ANISOTROPY AND DIMENSIONALITY IN UNTWINNED CRYSTALS OF $YBa_2Cu_3O_{7-\delta}$*

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ABSTRACT

A review of dc magnetization measurements of the superconducting transition in twinned and untwinned single crystals of $YBa_2Cu_3O_{7-\delta}$ is presented. The upper critical fields derived from the onset of diamagnetism are a factor of 3-4 times larger than those determined from the zero resistance temperature. The magnetic upper critical fields are linear in temperature with an anisotropy in slope of ~5 between the **c** direction and the **ab** plane. The angular dependence of the upper critical field implies three dimensional behavior. A crossover to layered behavior is estimated to occur at ~74 K. In untwinned crystals produced by a uniaxial strain technique, there is no detectable anisotropy in the upper critical field in the **ab** plane, implying that the one dimensional Cu-O chains do not play a significant role in determining the superconducting properties.

1. INTRODUCTION

The upper critical field is one of the most informative of the fundamental superconducting properties. It reflects the intrinsic symmetry and anisotropy of the superconducting state as well as the structure of the vortex state occurring at intermediate fields. The most important connection between the orbital upper critical field H_{c2} and the characteristic superconducting state parameters is given by

$$H_{c2} = \frac{\phi_o}{2\pi\xi^2} \qquad (1)$$

where ϕ_o is the quantum of flux and ξ is the Ginzburg-Landau (GL) coherence length. This formula can be derived from simple GL theory or from strictly geometrical arguments. The coherence length ξ is one of the two characteristic lengths in GL theory, the other being the magnetic penetration depth λ. Physically, ξ sets the length scale for several important features of the superconducting state. It is the minimum distance over which the superconducting order parameter can change, or, equivalently, the separation required between superconducting and non-superconducting parts of the crystal. The relative size of ξ and structural features, such as the layer spacing in the copper oxide systems, determines whether the anisotropy of those structural features may affect the superconducting behavior of the crystal. The coherence volume is primarily responsible for determining the importance of fluctuations at the superconducting phase transition. For applications where strong pinning is required to produce large critical currents, the coherence length determines the optimal size for defects to be effective pinning sites. Finally, the relative size of the coherence length and the mean free path in the normal state determines whether the sample is in the clean or dirty limit and, consequently, the quantitative response of the sample to external magnetic and electric fields.

In order to analyze the dimensionality of the superconducting state in the high T_c systems it is useful to reduce the crystal structure to a series of metallic copper oxide layers separated by relatively inert barium oxide layers. There are two important dimensions: the separation between the layers d and the thickness of the layers t, as shown in Figure 1. The dimensionality of the

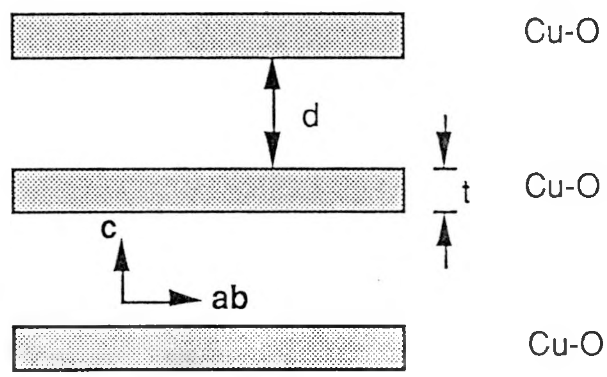

FIGURE 1. Schematic representation of layered Cu-O structure.

superconducting state is determined by the size of ξ compared to the layer separation d. If $\xi >> d$, the layers must all go superconducting together or not at all because the order parameter cannot change its value from layer to layer. This is the three dimensional limit where the usual 3D GL formulation applies. In the anisotropic form of GL theory[1)], there is only one anisotropic parameter, the effective mass m, which determines the anisotropy in both the coherence length and the upper critical field. H_{c2} is given by

$$H^{i}_{c2} = \frac{\phi_0}{2\pi\xi_j\xi_k} \qquad (2)$$

where the indices ijk indicate that H_{c2} in a given direction is given by the coherence lengths in the two perpendicular directions, and the anisotropies are related by

$$\frac{H_{c2}^{ab}}{H_{c2}^{c}} = \frac{\xi_{ab}}{\xi_c} = \left(\frac{m_c}{m_{ab}}\right)^{1/2} \qquad (3)$$

where $m_{\mathbf{ab}}$ and $m_{\mathbf{c}}$ are the effective masses in GL theory. Measurements of H_{c2} in the **c** and **ab** directions give the two coherence lengths $\xi_{\mathbf{c}}$ and $\xi_{\mathbf{ab}}$.

