely critical exponents. Remark the presence of the genuinely critical fluctuation regime ($\lambda_{cr}=0.32$) in-between the two transitions.

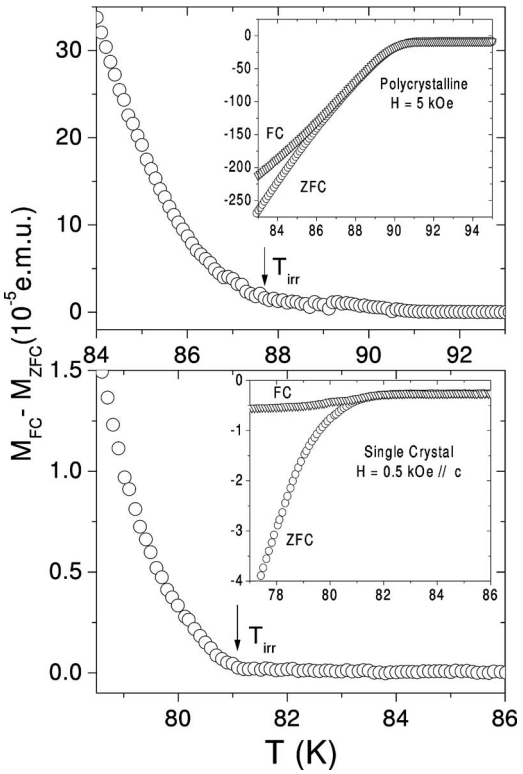

FIG. 3. Typical $\Delta M=M_{FC}-M_{ZFC}$ plots, used to locate the irreversibility limit. The insets show the respective M_{FC} and M_{ZFC} plots.

$$[\chi_\sigma(T)]^{-1}=\frac{1}{\lambda}(T-T_c). \tag{2}$$

The existence of asymptotically critical fluctuation regimes becomes evident if a linear behavior of the data over considerable temperature intervals occurs in this plot.

Figure 2 displays representative plots $[\chi_\sigma(T)]^{-1}$ for the polycrystalline $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ sample for the indicated applied fields, chosen among many others. The straight-line segments through the data are fittings with [Eq. (2)]. They represent well-defined fluctuation regimes predicted by theory and labeled by the respective values of the critical exponents λ. In the immediate vicinity above T_{c_1}, we identify the asymptotic genuinely critical (correlated) fluctuation regime [$\lambda_{cr}=0.18\pm0.01$] precursory of a weakly first-order transition.[25] Extrapolation of this regime to the temperature axis provides the closest approach for $T_{c_1}=92.3$ K. Above this regime the genuinely critical 3D-*XY* fluctuation regime [$\lambda_{cr}=0.32\pm0.02$] is found.[25] The same genuinely critical fluctuation regime, $\lambda_{cr}=0.32$, is also visible, for the first time, in the immediate vicinity above the lowest transition T_{c_2}, in-between the two T_c points. This regime provides $T_{c_2}=91.2$ K. The observation of the genuine critical fluctuation regime closely above T_{c_1} and also above T_{c_2} proves that these two superconducting transitions effectively occur independently from each other and in different regions of the sample. In temperatures beyond the genuinely critical fluctuation regimes we observe 3D, 2D, and 1D Gaussian (uncorrelated) fluctuation regimes.[24] Although the magnetoresistivity data of the single crystal provide a reliable picture of the superconducting morphology, they are not smooth enough for a consistent analysis of the fluctuation regimes.

C. Magnetic irreversibility

For low applied fields the magnetization of the polycrystalline sample shows in low fields two clear initial inflections, one near $T_{c_1}=92.3$ K and another one near $T_{c_2}=91.2$ K. In the single crystal, which was chosen from the same batch as the one used in the resistivity measurements, double inflections are less marked, but the upper one, T_{c_1}, occurs near 85 K, which corresponds to a concentration of about 15 at. % Pr.[8] In our method depicted in Fig. 3, the irreversibility limit $T_{irr}(H)$ is the temperature point where the difference data $\Delta M=M_{FC}-M_{ZFC}$ leaves the zero baseline, established by the data in the upper reversible temperature region. The *H-T* diagram of Fig. 4 displays the irreversibility data $T_{irr}(H)$ of the single crystal and polycrystalline samples. The magnetic irreversibility line $T_{irr}(H)$ of the single crystal exhibits the usual anisotropy for the $H\|c$-axis or $H\|ab$-plane. The continuous lines, labeled fc, through the $T_{irr}(H)$ data of the single crystal and the high field data of the polycrystalline sample are fittings to the power law predicted by the giant-flux-creep theories:[25–27]

