Magnetic phase diagram of the layered superconductor Bi$_{2+x}$Sr$_{2-x}$CuO$_{6+\delta}$ (Bi2201) with $T_c \approx 7$ K

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Abstract

We report on magnetization measurements performed on a single crystal of Bi$_{2+x}$Sr$_{2-x}$CuO$_{6+\delta}$ with $T_c \approx 7$ K for $H \parallel c$-axis. The isofield $M(T)$ curves show a large reversible region, with a pronounced rounding effect as $M$ approaches zero which prevents the determination of $T_c(H)$. Deviations from the linear behavior of magnetization near $T_c(H)$ are studied through the asymptotic behavior of $\sqrt{-M(T)} \propto (T - T_c)^m$, where $m$ is an exponent and $T_c$ an apparent temperature transition. Values of $m$ deviate from the expected mean field value, 1/2, suggesting the importance of phase fluctuations. Resulting values of $T_c$, interpreted as the onset of phase correlations, decrease as field increases, showing an upward curvature. Values of $T_c(H)$ are obtained through a two-dimensional critical scaling analysis obeyed by many $M(T)$ curves. The resulting phase diagrams do not show upward curvature and lie below the $T_c(H)$ line. The value of $H_{c2}(0)$ estimated from the initial slope $dH_{c2}/dT$ is twice the value suggested by the phase diagram. Amplitude fluctuations above $T_c(H)$ are explained in terms of a Ginzburg–Landau approach extended to high reduced temperatures and magnetic fields by the introduction of a total-energy cutoff in the fluctuation spectrum.

(Some figures may appear in colour only in the online journal)

1. Introduction

The monolayer Bi$_{2+x}$Sr$_{2-x}$CuO$_{6+\delta}$ system, Bi2201, has $T_c \leq 10$ K depending on the doping [1, 2]. The system possesses a layered structure and Cu–O bonds similar to other cuprate superconductors with much higher values of $T_c$ and upper critical fields at zero temperature, $H_{c2}(0)$. A motivation of the present study lies in the expected similarity between the phase diagram of Bi2201 [3] and its parent high-$T_c$ superconductors, such as Bi2212 (which displays the pseudo-gap [4–6]), for which one can only study the linear part (near $T_c$) of the phase diagram with magnetic fields commonly available in laboratories. Another motivation, is the apparent lacking in the literature of fluctuation magnetization studies of Bi2201, which is in contrast to extensive magnetization studies performed in Bi2212 [4]. Here, we have performed magnetization measurements as a function of magnetic field and temperature in a high-quality single crystal [2] of Bi$_{2+x}$Sr$_{2-x}$CuO$_{6+\delta}$, with $x = 0.1$ and $T_c \approx 7$ K for $H \parallel c$-axis. Among the studies presented in the literature for Bi2201, it is worth citing the works of [7] and [8] which obtained magnetic phase diagrams through resistivity measurements, and the work of [9] which studied fluctuation conductivity. Results of these works for the optimal doping
sample with $x = 0.1$ and $T_c \approx 7$ K, produced quite different values for $H_{c2}(0)$, and for $\partial H_{c2}/\partial T$ as well. For instance, values of $H_{c2}(0) \approx 60$ kOe and $\partial H_{c2}/\partial T = -16$ kOe K$^{-1}$ were found in [7] for $H \parallel c$-axis, while values about three times larger were found in [8] for the same quantities on a similar sample with the same field orientation. Regarding the times larger were found in [8] for the same quantities on a high-field in the phase diagram, has also been observed in other system [10]. Moreover, the upward curvature of the critical temperature due to phase fluctuations, produced values of deviations from the linear behavior of magnetization with phase coherence [9] point to the need for further studies in this systems.