The two dimensional case is given by $\xi_{\mathbf{c}} << d$, where each of the layers is allowed to change its superconducting behavior independently of the others. This case is similar to the thin film case[2] where the thickness of the layers t determines some of the superconducting properties. In particular, in the expression for the upper critical field in the **ab** plane, the coherence length $\xi_{\mathbf{c}}$ must be replaced by t,

$$H_{c2}^{ab} = \frac{\phi_0}{2\pi\xi_{ab} t} . \qquad (4)$$

This has consequences for both the temperature and angular dependence of H_{c2}. The temperature dependence is affected because the coherence length in GL theory is proportional to $(T-T_c)^{-1/2}$, so that the three dimensional behavior of H_{c2} is linear while the two dimensional behavior is parabolic. The angular dependence is affected because the replacement of ξ by t occurs only for the field in the **ab** plane, not for the field in the **c** direction, where the perpendicular coherence length $\xi_{\mathbf{ab}}$ is not influenced by the layering. We expect the angular dependence of H_{c2} for the layered case to be similar to that for an isolated thin film of finite thickness t as derived by Tinkham.[2] A comparison of the angular dependence of H_{c2} from the thin film formula and the usual 3D GL theory is given in Figure 2. Because $t < \xi_c$, one expects H_{c2} to be larger in the thin film case than in the 3D case for the field in the

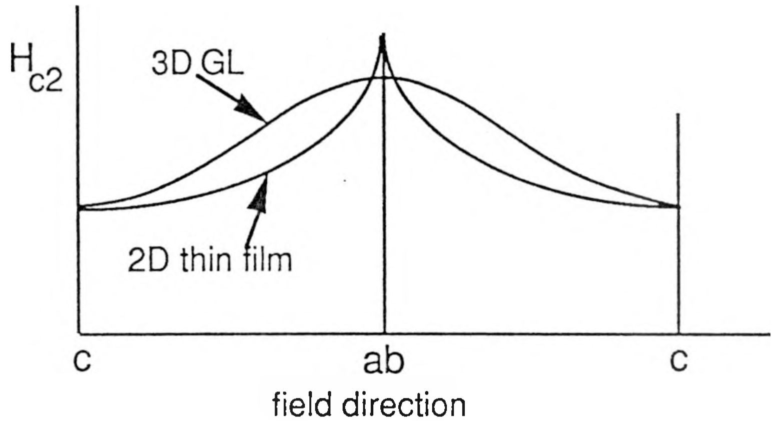

FIGURE 2. Schematic representation of the variation of the upper critical field with angle for the GL model and the thin film model.

ab plane. In addition, there is an obvious qualitative difference: the 3D case gives a smooth upper critical field with zero derivative as the field rotates through the **ab** plane, while the thin film case gives a cusp with a discontinuous derivative. Both the temperature and angular dependence of H_{c2} can be used to characterize the dimensionality of the system.

2. UPPER CRITICAL FIELD: TEMPERATURE DEPENDENCE

2.1 Resistive measurements

Resistive measurements of the superconducting transition in $YBa_2Cu_3O_{7-\delta}$ show an unusual broadening in the presence of a magnetic field,[3)] as illustrated in the lower panel of Figure 3. The onset of the transition remains nearly independent of the applied

