

## Temperature dependence of the second magnetization peak in a deoxygenated $\text{YBa}_2\text{Cu}_3\text{O}_{6.65}$ single crystal

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We report on magnetic measurements carried out in a  $\text{YBa}_2\text{Cu}_3\text{O}_{6.65}$  deoxygenated single crystal with superconducting transition temperature  $T_c = 62.5$  K. The so-called fishtail or second magnetization peak ( $H_p$ ) has been observed in magnetization curves from 60 K down to 1.8 K. In the region  $6 \text{ K} < T < 60 \text{ K}$ ,  $H_p$  increases with a positive curvature as the temperature is lowered. For  $T < 6$  K,  $H_p$  increases linearly with temperature and an extrapolation to  $T=0$  gives  $H_p(0) = 2.9$  T. This value occurs just above a decoupling field  $H_d$ , above which there is a change in the temperature dependence of the irreversibility line, a departure from the relation  $(1 - T/T_c)^m$ . Magnetic relaxation measurements for fields in the region of the second magnetization peak indicate a change in the mechanism originating the second peak below 5 K. At higher temperatures the peak position changes with time. For temperatures lower than 5 K there is no differences in relaxation below and above  $H_p$ , and the position of the peak does not change with time. [S0163-1829(99)14525-9]

The fishtail or second magnetization peak is a feature appearing in isothermal magnetization curves for intermediate fields which resembles a fishtail shape and has been observed in conventional<sup>1,2</sup> and high-temperature superconductors (HTS's).<sup>3</sup> Application of the Bean model to a magnetization curve exhibiting a fishtail produces a peak in the critical current density which is of major application importance. The understanding of the mechanism leading to the formation of the second magnetization peak is not yet clear and has been the subject of numerous investigations.<sup>3-7</sup> On the other hand, the literature lacks in flux dynamics studies of the temperature dependence of this feature in a wider range of the  $(H, T)$  phase diagram. Such a study will certainly contribute to a better understanding of the vortex dynamics in HTS's and is the subject of this work.

Among copper-based HTS's, deoxygenated  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  crystals are of particular interest, providing a continuous variation of most superconducting properties by varying the oxygen deficiency in the system.<sup>9,10</sup> Particularly, the reduction of the critical temperature  $T_c$  and upper critical field  $H_{c2}(0)$ , as compared with the fully oxygenated compound, allows a study of their rich magnetic phase diagram  $(H, T)$  down to low temperatures without requiring very large magnetic fields.<sup>11</sup> In this work we have studied the temperature, field, and time dependence of the second magnetization peak in the phase diagram of a deoxygenated  $\text{YBaCuO}$  crystal with  $T_c = 62.5$  K. The final purpose of the work is to extend the study presented here to lower  $T_c$  crystals. Our measurements allow the observation of the second magnetization peak  $H_p$ , in curves of magnetization against field  $M$  vs  $H$  from 60 K down to 1.8 K. At higher and intermediate temperatures  $H_p$  increases with a positive curvature

as  $T$  is lowered, as previously observed in the literature for  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  and other HTS's.<sup>5-8</sup> A change in curvature in the temperature dependence of  $H_p$  occurs at low temperatures, with  $H_p$  increasing linearly with temperature as  $T$  is lowered. Extrapolation of the linear behavior to  $T=0$  gives a field which falls just above a region of fields where a change of behavior in the irreversibility line (IL) is observed. Such change in the IL has been associated with a magnetic-field-induced decoupling of the vortex state. Magnetic relaxation measurements for fields in the region of the second magnetization peak show differences in relaxation behavior above and below  $H_p$ . These differences, which have been associated with the origin of the second magnetization peak at higher temperatures,<sup>6</sup> disappear below 5 K, evidencing that a different mechanism is responsible for the second magnetization peak at low temperatures.

