Equilibrium superconducting properties of grain-aligned HgBa₂Ca₂Cu₃O_{8+ δ}

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The upper critical field H_{c2} and other equilibrium properties of a magnetically aligned $HgBa_2Ca_2Cu_3O_{8+8}$ superconductor have been investigated. Experimental results for the reversible magnetization, both with H||c and H||ab, are analyzed using the theoretical formalism of Hao *et al.* [Phys. Rev. B 43, 2844 (1991)] for type-II superconductors. Near T_c , the slopes dH_{c2}/dT of the upper critical field are -1.6 T/K and -12.1 T/K for fields parallel and perpendicular to the *c* axis, respectively. These correspond to coherence lengths (extrapolated to T=0) of $\xi_{ab}(0)=15$ Å and $\xi_c=1.9$ Å. This gives a lower bound on the superconductive anisotropy parameter $\gamma = (m_c/m_{ab})^{1/2} \ge 8$.

INTRODUCTION

The equilibrium properties of high- T_c superconductors, such as the upper critical fields H_{c2} and the thermodynamic critical field H_c , are important, because they give relatively direct information about the microscopic parameters. These include the superconducting coherence length ξ and the magnetic penetration depth λ , particularly for the interesting case in which the magnetic field is applied perpendicular to the CuO layers in the superconductors. Studies of aligned materials also can provide lower bound estimates for the degree of superconducting anisotropy.

In earlier analyses of dc magnetization measurements, Welp et al.¹ obtained values for H_{c2} for YBa₂Cu₃O_{7- δ} by determining a nucleation temperature $T_c(H)$. This temperature was constructed by linearly extrapolating the temperature-dependent reversible magnetization M in the superconducting state (with H= fixed) to the normalstate base line. Hao et al.² pointed out, however, that much of the experimental magnetization data corresponded to fields H far below $H_{c2}(T)$. Under these conditions, the usual linear relation between M and H near $H_{c2}(T)$ [which follows from Ginzburg-Landau (GL) theory³] does not adequately describe their functional relationship. Indeed, the experiments showed that the slopes dM/dT changed with field, confirming that simple linear relationships between M and H are not correct. As described below, Hao and Clem developed a more sophisticated theory that enables one to determine many fundamental properties of a superconductor from studies of the reversible magnetization in the intermediate-field region.⁴ This formalism is most useful well below T_c , where fluctuation effects are relatively insignificant. Near T_c , fluctuations play a particularly pronounced role in high- T_c superconductors. For this regime, Bulaevskii, Ledvij, and Kogan⁵ and Kogan *et al.*⁶ have developed a theory incorporating such effects, which can complement other analyses in favorable cases.

In this study, we analyze the reversible magnetization of HgBa₂Ca₂Cu₃O_{8+ δ}, for both field orientations H||*c* and H||*ab*, using the formalism of Hao-Clem. We evaluate the characteristic fields $H_{c2}(T)$ and $H_c(T)$ to obtain values (extrapolated to T=0) for the microscopic parameters $\lambda(0)$ and $\xi(0)$ using standard Ginzburg-Landau expressions. Finally, we corroborate some of these results by analyzing the equilibrium magnetization near T_c using two-dimensional (2D) scaling theory.⁷

EXPERIMENTAL ASPECTS

Bulk samples of HgBa₂Ca₂Cu₃O_{8+ $\delta}$ were prepared by a solid-state reaction from stoichiometric mixtures of 99.998% HgO, 99.997% BaO, 99.97% CaO, and 99.999% CuO as described previously.⁸ X-ray diffraction indicates that >90% of the material comprise the desired three-layer Hg-cuprate phase. From these materials, an aligned composite sample was produced from monocrystallites of 7 μ m average size that were dispersed in "45 min" liquid epoxy and aligned in a 5 T field. The volume}

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fraction of superconductor in the epoxy matrix was about 9%. X-ray diffraction with the scattering vector parallel to the alignment field direction revealed strong (00*l*) reflections, but no (hkl) reflections with nonzero indices h or k. The width of the rocking curve full width at half maximum was 2.6° for the (006) line.

