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MAGNETIC MEASUREMENTS OF THE LOWER CRITICAL FIELDS AND
IRREVERSIBLE PROPERTIES OF OXYGEN DEFICIENT $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ SINGLE CRYSTALS*

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Magnetic Measurements of the Lower Critical Fields and Irreversible
Properties of Oxygen Deficient $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ Single Crystals*

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Abstract

We present results for the lower critical field, irreversibility field and hysteresis on an oxygen deficient single crystal of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ annealed to a T_C of 28 and 85 K. We compare the results for the off-plateau ($T_C = 28$ and $T_C = 85$ K) stoichiometries to our earlier results on the ~ 60 K T_C plateau crystals and the fully oxygenated ~ 92 K T_C crystals. We find a systematic decrease in the lower critical field with decreasing T_C , and a decrease in the remanent magnetization by a factor of 10, at $T = 5$ K, for the $T_C = 28$ K phase compared to the $T_C = 85$ K phase. Our irreversibility field versus reduced temperature data indicate much stronger curvature and hence a larger reversible region for the reduced oxygen stoichiometries. For crystals with $T_C = 62.2, 56, 50$, and 28 K, a crossover occurs in the slope of $\log(H_{irr})$ vs $\log(1-T/T_C)$. The field at which this crossover occurs is lower for the more oxygen deficient phases and may be

linked to their lower H_{C1} values and corresponding longer penetration depths.

Introduction

With the increasing availability of single crystals of $YBa_2Cu_3O_{7-\delta}$, many experiments are now possible which probe the intrinsic properties of this compound. Also, improved methods for producing homogeneous oxygen deficient crystals make further experiments feasible which can monitor these properties as a function of oxygen content. Most of the studies on oxygen deficient crystals have been done on samples with T_C 's near the 60 K plateau [1-6]. There still remains a shortage of experiments on oxygen deficient crystals with other oxygen contents. In this work, we present measurements of the lower critical field as well as the irreversibility line and magnetic hysteresis for an oxygen deficient single crystal annealed to T_C 's of 85 K and 28 K, on either side of the 60 K plateau. By obtaining these measurements on the same single crystal annealed to the different T_C 's, differences in the measured properties not attributable to variations in the oxygen stoichiometry were avoided. Combining these results with our previous data for the 60 K and 92 K crystals, we obtain a clearer picture of the effects of oxygen stoichiometry on the fundamental superconducting properties. Namely, there is a systematic decrease in the lower critical fields with decreasing oxygen content supporting interpretations that the chain oxygen concentration determines the charge carrier density in the planes. Also, there is a substantial reduction of the remanent magnetization as well as increased curvature in the irreversibility lines with reduced oxygen stoichiometry.

In addition to this increased curvature, we find that the dependence of $\log(H_{irr})$ on $\log(1-T/T_C)$ changes slope at a field which is strongly

dependent on the oxygen stoichiometry. This behavior has been seen before [7-10] and it has been proposed [11] that the crossover field represents a demarcation between weak and strong correlations in the flux line lattice. For $\text{Sm}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ [8], it was found that this crossover occurs at a field where the flux line spacing was $\sim \lambda/4$. By deriving λ from our H_{c1} results we find that, for our $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ crystals, this crossover field corresponds to a flux line spacing $\sim \lambda/8$. Our results qualitatively support the proposal that the crossover in slope of the $\log(H_{irr})$ vs. $\log(1-T/T_c)$ is due to vortex-vortex interactions [11].

Experimental Methods

Single crystals were prepared using a "self-flux" procedure similar to those described by Kaiser et al. [12] and Schneemeyer et al. [13]. The "as-grown" crystals were subsequently annealed in flowing oxygen for two weeks which produced homogeneous crystals with T_c 's ranging from 91-93 K. The samples ranged in size from 0.5-2 mm in width and length and 0.04-0.4 mm in thickness. In all instances, the samples were heavily twinned with the c crystallographic axis along the short dimension.