In this work, we address the above issue by performing precise magnetization measurements in a Bi2201 single crystal. The magnetic phase diagram of the Bi2201 studied sample is obtained through a critical scaling analysis of many isofield magnetization curves. The resulting mean field phase diagrams do not show any upward curvature, but an analysis of $M(T)$ curves near the transition, considering deviations from the linear behavior of magnetization with temperature due to phase fluctuations, produced values of an apparent temperature transition, $T_c(H)$, which represents the onset of phase correlations. Values of $T_c(H)$ lie above $T_c$, but below $T_c$, and show an upward curvature and a much higher value of the apparent $\partial H_{c2}/\partial T$. The resulting mean field phase diagram shows $\partial H_{c2}/\partial T = -12$ kOe K$^{-1}$, and $H_{c2}(0) \approx 30$ kOe, which reasonably agree with values obtained in [7] for a sample with similar $T_c$ and the same field orientation. The value of $H_{c2}(0) \approx 60$ kOe, estimated from the Werthamer–Helfand–Hohemberg (WHH) formula [11], $H_{c2}(0) = -0.69T_c(\partial H_{c2}/\partial T)$, is twice the value suggested by the phase diagram. We also observe that a 30 kOe field suppresses any signal of superconductivity above 3 K (the lowest temperature in this work), allowing us to obtain the temperature dependence of the normal state magnetization.

2. Experimental details

The studied single crystal of Bi2201 has a mass of 7 mg and approximate dimensions of $0.35 \times 0.3 \times 0.01$ cm$^3$. The single crystal shows a fully developed superconducting transition at $T_c \approx 7$ K with $\Delta T_c \approx 1.5$ K. The crystal was grown by a traveling-solvent-floating-zone method as described in [2]. The magnetization measurements were carried out by using a Cryogenics magnetometer system based on a superconducting quantum interference device, built with a 60 kOe superconducting magnet with a low-field feature. We only obtained measurements for the direction $H \parallel c$-axis. All data were obtained after a desired magnetic field was applied without overshooting and magnetization data were collected by heating the sample with fixed $\delta T \leq 0.1$ K increments from 3 K up to temperatures well above $T_c$ producing an isofield zero-field-cooling (ZFC) $M(T)$ curve. After that the sample was again cooled to 3 K, in the applied magnetic field, and another set of data was collected while heating the sample from 3 K to above $T_c$, producing an isofield field-cooling (FC) $M(T)$ curve. This procedure allows for determination of the reversible regime of each isofield $M$ versus $T$ curve. A few isothermic $M(T)$ curves were also obtained which exhibit a pronounced fish-tail (second magnetization peak). As mentioned, the suppression of superconductivity observed with a 30 kOe field allowed us to obtain the precise form of the normal state magnetization, $M_{back} = a + bT + c/T$. The background magnetization was then corrected for each $M(T)$ curve after selecting and fitting a wide region above $T_c$ to the form $a + bT + c/T$, where $a < 0$, $b$ is small, positive for low fields, decreases as field increases changing signal for higher fields, and $c > 0$.

3. Results and discussion

Figure 1 shows all the measured ZFC $M(T)$ curves obtained after the proper background correction. The arrows in figure 1 show the positions at which the ZFC and FC curves separate (producing a kind of hump) as $T$ is lowered below the irreversibility temperature, $T_{irr}$, which is only observed for fields $H \leq 1$ kOe. All curves obtained for fields above 1 kOe show only a reversible regime above 3 K. The inset of figure 1 shows selected $M(T)$ curves as measured, evidencing the behavior of the as-measured $M(T)$ curve for $H = 30$ kOe (for
which no superconductivity signal is detected) and for $H = 20$ kOe, both exhibiting the same normal state behavior which is well fitted to the form $a + bT + c/T$. This inset also shows the resulting magnetization curve for $H = 20$ kOe obtained after subtraction of the normal state magnetization extrapolated to the lower temperature region. For this particular curve ($H = 20$ kOe), the normal state region was obtained by fitting the data above $T = 10$ K, which produced the best fit. We mention that there are no visible changes in the resulting magnetization after using data above 12 K in the fitting. For all curves we used the same criterion, by selecting the normal state region that produced the best fit. It should be mentioned that a similar normal state background with a small linear temperature dependence term was previously observed in Bi2201 nanocrystalline phase [12]. As shown in the curves of figure 1, the resulting isofield $M(T)$ curves show a large reversible region displaying a linear region with $T_c$ near that the magnetization in the Abrikosov approximation, evidencing the linear extrapolation of these fittings to $M = 0$.

