Analysis of fluctuation conductivity of polycrystalline $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ superconductors

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In this work we report on conductivity fluctuation measurements in polycrystalline samples of the $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ superconductor ($x=0.00, 0.05$ and $0.10$). Samples were prepared by a standard solid-state reaction technique. The results were analyzed in terms of the temperature derivative of the resistivity and of the logarithmic temperature derivative of the conductivity that allowed the identification of power-law divergences of the conductivity. The results show that the transition proceeds in two stages: pairing and coherence transition. Also, our results, from the critical exponent analysis, show a two-peak splitting at pairing transition, indicating possibly a phase separation. On approaching the zero resistance state, our results show a power-law behavior that corresponds to a phase transition from para coherent to a coherent state of the granular array.

Keywords: High-$T_c$ Superconductor, Erbium, Praseodymium, Critical Phenomena.

1. INTRODUCTION

It is well known that rare earth (RE) substitution for $Y$ in $YBa_2Cu_3O_7$ (Y-123) has little effect on superconductivity, except for $Pr$, $Ce$, $Tb$ and $Pm$ [1]. $Pr$ forms a compound that is isostructural with $Y_{123}$, however it is not a superconductor [2]. On the other hand, $Ce$ and $Tb$ do not form the compound $RE_{123}$ [3-5], and $Pm$ is radioactive with a short half life. In particular, $RE_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ compound has become one of the most studied subjects in the field of high temperature superconductivity. From these studies, it has been observed that increasing $Pr$ substitution in $RE_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ causes a monotonically decrease in the critical temperature ($T_c$) [6]. Some mechanisms such as hole localization [7,8], hole filling [9], magnetic pair breaking [10] and percolation [11] have been proposed to explain the degradation of superconductivity. Also, it was observed an ion-size effect in experiments on the $RE_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ system for $RE = Nd, Eu, Gd, Y$, Er and Yb, indicating an approximately linear $T_c$ decrease with increasing the RE-site ions radius for a fixed dopant amount [12]. Yet, some of $REBa_2Cu_3O_{7-\delta}$ compounds (such as for $RE = Nd, Sm, Gd, Ho$ and $Er$) exhibit antiferromagnetic ordering below $T_c$ [13], showing that investigations about the interplay between superconductivity and magnetism can be of particular interest in this system.

Despite the large amount of research on this system, considerable controversy still persists over the mechanisms of $T_c$ suppression by $Pr$ in $RE_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$. Careful studies of the conductivity in the neighborhood of the critical temperature can yield information about the nature of the superconducting state [14-16] as well as on the influence of the granularity at microscopic and mesoscopic levels on the fluctuation regimes [17-19]. In this paper, we report on fluctuation conductivity measurements in polycrystalline samples of the $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ ($0 \leq x \leq 0.10$) superconductor. The structural characterization of the samples was obtained through Rietveld characterization of the XRD patterns and the results revealed that all samples were nearly single-phase, without dependence on the $Pr$ concentration. Also, using the temperature derivative of the resistivity, $d\rho/dT$, and the logarithmic temperature derivative of the conductivity, $-d\ln(\Delta\sigma)/dT$, we observed the evolution of the critical regimes as a function of praseodymium concentration.

2. EXPERIMENTAL DETAILS

Polycrystalline samples of $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ ($x=0.00, 0.05$ and $0.10$) were prepared by solid-state reaction technique, using $Er_2O_3$, $Pr_6O_{11}$, $BaCO_3$, and $CuO$. Appropriate amounts were mixed and calcinated in air at $850, 880$ and $920^0C$ for $24$ hours, and then slowly cooled through $700^0C$. Finally, the samples were heated in flowing oxygen at $400^0C$ for $48$ hours. The resistivity measurements were performed with a low-frequency-low-current AC technique that employs a lock-in amplifier as a null detector. The measurements current density was up to $600$ $mA/cm^2$. To provide electrical contact to the sample, four stripes of silver paint were painted onto the surface. The silver paint was fixed to the sample by heating it in flowing $O_2$ for $4$ h at $400^0C$. Then, copper wires were attached to the samples with silver paste, resulting in contact resistance of less than $1$ ohm. Temperature was measured with a Pt sensor, allowing a resolution better than $1$ mK, and the resistivity measurements were performed at a $3$ K/h rate or lower.

