Magnetic measurements of the upper and lower critical fields of oxygen-deficient $YBa_2Cu_3O_{7-\delta}$ single crystals

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We present dc-magnetization measurements in fields up to 5 T on oxygen-deficient ($T_c \simeq 60$ K) $YBa_2Cu_3O_{7-\delta}$ single crystals. The upper-critical-field slopes are -2.0 and -8.7 T/K and the lower critical fields at 5 K are 83 and 25 Oe for fields perpendicular and parallel to the Cu-O planes, respectively. The upper-critical-field slope anisotropy is not substantially different from the anisotropy found for the 90-K crystals. The H_{c1} values are, however, much smaller for the 60-K crystals. We explain these results through a model in which the chains do not contribute significantly to the coupling between the Cu-O planes but rather serve as a charge reservoir for the superconductivity that occurs in the planes.

INTRODUCTION

One of the most interesting properties of the $YBa_2Cu_3O_{7-\delta}$ superconductor is its strong dependence of transition temperature on oxygen concentration.[1] The outstanding feature of the T_c-versus-δ curve is the plateau occurring for $T_c \sim 60$ K and $\delta \simeq 0.5$.[2] The origin of the plateau is as yet unknown but electron- and x-ray-diffraction evidence suggests the possibility of a distinct phase occurring for $\delta \simeq 0.5$ with a structure where every second Cu-O chain is missing. This type of oxygen ordering has also been predicted by theory.[3] Therefore, a thorough study of the superconducting properties of this oxygen-deficient phase and comparison to the oxygen-rich compound could help in an understanding of the relative importance of the Cu-O chains and the Cu-O planes. However, due to the difficulty in producing oxygen-deficient crystals with sharp transitions comparable to their 90-K counterparts, little has been measured in the way of basic properties such as the upper and lower critical fields.

In order to better characterize this phase we have carried out magnetic measurements of H_{c1} and H_{c2} as well as the irreversibility temperature on high-quality $YBa_2Cu_3O_{7-\delta}$ single crystals. With our low-pressure oxygen-annealing procedure, we are able to produce crystals with any T_c, ranging from 0 to 93 K, with low-field magnetic-transition widths often less than 1 K.[4] These homogeneous crystals allow unique determinations of the upper and lower critical fields which would be difficult to ascertain in lower-quality crystals. For the upper-critical-field measurements on the 60-K T_c crystals, we observe that the magnetization is reversible and linear with temperature within a reasonable range below T_c (~ 20 K for $\mathbf{H} \| \mathbf{c} = 5$ T) similar to the behavior of the 90-K crystals.[5] A linear extrapolation of the reversible region to the normal-state base line allows a unique determination of the mean-field transition temperature as a function of field. The lower critical field was determined from magnetization-versus-field data where the first deviation from linear diamagnetism was used to define H_{c1}. This same procedure was previously used for the 90-K material.[6,7] For the 60-K crystals, we find that the upper-critical-field slopes, and consequently the effective-mass anisotropy, are close to those obtained for the 90-K crystals. However, we see significantly smaller H_{c1} values for the oxygen-deficient crystals. Therefore, the penetration depths are considerably greater for the 60-K material, implying diminished equilibrium supercurrents. Both the diminished supercurrents and unchanged effective-mass anisotropy can be explained through a model in which the chains act mostly as a charge reservoir for superconducting processes in the Cu-O planes.