The studied single crystal of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ , with  $x = 0.35$  and  $T_c = 62.5$  K, was grown by the self-flux procedure. Details of the sample preparation are described in Ref. 9. The sample dimensions are approximately  $1 \text{ mm} \times 1 \text{ mm} \times 0.2 \text{ mm}$ , with the  $c$  axis perpendicular to the larger planes. The sample exhibits a sharp transition width  $\Delta T_c \leq 1.0$  K. The experiment was conducted with the magnetic field  $H$  applied parallel to the  $c$  axis of the sample. Isofield  $M$  vs  $T$  curves were obtained with a 5-T superconducting quantum interference device (SQUID) magnetometer (Quantum Designs MPMS). Isothermal  $M$  vs  $H$  curves were obtained with the SQUID magnetometer at higher temperatures and at lower temperatures with an extraction magnetometer (Quantum Design PPMS), allowing magnetic fields up to 9 T. Magnetic relaxation measurements were made with the extraction magnetometer. All measurements were obtained by

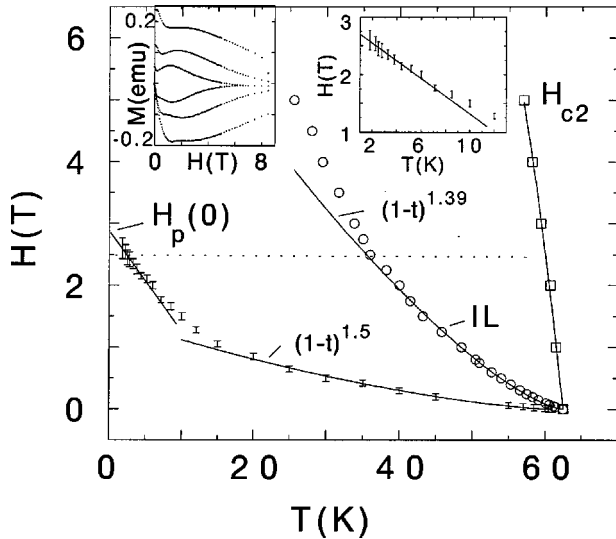


FIG. 1. Magnetic phase diagram of the studied sample, showing the second magnetization peak  $H_p(T)$ , irreversibility line  $IL(H_{irr}, T_{irr})$ , and upper critical field  $H_{c2}(T)$ . Solid lines on  $H_p(T)$  and  $IL$  correspond to fits performed in the data, where  $t = T/T_c$ . The solid line on  $H_{c2}(T)$  is only a guide to the eye. The dotted line at 2.5 T represents the expected decoupling line separating 3D (below) from quasi-2D (above) vortex states. The left inset shows isofield  $M$  vs  $H$  curves at 1.8, 5.2, and 12 K. The right inset shows an enlarged plot of  $H_p(T)$  at low temperatures.

cooling the sample from above  $T_c$  to the desired temperature in a zero magnetic field. Magnetic measurements performed for  $H||c$  axis allow the determination of the upper critical field  $H_{c2}(T)$ , irreversibility line  $T_{irr}(H)$ , and the second magnetization peak  $H_p(T)$ . The irreversible point ( $H_{irr}, T_{irr}$ ) is determined from isofield  $M$  vs  $T$  curves as the point where magnetization lost linearity,<sup>12</sup> which coincides with the onset of large deviations in the dipole fit of the SQUID signal.<sup>13</sup>

All high-field  $M$  vs  $T$  curves present a distinct rounding around  $M \approx 0$  that makes difficult the determination of  $T_{c2}(H)$ . This rounding has been associated with lowest Landau level (LLL) fluctuations.<sup>14</sup> Values of  $T_{c2}(H)$  were obtained by performing two-dimensional (2D) LLL fluctuation scaling on the high-field data, where  $M$  scales with  $(TH)^{1/2}$  and  $T$  is replaced by  $[T - T_{c2}(H)]/(TH)^{1/2}$ . The optimum temperature regions for the scaling are 59–64 K for 1 T, 55–71 K for 2 T, and 50–80 K for 3, 4, and 5 T data. The above temperature intervals give an estimate of the 2D vortex liquid phase region. The values of  $T_{c2}(H)$  obtained from the LLL scaling produced  $dH_{c2}(T)/dT = -0.90$  T/K for  $H||c$  axis. The Werthamer-Helf-Hohenberg formula at  $T = 0$  gives  $H_{c2}(0) = 0.693T_c |dH_{c2}(T)/dT|$ . In the anisotropic Ginzburg-Landau limit for  $H||c$ ,<sup>15</sup>  $H_{c2} = \Phi_0/2\pi\xi_{ab}^2$ , where  $\xi_{ab}$  is the coherence length in the  $ab$  plane, so we estimate  $H_{c2}(0) \approx 39$  T and  $\xi_{ab}(0) \approx 29$  Å for our studied sample. It is interesting to compare these values with the estimated values for fully oxygenated  $YBa_2Cu_3O_{7-x}$ , with  $T_c = 93$  K,  $H_{c2}(0) \approx 120$  T and  $\xi_{ab}(0) \approx 16$  Å.<sup>16</sup>