The crystals were deoxygenated in a tube furnace in a static partial pressure of oxygen [14, 15]. This procedure differs from previously reported deoxygenation methods in flowing oxygen/nitrogen atmospheres [1, 16]. The preparation consisted of a 2 hour vacuum anneal (< 20 mTorr) in a quartz tube at 650°C , to remove any surface impurities, followed by a 7 day anneal at 530°C in a 0.5 - 200 Torr oxygen environment, depending on the desired stoichiometry. The samples were then quenched to room temperature by pouring water over the evacuated tube containing the samples. Evacuation of the tube before quenching helped prevent the

absorption of oxygen as the samples cooled. With this procedure, crystals with any desired oxygen stoichiometry, for any δ between 0 and 1, could be produced with T_C 's ranging from 0 K to 93 K and low-field transition widths < 2 K.

The crystal used for this study was annealed in an oxygen partial pressure of 200 and 1.5 Torr which produced T_C 's of 85.0 and 28.0 K, respectively. The crystal had a mass of 3.09 mg and dimensions of $1.1 \times 1.1 \times 0.4$ mm³. By using a crystal with a relatively thick dimension along c , the problems associated with large demagnetization factor corrections for $H_{C1||c}$ were alleviated.

DC magnetization was measured with a commercial SQUID magnetometer in fields up to 5 T. In addition, low field magnetization measurements for initial sample characterization were performed in a non-commercial low-field SQUID magnetometer, described elsewhere [17].

Results

The upper and lower part of Figure 1 shows shielding data in a field of 1 Gauss applied perpendicular to the Cu-O planes of the single crystal after annealing to T_C 's of 85.0 and 28.0 K respectively. For both T_C 's, the transition widths are very narrow, about 0.8 K wide, and the susceptibilities in units of $4\pi(1-n)M/H$ are close to -1, indicating bulk superconductivity. The demagnetization factor is estimated to be 0.61 from the sample dimensions.

The lower critical field values were extracted using the same methods as outlined before [1]. Figure 2 shows $H_{C1}(T)$ data corrected for demagnetization factors for the 85.0 K T_C stoichiometry. The dotted lines are fits to the expression $H_{C1}(T) = H_{C1}(0)(1-(T/T_C)^2)$ for the data at

temperatures 35 K and greater. It is evident that there is an upturn in the H_{C1} data for both field orientations for temperatures less than 35 K. This upturn has been seen elsewhere for the 92 K material and its origin and interpretation are not clear [18, 19]. Assuming that the H_{C1} values at 5 K represent the saturated H_{C1} values at 0 K, the $H_{C1}(0)$ values are 95 and 380 Oe for fields parallel and perpendicular to the Cu-O planes, respectively. These values of H_{C1} are less than those reported for the 92 K single crystals [2, 18-24].

Figure 3 shows $H_{C1}(T)$ data, corrected for demagnetization factors, for the 28.0 K stoichiometry. The dotted lines are again fits to the expression $H_{C1}(T) = H_{C1}(0)(1-(T/T_C)^2)$. Assuming, as before, that the 5 K H_{C1} values represent the zero temperature saturation values, one gets $H_{C1}(0)$ values of 8 Oe and 33 Oe for fields parallel and perpendicular to the Cu-O planes, respectively. These values of H_{C1} are substantially smaller than for any of the higher T_C phases measured.

To investigate nonequilibrium effects, the irreversibility field (H_{irr}) was measured as a function of temperature. This was accomplished by finding the temperature of the separation of the field-cooled signal and the zero-field-cooled signal, as described previously [1]. Figure 4 shows the dependence of H_{irr} on the reduced temperature for field applied perpendicular to the Cu-O planes for the 85 K and 28 K T_C phases. The dotted lines are fits to the general relation $A(H)^m = (1-T/T_C)$, yielding values of $A = 9.2 \times 10^{-5} \text{ G}^{-0.77}$, $m = 0.77$ and $A = 8.5 \times 10^{-2} \text{ G}^{-0.21}$, $m = 0.21$, for the 85 K and 28 K T_C data, respectively. The weaker power dependence for the proportionality $H^m \sim (1-T/T_C)$, of $m=0.21$ for the 28 K T_C data as opposed to $m = 0.77$ for the 85 K data, is manifested graphically as a much stronger curvature in the irreversibility line for $T_C = 28$ K.

There is therefore an appreciable increase in the normalized width of the reversible region with reduced oxygen stoichiometry. These results are qualitatively consistent with irreversibility line data measured by the peak in the out of phase signal of the ac susceptibility for various oxygen deficient single crystals [25]. Our results are also consistent with measurements on grain aligned oxygen deficient samples [26]. Results from room temperature annealing experiments find a similar dependence in that the curvature in the irreversibility line decreases systematically as T_C is increased due to oxygen vacancy ordering in the chains [7]. Our current data are also consistent with our previous results [1] on the $T_C=62.2$ K crystal where we obtained a value of $m = 0.67$, intermediate to the m values for the $T_C=28$ K and $T_C=85$ K phases.

