

Magnetic phase diagram of underdoped $\text{YBa}_2\text{Cu}_3\text{O}_y$ inferred from torque magnetization and thermal conductivity

Fan Yu^a, Max Hirschberger^b, Toshinao Loew^c, Gang Li^a, Benjamin J. Lawson^a, Tomoya Asaba^a, J. B. Kemper^{d,e,2}, Tian Liang^b, Juan Porras^c, Gregory S. Boebinger^{d,e}, John Singleton^f, Bernhard Keimer^c, Lu Li^a, and N. Phuan Ong^{b,1}

^aDepartment of Physics, University of Michigan, Ann Arbor, MI 48109; ^bDepartment of Physics, Princeton University, Princeton, NJ 08544; ^cMax Planck Institute for Solid State Research, 70569 Stuttgart, Germany; ^dDepartment of Physics, Florida State University, Tallahassee, FL 32310; ^eNational High Magnetic Field Laboratory, Florida State University, Tallahassee, FL 32306; and ^fNational High Magnetic Field Laboratory, Los Alamos National Laboratory, Los Alamos, NM 87545

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Strong evidence for charge-density correlation in the underdoped phase of the cuprate $\text{YBa}_2\text{Cu}_3\text{O}_y$ was obtained by NMR and resonant X-ray scattering. The fluctuations were found to be enhanced in strong magnetic fields. Recently, 3D charge-density-wave (CDW) formation with long-range order (LRO) was observed by X-ray diffraction in $H > 15$ T. To elucidate how the CDW transition impacts the pair condensate, we have used torque magnetization to 45 T and thermal conductivity κ_{xx} to construct the magnetic phase diagram in untwinned crystals with hole density $p = 0.11$. We show that the 3D CDW transitions appear as sharp features in the susceptibility and κ_{xx} at the fields H_K and H_p , which define phase boundaries in agreement with spectroscopic techniques. From measurements of the melting field $H_m(T)$ of the vortex solid, we obtain evidence for two vortex solid states below 8 K. At 0.5 K, the pair condensate appears to adjust to the 3D CDW by a sharp transition at 24 T between two vortex solids with very different shear moduli. At even higher H (41 T), the second vortex solid melts to a vortex liquid which survives to fields well above 41 T. de Haas–van Alphen oscillations appear at fields 24–28 T, below the lower bound for the upper critical field H_{c2} .

cuprate superconductivity | high-field phase diagram | vortex liquid | torque magnetometry | thermal conductivity

The existence of static charge order in underdoped $\text{YBa}_2\text{Cu}_3\text{O}_y$ (YBCO) in an intense magnetic field was reported by Wu et al. (1) using NMR. Subsequently, Ghiringhelli et al. (2) uncovered temperature-dependent charge-density-wave (CDW) correlations in zero magnetic field by resonant X-ray scattering (RXS). The RXS signal onsets near 180 K and peaks at the superconducting critical temperature T_c before falling. Several groups (3–5) recently showed that the RXS intensity is enhanced in finite field H . At low temperature T , several experiments have uncovered field-induced transitions. These include a transition at 18 T from ultrasonic measurements (6), features in the high-field thermal conductivity (7), and the onset of line splitting in the NMR spectra starting at the charge-ordering field $H_{ch} = 10$ –15 T and saturating at around 18–20 T (depending on the hole density p) (8). Recent X-ray diffraction experiments (9–11) show that the transition to a 3D CDW with long-range order (LRO) onsets near $H = 15$ T.