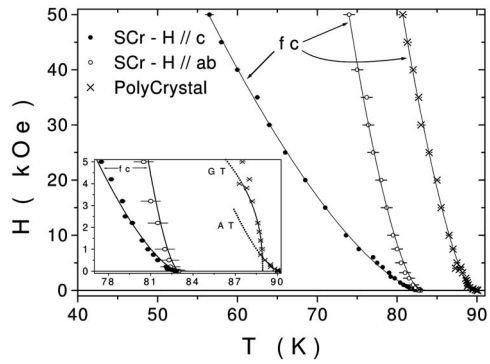

FIG. 4. The magnetic irreversibility data $T_{irr}(H)$ of the $Y_{0.85}Pr_{0.15}Ba_2Cu_3O_{7-\delta}$ single crystal (SCr) for $H\|c$ and $\|ab$ and of the $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ polycrystalline sample (PolyCrystal). The continuous lines through the data labeled fc are fittings to the power law, predicted by the giant-flux-creep theories, [Eq. (3) with $\alpha=\frac{3}{2}$]. The inset shows that while this power law fits well the data of the single crystal down to the lowest fields, the $T_{irr}(H)$ data of the polycrystalline sample follow in a low field region AT $\left[\alpha=\frac{3}{2}\right]$ and GT $\left[\alpha=\frac{1}{2}\right]$ power laws.

$$H_{irr}(T) = H_0(1-t)^{\alpha} \quad \left(\alpha = \frac{3}{2}\right). \tag{3}$$

Here $H_{irr}(T)$ is the irreversibility field as a function of the temperature, $t=T_{irr}(H)/T_{irr}(H=0)$ is the reduced temperature, and H_0 and $T_{irr}(0)$ are fitting parameters (see fitting values in Table I). The inset in Fig. 4 shows that the "fc" power law fits well the data of the single crystal in the whole field range. Nevertheless, in the case of the polycrystalline sample, this equation fits well the data only in fields above 4 kOe. In fields below 0.8 kOe the $T_{irr}(H)$ data are well described by a de Almeida-Thouless (AT)-like power law.[28] Although this power law is mathematically identical to the fc power law, its physical background and the fitting parameters are different,[25,30,31] see Table I. For fields 0.8 kOe$<H<$4 kOe the $T_{irr}(H)$ data follow a Gabay-Toulouse (GT)-like power law, with $\alpha\approx\frac{1}{2}$,[29] see Table I. The possible origin of the AT and GT like irreversibility regimes in the HTSC and of the crossover between these regimes is extensively discussed in Refs. 25 and 30–32 with base in the analogous regimes and crossover occurring in the spin-glass systems.

TABLE I. The power-law exponents α, the parameters H_0 and $T_{irr}(0)$ from fittings of the $T_{irr}(H)$ data of the single crystal (SCr) and the polycrystalline sample (PCr) to the flux-creep (fc), de Almeida-Thouless (AT) and the Gabay-Toulouse (GT) power laws.

Samples	Fit	α	H_0 (kOe)	$T_{irr}(0)$ (K)
SCr ($H\|ab$)	fc	1.56±0.11	1650.43	82.80
SCr ($H\|c$)	fc	1.54±0.04	277.02	82.98
	fc	1.54±0.09	1706.14	89.77
PCr	GT	0.44±0.07	26.03	88.94
	AT	1.31±0.20	204.54	90.13

Entirely similar irreversibility data have been found for pure $YBa_2Cu_3O_{7-\delta}$ single crystals,[33] where, however, the low field behavior of the data was interpreted as a thermal softening.

IV. DISCUSSION

The magnetic irreversibility of superconductors originates from the plastic and dissipative motion of the magnetic flux within the sample. As this plastic motion strongly depends on the flux pinning, it provides information on the physics of the flux pinning mechanism acting within the sample spaces where this motion effectively takes place. The power law, Eq. (3), has been established within the scenario of giant-flux-creep in homogeneous superconducting media. The fact that this power law describes well the high field magnetic irreversibility data of our polycrystalline $Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ sample, which clearly is a granular superconductor, indicates that, under high fields, the magnetic flux does not perceive this granularity. However, the conformity of the low field data to the AT and GT power-law regimes shows that, under low fields, the magnetic flux discerns the superconducting grains and restricts its motions to the intergrain spaces. Therefore it exhibits the physics of the superconducting grain couplings that are strongly affected by the applied field and largely dominated by frustration.[25,30,31] It is hardly imaginable that a thermal softening[33] can lead to such specific and exclusive power law behaviors.