EXPERIMENTAL METHODS

Single crystals were prepared using a "self-flux" procedure similar to those described by Kaiser *et al.*[8] and Schneemeyer *et al.*[9] Before deoxygenation these crystals exhibited sharp low-field magnetic transitions less than 0.3 K wide (10 and 90% values of the full diamagnetic signal) with $T_c \geq 92$ K. The oxygen-deficient samples were prepared by equilibrating in a controlled O_2-N_2 atmosphere at 520 °C followed by a quench to liquid nitrogen. Complete details of the deoxygenation procedure as well as methods for the determination of oxygen stoichiometry are reported elsewhere.[4] The data presented in this paper apply to one crystal which had a mass of 4.05 mg and dimensions of $1.1 \times 1.1 \times 0.6$ mm^3 with the short dimensions along the c axis. The sample was twinned with an estimated oxygen stoichiometry of $\delta = 0.35$. A second, much smaller, crystal gave similar

results to the large crystal, but the data are not reported here because of the significantly lower signal-to-noise ratio.

dc magnetization was measured with a commerical superconducting quantum interference device (SQUID) magnetometer in fields up to 5 T. In addition, low-field magnetization measurements for initial sample characterization were performed in a noncommercial low-field SQUID magnetometer.

RESULTS

Figure 1 shows a zero-field-cooled magnetization (shielding) curve for the oxygen-deficient single crystal. In this measurement the sample was cooled in zero field to well below T_c, a field of 1 Oe was applied perpendicular to the Cu-O planes, and the dc magnetization was measured on warming. The transition width is very narrow, about 1 K wide, and T_c, determined by the onset of diamagnetism, is 62.2 K. The data are plotted in units of $4\pi(1-n)M/H$ where the demagnetization factor, n, is estimated from the sample dimensions to be 0.56. The complete flux exclusion indicates that the sample is a bulk superconductor. The small deviation of the diamagnetic signal $4\pi(1-n)M/H$ from -1, for $T<T_c$, is within the uncertainty in the demagnetization factor.

Figures 2 and 3 show shielding data for field parallel and perpendicular to the Cu-O planes, respectively. A small normal-state background, following a Curie law with $C=7.4\times10^{-4}$ K and 6.7×10^{-4} K for H parallel and perpendicular to the Cu-O planes, respectively, has been subtracted from the data. In later experiments, where this crystal was deoxygenated to produce a T_c of 20 K, the normal-state background was described by the same Curie constant. This suggests that the background is due to impurities rather than the oxygen environment of the Cu ions. The most probable source of impurities was some excess flux partially covering the surface of the sample as seen through an optical microscope. Plotted along with the shielding data for **H**||**c** are three magnetization curves taken as the sample was cooled in fields of 1, 3, and 5 T. These results show that the magnetization is reversible in the temperature range shown. As was

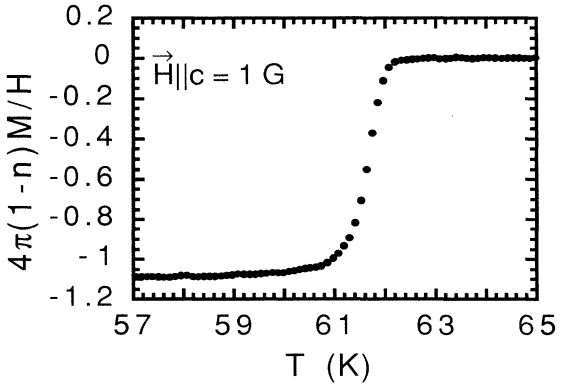

FIG. 1. Shielding measurement in 1 G with field perpendicular to the Cu-O planes.

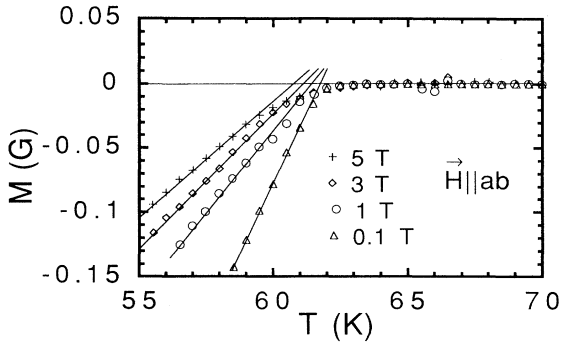

FIG. 2. Shielding measurements in fields of 0.1, 1, 3, and 5 T with field parallel to the Cu-O planes.