X-ray powder diffraction patterns were collected from $5^0$ to $100^0$ in the $2\theta$ range with $0.02^0$ step and $4$ s counting time. Rietveld refinements of crystal structures were performed using the GSAS and EXPGUI software [20,21]. The X-ray powder diffraction patterns of $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ for $x=0.00, x=0.05$ and $x=0.10$ are displayed in Fig. 1. The X-ray pattern almost completely matches the orthorhombic $Er_{123}$ structure (compared with JCPSD files), and belongs to the $ErBa_2Cu_3O_{7-\delta}$ orthorhombic unit cell with symmetry $Pmmm$. Also, all samples were found to be nearly single-phase, and show no dependence on the $Pr$ concentration or the presence of trace impurities of $BaCuO_2$, $PrBaO_3$ and $CuO$. The presence of $BaCuO_2$ phase is a result of substitution of $Pr$ at Ba site, while the observation of small quantities of the $BaCuO_2$ and $PrBaO_3$ impurity phases is a...
characteristic of Pr-rich samples [22,23].

The unit cell parameters, atomic parameters, and agreement factors for \(Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}\) (\(x = 0.00, x = 0.05\) and \(x = 0.10\)) obtained through Rietveld refinements at room temperature are summarized in Table I. The value of the lattice parameter \(a\) decreases from 3.8236 (1) Å for \(x = 0.00\) to 3.8165 (1) Å for \(x = 0.10\) while \(c\) also decreases from 11.6778 (2) Å for \(x = 0.00\) to 11.6730 (2) Å for \(x = 0.10\). Meanwhile, \(b\) increases from 3.8792 (1) Å to 3.8839 (2) Å. Also, the volume decreases from 173.213 (8) Å\(^3\) for \(x = 0.00\) to 173.03 (1) Å\(^3\) for \(x = 0.10\). In a general manner, the lattice parameters and volume derived from least squares refinement are in agreement with published results obtained in others studies [24,25]. From Table I we can observe that \(R_p\) (\(R\)−pattern), \(R_{wp}\) (\(R\)−weighted pattern), \(R_e\) (\(R\)−expected) and \(\chi^2 = \sqrt{R_{wp}/R_e}\) (goodness-of-fit) systematically increase with increasing \(x\). However, as the \(R_i\) factors are small, the results are reliable [26,27].

### TABLE I: Unit cell parameters, atomic parameters, and agreement factors for \(Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}\); \(x = 0.00, x = 0.05\) and \(x = 0.10\); obtained through Rietveld refinements at room temperature.

| \(x\)          | \(a(\text{Å})\) | \(b(\text{Å})\) | \(c(\text{Å})\) | \(V(\text{Å}^3)\) | \(R_p\) (%) | \(R_{wp}\) (%) | \(\chi^2\) | \(R_e\) (%) |
|---------------|----------------|----------------|--------------|----------------|-------------|--------------|-----------|-------------|
| \(x = 0.00\)  | 3.8236 (1)     | 3.8792 (1)     | 11.6778 (2)  | 173.213 (8)    | 8.91        | 11.51        | 1.431     | 9.69        |
| \(x = 0.05\)  | 3.8183 (1)     | 3.8855 (1)     | 11.6739 (2)  | 173.197 (8)    | 9.59        | 12.48        | 1.766     | 9.39        |
| \(x = 0.10\)  | 3.8165 (1)     | 3.8839 (2)     | 11.6730 (2)  | 173.03 (1)     | 11.60       | 15.04        | 1.255     | 11.98       |