The magnetic properties were measured with a Quantum Design model MPMS-7 superconducting quantum interference device magnetometer. Scan lengths of 3 cm were used to maintain a field uniformity of < 0.005% in the 7 T magnet during measurement. Before application of the magnetic field, the temperature was stabilized to within ± 0.05 K of the target temperature. Measurements of the isothermal magnetization M(H) were made for a set of temperature T between 5 and 140 K, with fields $H \| c$ and $H \| ab$ orientation. Before each new T setting, the sample was brought to a temperature well above T_c and then cooled under zero applied field. Once the temperature was stabilized, the magnetic moment was measured at various applied magnetic fields H, increasing from 0.01 to 6.5 T and decreasing back to 0.01 T. After each field increment, the system paused for 10 sec to allow rapid instrumental transients to decay. In order to correct for magnetic background signals and obtain reliable information for the Hao-Clem analysis, the normalstate magnetic susceptibility was measured for temperature up to 250 K. A Curie-like behavior was found. After subtracting the background signal, the equilibrium (reversible) magnetic moment was determined as the mean value between field-increasing and -decreasing measurements. In most of the experimental range, however, both signals coincided since the (T,H) settings were mostly in the reversible region above the irreversibility line of the magnetic phase diagram.

The equilibrium magnetization M(T,H) of the sample was calculated as the reversible magnetic moment per unit of volume of superconductor. The superconducting volume was obtained by dividing the total mass of the sample by the x-ray density (in our case, 6.3 g/cm³). The process assumes that all of the HgBa₂Ca₂Cu₃O_{8+δ} powder superconducts; the following results for the lowfield susceptibility justify this assumption.

EXPERIMENTAL RESULTS

Figure 1 shows the low-field dc susceptibility $4\pi\chi$, using a static measuring field of 4.0 Oe. Prior to these measurements, the magnet in the magnetometer was "reset" by heating it above its T_c , so that trapped magnetic flux in the windings was released. The data in Fig. 1 exhibit no significant structure, such as an additional signal near 95 K arising from a Hg-1201 impurity phase. The transition (10-90%) is narrow for $\mathbf{H} \parallel c$, which give a qualitative measure of the sample homogeneity. The conventional superconductive transition temperature T_c , defined by the low-field diamagnetic onset, is 133.5 K.

At low temperature, the zero-field-cooled (ZFC) susceptibility for $\mathbf{H} \| c$ is $4\pi \chi = -0.94$, corresponding to nearly complete screening of the grains interior; for $\mathbf{H} \| ab$, the corresponding ZFC value is -0.26. In each case, the values have been corrected for demagnetizing

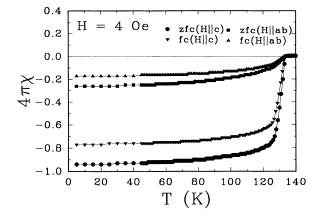


FIG. 1. Field-cooled and zero-field-cooled transition curves for both field orientations in aligned $HgBa_2Ca_2Cu_3O_{8+\delta}$.

effects using an effective demagnetizing factor $D \approx \frac{1}{3}$, as appropriate for a dilute array of roughly equiaxed particles.

When cooling from above T_c in the same 4 Oe applied field, we observed a large Meissner flux expulsion, with a field-cooled (FC) susceptibility $4\pi\chi = -0.76$ for $\mathbf{H} \parallel c$. This value, 76%, is a lower bound estimate for the true superconducting volume fraction. In this field configuration, the near surface of a grain is penetrated by the field to a depth $\sim \lambda_{ab}$, the London penetration depth for screening by supercurrent flow in the ab direction. Considering the small particle size, which reduces the magnitude of the susceptibility, the observed $4\pi\chi$ implies that most (if not all) of the powder sample is superconducting, as assumed earlier. At 5 K, the corresponding FC value with $H \parallel ab$ was -0.17. Thus the ratio of susceptibilities at low temperature for different field directions, $\chi(H||c)/\chi(H||ab)$, is 4.5 for the FC case and 3.6 for the ZFC case, respectively. Comparing the FC with the ZFC responses gives susceptibility ratios $\chi(FC)/\chi(ZFC)$ of 0.82 for H||c and 0.64 for H||ab. Thus flux was trapped more strongly at low field with $H \parallel ab$.