seen in the 90-K phase,[5] there is a linear temperature dependence of the magnetization, as expected from three-dimensional Ginzburg-Landau (3D-GL) theory, accompanied by a rounded region very close to T_c. Here as before we attribute this rounding to either diamagnetic fluctuations or sample inhomogeneities. The transition temperature as a function of field is derived from the intersection of the linear fit with the normal-state base line, as illustrated. We note that the variation of the slopes $\partial M/\partial T$ with field in Figs. 2 and 3 is not predicted through 3D-GL theory.[10] However, an analysis of this problem[11] suggests that the H_{c2} curves derived from our procedure are not strongly affected by this behavior.

Figure 4 shows $H_{c2}(T)$ data derived from the $T_c(H)$ values in Figs. 2 and 3. As we observed for the 90-K phase there is a strong suppression of T_c with low fields which is best seen by the fact that $T_c(H=1\text{ G})$ is 62.2 K while $T_c(H=0.1\text{ T})\sim61.5$ K for either field direction. For higher fields the $H_{c2}(T)$ curves steepen considerably. A linear fit to the high-field behavior yields critical-field slopes of -8.7 and -2.0 T/K for fields parallel and perpendicular to the Cu-O planes, respectively. These slopes are not significantly different from those found for the 90-K material.[5] The orbital critical fields extrapolated to $T=0$ K using the Werthamer-Helfand-Hohenberg[12] (WHH) formula

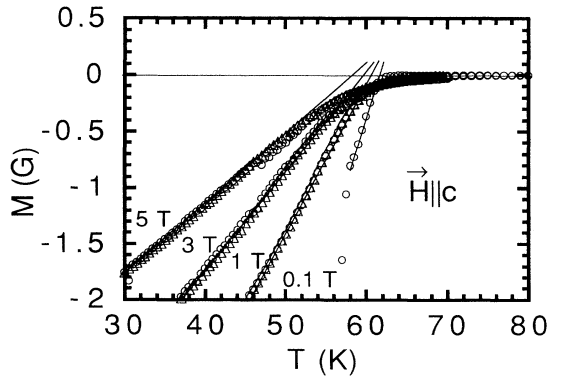

FIG. 3. Shielding measurements (circles) in fields of 0.1, 1, 3, and 5 T, and Meissner measurements (triangles) in fields of 1, 3, and 5 T for field orientated perpendicular to the Cu-O planes.

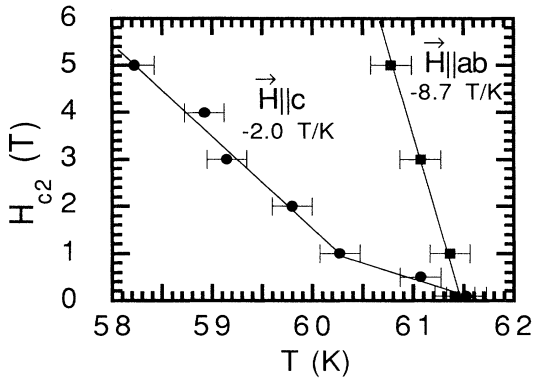

FIG. 4. Temperature dependence of the upper critical field for both field orientations. The slopes of the linear extrapolations are indicated.

$$H_{c2}(0)=0.7\left[\frac{\partial H_{c2}}{\partial T}\right]\Bigg|_{T_c} T_c$$

are $H_{c2}^{c}(0)=87$ T and $H_{c2}^{ab}(0)=380$ T. According to the relations $H_{c2}^{c}=\phi_0/2\pi\xi_{ab}^2$ and $\xi_c/\xi_{ab}=H_{c2}^{c}/H_{c2}^{ab}$, this corresponds to coherence lengths of $\xi_{ab}=20$ Å and $\xi_c=4.5$ Å.