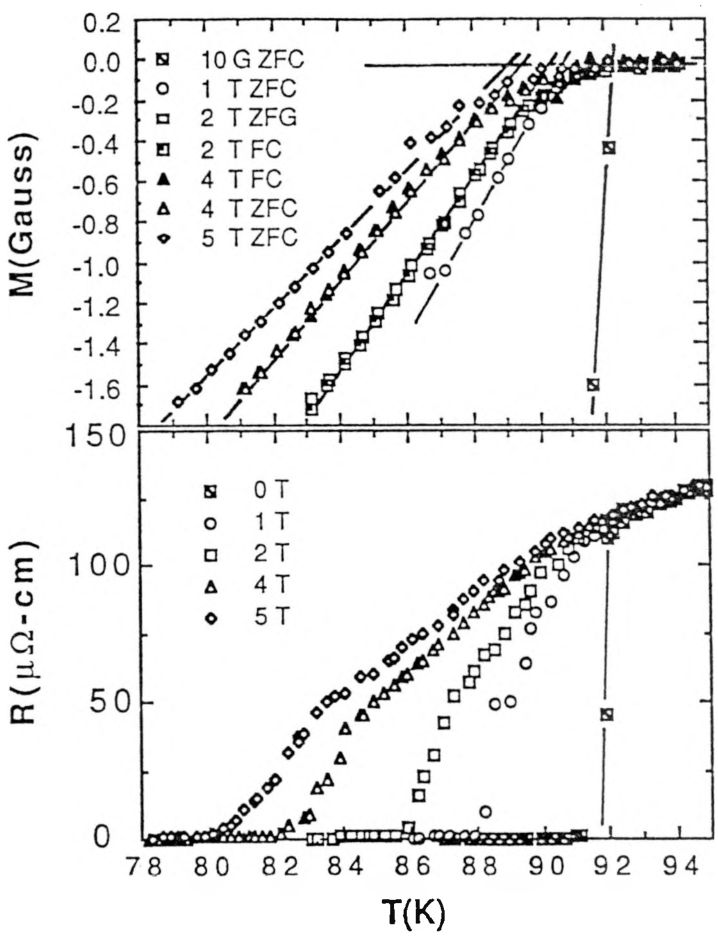

FIGURE 3. Upper panel: Temperature dependence of the magnetization in different magnetic fields oriented perpendicular to the Cu-O planes for zero field cooled (ZFC) and field cooled (FC) measurements. Lower panel: Resistive transition curves in the same fields as in the upper panel.

field, while the zero resistance point moves steadily downward as the field strength is increased. This feature is prominent in both the polycrystal and single crystal data.[3)] Conventionally, one expects a sharp drop in the resistivity occurring at the superconducting transition temperature. The absence of such a sharp drop in the resistivity data makes any conclusion about the size of H_{c2} somewhat suspicious. In particular, very different values of H_{c2} result from different choices of criteria for T_c, such as 50% of the normal state resistivity or the zero resistance point.

2.2 Magnetic measurements

In order to find an alternate, unambiguous measurement of H_{c2}, we have carried out dc magnetic measurements[4)] of the superconducting transition in fields up to 5 T using a SQUID magnetometer. In this experiment, the sample is cooled in an applied field while the magnetization is monitored (FC), or cooled in zero field, after which a magnetic field is applied, and the magnetization is monitored on warming (ZFC). One expects a zero or small response in the normal state, and a sharp downturn with the onset of diamagnetism when the sample enters the superconducting state. If the field is above H_{c1}, the diamagnetism increases continuously as the temperature is lowered all the way to T=0 K. For such fields the concept of a transition width does not exist, and the signature of T_{c2}, the temperature at which superconductivity sets in for the particular applied field, is the downturn in the magnetization at the onset of diamagnetism. There are two advantages to this method over resistance measurements. First, dc magnetization in the reversible state is a thermodynamic quantity which can be related to the specific heat and condensation energies. Second, dc magnetization is a bulk measurement, requiring a large fraction of the sample to respond before any measurable signal is observed. Thus, superconducting filaments or second phases will not confuse the interpretation.

Magnetization data for the field in the **c** direction are shown in the upper panel of Figure 3. The expected features are observed, in particular an onset of diamagnetism at a well-defined temperature for each applied field. The linear behavior of the magnetization below the onset allows one to extrapolate back to zero magnetization to define the onset temperature T_{c2}. It will be shown below that T_{c2} defined in this way is the mean field transition temperature of GL theory. As the applied field is increased, T_{c2} moves systematically down in temperature, allowing $H_{c2}(T)$ to be traced out.