Figure 2 shows experimental results for the magnetization M versus T near T_c , in various applied magnetic fields for both $H \parallel c$ and $H \parallel ab$. In a field of 1 T, the magnetization is reversible for temperatures above approximately 62 K for $H \parallel c$ and 95 K for $H \parallel ab$, respectively. We first analyze the data with $H \parallel c$, using the theory of Hao et al.² This formalism relates H to B by accounting for the kinetic energy and the condensation energy terms arising from a suppression of the order parameter in vortex cores, which is neglected in the usual London model. Operationally, the diamagnetism M(T,H) is calculated numerically and fitted to the experimental data. The fitting procedure yields two important properties, the Ginzburg-Landau parameter $\kappa(T) = \lambda/\xi$ and the thermodynamic critical field $H_c(T)$. The product of these two gives $H_{c2}(T) = \sqrt{2\kappa}(T)H_c(T)$, and $\lambda(T)$ and $\xi(T)$ can be obtained, also.

The fitting procedure is as follows. For a fixed value of T within the reversible regime, the experimental values $-4\pi M_i/H_i$ have to be compared with the theoretical ra-

tio $-4\pi M'/H'$ (where M' and H' are dimensionless quantities) as obtained from the simultaneous solution of Eqs. (20) and (21) of Ref. 2; here $B' = H' + 4\pi M'$ and κ as adjustable parameters. Primes denote dimensionless units in which fields are measured in units of $\sqrt{2H_c(T)}$. The subindex i denotes values in different fields with Tfixed. For each value of κ , there will be one solution B'. From this, M' and H' are derived. The unit of magnetic field is determined from $\sqrt{2H_{ci}(T)} = H_i/H' = M_i/M'$. This has to be done for each data point i. If the value chosen for κ is not right, the resulting values for H_{ci} will not be constant (as they should be, since T is fixed). The best choice for κ gives the minimum variance in the set of values for $H_{ci}(T)$. The resulting values for $\kappa(T)$ at each temperature are shown in Fig. 3, for both field orientations.

In this analysis, the temperature range should extend not too far below T_c (since the system must be reversible), but not too close to T_c either, where the Ginzburg-Landau mean-field theory becomes invalid and superconducting fluctuations play an increasingly important role. In principle, κ should be almost constant, particularly near T_c ; in practice, the best κ values increase with temperature and, for $H \parallel c$, reach apparent values $\kappa_c \approx 400-600$ near T_c , due to the fact that fluctuation effects are not included in the theoretical formalism. Fortunately, this material in its "as prepared" form has relatively weak vortex pinning and is reversible well below

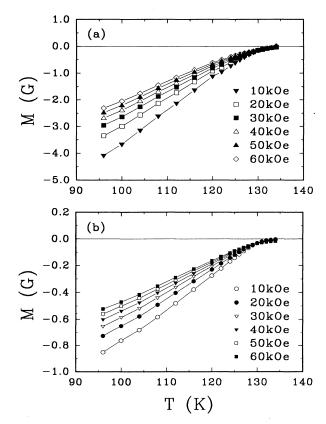


FIG. 2. Equilibrium magnetization M versus T for aligned HgBa₂Ca₂Cu₃O_{8+ δ} with (a) H||*c* and (b) H||*ab*.

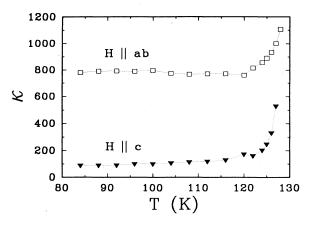


FIG. 3. GL parameter $\kappa_c(H||c)$ and $\kappa_{\text{eff}}(H||ab)$ as a function of temperature from Hao-Clem analysis.

 T_c , so we can work in a wide temperature range; see Fig. 3. The Ginzburg-Landau parameter $\kappa_c = \lambda_{ab} / \xi_{ab}$ is nearly temperature independent, with average value 102 (**H** $\parallel c$), in the temperature range 84–112 K.

To illustrate the underlying theory, Fig. 4 shows the reduced (dimensionless) quantities M' versus H', for HgBa₂Ca₂Cu₃O_{8+ δ} with H||*c*. The figure includes magnetization data (symbols) for a range of temperatures, all computed using the average value of the parameter κ_c . The theoretical model of Hao *et al.* (solid line) describes the experimental results well.