As a check on the intrinsic nature of the above H_{c2} values, high-field measurements were made on a second single crystal, with $T_c=58.1$ K, grown by a different sample grower and deoxygenated in a different manner. The sample was a small plate weighing 100 μg and of dimensions $0.8\times0.5\times0.05$ mm^3. The H_{c2} curves up to 1 T agree with the above results. However, because the crystal was relatively small, the H_{c2} values for higher fields could not be extracted due to noise.

To completely characterize the 62.2-K T_c sample and extract the Ginzburg-Landau parameter κ, H_{c1} was measured for both field orientations and at various temperatures. Figure 5 shows magnetization-versus-field data for three temperatures with **H**||c. Also shown is a straight-line fit to the low-field diamagnetic regime. It is clear that the deviation of the magnetization from linearity is more gradual for the low-temperature data which we attribute to increased pinning. To better facilitate the determination of the entry field we graphed the magnetic induction as a function of field, as shown in Fig. 6. Plotted in this manner, the transition from the Meissner state ($B=0$) to the mixed state ($B>0$) is more easily seen. The entry field (H_{c1}) was determined as the field at which B deviated obviously from zero. We were careful to choose an entry field that represented the general trend of the magnetic-induction data departing from zero rather than assigning H_{c1} to a small deviation in B due to noise. An enlarged section of the B-versus-H data, for $T=40$ K and **H**||c, is shown in Fig. 7, which illustrates our method for defining H_{c1}. The line at $B=0$ represents a linear fit to the low-field diamagnetic region while the two lines on either side represent the scatter width of the data. H_{c1} is defined as the field at which B deviates beyond the scatter. This approach is analogous to the method we used previously for the determination of H_{c1} for the 90-K crystals.[6,7]

We did not find it advantageous to fit the data for $B>0$ to either $B\propto(H-H_{c1})^2/(H_s-H_{c1})$ or $B\propto(H-H_{c1})^3/(H_s-H_{c1})^2$ (where H_s is the field at which the flux fronts meet in the center of the sample[13]) as did Martinez *et al.*[14] for M-versus-H data for the $Y_2Ba_4Cu_8O_{16}$ compound. To illustrate why such a fit is not appropriate for our data, one must look at an extensive range of magnetization-versus-field data as is shown in Figure 8 for $T=5$ K and **H**||c. Since the minimum in the magnetization corresponds approximately to $H_s=5000$ G while $H_{c1}\sim32$ G (uncorrected for demagnetization factor), the fitting region for B, where $H_{c1}<H<H_s$, is extremely large compared to H_{c1}. The extrapolated H_{c1} [defined as the intersection of the $B=0$ line and the $B\propto(H-H_{c1})^2/(H_s-H_{c1})$ or $B\propto(H-H_{c1})^3/(H_s-H_{c1})^2$ fitted curve] is extremely sensitive to the number of data points fitted, even changing sign as the fitting range is varied. This sensitivity is probably due to the fact that the linear flux density profile assumed in Bean's model is too simple. A more thorough treatment of the critical state in a superconducting disk shows this to be the case.[15]

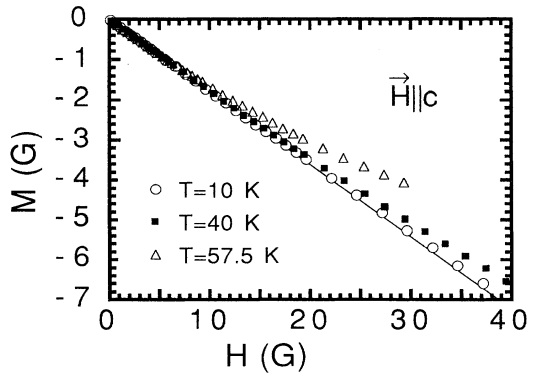

FIG. 5. Magnetization as a function of field for three temperatures with field perpendicular to the Cu-O planes.