If the data from Figure 4 is plotted in a different manner, an interesting crossover feature in the irreversibility line can be seen. Figure 5 shows the irreversibility data from Figure 4 plotted along with data from a crystal with $T_C = 56$ K on log-log axes. There is a change in the $\log H$ vs $\log(1-T/T_C)$ slope corresponding to a crossover in the power dependence, from $m = 0.53$ to $m = 0.36$ and $m = 0.45$ to $m = 0.19$, for the 56 K and 28 K data, respectively. This crossover feature is not seen for the $T_C = 85$ K data where m is constant at 0.77. A crossover in the slope has also been seen in the irreversibility data for single crystals of $\text{Sm}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ [8] and $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ [9] as well as polycrystals of $\text{Pr}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ [10] and for an oxygen deficient $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ single crystal, room temperature annealed to T_C 's between 7 and 17 K [7]. The field at which this crossover occurs, H_{co} , is larger for the 56 K T_C , $H_{co} = 1.5$ Tesla, than for the 28 K T_C , $H_{co} = 0.6$ Tesla, and presumably occurs at an experimentally unattainable field for the 85 K T_C phase. Two other crystals (not plotted for clarity)

with T_C 's of 62.2 [1] and 50 K had crossover fields of 1.8 and 1.3 Tesla, respectively. Our data is consistent with suggestions [11] that the crossover field is related to H_{C1} such that superconductors with larger penetration depths (lower H_{C1} 's) will have correspondingly lower crossover-fields.

To further probe the irreversible nature of the two oxygen deficient stoichiometries, the remanent magnetization was measured at $T = 5$ K after cycling the field from zero to five Tesla and back. In the upper and lower portions of Figure 6, the hysteresis data is shown for the field parallel and perpendicular to the Cu-O planes, respectively. For the lower plot, data for the 85 K phase was only obtained up to a field of 3.5 Tesla, since at $H = 4$ Tesla, the crystal shattered after the strong magnetic force caused it to demount from the sample holder. Though the complete remanent data does not exist for H_{llc} and $T_C = 85$ K, comparison with the H_{llab} data implies a remanent magnetization, M_{rem} , on the order of $3000 \text{ erg}/(\text{Oe}\cdot\text{cm}^3)$ at $H=0$. Therefore, for both field directions, the remanent magnetization (at $H=0$) for the 28 K T_C phase is about a factor of 10 smaller than for the 85 K T_C phase. Specifically, the H_{llab} values are $M_{rem} = 850$ and $50 \text{ erg}/(\text{Oe}\cdot\text{cm}^3)$ and the H_{llc} values are $M_{rem} = 3000$ and $300 \text{ erg}/(\text{Oe}\cdot\text{cm}^3)$ for the $T_C = 85$ K and $T_C = 28$ K phases, respectively.

As expected, the hysteresis measurements for H_{llc} in Figure 6 confirm the irreversibility data of Figure 4. The hysteresis data were taken at $T = 5$ K representing the reduced temperatures of 0.18 for $T_C = 28.0$ K and 0.06 for the $T_C = 85.0$ K phase. For the $T_C = 28$ K phase, the reduced temperature, 0.18, crosses the irreversibility line somewhere between 3 and 4 Tesla which corresponds roughly to the field at which the hysteresis loop collapses to zero. For the 85.0 K T_C phase, the reduced

temperature, 0.06, is far below the irreversibility line for the highest field measured of 5 Tesla and, correspondingly, the hysteresis loop does not collapse in the fields attainable.