The other key information provided by the Hao-Clem analysis is the temperature-dependent thermodynamic critical field $H_c(T)$, as shown in Fig. 5. We extrapolate these results to obtain the value $H_c(0)$ at T=0 by fitting the BCS temperature dependence⁹ for $H_c(T)$,

$$\frac{H_c(T)}{H_c(0)} = 1.7367 \left[1 - \frac{T}{T_c} \right] \left[1 - 0.2730 \left[1 - \frac{T}{T_c} \right] -0.0949 \left[1 - \frac{T}{T_c} \right]^2 \right].$$
 (1)

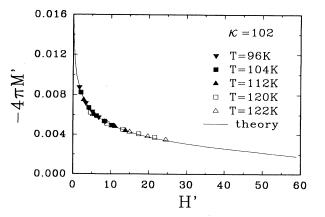


FIG. 4. Magnetization versus applied field in reduced (dimensionless) units for aligned $HgBa_2Ca_2Cu_3O_{8+\delta}$ with H||c.

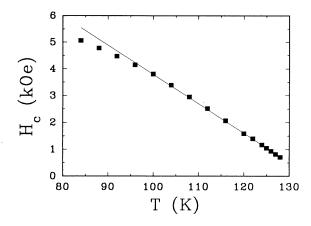


FIG. 5. Temperature dependence of H_c for aligned HgBa₂Ca₂Cu₃O_{8+ δ}. The solid line is fitted to the BCS temperature dependence (for T > 95 K).

The expression is valid for $T/T_c > 0.7$, i.e., for $T > \sim 93$ K, which is interval used. From this procedure, we obtain the result $H_c(0)=9.3$ kOe. The solid line in Fig. 5 is a plot of Eq. (1), fitted to the data (symbols). From H_c , we obtain the condensation energy density $F_c = H_c^2/8\pi$. This gives $F_c(0)=0.34$ J/cm³ at T=0, which is smaller than the value (0.45 J/cm³) found⁴ for YBa₂Cu₃O₇.

Let us now consider the field orientation H||ab. Again, the Hao-Clem analysis can be used, as described above. In this case, we impose the additional constraint that the condensation energy $F_c = H_c^2/8\pi$ must be the same as with H||c; certainly this thermodynamic quantity should be independent of orientation and field magnitude. The detailed values are shown in Fig. 3. Once again, κ is nearly independent of temperature up to ~120 K, with an average value $\kappa(H||ab|) \sim 784$.

From κ and H_c , one can obtain much useful information. For example, we construct the $H_{c2}(T)$ lines near T_c , as shown in Fig. 6. An extrapolation to zero field intersects the horizontal axis at the respective values $T_{c,Hc2}$ of 134.7 K for H||c and 133.9 K for H||ab. These values are

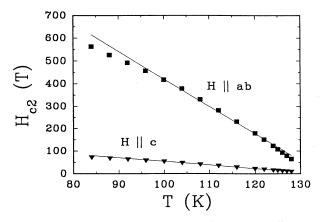


FIG. 6. Temperature dependence of the magnetically determined upper critical field near T_c in aligned HgBa₂Ca₂Cu₃O_{8+ δ} with **H** $\|c$ and **H** $\|ab$.

(slightly) higher than the T_c onset of 133.5 K determined from the low-field susceptibility. In earlier studies on Tl-2223 and Hg-1201 superconductors,¹⁰ similar effects were observed, but with a larger depression from the meanfield value as estimated from $T_{c,Hc2}$. These experimental results are consistent with the theoretical idea¹¹ that the mean-field T_c can be slightly higher than the experimentally observable T_c onset measured in small magnetic fields. Continuing then, Werthamer, Helfand, and Hohenberg¹² have shown that the upper critical field $H_{c2}(0)$ extrapolated to T=0 is proportional to the slope dH_{c2}/dT near T_c and is given by

$$H_{c2}(0) = -0.7T_c [dH_{c2}/dT]_{\approx T_c} .$$
⁽²⁾

Thus we obtain the values $H_{c2}^{H\parallel c}(0) = 149$ T and $H_{c2}^{H\parallel ab}(0) = 1150$ T, respectively. To estimate the coherence length at zero temperature, we use the GL expression $H_{c2}(0) = \phi_0/2\pi\xi_{eff}^2$; here ξ_{eff}^2 is ξ_{ab}^2 for $H\parallel c$ (ignoring *ab* plane anisotropy) and $\xi_{ab}\xi_c$ for $H\parallel ab$. The corresponding lengths are $\xi_{ab} = 15$ Å and $\xi_c = 1.9$ Å. In the latter case, the length is much smaller than the spacing "s" between trilayer sets, implying that the system should have substantial 2D character.