Figure 2(a) shows details of the reversible region of selected $M(T)$ curves, where in this figure both the ZFC and FC data are plotted. It is possible to see in figure 2(a) that the extrapolation of the linear region of each curve to $M = 0$, a method [17] commonly used to estimate the mean field temperature transition, $T_c(H)$, produces inconsistent values of $T_c(H)$, values which even increase with field. Deviations from the linear behavior of $M(T)$ curves near $T_c(H)$ are examined by studying the asymptotic behavior [18–20] of $\sqrt{-M} \propto (T - T_c)^m$, where $m$ is an exponent (the mean field value of which is 1/2 [21]) and $T_c(H)$ an apparent temperature transition. The approach is based on the fact that the magnetization in the Abrikosov approximation, near $T_c(H)$, is given by $M = -\frac{\hbar}{m} |\psi_0|^2$ where $\psi_0$ is the superconducting order parameter [21]. It follows that near $T_c(H)$, $\sqrt{-M} \propto (T_c(H) - T)^m$ where $m = 1/2$ for s-wave BCS superconductors [21]. The fitting is performed by selecting a reversible region of data below the inflection point of each $\sqrt{-M}$ versus $T$ curve. The inflection point occurs near a change in the concavity of the curve, above which amplitude fluctuations are dominant. Results of this analysis are shown in figure 2(b) for selected $M(T)$ curves. Resulting values of $T_c$ decrease as field increases, but, as will be seen, lie above $T_c(H)$. Resulting values of the exponent $m$ deviate considerably from the expected mean field value, 1/2, which suggests the existence of phase fluctuations [22] (as discussed below), which extends phase correlations above $T_c(H)$ up to $T_a(H)$. The resulting values of $T_a(H)$ will be plotted below in a phase diagram of the studied sample. It is worth mentioning that deviations from the mean field exponent value, 1/2, are expected for order parameters with d-wave symmetry (as is the case for cuprates), for which phase and amplitude fluctuations are predicted to have distinct contributions at the node and anti-node [22]. It is shown in [22] that in that case (the existence of a node in the order parameter) the effect of phase fluctuations is to reduce the density of states changing the shape of the gap near $T_c(H)$, and consequently the value of the exponent $m$. Within this scenario, $T_a(H)$ represents the onset of phase correlations which may occur above $T_c(H)$. It is important to mention that the possible existence of a pseudo-gap phase above $T_c$ in Bi2201 would make one expect values of $T_a(H)$ lying above $T_c$ [23], such as for instance observed in deoxygenated $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ and Bi2212 (see [18] and [19]), which is not the case here since the $T_a(H)$ line lies below $T_c$. This fact does not rule out the existence of a pseudo-gap above $T_c$ in the studied system, but with no phase correlations. It should mentioned that, due to the similarities between Bi2201 and Bi2212, the superconducting fluctuations observed here, could be explained in terms of uncorrelated pairs existing above $T_c$ due to charge inhomogeneity, as observed in Bi2212 within the pseudo-gap region [5, 6]. The inset of figure 2(a) shows a selected $M(H)$ curve obtained at 3 K, where it is possible to
identify a Meissner region up to 3 Oe, and the field $H_{\text{c}0}$ above which a pronounced second magnetization peak sets in. The Meissner region allows us to obtain $M$ in Gauss units.

Figure 3 shows the analysis of the large temperature region above $T_c(H)$ which corresponds to amplitude fluctuations without phase correlations. The magnetization in this region is extended to the case of high reduced temperatures, $\varepsilon \equiv \ln(T/T_c)$, and high reduced-magnetic-fields $h \equiv H/H_{\text{c}2}^*T_c$ (here $-H_{\text{c}2}^*$ is the $H_{\text{c}2}(T)$ slope at $T_c$), by introducing a cutoff in the fluctuation spectrum to eliminate the contribution of the high-energy fluctuation modes [24]. According to this model, the fluctuation magnetic moment of single-layer two-dimensional superconductors is given by [25]

$$M = -\frac{k_B T V}{\phi_0 s} \left[ -\frac{c}{2h} \psi \left( \frac{h + c}{2h} \right) - \ln \Gamma \left( \frac{h + \varepsilon}{2h} \right) + \ln \Gamma \left( \frac{h + \varepsilon}{2h} \right) + \frac{c - \varepsilon}{2h} \right].$$

Here $\Gamma$ and $\psi$ are the gamma and digamma functions, $k_B$ the Boltzmann constant, $\phi_0$ the flux quantum, $V \approx 1$ mm$^3$ the sample volume, and $c \approx 0.5$ the total-energy cutoff constant [24]. This expression is applicable up to $\varepsilon = c$ (which corresponds to the reduced temperature at which fluctuation effects vanish), and up to $h \approx c/2 \approx 0.3$ [25]. In the low magnetic field limit ($h \ll \varepsilon$) equation (1) may be approximated by

$$M = -\frac{k_B T V}{\phi_0 s} \left( \frac{1}{\varepsilon} - \frac{1}{c} \right).$$

which is linear in $h$. In turn, in the absence of a cutoff (i.e. for $c \to \infty$) it reduces to the well known Schmidt-like expression for 2D materials, proportional to $e^{-1}$.