Discussion

In Table I, a comparison of the present $H_{c1}(0)$ and crossover field results are shown beside our results for the oxygen rich material [2] and $T_C \sim 60$ K oxygen deficient material [1]. For both field orientations, there is an obvious decrease in H_{c1} with decreasing T_C . For high κ superconductors, the lower critical field is related to the penetration depth through the relations [27],

$$H_{cl}^{ab} = \frac{\phi_0}{4\pi\lambda_{ba}^{ab}\lambda_c^{ab}}(\ln\kappa^{ab} + .497) \quad \text{and} \quad H_{cl}^c = \frac{\phi_0}{4\pi(\lambda_{ba}^c)^2}(\ln\kappa^c + .497) \quad (1)$$

where the superscripts denote the field direction and the subscripts denote the direction of the field penetration, and $\lambda_{ba}^c = \lambda_{ab}^c$ [28]. Therefore, the present results indicate a dramatic increase in the penetration depths with reduced oxygen stoichiometry. Noting that, from equation (1), the dependence of λ on κ is weak, since κ is evaluated as a natural log, we can estimate the penetration depths for the oxygen deficient crystals using the values of κ from the fully oxygenated crystals. In the above relations, if one substitutes the values of $\kappa^c = 52$ and $\kappa^{ab} = 370$, as derived from reversible magnetization measurements [2] on the fully oxygenated crystals, one obtains large values of $\lambda_{ab}^c(0) = 4700 \text{ \AA}$ and $\lambda_{ba}^{ab}(0) = 28000 \text{ \AA}$ for the 28 K T_C stoichiometry. These penetration depths are substantially larger than those which can be derived for the $T_C \sim 92$ K

crystals of $\lambda_{ab}^c(0) = 970 \text{ \AA}$ and $\lambda_{ba}^{ab}(0) = 6300 \text{ \AA}$ [2]. Also, since the penetration depth is linked to the charge carrier density, n_s^* , through the relation, $\lambda^2 = m^*c^2/4\pi e^*n_s^*$, the results are consistent with the assumption that the decreased chain oxygens are associated with a reduction of charge carriers in the planes.

We did not find a substantially larger H_{c1} anisotropy for the $T_c = 28.0$ than for the $T_c = 85 \text{ K}$ phase. However, torque measurements [29] suggest effective mass anisotropies (m_c/m_{ab}) as large as 1000 for oxygen deficient $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ grain aligned samples, with $T_c \sim 30 \text{ K}$. Since the effective mass anisotropy is related to the anisotropy in κ and λ by $(m_c/m_{ab})^{1/2} = \kappa^{ab}/\kappa^c = \lambda_{ba}^{ab}/\lambda_{ba}^c$ [27], the anisotropy in H_{c1} can be written as,

$$\frac{H_{c1}^c}{H_{c1}^{ab}} = \left[\frac{\kappa^{ab}}{\kappa^c} \right] \frac{(\ln \kappa^c + .497)}{(\ln \kappa^{ab} + .497)} = \left[\frac{m_c}{m_{ab}} \right]^{\frac{1}{2}} \frac{(\ln \kappa^c + .497)}{(\ln \kappa^{ab} + .497)} \quad (2).$$

For an effective mass ratio $m_c/m_{ab} = 1000$, the anisotropy in H_{c1} can be found by substituting into equation (2) the value $\kappa^{ab} = 370$, from above, and choosing κ^c to be 12, so that the ratio $\kappa^{ab}/\kappa^c = (m_c/m_{ab})^{1/2} = (1000)^{1/2}$. Using these values, the corresponding anisotropy, H_{c1}^c/H_{c1}^{ab} , is found to be 14. Our measured anisotropy for the 28 K T_c phase is 4.1, however, due to the large percentage uncertainty in the H_{c1}^{ab} value of $8 \pm 4 \text{ Oe}$, an H_{c1} anisotropy as large as 10 would be possible. Therefore, our anisotropy in H_{c1} is not necessarily significantly different than that derived from the torque measurements.

It is interesting to examine our irreversibility results using the model first proposed for $\text{Sm}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ [8, 11]. There it was suggested that the crossover in the irreversibility data should occur at a field where the vortex separation is small enough to allow a significant overlap of the

vortex currents where strong correlations between the vortices become important. Empirically, it was found that for $\text{Sm}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ this crossover occurred at a vortex separation $\sim \lambda/4$. Examining our results in the same light, we can compare the flux line spacings for the crossover fields, $a_0 = (\frac{2\phi_0}{\sqrt{3}H_{c0}})^{1/2}$, from Table I, to the penetration depths as derived from H_{c1} . Using the penetration depths, λ_{ab}^c , for the 62.2 K and 28 K crystals of 2800 and 4700 Å, as derived from equation (1), the flux line spacings for the crossover fields represent values of $\lambda/7.7$ and $\lambda/7.5$, respectively. For the 85 K and 92 K T_c phases, a flux line spacing of $a_0 \sim \lambda/8$ would correspond to crossover fields of 9 and 16 Tesla, respectively, outside the experimentally attainable range. This might explain the absence of reports of a crossover behavior for fully oxygenated crystals where, because of the relatively short penetration depths as compared to other high T_c compounds or oxygen deficient $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$, the crossover field is never attained in most measurements. Other methods than SQUID measurements for determining the irreversibility field, such as the ac susceptibility, would be necessary to determine if this crossover behavior exists for the more oxygenated crystals.