In order to establish that H_{c2} measured in this way is an intrinsic property of the sample, it is necessary to show that the magnetization curves in Figure 3 are the equilibrium values, unaffected by flux pinning. This was verified in two ways. At 2 T and 4 T data were taken by both field cooling and zero field cooling, with the results shown in Figure 3. There is no detectable difference between the FC and ZFC data, indicating that the measured magnetization is independent of the sample history. In addition, magnetization curves were measured in increasing and decreasing field at several temperatures in the range shown on Figure 3. No hysteresis was found, showing directly that the magnetization is reversible.

The linear construction shown in Figure 3 for defining T_{c2} can be given a precise interpretation using GL theory. In GL theory, the magnetization near H_{c2} is given by the linear expression[5)]

$$M(H,T) = \frac{H - H_{c2}(T)}{4\pi\beta_A(2\kappa^2-1)} \qquad (5)$$

where H is the applied field, β_A is a numerical constant dependent on the structure of the vortex lattice, and κ is the GL parameter λ/ξ. Taking the temperature derivative of (5) and ignoring the small temperature dependence of κ,

$$\frac{\partial M}{\partial T} = \frac{-1}{4\pi\beta_A(2\kappa^2-1)}\frac{\partial H_{c2}}{\partial T} \ . \qquad (6)$$

In GL theory, H_{c2} is linear in T, so the magnetization below T_{c2} is also linear, as is observed. The intersection of the linear portion of M below T_{c2} with the normal state value then gives the GL transition temperature. This is the mean field transition temperature which would occur in the absence of fluctuations. In all real systems one expects some rounding of the transition due to sample inhomogeneity or to fluctuations, and such rounding is observed in Figure 3. Nevertheless, the GL mean field behavior is recovered below the transition, allowing the mean field transition temperature to be inferred from the data by the linear construction of Figure 3. Thus, the difficult problem of defining a transition temperature in the presence of fluctuations or sample inhomogeneities is avoided by our method.

The upper critical fields derived from the resistive and magnetic measurements are shown in Figure 4. The symbols show the magnetic measurements for the field in the **c** direction and in the **ab** plane and the dotted lines show the temperatures where the resistivity goes to zero. The magnetic data can be fit well by a straight line with the slopes indicated, giving an anisotropy of about 5 in the critical field. The resistive zeros occur at much lower fields, by a factor of 3 to 4, and show upward curvature which is characteristic of the onset of dissipation. Comparing the resistive and magnetic data, one sees that the resistive data are extremely misleading as to the values of H_{c2}. This point can be made even more dramatically by comparing the original data in Figure 3. At the onset of diamagnetism, there is no visible feature in the resistivity curves. The onset of diamagnetism occurs consistently at about 85% of the normal state resistivity; that is, there is a large resistivity in the superconducting state. The origin of this resistivity, often attributed to flux flow effects, is currently controversial.

In the region within about 1 K of T_c, the magnetic upper critical fields in both directions flatten considerably, approaching T_c

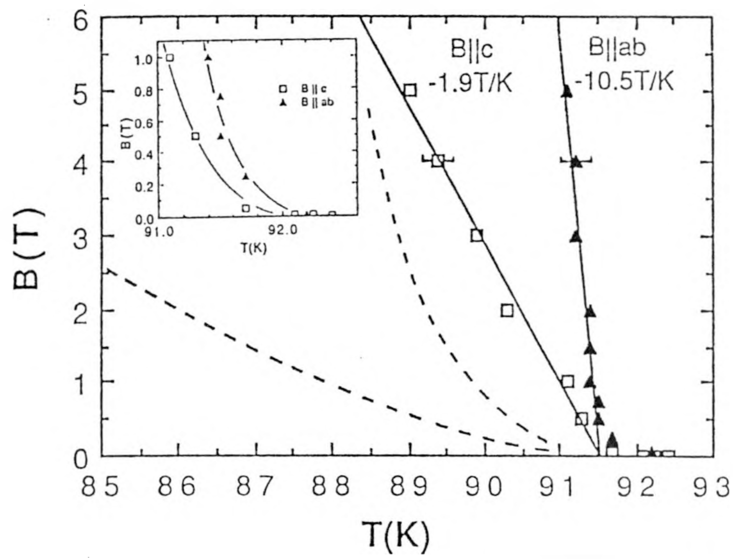

FIGURE 4. Temperature dependence of the magnetically determined upper critical field for both field orientations. The slopes of the linear extrapolations are indicated. The dashed lines represent the zero resistance temperatures. Inset: Low field part of the magnetic phase diagram. The lines are guides to the eye.