The lines in figure 3 are the best fit of equation (1) to the $M(T)$ data for fields up to 5 kOe, well within the applicability range of equation (1) (see below). All curves were obtained by using $T_c$ and $H_{\text{c}2}$ as free parameters. The fit is reasonably good in all the accessible temperature range above $T_c$, and leads to $T_c = 6.6$ K and $H_{\text{c}2}^* = 4.5$ kOe K$^{-1}$. In the inset is presented an example, corresponding to $T = 8$ K, of the field dependence of the fluctuation magnetic moment. The line corresponds to equation (1) evaluated with the same parameters as in the main figure. It is interesting that, after the initial (roughly linear) increase of $|M|$ and the subsequent rounding associated with finite field effects, it begins to decrease and ends up vanishing at $H \approx 30$ kOe. This field is consistent with the $H_{\text{c}2}$ value extrapolated to $T = 0$ K, as may be estimated from the above $T_c$ and $H_{\text{c}2}$ values, and from the analysis in the critical region (see below).

The existence of a line in the $H$–$T$ phase diagram at which fluctuation effects vanish was shown a few years ago in the magnetization of low-$T_c$ alloys [26]6. In agreement with the present results, such a line was found to tend to $H \approx H_{\text{c}2}(0)$ (i.e. $h \approx 1$) at low temperatures and to $T \approx 1.7T_c$ (i.e. $\varepsilon \approx 0.5$) at low-field amplitudes [26]. It was suggested that the vanishing of fluctuation effects at these high $h$ or $\varepsilon$ values is related to the shrinking of the superconducting coherence length to its minimum possible length, the Cooper pairs size $\xi_0$, and that should be accounted for by the introduction of a total-energy cutoff in the fluctuations’ spectrum [26]. In the presence of large $h$ values such an introduction presents some difficulties [26, 28] and, in fact, equation (1) is not applicable above $h \approx 0.3$. However, very simple arguments [26] indicate that fluctuation effects should vanish at $h \approx 1.1$, in good agreement with the present observations.

An inspection of figure 2(a) shows in better detail the existence of a crossing point of $M(T)$ curves occurring at $T^* \approx 5.9$ K. Crossing points appearing in plots of many isofield $M(T)$ curves have been observed in other high-$T_c$ systems [13] and interpreted in terms of vortex fluctuations treated in a Ginzburg–Landau, GL, theory under a lowest-Landau-level, LLL, approximation which takes into account fluctuations correlations [29, 16, 30–32]. As discussed in [16], vortex fluctuations are expected for low magnetic fields within the London region, while, for high fields amplitude fluctuations dominate, explaining why the curves with 20 and 25 kOe do not cross the same point (see figure 1). The LLL critical theories under the GL formalism predict universal expressions for magnetization and other thermodynamic quantities, depending on the dimensionality, $D$, of the system. While the universal expressions allow one to fit experimental data and obtain values of intrinsic parameters, the associated scaling laws allow us to obtain values of the