Values of  $H_{c2}(T)$ ,  $IL(H_{irr}, T_{irr})$ , and  $H_p(T)$  are plotted as a function of temperature in Fig. 1. The left inset of this figure shows isothermal  $M$  vs  $H$  curves at selected temperatures. The values of  $H_p(T)$  for each data curve are extracted

from the maximum diamagnetic value of the magnetization second peak, measured in the field increasing branch. This maximum is obtained by fitting a polynomial to the peak region and the error bars are estimated by eye. This procedure is carried out over estimating the actual error, which is the largest at 1.8 K ( $\pm 0.17$  T) where the peak is less pronounced. The low-field region of the IL follows the relation  $H_{irr} \approx (1 - T_{irr}/T_c)^n$  with  $n = 1.39$ . The power law region of  $H_{irr}$  is obtained by plotting  $\ln[(1 - T_{irr}/T_c)]$  against  $\ln[H]$ , which is valid for fields in the region  $0 < H < 2.0$  T. Above  $H_d = 2.5$  T the IL deviates from the power law fit. A similar behavior has been found in deoxygenated  $YBaCuO$  single crystals with  $n = 1.5$ ,<sup>11</sup> deoxygenated  $Y123$  thin films,<sup>17</sup> and various other anisotropic HTS's.<sup>18</sup> The departure from the experimentally observed power law has been associated with a theoretically predicted field-induced decoupling of the vortex state in to quasi 2D vortex pancakes.<sup>19</sup>

The main fact in the magnetic phase diagram of Fig. 1 is the behavior of the  $H_p(T)$  line showing a curvature change at  $T \approx 6$  K. It will be shown with relaxation measurements that the flux dynamics changes below this temperature. For  $T \geq 6$  K,  $H_p$  follows the common trend previously observed,<sup>5–8</sup> showing a positive curvature with decreasing temperature. For  $T \leq 6$  K,  $H_p$  increases linearly as the temperature is lowered. This low-temperature behavior is enlarged in the right inset of Fig. 1. Assuming that the second magnetization peak exists below 1.8 K we perform an extrapolation of the linear behavior to  $T = 0$  to obtain  $H_p(0) = 2.9 \pm 0.07$  T. For temperatures above 20 K,  $H_p(T)$  follows the relation  $(1 - T/T_c)^m$  with  $m = 1.5$ . In the temperature region  $6 \text{ K} < T < 20 \text{ K}$  the curvature of  $H_p(T)$  is positive but does not follow the power law. It is important to point out that the breakdown of the power law behavior below 20 K as well as the linear behavior of  $H_p(T)$  below 6 K were not previously reported in the literature.

Another important point to be noted in Fig. 1 is that the linear portion of  $H_p(T)$  crosses the decoupling field,  $H_d = 2.5$  T. This represents an expected decoupling line above which vortex lines behave as quasi-2D pancakes. In relation to  $H_p(T)$ , the second magnetization peak observed in the 3D system  $Ba_{1-x}K_xBiO_3$  (Ref. 7) follows a temperature behavior similar to that observed in  $YBa_2Cu_3O_{7-x}$ , suggesting that the latter is also 3D. Thus, observation of the second magnetization peak just above the expected decoupling line opens a question about the interpretation made for the deviation from the power law observed in the IL. Measurements of the second magnetization peak at low temperatures for other anisotropic samples could clarify this point.

Figure 2(a) shows relaxed magnetization curves, measured 600 s after the field is stabilized. The data are shown at selected temperatures, for fields above and below  $H_p$ . It is worth mentioning that the data at 1.8, 2.5, 3.3, 5.2, 8.5, and 12 K were measured twice and the reproducibility is within the equipment sensitivity. Large relaxation effects are observed down to the lowest temperature  $T = 1.8$  K. Measurements at 1.8 and 12 K confirmed that relaxation effects are the same on the field increasing and decreasing branches. The curves in Fig. 2(a) clearly show differences in relaxation for fields above and below  $H_p$  at  $T > 8$  K, which indicates that there are two distinct mechanisms controlling flux creep. Most important, there are no apparent differences in relax-