3. RESULTS AND DISCUSSION

It is well-known that sintered samples of high temperature superconductors show a pronounced granular character that plays an important role in their resistive transition. As the temperature decreases from the normal state, it is first observed a pairing transition and then a coherence transition. At the pairing transition, the superconductivity is stabilized in some small and homogeneous regions of the sample at a temperature which virtually coincides with the critical temperature of the bulk, \(T_C\). These superconducting grains may not necessarily be coincident with the crystallographic grains. At a lower temperature, \(T_{CO}\), the superconductivity is established among the grains. This is the coherence transition, a long-range superconducting state that is achieved through a percolation-like process which controls the activation of weak links between grains. This behavior is very common in polycrystalline samples and it is associated with disorder at mesoscopic level.

In Fig. 2 we show the superconducting transition for three \(Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}\) samples: \(x = 0.00\) (open circle), \(x = 0.05\) (open square) and \(x = 0.10\) (closed circle).

The measurements were performed with current density of \(J = 60 \text{ mA/cm}^2\) and the room-temperature resistivities were \(\rho(300 \text{ K}) = 3.3 \text{ m } \Omega \text{ cm} (x = 0.00), 6.0 \text{ m } \Omega \text{ cm} (x = 0.05)\) and \(2.4 \text{ m } \Omega \text{ cm} (x = 0.10)\). The presented data are normalized to unity at 95 K. The results (not shown) also indicate that our samples present a metallic-like behavior at high temperatures. The panels (a) and (c) show the resistivity versus temperature plots whereas panels (b) and (d) show the plot of \(d\rho/dT\) versus temperature in the corresponding temperature range. The plots of \(d\rho/dT\) versus temperature, shown in panels (b) and (d), present two maxima denoted by \(T_{P1}\) and \(T_{P2}\). These two peaks are associated with a splitting of the pairing transition. The maxima \(T_{P1}\) and \(T_{P2}\) correspond approximately to the pairing critical temperatures \(T_{C1}\) and \(T_{C2}\), respectively. \(T_{P1}\) is approximately 91.8 K \((x = 0.00)\), 93.0 K \((x = 0.05)\) and 88.2 K \((x = 0.10)\) and \(T_{P2}\) is about 91.2 K \((x = 0.05)\) and 85.6 K \((x = 0.10)\). These results indicate that the superconducting pairing transition occurs in two steps of about 1.8 K apart \((x = 0.05)\) sample and with about 4 K \((x = 0.10)\) sample. It is interesting to note that for low concentration of praseodymium \((x = 0.05)\), the value of \(T_{P2}\) is higher than that for the pure system. This effect was also observed for other doped samples [28]. From panels (c) and (d) we can observe that 10 % Pr substitution in the Er sites causes a strong depression of the superconducting transition temperature and a significant enlargement of the width of the resistive transition (10 % to 90 % of the transition) of approximately 2.0 K \((x = 0.00)\) to 6 K \((x = 0.10)\). For the \(x = 0.10\) sample, the whole transition broadens.

3.1. Method of Analysis

We study the effect of thermal fluctuations to the conductivity by using a Kouvel-Fischer-type method [29]. It is assumed that the fluctuation conductivity \(\Delta \sigma\) diverges as a power-law given by

\[
\Delta \sigma = A \varepsilon^{-\lambda},
\]

where \(\Delta \sigma = \sigma - \sigma_0\), \(A\) is the critical amplitude, \(\varepsilon = (T - T_C)/T_C\) is the reduced temperature and \(\lambda\) is the critical exponent. From the resistivity measurements, we calculate
the conductivity $\sigma = 1/\rho$. $\sigma_R$, the regular conductivity, is obtained by linear extrapolation of the resistivity data in the range from $2T_C$ to room temperature.