The field-induced CDW state raises several intriguing questions regarding its relation to superconductivity. Does CDW formation with LRO suppress the superconducting condensate? Where is the true upper critical field H_{c2} ? How does it affect the stability of the vortex solid? Although NMR and X-ray diffraction are incisive probes of charge modulation and CDW formation, they do not couple directly to the fundamental excitations of the pair condensate (quasi-particles and vortices), so they are less sensitive to the pairing correlations which reflect superconductivity. By contrast, the diamagnetic magnetization \mathbf{M}_d (which dominates the observed magnetization \mathbf{M}_{obs} below the critical temperature T_c)

couples directly to the vortex excitations because it measures the current–current correlation. In the magnetic phase diagram, hysteretic behavior of the \mathbf{M}_d curves readily identifies the stability region of the vortex solid. Above the melting field of the solid H_m , \mathbf{M}_d also identifies the vortex liquid which displays a unique reversible diamagnetism. Surprisingly, we find that the magnetic susceptibility χ_d also detects the onset of charge ordering as a weak peak at the field H_K . Both H_K and a higher cross-over field H_p are apparent in the thermal conductivity. We compare H_K and H_p with field scales reported from NMR and ultrasonic experiments. Taken together, these features fill out the magnetic phase diagram, and relate the charge-ordering fields to the phase boundaries in the vortex system.

We have measured by high-resolution torque magnetometry the magnetization and thermal conductivity κ_{xx} in high-purity, detwinned ortho-II crystals of $\text{YBa}_2\text{Cu}_3\text{O}_y$ ($y = 6.55$, $T_c = 61$ K, $p = 0.11$) in a dc field H up to 45 T. Sample 1 has a large volume to facilitate accurate measurements at T up to 210 K. Sample 2 and sample 3 were picked to have smaller volumes (0.184 mm³ and 0.344 mm³, respectively) to avoid overloading the cantilever in the vortex solid regime below T_c (see ref. 4 for details on the crystals). Samples 4 and 5 were used for the κ_{xx} measurements. From

Significance

We obtain the magnetic phase diagram in the underdoped cuprate $\text{YBa}_2\text{Cu}_3\text{O}_y$ using torque magnetometry at temperatures 0.3–70 K and magnetic fields up to 45 T. At low fields, vortices (quantized flux tubes) form a vortex solid that is strongly pinned to the lattice. At large fields, melting of the solid to a vortex liquid produces nonzero dissipation. However, the vortex liquid persists to fields above 41 T. We have also mapped out the “transition” fields at which the charge-density-wave state (observed in X-ray diffraction experiments) becomes stable. Our results show that, in intense fields, superconductivity adjusts to coexist with the charge-density wave, but the Cooper pairs, which define the superconducting fluid, survive to fields well above 41 T.

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¹To whom correspondence should be addressed. Email: npo@princeton.edu.

²Present address: Department of Physics, Gonzaga University, Spokane, WA 99258.

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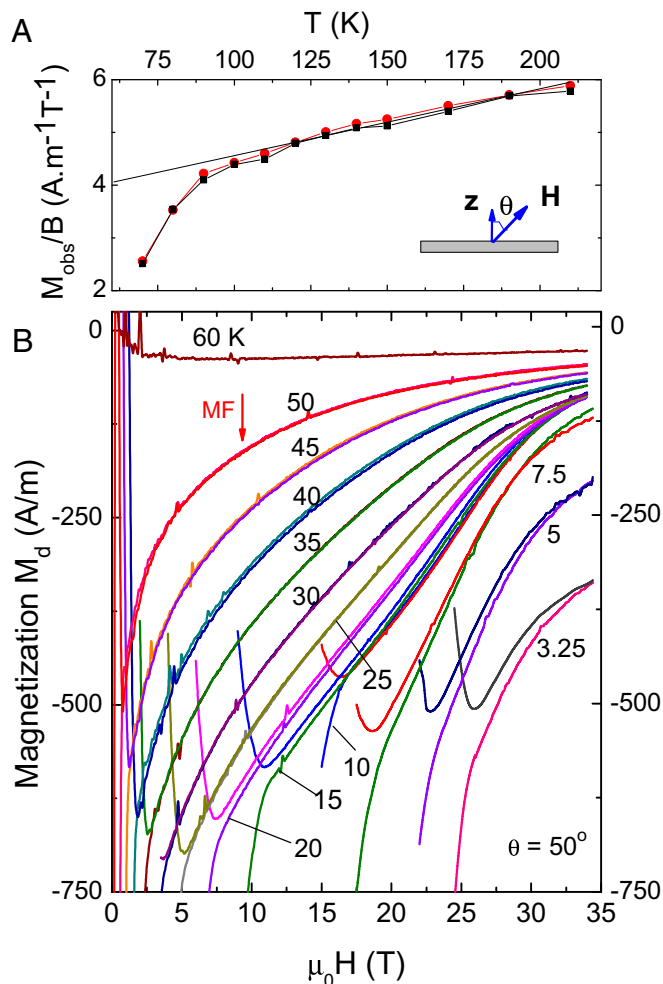