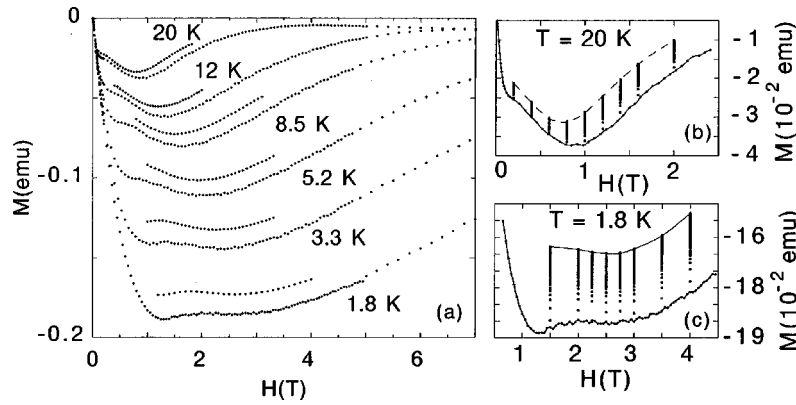


FIG. 2. (a) Isofield  $M$  vs  $H$  curves and the respective relaxed curves obtained in the region of the second magnetization peak at several temperatures. The relaxed curves are measured 600 s after  $H$  is stabilized. (b), (c) magnetic relaxation measured during 4500 s for fields in the region of  $H_p$ , plotted with the  $M$  vs  $H$  curves for  $T = 20$  and 1.8 K.

ation for fields above and below  $H_p$  for temperatures below 5 K (1.8, 2.5, and 3.3 K). At 5.2 K the differences are very small, while for the curves at 8.5, 12, and 20 K relaxation is larger above  $H_p$ . As observed in Ref. 6, plastic pinning controls creep for fields above  $H_p$  while collective pinning is the dominant mechanism for fields below  $H_p$ . The absence of differences in relaxation in the  $M$  vs  $H$  curves below 5 K indicates that only one mechanism, possibly plastic pinning, is controlling creep on the second magnetization peak. This fact suggests that the pinning mechanism changes below the  $H_p(T)$  line as the temperature is lowered for a fixed value of field.

We have verified this point by conducting magnetization measurements as a function of time during 4500 s in the temperature region 1.8–20 K, with applied fields of 1, 1.5, 2, and 3 T. The results plotted as  $\ln(M)$  vs  $\ln(t)$  show a linear behavior. The magnetization rate  $S = d[\ln(M)]/d[\ln(t)]$  is obtained for each curve, and plotted in Fig. 3 as a function of temperature for  $T < 8$  K. The inset shows the same plot extended to 20 K. An increase of the rate for 1 and 1.5 T data is observed at lower temperatures, which confirms the change in the mechanism of creep mentioned above, occurring below 8 K for 1 T and below 5 K for 1.5 T. By tracing a vertical line at  $H = 1$  T in Fig. 2(a) crossing all  $M$  vs  $H$

curves it is possible to see that the relaxation regime changes twice as temperature drops below 20 K. At 20 K, a 1 T field is above  $H_p$  and as claimed in Ref. 6 this corresponds to a region of plastic pinning. At 12 K, 1 T is below  $H_p$ , corresponding to a region of collective pinning.<sup>6</sup> At 3.3 K relaxation at 1 T increases when compared to the 8.5 K curve and also there is no differences in relaxation below and above  $H_p$ . Thus the increase in the relaxation rate at approximately 8 K evidences a new change in the pinning mechanism. The anomaly observed in the magnetization rate  $S$  is shifted to lower temperatures at 1.5 T and is not observed for the 2 and 3 T. In fact, for  $H = 2$  T the  $H_p(T)$  line is crossed in the linear region and  $H = 3$  T lies above  $H_p(0)$ , a region where plastic creep is expected.<sup>6</sup> The latter fact is in accordance with the absence of differences in relaxation below and above  $H_p$  for temperatures below 5 K in the  $M$  vs  $H$  curves, and suggests that plastic creep is dominant for  $T \lesssim 5$  K below the  $H_p(T)$  line.