6 It is worth noting that recent measurements of the fluctuation conductivity in the presence of large applied magnetic fields in $\text{La}_{2−x}\text{Sr}_x\text{CuO}_4$ and $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$, with different doping levels fully confirm the existence of a line in the $H$–$T$ phase diagram at which fluctuation effects vanish, in good qualitative agreement with the one in [26]. See [27].
of $M(T)$ curves, the wide range of magnetic fields values (1–25 kOe), and the separation of the curves as shown in figure 1, the collapse shown in figure 4(a) is impressive. We mention that we also tried to apply the 3D version of the scaling law for magnetization to the curves of figure 1, which failed. The values of $T_c(H)$ obtained from the 2D scaling are used to plot a phase diagram of the system, which is shown in figure 4(b), producing $dH_{c2}/dT = -12$ kOe K$^{-1}$, and a value of $H_{c2}(0) \approx 30$ kOe. It is important to point out that the values of $T_s(H)$ could only be obtained through the 2D scaling procedure. We also plot in the phase diagram, values of $T_s(H)$ and values of $T_{irr}$. It is important to note that the phase diagrams do not show any upward curvature. On the other hand, an upward curvature is present in the plot of $H$ versus $T_s(H)$, which also would show a d$H_{c2}/dT$ higher than $-12$ kOe K$^{-1}$. As discussed above, $T_s(H)$ represents the onset of phase correlations, which has a value larger than the corresponding $T_c(H)$ but smaller than $T_c$. It is also important to mention that the value of $H_{c2}(0)$ estimated from the WHH formula is about twice the value suggested by the phase diagram. This fact may suggest that the WHH formula overestimate the values of $H_{c2}(0)$ for high-$T_c$ systems. The resulting phase diagram shown in figure 4(a), produced quite different values than those found in [8] for a sample with $T_c \approx 7$ K, where $dH_{c2}/dT$ values varies from $\approx -27$ to $-50$ kOe K$^{-1}$ and $H_{c2}(0)$ from $\approx 150$ to 240 kOe (depending on the criterion used to determine the onset of superconductivity). Also, the authors of [8] found that a suppression of superconductivity for a sample with $T_c \approx 7$ K is reached with a 250 kOe field, while here no superconductivity was observed for a 30 kOe field. Our finding roughly agrees with the suppression of superconductivity observed with a 50 kOe field found in [7] for a Bi2201 sample with $T_c \approx 7$ K. Similarly large values of $H_{c2}(T)$ (as the values found in [8]), were found in [9] from an analysis of magnetoconductivity fluctuations above $T_c$ in Bi2201. It is worth mentioning that the agreement between our work and that of [9] lies in the two-dimensional character of the fluctuations above $T_c$.

We also fit a selected region of data in figure 4(a) (see the dashed line) by the corresponding expression for magnetization developed for two-dimensional systems in [16]. We used equations (7) and (10) appearing in [16], where equation (10) is written as $(T_c - T^*)/T^* = b(dH_{c2}/dT)/(\rho s(U_0)^2)$ where $\rho$ and $s$ are the original GL parameters, $\phi_0$ is the quantum flux, $s$ is the interlayer distance, $U_0 \approx 0.8$, and $T^* = 5.9$ K is the crossing point temperature. The fitting is conducted to data selected in a region of scaled temperatures limited by $T_{irr}$ and $T_{irr}^+$ as indicated by the arrows in figure 4(a), producing the following values for the fitting parameters: $s = 11.9$ Å, $T_c = 6.2$ K, and $dH_{c2}/dT = -9800$ Oe K$^{-1}$. The value of the interlayer distance $s$ is in good agreement with the listed value [2], $s \approx 12$ Å, and the value of $dH_{c2}/dT$ is in good agreement with the experimental value obtained here; however, the value of $T_c$ is lower than the experimental value. The values of $T_{irr}$ and $T_{irr}^+$, which represent the temperature region where the 2D-LLL equations well fitted the data, are obtained for each original $M(T)$ curve.

![Figure 4.](image)

(a) Reversible isofield $M$ versus $T$ curves for $H \geq 1$ kOe are plotted following the 2D-LLL scaling law for magnetization. The dashed line represents a fitting of the selected region to the equation (7) of [16]. (b) The resulting phase diagram, where the lines (dashed and dotted) are only a guide to the eyes.

mean field temperature transition of the scaled curves [13]. Following the general scaling law for magnetization curves, plots of $M/(TH)^{(D-1)/D}$ versus $(T - T_c(H))/(TH)^{(D-1)/D}$ where $D$ is the dimensionality and $T_c(H)$ an adjusted parameter, should collapse into a universal curve. The existence of only one crossing point in figure 1 (better shown in figure 2), suggests the absence of dimensional crossover induced by magnetic field, as observed in deoxygenated YBa$_2$Cu$_3$O$_{7-\delta}$ [32, 33]. The results of figure 3 above, as well as the similarity between $M(T)$ curves of Bi2201 and Bi2212 [15] is evidence of the 2D character of the studied system.