with very small slopes. This behavior is systematic and reproducible, as shown in the inset to Figure 4. Such behavior has been documented[6)] earlier on other crystals of $YBa_2Cu_3O_{7-\delta}$. At present we offer no explanation for this curvature, but we note that it seems to depend on the presence or absence of twin boundary planes[7)], as described below.

The upper critical field slopes can be used to infer the zero temperature critical fields and coherence lengths according to

$$H_{c2}(0) = 0.7\, T_c \frac{\partial H_{c2}}{\partial T} \tag{7}$$

and Equation (2). The results are

$$H^{c}_{c2}(0) = 120\,\mathrm{T} \qquad H^{ab}_{c2}(0) = 670\,\mathrm{T}$$

$$\xi_{ab} = 16\,\text{Å} \qquad \xi_{c} = 3.0\,\text{Å}\,.$$

The coherence length in the **ab** plane is much larger than the unit cell dimension, ~3.8 Å, while the coherence length in the **c** direction is much smaller than the unit cell dimension, ~12 Å. Thus one expects that layering will play a role in determining the superconducting properties at low temperature. The linear behavior of H_{c2} implies that the superconductivity is three dimensional at the measurement temperature.

3. UPPER CRITICAL FIELD: ANGULAR DEPENDENCE

Further insight into the dimensionality of the system can be obtained from the angular dependence of H_{c2} between the **c** direction and the **ab** plane. Complete measurements of the temperature dependence of H_{c2} at many field directions between **c** and **ab** would be too time consuming to carry out. Instead, we adopted a simpler procedure: measurement of the variation in T_{c2} with field direction in a fixed field strength of 5 T. The variation in T_{c2} expected from the 3D GL model and from the thin film formula can be derived[8] and show qualitatively the same behavior as H_{c2}, illustrated in Figure 2. The field direction was varied *in situ* by changing the sample orientation in the magnetic field using a special rotator[8] built for this experiment. The values of T_{c2} were determined from dc magnetization experiments as described above.

The measured variation of T_{c2} is shown in Figure 5. There are many data near Θ=90° because that is the region where the 3D GL and thin film behaviors differ the most. A qualitative assessment of the data suggest they are more consistent with a flat behavior at

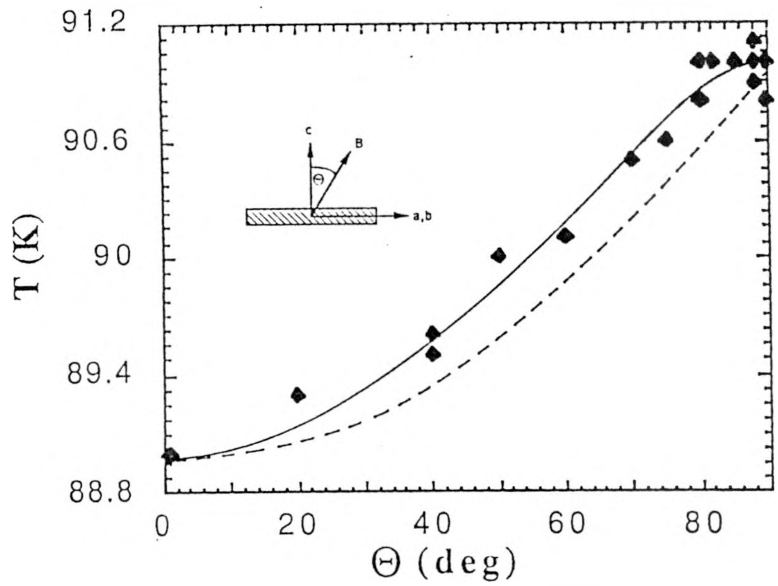

FIGURE 5. Angular dependence of T_{c2}. The solid line is a fit to the anisotropic GL theory with a mass anisotropy of 25. The dashed line is a fit to the thin film model.