To obtain the values for $\lambda$ e $T_C$, we determine numerically the logarithmic derivative of $\Delta\sigma$ from experimental data and define

$$\chi_\sigma = -\frac{d}{dT} \ln \Delta\sigma.$$  \hfill (2)

Combining eqs. (1) and (2) we obtain

$$\frac{1}{\chi_\sigma} = \frac{1}{\lambda} (T - T_C).$$  \hfill (3)

FIG. 1: Room temperature XRD patterns of polycrystalline samples of $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$: (a) $x = 0.00$, (b) $x = 0.05$ and (c) $x = 0.10$. The lower curve is the difference between the observed and the calculated intensities.

FIG. 2: The panels (a) and (c) show the resistivity versus temperature plot while the panels (b) and (d) show the temperature derivative of the resistivity versus temperature, around of the critical temperature, for three $Er_{1-x}Pr_xBa_2Cu_3O_{7-\delta}$ samples: $x = 0.00$ (open circle), $x = 0.05$ (open square) and $x = 0.10$ (closed circle). The temperature $T_P$ corresponds approximately to the intragrain pairing transition. The current density was 60 mA/cm$^2$.

FIG. 3: Panels (a) and (b) show representative results of the resistive transition ($J = 60$ mA/cm$^2$) for a $ErBa_2Cu_3O_{7-\delta}$ sample and panels (c) and (d) for a $Er_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ sample. Panels (a) and (c) show $d\rho/dT$ versus temperature and panels (b) and (d) the inverse of the logarithmic derivative of the conductivity ($\chi_\sigma^{-1}$) as a function of $T$. The straight lines correspond to fits to Eq. (3) and the respective exponents are quoted. The inserts show an expanded view of the asymptotic regime of the coherence transition.
The respective exponents are quoted. The insert shows an expanded
measurements, we obtained the mean values of the critical
plotting $\chi$ versus $\sigma$ as a function of $\sigma$. Performing averages over several
with $J = 60 \text{ mA/cm}^2$ and $J = 600 \text{ mA/cm}^2$. The respective exponents are quoted. The insert shows an expanded
view close to $R = 0$ state.

Then, it is possible to determine simultaneously $T_C$ and $\lambda$ by
plotting $\chi^{-1}_a$ versus $T$ [14]. Performing averages over several
measurements, we obtained the mean values of the critical
exponents.

### 3.2. Critical Fluctuation Regimes

In Fig. 3 we compare the resistive transitions of $ErBa_2Cu_3O_{7-\delta}$ ($x = 0.00$) and $Er_{0.95}Pr_{0.05}Ba_2Cu_3O_{7-\delta}$ ($x = 0.05$) measured with current density of $J = 60 \text{ mA/cm}^2$. In panels (a) and (c), $dp/dT x T$ is plotted. In panels (b) and (d), the transition is shown as $\chi^{-1}_a x T$ in the corresponding temperature interval. The figure allows the identification of the asymptotic regime of the coherence critical region (between $T_{CO}$ and $T_P$ for $x = 0.00$ sample and between $T_{CO}$ and $T_P$ for $x = 0.05$ sample) and of the pairing critical region (above $T_P$ for $x = 0.00$ sample and above $T_{P2}$ for $x = 0.05$ sample). The pairing critical region has an internal structure [16], and two power-law regimes are observed in sequence when $T$ approaches $T_{C1}$ from above corresponding to $\lambda_P^{a} \approx 0.34$ and $\lambda_P^{b} \approx 0.15$. From panel 3(b) we can observe, above $T_P$, a full dynamic regime dominated by genuine critical fluctuations labeled by the exponent $\lambda_P^{a} = 0.35 \pm 0.03$. The value of the exponent $\lambda_P^{b}$ is consistent with the predictions for the 3D-XY universality class [14]. Still closer to $T_{C3}$, it is observed a fluctuation regime beyond 3D-XY with an exponent $\lambda_P^{b} = 0.16 \pm 0.03$. This scaling might represent a crossover to an ultimate weakly first-order character of the normal superconducting transition of high temperature cuprates [16]. This critical regime was previously observed in polycrystalline and single-crystal samples [16,30]. Yet, from panel 3(b), we can observe near the zero resistance state ($T_{CO}$) that the fluctuation conductivity can be described by an another power law, given by the equation $\Delta G \propto (T - T_{CO})^{-\lambda}$, with exponent $s = 2.9 \pm 0.3$. The value $s$ is expected value for critical conductivity fluctuations in artificially prepared granular arrays, $s \approx 3$ [32]. The same exponent was also observed to characterize the resistive paracohesence-coherence transition of Y-123 ceramics [14,33].