The derived quantities $\xi(0)$, $\lambda(0)$, and $H_{c1}(0) = H_c \ln \kappa / (\sqrt{2}\kappa)$, as readily calculated from $\kappa(0)$, $H_c(0)$, and $H_{c2}(0)$, are tabulated in Table I. In the effective-mass approximation,¹³ one has λ_{ab} and λ_c incorporated in an effective penetration depth for $\mathbf{H} \| c$ and $\mathbf{H} \| ab$ according to the following relations:

$$\lambda_{\text{eff}}(H\|c) = \lambda_{ab}, \quad \lambda_{\text{eff}}(H\|ab) = \sqrt{\lambda_{ab}\lambda_c} \quad . \tag{3}$$

We obtain the results $\lambda_{ab}(0) = 1700$ Å and $\lambda_{eff}(0) = 4670$ Å with $\mathbf{H} || ab$. Combining these gives the value $\lambda_c(0) = 13000$ Å.

Given the small values of ξ_c , it is reasonable to expect that the superconductive system is substantially two dimensional. For a 2D superconductor, theory predicts that the temperature and field dependence of physical quantities near T_c should scale in the variable $t_G = g[T - T_c(H)]/(TH)^{1/2}$, where g is a field- and temperature-independent function.^{7,14} Consequently, when $M/(TH)^{1/2}$ is plotted versus t_G , then the mixed-

TABLE I. Summary of physical characteristics measured for both orientations in aligned $HgBa_2Ca_2Cu_3O_{8+\delta}$. (For units conversion, note that $1 T = 10^4 G = 10^4 Oe$ in free space.)

Property	$\mathbf{H} \ c$	$\mathbf{H} \ ab$
$H_{c2}(0)$ (T)	149	1150
$\xi(0)$ (Å)	15	1.9
$(dH_{c2}/dT)_{T \approx T_c}$ (T/K)	-1.6	-12.1
$(\kappa)_{T\approx 0}$	110	870
$(\kappa)_{T \approx T_c}$	100	780
$H_{c1}(0)$ (Oe)	280	50
λ (nm)	170	$\lambda_c = 1300$
		$\lambda_{\rm eff} = 470$
$H_c(0)$ (kOe)	9.3	\equiv 9.3
$T_{c0,Hc2}$ (K)	134.7	133.9

state data should collapse onto a single curve, independent of H. (In the 3D case, the functional model is similar except that the exponent $\frac{1}{2}$ in the above expression is replaced by $\frac{2}{3}$.) The transition temperature $T_c(H)$ can be taken as a linear function, with slope dH_{c2}/dT and intercept T_{c0} on the T axis. Adjusting the two parameters provides the 2D scaling shown in Fig. 7(a) (for H||c) and 7(b) (for H||ab). It is evident that the 2D model provides good scaling of the data. The resulting values for the slope dH_{c2}/dT are -1.8 T/K for H||c and -12.2 T/K for H||ab. These independent results compare well with the values from the Hao-Clem analysis given in Table I.

DISCUSSION

The superconducting material HgBa₂Ca₂Cu₃O_{8+ δ} has a range of magnetic reversibility extending well below T_c . Consequently, it is relatively straightforward to apply thermodynamics to its magnetization, in the form of the Hao-Clem formulation. In this way, we have obtained useful information regarding this superconductor. We now consider some features and limitations of this study.

An interesting aspect of the magnetization data in Fig. 2 is that the mixed-state magnetization persists to temperature above the low-field diamagnetic onset T_c . This is due to vortex fluctuations, which give an entropic

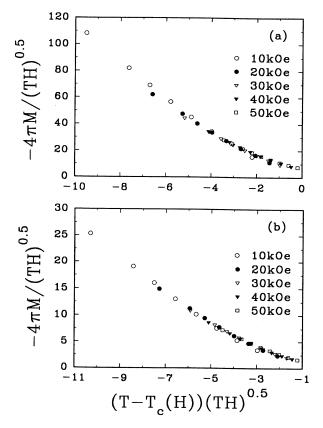


FIG. 7. Fluctuation analysis of M(T) using 2D model for aligned HgBa₂Ca₂Cu₃O_{8+ δ} with (a) H||*c* and (b) H||*ab*.