Θ=90° than with a cusp. A test of the 3D GL form (solid curve) was made by requiring the curve to go through the datum at Θ=0° and using the critical field anisotropy of ~5 (mass anisotropy of ~25) obtained from the temperature dependence of H_{c2} in the **c** direction and in the **ab** plane. This procedure gives a rather good account of the data. The thin film formula (dotted line) was tested by requiring it to describe T_{c2} at both Θ=0° and Θ=90°, and observing the agreement at intermediate angles. The data follow the 3D GL behavior significantly better than the thin film behavior. We conclude from the angular dependence of H_{c2} that, in the temperature range near T_c, the superconductivity is three dimensional and well described by GL theory with a mass anisotropy of ~25.

The crossover from 3D behavior near T_c to layered behavior at low temperature is governed by the relative sizes of the coherence length ξ_c and the layer separation d. Assuming the GL temperature dependence $\xi_c(T)=\xi_c(0)/(T-T_c)^{1/2}$ and assigning a value to d, the crossover temperature T* can be estimated from the condition $\xi_c(T^*)=d$. Letting d=8 Å, the separation of Cu-O double layers in $YBa_2Cu_3O_{7-\delta}$, and using $\xi_c(0)=3.0$ Å, the crossover temperature is estimated to be 74 K. Thus, in $YBa_2Cu_3O_{7-\delta}$ there is a rather wide temperature range where 3D behavior is expected.

4. ANISOTROPY IN THE **ab** PLANE

While the existence of Cu-O layers in $YBa_2Cu_3O_{7-\delta}$ is the most dominant feature of the structure, there is another structural feature which may play an important role in determining the superconducting properties. Between each double layer of planes is a layer of Cu-O chains along the **b** direction. According to band structure calculations[9)] these chains are metallic and therefore may make a potentially important contribution to the superconducting behavior. Experimentally, the chains are known to play a role in determining T_c, since the transition temperature can be reduced to zero simply by depleting the oxygen in the chain sites.[10)] However, whether the chains are important for the pairing of electrons or simply act as a reservoir of electrons for pairing that takes place in the planes is at present an unresolved issue.

A direct confirmation of the fundamental importance of the chains for superconductivity could be provided by their influence on the upper critical field. If the chains play a strong role in superconductivity, one may expect an anisotropy in H_{c2} in the **ab** plane depending on whether the field is aligned parallel or perpendicular to the chains, in analogy to the anisotropy in H_{c2} induced by the Cu-O planes. Measurements of the upper critical field anisotropy in the **ab** plane are complicated by the presence of twins in the $YBa_2Cu_3O_{7-\delta}$ structure. These twins can be removed by a strain-anneal process described below and the anisotropy of H_{c2} in the **ab** plane can be measured.

4.1 Twins

Symmetry related twins occur in the orthorhombic $YBa_2Cu_3O_{7-\delta}$ structure by the interchange of the **a** and **b** axes in the basal plane, maintaining a common **c** direction. The boundary between the twin domains is either a (110) or (1$\bar{1}$0) plane at ~45° to the **a** and **b** axes. The twin boundary planes extend macroscopically throughout the crystal, often traversing the entire sample. Depending on how the sample is prepared, the spacing between twin boundary planes ranges from 500 Å to 10,000 Å.