In panel 3(d), it is observed in sequence, again, two power-law regimes when $T$ approaches $T_{C1}$ from above corresponding to $\lambda_P^{a} = 0.34 \pm 0.03$ and $\lambda_P^{b} = 0.15 \pm 0.02$. The value $\lambda_P^{a} = 0.34$ reveals the effects of genuine fluctuations which belong to the 3D-XY universality class. In the immediate vicinity above $T_{P1}$ we identify a genuinely critical fluctuation regime described by the exponent $\lambda_P^{b} = 0.15$, which is precursor of a weakly first-order transition [16]. Another genuinely critical regime represented by the exponent $\lambda_P^{b} = 0.35 \pm 0.03$ is also visible below $T_P$, in the immediate vicinity above $T_{P2}$. This regime, that was not observed in the $x = 0.00$ sample, is in agreement with the predictions of the full dynamic 3D-XY model [14]. The splitting of the pairing transition, verified by observation of critical regimes $\lambda_P^{a1}$ and $\lambda_P^{b1}$ (above $T_P$) and $\lambda_P^{b2}$ (below $T_P$), could be produced by the presence of two superconducting phases with different temperature transition induced by microscopic granularity. The results displayed in Fig. 2(b) suggest that Pr doping in-
duces a superconducting phase with higher critical temperature (\( \sim 93.1 \) K). A genuine critical fluctuation regime in the temperature range between \( T_{P2} \) and \( T_{P1} \) was first observed by Barros et al. in \( Y_{1-x}Pr_2Ba_2Cu_3O_{y-5} \) single crystals and polycrystalline samples [19,30], and it was associated with a peculiar phase separation related with oxygen doping. Panel 3(d) also shows that, between \( T_{P2} \) and \( T_{C0} \), the variation of \( \chi_0^{-1} \) as a function of temperature is well described by a power law regime with corresponding exponent \( s = 2.9 \pm 0.3 \). The inset shows an expanded view of the paracoherence temperature region. This value is the same observed in the pure system and is interpreted as an indication of a paracoherent-coherence transition of the granular array [14,30].

Fig. 4 displays representative plots of \( \chi_0^{-1} \times T \) for the \( E_{R10}0.90Pr_{0.10}Ba_2Cu_3O_{y-5} (x = 0.10) \) sample for \( J = 60 \) mA/cm\(^2\). Above \( T_{P1} \), the results show the occurrence of a genuine critical regime, with exponent \( \lambda_{P1} = 0.32 \pm 0.01 \) that is consistent with the predictions for the 3D \(- XY\) universality class. It is important to point out that, in this case, a scaling beyond 3D \(- XY\) precursory of a weakly first-order transition [16] was not observed, as noticed for the \( Y_{0.95}Pr_{0.05}Ba_2Cu_3O_{y-5} \) and pure samples. Between \( T_{P2} \) and \( T_{P1} \), we can clearly observe another fluctuation regime with exponent \( \lambda_{P2} = 1.3 \pm 0.2 \). This exponent does not correspond to an integer dimensionality. In this case we suppose that the fluctuations develop in a space having fractal topoloy. According to Char and Kapitulnik [34], the conductivity exponent should be written as \( \lambda = 2 - \frac{d}{T} \) where \( \tilde{d} \) is the fractal dimension of the fluctuation network. Our exponent \( \lambda_{P2} \approx 1.3 \) is consistent with \( \tilde{d} \approx \frac{4}{3} \), which is the well-known fractal dimensionality of the percolation network [35] that corresponds to a crossover regime between 1D and 2D geometry. A similar exponent was also observed in Bi based cuprates [14, 36]. We expected the presence of the 3D \(- XY\) scaling closer to \( T_{P2} \) in the \( x = 0.10 \) sample, as observed for the \( x = 0.00 \) and \( x = 0.05 \) samples. The disappearance of this regime is clear associated with the increase in Pr doping. The observation of the Gaussian regime is related with its robustness against the Pr doping. Again, in approaching the zero resistance state, our results show a power-law behavior with critical exponent \( s = 2.7 \pm 0.1 \) that corresponds to a phase transition from a paracoherent to a coherent state of the granular array [31]. These results indicate that an increasing Pr concentration destroys the pairing critical regimes. On the other hand, 10% of Pr substitution does not affect the coherent transition.