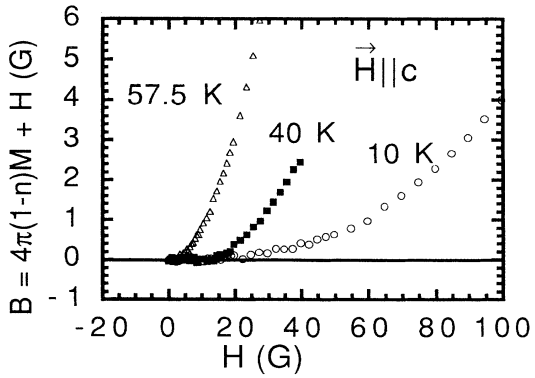

FIG. 6. Magnetic flux density as a function of field for the same field orientation and temperatures as in Fig. 5.

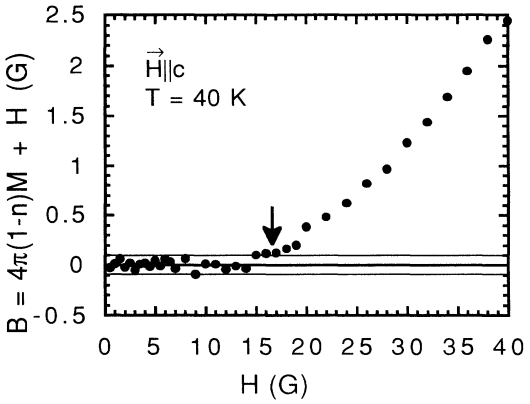

FIG. 7. Magnetic flux density at 40 K as a function of field along c. The arrow denotes H_{c1} uncorrected for demagnetization factor.

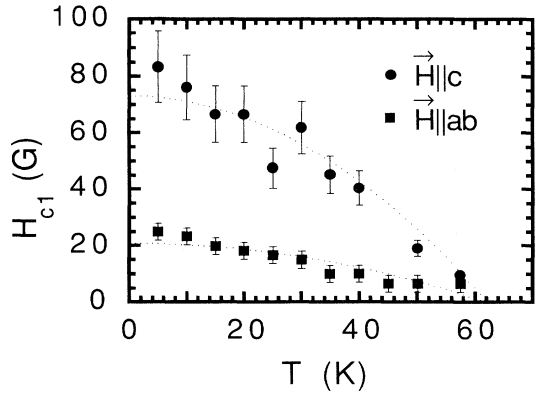

FIG. 9. Temperature dependence of the lower critical field for both field orientations.

Using our procedure, H_{c1} values (corrected for demagnetization factors) are plotted versus temperature in Fig. 9 for both field orientations. The dotted lines are fits to the expression $H_{c1}(T)=H_{c1}(0)[1-(T/T_c)^2]$. The data seem to roughly follow this equation although, within the scatter, an upturn in H_{c1}, as seen by some[6,16] for the 90-K material at low temperatures, cannot be ruled out. The values of H_{c1} at 5 K are 25 and 83 Oe for fields parallel and perpendicular to the Cu-O planes, respectively. Because of the relatively low aspect ratio for this sample (ratio of width to thickness ~ 2) these H_{c1} values, especially for **H**||**c**, were not subject to the severe demagnetization factor corrections normally encountered for the more common platelike single crystals. These low-temperature values of H_{c1} are much lower than those reported for the 90-K material.[6,16–18] Evaluating the relations[19]

$$H_{c1}^{ab}=\frac{\phi_0}{4\pi\lambda_{ba}^{ab}\lambda_c^{ab}}\ln\kappa^{ab} \quad \text{and} \quad H_{c1}^{c}=\frac{\phi_0}{4\pi(\lambda_{ba}^{c})^2}\ln\kappa^{c}$$

yields large penetration depths of $\lambda_{ba}^{c}=3180$ Å for fields along c with penetration into ba and $\lambda_{ba}^{ab}=13\,600$ Å for fields along ab with penetration into ba.