It has been proposed that the crossover behavior in the irreversibility line represents a transition in the vortex lattice from semi-rigid three dimensional (3D) vortex lines to 2D pancake vortices that can move independently within the Cu-O layers [30, 31, 32]. The 3D limit can be characterized as columns of 2D pancake vortices where the Josephson coupling between the pancakes in adjacent layers is strong. The 2D limit is characterized by pancake vortices interacting only with vortices within their own Cu-O layer. The 3D to 2D crossover should occur when the

vortex-vortex intralayer interaction energy becomes stronger than the interlayer Josephson coupling energy. Physically, the greater the overlap of the vortex currents the higher the tendency to decouple an individual pancake vortex from the Josephson coupled column of vortices which comprise a vortex line in the 3D limit. Our results are consistent with this analogy in that, for the more oxygen deficient crystals with the larger penetration depths and greater vortex current overlap, the crossover occurs at a lower field due to the stronger intralayer interactions between vortices.

In conclusion, we have measured the lower critical fields, the remanent magnetization, and the irreversibility line for two oxygen deficient stoichiometries, $T_C = 28.0$ K and $T_C = 85.0$ K, and compared our results to data for 60 K and 92 K $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ single crystals. We find reduced lower critical fields, increased curvature of the irreversibility line and a reduction in the remanent magnetization with decreased oxygen stoichiometry. We also find a crossover in the $\log(H_{irr})$ vs $\log(1-T/T_C)$ dependence of the irreversibility line which occurs at a field that is linked to the penetration depth, and this field decreases with decreasing oxygen stoichiometry. The decrease in H_{c1} with decreasing oxygen stoichiometry supports the assumption that the decrease in chain oxygen occupation is associated with a reduction of charge carriers in the planes. Our findings linking the penetration depth to the crossover in the irreversibility line are consistent with suggestions of a 3D to 2D transition in the vortex lattice.

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Figure Captions

- Figure 1: Diamagnetic shielding data for a field of 1 Gauss applied perpendicular to the Cu-O planes for the 85.0 K T_C (upper plot) and the 28.0 K T_C (lower plot) phases.
- Figure 2: Temperature dependence of the lower critical field for both field orientations for the 85.0 K T_C phase.
- Figure 3: Temperature dependence of the lower critical field for both field orientations for the 28.0 K T_C phase.
- Figure 4: Dependence of the irreversibility field on reduced temperature for the field oriented perpendicular to the Cu-O planes for the 85 K and 28 K T_C phase.
- Figure 5: Dependence of the irreversibility field on reduced temperature as graphed on a log-log plot for the field oriented perpendicular to the Cu-O planes for the 85 K and 28 K T_C phase and for a crystal with $T_C = 56$ K.
- Figure 6: Magnetization versus applied field, at $T = 5$ K, for the 85 K and 28 K T_C phases with the field parallel (upper plot) and perpendicular (lower plot) to the Cu-O planes.

Table Captions

Table 1: Comparison of the lower critical fields, crossover fields and corresponding vortex lattice spacings for the crossover fields for the various oxygen stoichiometries.

Fig 1

Fig 2

Fig 3

Fig 4

Fig 5

Fig 6

TABLE I

COMPARISON OF PARAMETERS

Parameter	28.0 K T_c Crystal		62.2 K T_c Crystal		85.0 K T_c Crystal		92 K T_c Crystal	
	Field Hllab	Directions Hllc	Field Hllab	Directions Hllc	Field Hllab	Directions Hllc	Field Hllab	Directions Hllc
$H_{c1}(T=0)$ (Gauss)	8	33	25	83	95	380	170	780
H_{c0} (Tesla)		0.6		1.8		> 5.0		> 5.0
a_0 (Å)		630		360		< 220		< 220

Table I

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END