The presence of twin boundary planes may have three important consequences. First, the twins prevent any measurement of the anisotropy in the **ab** plane because it is impossible to find a unique **a** or **b** direction for the crystal as a whole. Second, they may pin flux lines effectively, since they are capable of interacting with a flux line over its entire length.[11)] Third, they may alter the intrinsic behavior of the crystal near T_c, where the two characteristic lengths ξ and λ are diverging. If the twin boundary spacing is comparable to ξ or λ, deviations in the superconducting behavior at the twin boundary planes can propagate throughout the crystal.[12)]

We have devised a method[7)] for removing twins from single crystals of $YBa_2Cu_3O_{7-\delta}$ using a uniaxial strain technique similar to those reported earlier.[13)] The method is based on the difference in length between the **a** and **b** axes in the orthorhombic structure. If a uniaxial pressure is placed along one of the two **a**/**b** directions in the basal plane of a twinned crystal, the strain can be removed if twin boundaries move out of the crystal and leave the shorter **a** axis as the compressed direction. The twin boundaries move relatively easily by the motion of an oxygen atom diagonally across the unit cell. Thus, a period of a few hours at ~400 C under uniaxial stress in flowing oxygen is sufficient to remove the twins from a crystal of mm dimensions. The complete details of the procedure are given elsewhere.[7]

The twins can be observed most easily with polarized light, where characteristic color patterns arising from the relative orientation of the **a** and **b** axes of the crystal with the polarization of the light can be seen. Direct observation of the fractional volume contained in each twin domain can be made with x-ray diffraction. Crystals subjected to the uniaxial strain and annealing process show Bragg peaks corresponding to only one twin domain,[7] providing strong evidence that the de-twinning procedure works well. Further evidence of the absence of twins comes from measurements of the elastic anisotropy[14] of single crystals in the **ab** plane. No anisotropy is seen in twinned crystals, while a variation of ~10% is seen in untwinned crystals.

4.2 Upper critical field anisotropy

Using an untwinned crystal produced by the method described above, dc magnetization measurements[7] were made of the superconducting transition for the field in the **a** and **b** directions. Results for 1, 2 and 4 T are shown in Figure 6. As is evident, there is no measurable difference between the values of T_{c2} for the two directions. Thus, within the experimental resolution, there is no difference in H_{c2} for the **a** and **b** directions. However, the minimum detectable difference depends on how well the temperature T_{c2} can be determined, which, in turn, depends on the temperature interval between data points in Figure 6. Differences in T_{c2} of as little as 70 mK cannot be detected, which corresponds to a critical field anisotropy of order 15%. Thus, recent Bitter pattern experiments[15] of trapped flux in $YBa_2Cu_3O_{7-\delta}$ indicating an

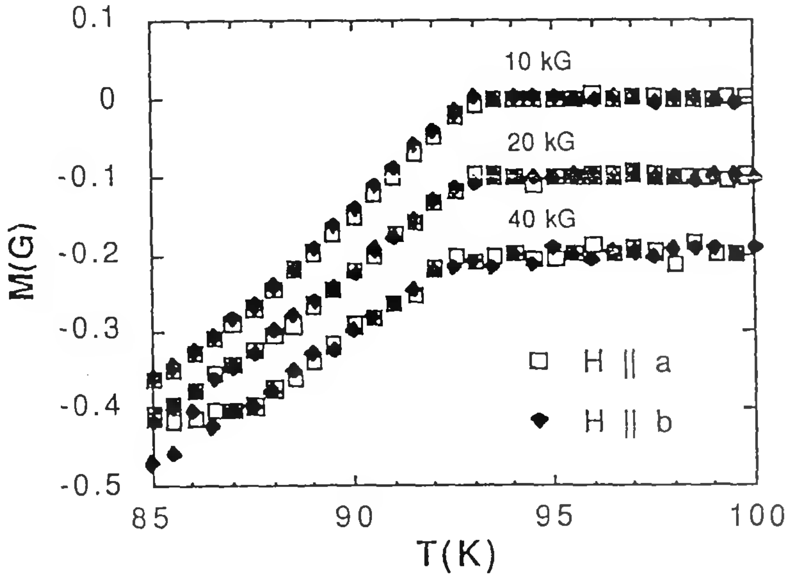

FIGURE 6. Temperature dependence of the magnetization in various fields parallel to **a** and **b**. Between the different sets of results the zero-point has been shifted by -0.1 G for clarity.

effective mass anisotropy of 1.3, corresponding to a critical field anisotropy of 14%, are not inconsistent with the present results.