To investigate nonequilibrium effects for the 60-K superconductor, the irreversibility temperature was measured as a function of field. The departure of the field-cooled (Meissner) from the zero-field-cooled (shielding) signal is often defined as the irreversibility temperature ($T_{\rm irr}$) which is generally a function of field.[20] Figure 10 shows Meissner and shielding data for a field of 0.1 T applied along c. Irreversibility with decreasing temperature is illustrated by the separation of the zero-field-cooled and field-cooled signals (thus, the linear extrapolation shown in Fig. 3, for $H=0.1$ T, is justified only in the limited temperature range where the behavior is reversible). Figure 11 shows the **H**||**c** irreversibility field plotted as a function of temperature as derived from curves similar to Fig. 10. Also shown are the two lines which define the $H_{c2}(T)$ slopes for each field orientation. The **H**||**c** depression of the irreversibility temperature with increasing field is quite drastic compared to the corresponding depression of T_c. The dotted line represents the fitted function $(6.1\times10^{-4}\,\mathrm{G}^{-2/3})H^{2/3}=(1-T/T_c)$, which has the form of a "quasi–de Almeida–Thouless line."[21] This dependence is surprisingly close to the fitted function obtained by Yeshurun and Malozemoff[22] of $(4.6\times10^{-4}\,\mathrm{G}^{-2/3})H^{2/3}=(1-T/T_c)$ on a 90-K single crystal also

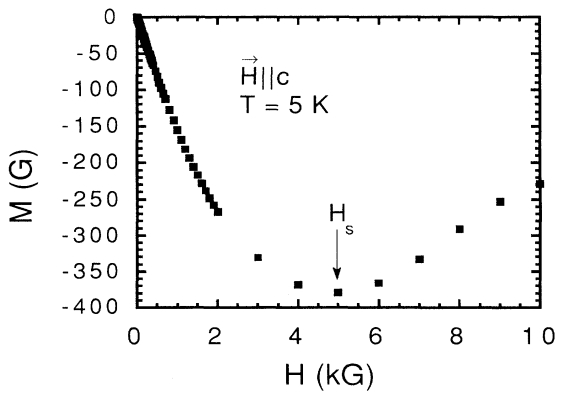

FIG. 8. Magnetization at 5 K as a function of field along the c axis.

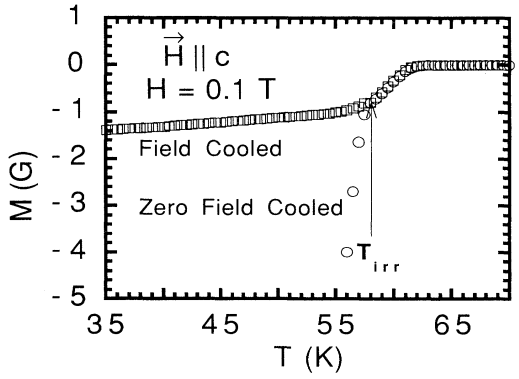

FIG. 10. Meissner (field-cooled) and shielding (zero-field-cooled) curves in a field of 0.1 T along the c axis.

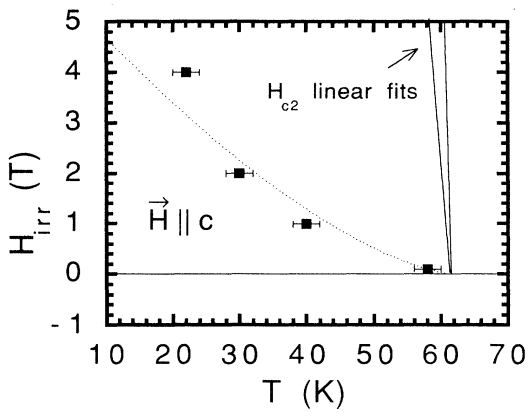

FIG. 11. Temperature dependence of the irreversibility field for field along the c axis.

measured in a SQUID with field oriented along c. It is also interesting that the T_{irr} data shown here closely resemble data measured with rf techniques on an oxygen-reduced crystal with $T_c \sim 60$ K.[23]