correction to the superconductive free energy that is particularly significant near T_c . As shown earlier by Tesanovic et al.14 and later by Bulaevskii, Ledvij, and Kogan⁵ and Kogan et al.,⁶ the equilibrium magnetization M(T)exhibits a "crossing point" at which M is independent of field. In fact, the various curves in Fig. 2 cross at a characteristic temperature $T^* = 129.5$ K. Unfortunately, we are not able in this case to make a reliable analysis using the fluctuation formalism. For example, the value of the crossing point magnetization M^* (for H||c) is small. Theoretically, one has $M^* = k_B T^* / \phi_0 s$, where "s" = 16 Å is the spacing between sets of CuO trilayers. Using this relation gives a calculated magnetization that is larger than the experimental value in Fig. 2 by a factor of 3. Previously, such an effect has been interpreted in terms of a superconducting volume fraction;⁶ in fact, we found this interpretation to be consistent with the measured Meissner signal and the crystallographic phase abun-dance in Hg-1201 and Tl-2223 materials.¹⁰ A parallel analysis here would imply that the superconducting volume fraction is only $\sim 30\%$. However, this value is unphysical, being much smaller than either the low-field shielding signal or the Meissner signal. This discrepancy may originate from background effects near T_c where the overall signals are small and the corrections are significant. Alternatively, the effect may arise from some more fundamental (but as yet unidentified) limitation. Much further from T_c where we have applied the Hao-Clem formalism, the background signals are relatively smaller and more easily removed.

With data for the two orthogonal field orientations, one can establish some bounds on the superconductive anisotropy parameter $\gamma = 1/\epsilon$. In an anisotropic London model,¹³ this quantity is related to the components of the normalized mass tensor m_i (with i=1,3) as follows: $\gamma^2 = m_3/m_1 = m_1^{-3} = m_3^{3/2}$ with $m_1^2 m_3 = 1$. In terms of fundamental lengths, one has $\gamma = \xi_{ab} / \xi_c = \lambda_c / \lambda_{ab}$. From the data in Table I, we obtain $\gamma \ge 7.7$, which is larger than that observed ($\gamma = 5.5$) in YBa₂Cu₃O₇, for example.^{1,15} We regard the present value as only a lower bound, since it directly depends on measurements with the field $H \parallel ab$. For this approximate orientation, M changes very rapidly with angle, making the measurement quite sensitive to any macroscopic sample misalignment. Also, the angular spread in the alignment of crystallites, 2.6° in this case, can be very significant when γ is large. Note, however, that *M* is *much* less sensitive to angular deviations when the orientation is nearly $\mathbf{H} \| c$. Consequently, the results for the latter case are unaffected. An alternative estimate of the anisotropy parameter, $\gamma \sim 16$, was obtained from an analysis of ac response studies¹⁶ on random polycrystals of Hg-1223. From a study¹⁷ of the irreversible properties of magnetically aligned Hg-1223, we obtained the estimate that $\gamma \approx 35-70$, as based on a 2D to 3D crossover in the magnetic response with $\mathbf{H} \| c$. The crossover field is $H_{cr} \sim 2$ kOe, well below the fields used for the scaling analysis in Fig. 7. Consequently, it is internally consistent that the scaling follows the 2D model.

Finally, we note that the present value for λ_{ab} , as shown in Table I, agrees well with the result of Bae

et al.¹⁸ for grain-aligned Hg-1223 and is quite comparable with the value of 1500 Å obtained by Huang et al.¹⁹ for polycrystalline Hg-1212. In both cases, the authors analyzed the equilibrium magnetization using the fluctuation formalism discussed above. These studies obtained κ values (**H**||c) of ~60 and (77±13), respectively, which lie between the value $\kappa \approx 50$ for Hg-1201 (Ref. 10) and our value of 100 for Hg-1223.

CONCLUSIONS

Using the Hao-Clem theory, we have analyzed the equilibrium magnetization of a grain-aligned sample of the high- κ type-II superconductor HgBa₂Ca₂Cu₃O_{8+ δ}. With the magnetic field applied parallel to the *c* axis, we deduce that the slope of the upper critical field is -1.6 T/K, the value of κ is 100, and the associated coherence length is $\xi_{ab}(0)=15$ Å. Comparison with the values for the case with H||ab gives a lower bound estimate for the

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anisotropy parameter $\gamma \geq 8$. Two-dimensional scaling theory describes the measured M(H,T) well and yields slopes dH_{c2}/dT that coincide with those deduced from the Hao-Clem analysis.

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