Fig. 5 compares representative plots of \( \chi_0^{-1} \times T \) for the \( x = 0.05 \) sample for two different currents density, \( J = 60 \) mA/cm\(^2\) and \( J = 600 \) mA/cm\(^2\). We can observe that the regimes \( \lambda_{P1} \) and \( \lambda_{P2} \) are stable against \( J \) within the studied range. This behavior is consistent with the interpretation that the observed \( \lambda_{P2} \) regime is related with a pairing (intragrain) transition. Below \( T_{P2} \), and down to the zero-resistance state, the resistivity is current dependent. The figure shows that the higher current intensity shifts \( T_{C0} \) to lower temperatures. Thus, the enlargement of the resistive transition induced by current in our samples is an effect directly related to granularly. Indeed, the self-field is expected to enhance the granular character of the samples by weakening the junctions between grains. The effect of increasing current for the \( x = 0.10 \) sample (not shown) is similar to that observed for the \( x = 0.05 \) sample.

The comparison of the results obtained in the pure and doped systems shows that the Pr doping (until 10%) alters significantly the critical behavior of the \( E_{R10-x}Pr_xBa_2Cu_3O_{y-5} \) compound above \( T_{P2} \). Two possible Pr doping effects can be inferred from our experiments and analysis. The first one is a phase separation, showed in Figs. 3, 4 and 5, and that is indicated by the presence of two genuinely critical regimes above \( T_{P2} \). These critical regimes are not affected by the studied currents. The second effect is the weakness of the superconductivity, which can be observed by the suppression of the pairing critical regimes. The regime beyond 3D \(- XY\) (\( \lambda \approx 0.13 \)) observed above \( T_{P1} \) for \( x = 0.00 \) and \( x = 0.05 \) samples, was not observed for the \( x = 0.10 \) sample. At the same time, the critical regime 3D \(- XY\) (\( \lambda \approx 0.33 \)) observed between \( T_{P1} \) and \( T_{P2} \) for the \( x = 0.05 \) sample, was also suppressed in the \( x = 0.10 \) sample. On the other hand, below \( T_{P2} \), in the paracoherent regime, it was not observed any influence of the Pr doping on the critical exponents. These results indicate that Pr doping induces structural disorder at a microscopic level in the studied system.

4. CONCLUSIONS

In conclusion, our conductivity experiments on granular \( Er_{1-x}Pr_xBa_2Cu_3O_{y-5} (0 \leq x \leq 0.10) \) samples show that the superconducting transition proceeds in two stages: pairing and coherence transition. From the temperature derivative and power-law divergences of the conductivity, our results show a two-peak splitting at pairing transition, indicating possibly a peculiar phase separation associated with Pr doping. Another Pr doping effect is the weakness of the superconductivity, which can be observed through the suppression of genuine critical regimes. On approaching the zero resistance state, our results show a power-law behavior that corresponds to a phase transition from paracoherent to a coherent state of the granular array with exponents that are doping independent.

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