DISCUSSION

Table I lists the oxygen-deficient crystal H_{c2} and H_{c1} results along with H_{c2} (Ref. 5) and H_{c1} (Ref. 7) for a fully oxygenated crystal. The H_{c1} values, for $\mathbf{H}\|ab$, for the 92-K untwinned and twinned samples represent a range which contains many of the other measured H_{c1} values in the literature.[6,17,18,24] Because of the uncertainty in H_{c1} values, and questions relating to the severe demagnetizing corrections which often must be made for the c direction, we do not quote H_{c1}, $\mathbf{H}\|\mathbf{c}$ results for the 90-K crystals. Also shown are derived values ξ, the GL parameter κ, the anisotropic mass ratio, and the thermodynamic critical field H_c. κ was derived from the relation $H_{c1}/H_{c2}=\ln(\kappa_3)/2\kappa_1\kappa_3$, where κ_1 and κ_3 are the first and third GL parameters[25] and were taken to be equal in this analysis. H_c was extracted from the expressions $H_{c2}=2^{1/2}\kappa_1 H_c$ and $H_{c1}=H_c \ln\kappa_3/2^{1/2}\kappa_3$ again assuming κ_1 and κ_3 to be equal.[10] The anisotropic mass ratio can be calculated from either $H_{c2}^{ab}/H_{c2}^{c}=(m_c/m_{ab})^{1/2}$ or $\kappa^{ab}/\kappa^{c}=(m_c/m_{ab})^{1/2}$,[26] with m_c/m_{ab} values of 19.0 and 18.5, respectively. Since the former equation involves only the upper-critical-field anisotropies while the latter involves both upper and lower critical fields, the close agreement in calculated anisotropy ratios lends confidence to the analysis.

TABLE I. Comparison of various superconducting parameters, derived from the upper- and lower-critical-field measurements, between the oxygen-deficient and fully oxygenated crystals.

	62-K T_c crystal field directions		92-K T_c crystal field directions	
	$\mathbf{H}\|ab$	$\mathbf{H}\|\mathbf{c}$	$\mathbf{H}\|ab$	$\mathbf{H}\|\mathbf{c}$
$H_{c2}(T=0)$ (T)	380	87	674	122
$\xi(T=0)$ (Å)	20	4.5	16	3
dH_{c2}/dT (T/K)	−8.7	−2.0	−10.5	−1.9
H_{c1}(5 K) (G)	25	83	70 (untwinned) 210 (twinned)	
κ	700	160	540 (untwinned) 300 (twinned)	
m_c/m_{ab}	19		30	
H_c (T)	0.38		0.85 (untwinned) 1.55 (twinned)	

At first glance one might expect the effective-mass anisotropy for $YBa_2Cu_3O_{7-\delta}$ to increase with increasing oxygen deficiency. This would be expected if the coupling between plane layers depended on the oxygen occupancy in the intervening chain layers. That the mass anisotropy does not change, but is actually slightly smaller for the 60-K material, indicates that the chain oxygens apparently play no essential role in bridging the neighboring Cu-O planes. An alternative coupling mechanism involves the Cu(1)—O(4)—Cu(2) bonds between the chain and plane layers. It has been suggested that the position of the O(4) oxygen may be important in determining the superconducting properties in $YBa_2Cu_3O_{7-\delta}$.[27,28] The Cu(1)—O(4) [Cu(2)—O(4)] bond length changes from 1.86 Å (2.30 Å) for the fully oxygenated ($\delta=0$) material to 1.84 Å (2.35 Å) for the oxygen-deficient ($\delta=0.35$) material.[27] However, since the small change in mass anisotropy indicates that the coupling between the planes remains essentially constant, we conclude that neither the chain oxygens nor the Cu(1)—O(4) or Cu(2)—O(4) bond lengths appear to significantly affect the coupling of the order parameter along the c direction. This suggests that a more accurate description for the coupling along c might be that of a direct overlap of the superconducting wave functions from adjacent planes, or coupling through the Cu(1)—O(4)—Cu(2) bond system where the bond lengths do not play a role.