An important difference between the low- and high-temperature behavior in  $M$  vs  $H$  relaxed curves refers to the position of the second magnetization peak. At  $T \geq 8.5$  K, the second peak position in the relaxed curves is clearly shifted to a lower field value as compared to the unrelaxed curves. At 1.8, 2.5, 3.3, and 5.2 K the position of  $H_p(T)$  is un-

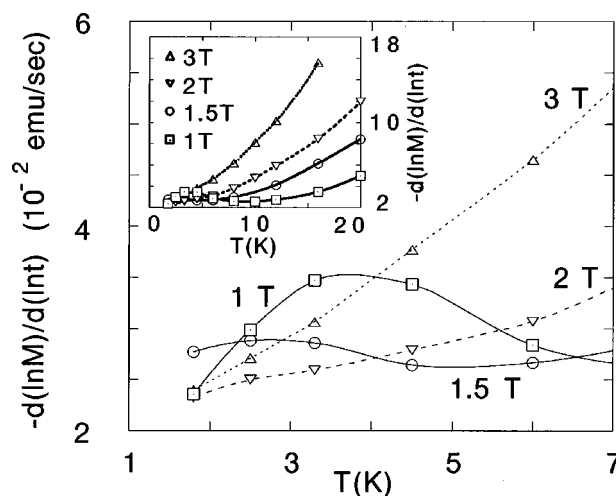


FIG. 3. Magnetization rate  $d[\ln(M)]/d[\ln(t)]$ , plotted in the low-temperature region for fields  $H = 1, 1.5, 2,$  and  $3$  T. The inset shows the same data extended to higher temperatures.

changed in the relaxed curves. These differences can be better visualized in Figs. 2(b) and 2(c), showing relaxed magnetization measurements for 4500 s at 20 and 1.8 K. The differences in  $H_p$  due to relaxation in Fig. 2(a) are  $\approx 0.12$  T. This value is much higher than the error in  $H_p$  for  $T > 3.8$  K. The values of  $H_p$  are the same at 1.8 K [Fig. 2(c)] and differ by 0.21 T at 20 K [Fig. 2(b)]. The time dependence of the peak position at 20 K indicates that the second magnetization peak at higher temperatures is determined by dynamic effects, and not by the critical current density  $J_c(H)$ . This effect was first observed in Ref. 6, where the authors assumed a plastic creep model occurring for fields  $H > H_p$ , and obtain a time dependence for the second magnetization peak,  $H_p \approx 1/\ln^2(t/t_0)$ , which explains the shift of  $H_p$  to lower fields with time. Based on this model, they also obtain a temperature dependence of the type  $H_p \approx [1 - (T/T_c)^4]^{1.4}$  not followed by our data. The error due to the time dependence of  $H_p(T)$  is  $\leq 0.05$  T for temperatures above 20 K, estimated from a combination of delay measuring time and increasing magnetization rate at higher temperatures. With this error added to the values of  $H_p$  the power law expression for  $H_p(T)$  still holds, with the exponent  $m$  varying from 1.3 to 1.5.

It should be pointed out that based on the observation that  $H_p(T)$  is time dependent, to obtain a precise temperature dependence of  $H_p(T)$  loses its meaning. The time window probing the magnetization measurement depends on temperature; therefore  $H_p(T)$  determined at different temperatures comes from values of magnetization measured at different times. Below 5 K the peak position is not time dependent, implying that the second magnetization peak is related to the critical current density  $J_c(H)$ . Thus, this result

presents evidence that there is a change in the origin of the second magnetization peak at lower temperatures. The region in temperatures where this change occurs corresponds to the linear part of  $H_p(T)$ , which is not time dependent. It may also be noted that the rate of relaxation in Fig. 3 has approximately the same value for all fields as the temperature approaches 1.8 K. This behavior suggests that below 1.8 K the system may enter a regime of quantum creep.<sup>20</sup>

In conclusion, the magnetic phase diagram of a deoxygenated  $\text{YBa}_2\text{Cu}_3\text{O}_{6.65}$  single crystal with  $T_c = 62.5$  K was determined from magnetic measurements. Isothermal magnetic curves showed the existence of the second magnetization peak from 60 K down to 1.8 K. An extrapolation gives a value for  $H_p(T=0)$  just above the expected decoupling field. Relaxed  $M$  vs  $H$  curves measured for fields in the region of the second peak show that at higher temperatures the magnitude of the relaxation is higher above  $H_p$ , whereas the relaxation at lower temperatures is the same for fields above and below  $H_p$ . The position of  $H_p(T)$  is found to be time dependent at higher temperatures, but time independent below 5.2 K. This time independence of  $H_p(T)$  at low temperatures and the differences in relaxation above and below  $H_p$  at higher temperatures indicate a change in the origin of the second magnetization peak at low temperatures. The relaxation data also give evidence for another change in the mechanism of pinning in the region below the  $H_p(T)$  line, possibly from collective to plastic, occurring below 8 K.

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