We finally show in figure 4(a) the results of the 2D-LLL scaling applied to the reversible region of the curves of figure 1 with $H \geq 1$ kOe. Figure 4(a) is obtained by only adjusting a value of $T_c(H)$ for each curve in a way that all curves collapse together. Considering the large number of $M(T)$ curves, the wide range of magnetic fields values (1–25 kOe), and the separation of the curves as shown in figure 1, the collapse shown in figure 4(a) is impressive. We mention that we also tried to apply the 3D version of the scaling law for magnetization to the curves of figure 1, which failed. The values of $T_c(H)$ obtained from the 2D scaling are used to plot a phase diagram of the system, which is shown in figure 4(b), producing $dH_{c2}/dT = -12$ kOe K$^{-1}$, and a value of $H_{c2}(0) \approx 30$ kOe. It is important to point out that the values of $T_s(H)$ could only be obtained through the 2D scaling procedure. We also plot in the phase diagram, values of $T_s(H)$ and values of $T_{irr}$. It is important to note that the phase diagrams do not show any upward curvature. On the other hand, an upward curvature is present in the plot of $H$ versus $T_s(H)$, which also would show a d$H_{c2}/dT$ higher than $-12$ kOe K$^{-1}$. As discussed above, $T_s(H)$ represents the onset of phase correlations, which has a value larger than the corresponding $T_c(H)$ but smaller than $T_c$. It is also important to mention that the value of $H_{c2}(0)$ estimated from the WHH formula is about twice the value suggested by the phase diagram. This fact may suggest that the WHH formula overestimate the values of $H_{c2}(0)$ for high-$T_c$ systems. The resulting phase diagram shown in figure 4(a), produced quite different values than those found in [8] for a sample with $T_c \approx 7$ K, where $dH_{c2}/dT$ values varies from $\approx -27$ to $-50$ kOe K$^{-1}$ and $H_{c2}(0)$ from $\approx 150$ to 240 kOe (depending on the criterion used to determine the onset of superconductivity). Also, the authors of [8] found that a suppression of superconductivity for a sample with $T_c \approx 7$ K is reached with a 250 kOe field, while here no superconductivity was observed for a 30 kOe field. Our finding roughly agrees with the suppression of superconductivity observed with a 50 kOe field found in [7] for a Bi2201 sample with $T_c \approx 7$ K. Similarly large values of $H_{c2}(T)$ (as the values found in [8]), were found in [9] from an analysis of magnetoconductivity fluctuations above $T_c$ in Bi2201. It is worth mentioning that the agreement between our work and that of [9] lies in the two-dimensional character of the fluctuations above $T_c$.

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and are plotted in figure 4(b). We mention that the temperature region where the rounding effect is observed in each curve virtually matches with the corresponding region delimited by the $T_{a \text{LLL}}$ and $T_{\text{LLL}}$ lines in figure 4(b), emphasizing the role of LLL-critical fluctuations on the pronounced rounding effect of the $M(T)$ curves.

4. Conclusions

In conclusion, we obtained the magnetic phase diagram of a Bi2212 sample with $T_c \approx 7$ K for the $H \parallel c$-axis, showing a $k H_{c2}/dT = -12$ kOe K$^{-1}$, a $H_{c2}(0) \approx 30$ kOe, and no upward curvature. The value of $H_{c2}(0) \approx 60$ kOe, estimated from the WHH formula, is twice the value suggested by the phase diagram. Due to the pronounced rounding effect on $M(T)$ curves, values of $T_a(H)$ could only be obtained through a 2D-LLL-critical scaling analysis of many isofield magnetization curves. An analysis considering deviations from the linear behavior of $M(T)$ with temperature, interpreted as due to phase fluctuations of the order parameter, produced values of an apparent temperature transition $T_a(H)$ in each curve, values of which is larger than the corresponding $T_c$. The temperature $T_a(H)$ is interpreted as the onset of phase correlations, and a plot of $H$ versus $T_a(H)$ shows an upward curvature. The large amplitude fluctuations observed above $T_a(H)$ on $M(T)$ curves are explained in terms of a Gaussian-GL approach by the introduction of a total-energy cutoff in the fluctuation spectrum. We also observed that a 30 kOe field suppresses any signal of superconductivity above 3 K allowing us to obtain the precise form of the normal state magnetization used to correct all $M(T)$ curves.

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