Fig. 1. Magnetization in ortho-II $\text{YBa}_2\text{Cu}_3\text{O}_y$ ($y = 6.55$, $T_c = 61$ K, $P = 0.11$) measured by torque magnetometry in dc field. (A) Observed susceptibility M_{obs}/B from $T = 80\text{--}210$ K measured at 8 T (black circles) and 11 T (red) in sample 1 ($B = \mu_0 H$, with μ_0 the permeability). The straight line is the Van Vleck anisotropy background $\Delta\chi^v = a(T + T_0)$ with $a = 1.18 \times 10^{-2}$ A(mTK) $^{-1}$ and $T_0 = 305$ K. The diamagnetic term M_d onsets near 110 K. (Inset) Tilt angle θ between H and the normal $\hat{z} \parallel \hat{c}$. (B) $M_d(T, H) = M_{\text{obs}}(T, H) - a(T + T_0)H$ vs. the magnetic field $\mu_0 H$ measured at selected T in sample 2 with $\theta = 50^\circ$. Splitting of the sweep-up from the sweep-down curves occurs at the melting field $H_m(T)$. At each T , the sample is in the vortex liquid state for $H > H_m(T)$. Diamagnetism persists to fields significantly above 32 T. For the curve at 50 K, the red arrow (labeled “MF”) marks the $H_{c2, \text{MF}}/\cos \theta$ using the mean-field value suggested for $H_{c2}(T)$.

120 to 210 K (Fig. 1A), the torque magnetization M_{obs} arises predominantly from the anisotropy $\Delta\chi^v = \chi_c^v - \chi_a^v$ of the paramagnetic Van Vleck term (χ_i^v is the susceptibility along axis i) (12, 13). $\Delta\chi^v(T)$ has a weak T dependence $\Delta\chi^v = a(T + T_0)$, with $T_0 = 305$ K (SI Appendix, section S1). At 110 K, we start to resolve a negative contribution caused by fluctuating diamagnetism. As $T \rightarrow T_c$, the diamagnetic component M_d grows rapidly in magnitude. In Sample 2, we focused on measurements of M_{obs} to 34 T at selected T between 10 and 80 K. In nonsuperconducting $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ ($x=0.050$), the Van Vleck term retains the form $a(T + T_0)$ down to 10 K (12). Hence, to isolate M_d we subtract this term from M_{obs} , i.e., $M_d(T, H) = M_{obs}(T, H) - a(T + T_0)H$.

Diamagnetism in the Vortex Liquid State

Fig. 1B displays the curves of M_d vs. H measured at temperatures from 60 K down to 3.25 K in fields up to 34 T with tilt angle $\theta = 50^\circ$. At all $T < 60$ K, M_d is negative (diamagnetic) but increases rapidly

with H . Throughout most of the field scale shown, the curves are reversible (sweep-up and sweep-down traces coincide). The reversible, strongly T -dependent diamagnetic response is the hallmark of the vortex liquid. Throughout the vortex liquid regime, $|M_d|$ is far too large to be attributed to Gaussian fluctuations (100 A/m corresponds to 1.8×10^{-3} Bohr magneton per unit cell). The diamagnetic signal reflects the array of small, diamagnetic supercurrent loops occupying the interstitial space between vortex cores. At each T , bifurcation of the sweep-up and -down curves occurs at the melting field $H_m(T)$ which separates the vortex solid from the liquid (at the scale shown, this first becomes apparent at 45 K at ~ 1.5 T). As T decreases, $H_m(T)$ rises rapidly, reaching 25 T at 3.25 K. (As sketched in Fig. 1A, \mathbf{H} is aligned at an angle θ to the c axis of the crystal. We define $\mathbf{H} \cdot \hat{\mathbf{c}} = H_z$.)