In the magnetoresistance measurements a characteristic signature of a weak linked superconducting grain aggregate is the coherence transition caused by grain coupling. This coherence transition is very sensitive to an applied magnetic field and therefore can thoroughly decide if a given sample is a weak linked aggregate of superconducting grains or a homogeneous superconducting medium.[25,30–32] While our polycrystalline sample displays a clear coherence transition between T_{c_2} and the point of zero resistance, the resistivity of the single crystal falls to zero at once. This strongly excludes the occurrence of a grain coupling process in the single crystal.

Considering the very short coherence length of the HTSC, it is not difficult to relate superconducting granularity with the polycrystallinity of ceramic samples. However, quite often the superconducting granularity features are observed in doped or oxygen depleted single crystals as well. Usually they have been imputed to structural inhomogeneities.[20,25,30,31] The complete absence of the characteristic signature of the Josephson-flux-dynamics in the magnetic irreversibility and the absence of a coherence transition in the magnetoresistive transition of our single crystals strongly exclude the existence of metallurgical grain junctions, but not necessarily that of isolated superconducting grains. On the other hand, all the performed structural analyses consistently show that the single crystals are clean and homogeneous samples. Confrontation of this apparent structural homogeneity and the absence of superconducting grain junctions with the occurrence of two close and sharp resistivity drops about 1 K apart strongly excludes the possibility of a conventional superconducting inhomogeneity in these samples.

Detailed magnetoresistance data are able to display a very detailed picture of granular superconductors. Our results for the single crystals are consistent with a superconducting morphology in which isolated grains or regions, all with closely similar higher superconducting transition temperatures, are imbedded in a matrix with an equally sharp but lower superconducting transition temperature. It is, moreover, important to add that this superconducting morphology establishes itself independently in each single crystal and is present in samples with very different T_c. Effectively, the resistivity of a conductor containing many isolated superconductor grains undergoes a drop at the superconducting transition temperature of the grains. However, as long as these grains do not percolate, this drop is only partial. Only when the matrix becomes superconducting too does the resistivity vanish. Moreover, the fact that the resistivity of our single crystals vanishes at once below the lower superconducting transition (T_{c_2}) shows that below this temperature the sample is not granular but a well-connected and a continuous superconducting medium. Feeble features of split superconducting transitions are often present in experimental data.[8,20–22] However, in our $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ samples they are so explicit and clear-cut that it is impossible to ignore them. Even seeming somewhat exotic they constitute an important morphological possibility that should be known by the experts in superconducting materials. In the most acceptable morphology for the polycrystalline sample, the phase with the higher T_c occurs in the grain cores, while that with the lower T_c occupies the grain boundaries. This sample, however, exhibits a clear coherence transition related with the grain coupling process.

Recently a micro Raman study[34] of a fully oxygenated $Y_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$ compound has evidenced that oxygen overdoping leads to a phase separation in which an overdoped phase with a well-defined but somewhat lowered T_c coexists with a phase of optimally doped material with a higher T_c. Oxygen overdoping in the $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ compound is even more favorable than for the Ca doped compound.[35] Underdoping, however, may lead to a phase separation too. The split resistive transitions of our Pr doped single crystal and polycrystalline samples are the signature of a phase separation in electric transport. In the case of our polycrystalline sample the phase with the lower T_c may occur at the grain boundaries, while the optimally doped material with the higher T_c occupies the grain cores. The morphology of the single crystals may be a matrix of over or underdoped material with a slightly decreased T_c that incorporates isolated regions of optimally doped material. When the temperature is lowered, the optimally doped regions within the single crystal and/or the cores of the grains in the polycrystalline sample become superconducting at T_{c_1}. At this stage the resistivity undergoes a partial drop. At about one degree lower temperature the matrix and/or the grain boundaries become superconducting too and the resistivity falls again. Since the matrix of the single crystals has no metallurgical granularity, in that case the resistivity falls to zero at once. However, in the polycrystalline sample the resistivity only vanishes after a coherence transition that follows with the crystallite grain coupling. This superconducting morphology can explain quite well the experimental features of our samples. We nevertheless agree that the role of Pr and the oxygen stoichiometry in the doublet transition of $YBa_2Cu_3O_{7-\delta}$ is far from solved and needs to be investigated by other techniques.

In summary, our $Y_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ samples, besides the usual magnetic irreversibility and fluctuation magnetoconductivity features, exhibit a curious split resistive transition that reflects two close and narrow superconducting transitions. In our view this split resistive transition arises from a highly unconventional superconducting morphology resulting from a phase separation caused by two well-defined oxygen stoichiometries. While in the single crystals isolated regions of optimally doped material are embedded in a homogeneously over or underdoped superconducting matrix, in the polycrystalline sample the optimally doped grain cores are coated by an oxygen over or underdoped layer with a slightly lower T_c. Although this morphology can in principle explain our results, it should be checked by specific microscopic techniques.

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