The viewpoint that the chains play little or no role in the c-axis coupling is consistent with the assumption that the chains act mainly as a charge reservoir for the superconductivity which occurs in the planes. That the superconducting processes occur mainly in the Cu-O planes is supported by studies involving substitutions on the Y and Ba sites in $YBa_2Cu_3O_{7-\delta}$ which suggest an interdependence of T_c on the number of holes in the planes.[29] In fact, cation doping experiments on the yttrium site of the 1:2:3 system have also shown that, while maintaining constant oxygen stoichiometry, changes in T_c could be directly influenced through the change in hole concentration exclusively in the Cu-O planes.[30] That the chains play a small role in superconducting processes is also supported through measurements on untwinned single crystals which show little or no H_{c2} (Ref. 31) or H_{c1} (Ref. 7) ab plane anisotropy between fields aligned perpendicular and parallel to the b-axis chains. This is consistent with infrared data which shows evidence of a superconducting energy gap for the Cu-O planes but no such gap for the chains.[32] Also, normal-state resistivity measurements show superconducting fluctuations which can be modeled as residing in the plane but not in the chains.[33] Therefore, there is strong evidence supporting our interpretation that the Cu-O chains play a minor role (other than as

a reservoir of charge) in determining the superconducting properties of $YBa_2Cu_3O_{7-\delta}$.

One of the most striking results in Table I is the relatively small lower critical fields for the oxygen-deficient crystal. This indicates a weakening of the equilibrium screening currents in the fully diamagnetic state which leads to larger penetration depths for the 60-K material. This may be a consequence of the decreased charge-carrier density, n_s, due to less hole doping from fewer oxygen atoms in the chains. Reduced carrier density leads to a longer penetration depth through the relation $\lambda^2 = m^* c^2/4\pi e^{*2} n_s^*$ from either London or GL theory.[10] This is consistent with normal-state resistivity measurements[34] which indicate a decrease of carrier concentration with increased oxygen deficiency. The reduced H_{c1} values for the oxygen-deficient crystal also lead to lower derived values of H_c. From BCS theory,[10] the expression $H_c^2(0)/8\pi = -\frac{1}{2}\Delta^2(0)N(0)$, where $\Delta \propto kT_c$, relates the condensation energy to the superconducting energy gap, Δ, and the density of states at the Fermi level, $N(0)$, so that $H_c^2 \propto T_c^2 N(0)$. The scaling in H_c^2 between the oxygen-deficient 62-K and the fully oxygenated 92-K superconductor is ~ 0.20 (~ 0.06) for the untwinned (twinned) crystal while the scaling in $T_c^2 \sim 0.45$. This indicates that the substantial drop in H_c as oxygen is depleted may be partially accounted for by a reduction in $N(0)$. This view is supported by photoemission experiments which find such a decrease in the Fermi-level density of states with increased oxygen deficiency.[35] Therefore a reduction in both $N(0)$ and n_s may account for the measured reductions in H_c and H_{c1}. This is consistent with the assumption that the chain oxygens provide a reservoir of charge carriers for the Cu-O planes.

In conclusion, we have measured H_{c1}, H_{c2}, and T_{irr} on an oxygen-deficient $YBa_2Cu_3O_{7-\delta}$ single crystal. The H_{c2}-versus-temperature slopes near T_c as well as effective-mass anisotropies do not differ significantly from those measured for fully oxygenated crystals. This implies that the coupling between Cu-O planes near T_c is not affected by the removal of oxygen in the chains. The H_{c1} values are much smaller for the oxygen-deficient crystal which we attribute to decreased superconducting screening currents stemming from fewer charge carriers. We explain our results through a picture in which the chains act mainly as a charge reservoir for the Cu-O planes.

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