The full temperature dependence of H_{c2} for all three directions in an untwinned crystal can be measured using the dc magnetization technique.[7)] The results are shown in Figure 7. The critical field slopes for the **c** direction and the **a** and **b** directions are the same as found for the twinned crystal, within experimental uncertainty: -1.8 T/K for the **c** direction, and -10 T/K for the **a** and **b** directions. There are two important differences from the twinned crystal. First, the value of T_c in zero field is higher, about 93 K compared to 92.5 K in the twinned crystal. Whether this increase is related to the absence of twins is not clear at present. Second, the strong upward curvature seen in twinned crystals within 1 K of T_c is not seen in the untwinned crystal. Figure 7 shows that the linear

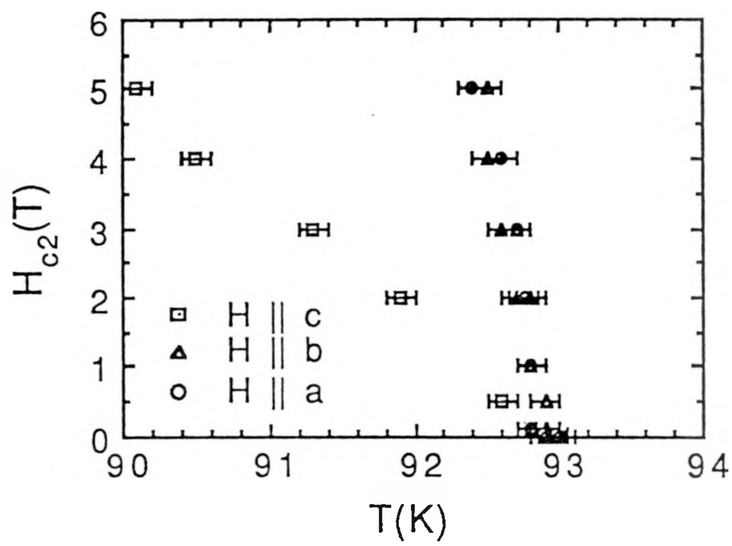

FIGURE 7. Temperature dependence of the upper critical field for an untwinned crystal along the three crystallographic orientations.

behavior at high fields extrapolates well all the way to T_c. Thus the strong upward curvature in H_{c2} near T_c in twinned crystals is not intrinsic to the $YBa_2Cu_3O_{7-\delta}$ materials and is likely to be caused by the presence of twins. We have no detailed explanation for this effect at present.

5. CONCLUSIONS

High field dc magnetization measurements of the superconducting transition in $YBa_2Cu_3O_{7-\delta}$ reveal a number of features not seen in resistive experiments. The onset of diamagnetism occurs at much higher temperatures than the zero of resistivity, implying there is a substantial resistivity, up to ~85% of the normal state value, in the superconducting state. The upper critical fields measured magnetically are a factor of 3 to 4 times larger than those derived from the zero resistance temperature. H_{c2} is linear in temperature in both the **c** direction and in the **ab** plane, with a factor of ~5 anisotropy in the slopes. The zero temperature value of the coherence length in the **c** direction is 3 Å, significantly shorter than the Cu-O layer separation. Therefore, the layered structure is expected to strongly affect the superconducting behavior at low temperatures. Near T_c, the angular dependence of the upper critical field between the **c** direction and the **ab** plane implies three dimensional superconductivity and is well described by a Ginzburg-Landau model. The crossover temperature between three dimensional and layered behavior is estimated to be ~74 K.

The presence of Cu-O chains in the $YBa_2Cu_3O_{7-\delta}$ structure suggests there may be anisotropy in H_{c2} between **a** and **b** in the basal plane if the chains play a role in the pairing process. The search for such an anisotropy is frustrated by the existence of twins in which the **a** and **b** axes are interchanged with respect to each other. We have developed a procedure to remove the twins from single crystals using uniaxial stress and low temperature annealing. DC magnetization measurements of the upper critical field in untwinned crystals show no measurable anisotropy between **a** and **b**, although a difference of ~15% would not be detectable in our experiment. This implies that the chains do not play a significant role in determining the superconducting properties. Untwinned crystals show the same critical field slopes as twinned crystals, but do not show any upward curvature close to T_c.

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