Peak in the Differential Susceptibility

An important feature of the M_d – H curves is the inflection (or kink) near $H = 25$ T for T below 20 K. The inflection is observed as a weak maximum in the differential susceptibility $\chi_d = dM_d/dB$, with B the flux density. Fig. 2 shows the curves of χ_d vs. H in measurements taken up to 45 T (sample 3) at selected values of the field tilt angle θ from 9° to 65° with T fixed at 15 K. The peaks are observed in the reversible part of the vortex phase diagram (solid and dashed curves were recorded in the field sweep-up and -down directions, respectively). We refer to the maxima in χ_d at each θ as the field $H_{K,\theta}$ (identified by vertical arrows in Figs. 2, 3B, and 4). (In the curves of χ_d vs. H plotted in Fig. 3B, the broad peak sits on a gentle linear background. The background does not affect

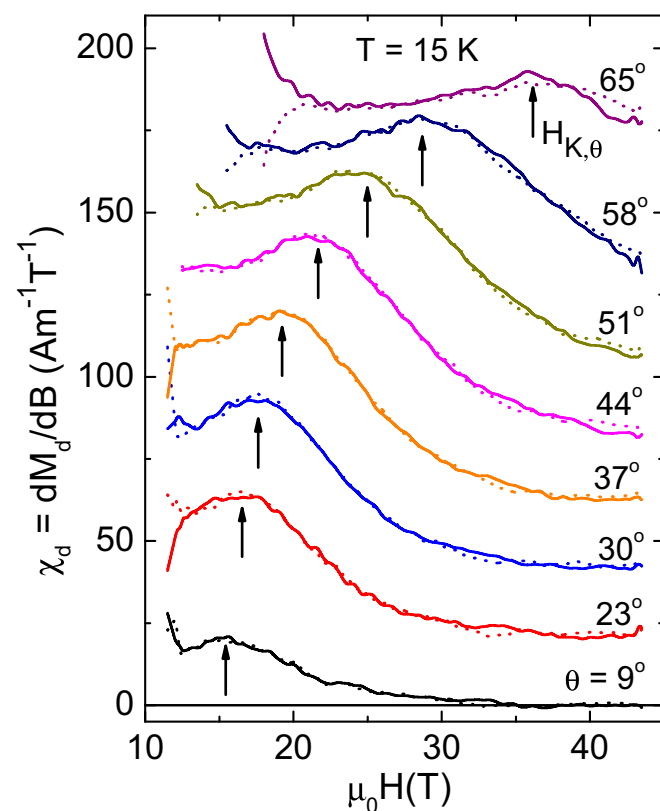


Fig. 2. Differential magnetic susceptibility $\chi_d \equiv dM_d/dB$ of YBCO (sample 3) is plotted against the magnetic field H at selected tilt angles θ at $T = 15$ K. The curves have been shifted vertically for clarity. At each θ , the solid (dashed) curve was recorded while the field H was slowly swept up (down). The bifurcation of the curves at low fields defines the melting field H_m of the vortex solid to the liquid state. In the liquid state, the χ_d - H curves display a broad peak which defines the field $H_{K\theta}$ (arrows).

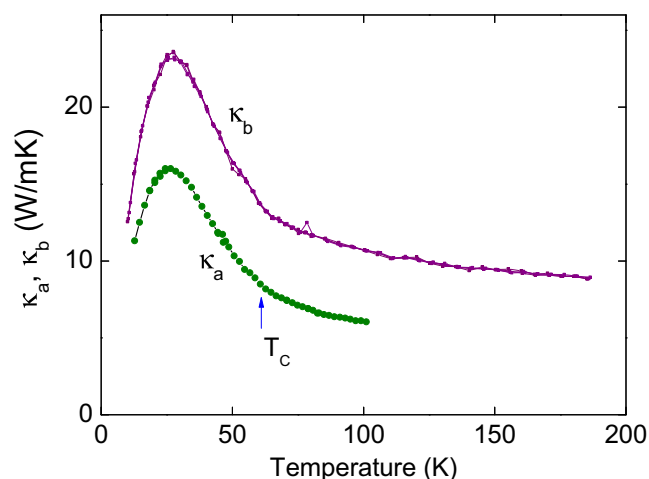


Fig. 5. The thermal conductivities κ_a (measured in sample 5) and κ_b (sample 4) in zero H .

CX1030 bare chip) were heat sunk to the crystal (using epoxy) via thick gold wires. Four thin phosphor-bronze wires were attached to each thermometer for a four-point resistance measurement. This configuration ensures good thermal contact and fast equilibration between the thermometers and the sample. The thermometers were carefully calibrated in situ, both in zero H and large H . The low-temperature setup including two reference thermometers on the sample stage was contained in a custom-made vacuum can inside the variable-temperature insert in a 35-T resistive magnet (*SI Appendix, section S4*).

The curves of κ_a and κ_b vs. H , plotted in expanded scale in Fig. 6, reveal two distinctive features (breaks in slope) that are nearly T -independent below 40 K (arrows). The feature at lower H may be identified with H_K because it occurs at the same field value to our resolution. The upper feature is called H_p . As T is raised, the features at H_K and H_p are thermally broadened, becoming unresolvable above 35 K. The step changes in κ_a and κ_b in the field interval (H_K , H_p) are quite small, accounting for <5% of the electronic thermal conductivity (*SI Appendix, Fig. S6*).

Magnetization to 45 T and de Haas–van Alphen Oscillations

The foregoing experiments, carried out to maximum fields of 35 T, pointed to intriguing features in the magnetic phase diagram which become better resolved at lower T and higher magnetic fields. We have extended the torque experiments to 45 T at temperatures down to 0.3 K and uncovered a distinct phase of the vortex system (vortex solid 2) that survives to 41 T. In addition, we have resolved de Haas–van Alphen (dHvA) oscillations (14) which onset in the vortex solid 2 phase [with amplitude and Dingle temperature closely similar to those in a previous dHvA experiment (15–17)]. We show that the vortex liquid appearing above the melting field is robust to $H_z = 41$ T (and well beyond judging from the trend vs. H).

Fig. 7 shows traces of the torque τ vs. H in the field region where the hysteretic loops begin to close at $T = 0.5, 1.8$, and 5 K. A number of important conclusions may be drawn from Fig. 7. First, the rapid closing of the hysteresis loop suggests that we can define a “lower” melting field H_{m1} (the nexus of the linear extrapolations of the upper and lower branches of τ). Above H_{m1} , there exists a second vortex solid phase (“vortex solid 2,” in the shaded region) that has a much weaker shear modulus but survives to a higher melting field H_{m2} . As we cool from 5 to 0.5 K, the small shaded region expands rapidly ($H_{m1} = 28$ T and $H_{m2} \sim 42$ T at 0.5 K). The rapid growth suggests that the vortex solid 2

constitutes a distinctive, stable phase of the overall vortex solid in the limit $T \rightarrow 0$ (instead of a fluctuation tail).

Secondly, when the curves are nonhysteretic ($H > H_{m2}$), the magnetic response is strongly diamagnetic at all fields up to 45 T ($\tau < 0$). From the trends of the curves, the diamagnetic response extends to fields considerably above 45 T. Because reversible diamagnetism is a hallmark of the vortex liquid, we conclude that the vortex liquid and the underlying pair amplitude are stable to extremely high magnetic fields, well beyond either melting field. These observations confirm the conclusion from previous Nernst and torque experiments that the pairing strength in cuprates is extremely robust.

Finally, dHvA oscillations can be resolved even in the curves at 5 K (*SI Appendix, section S3, Fig. S4*). The onset of the oscillations at 0.5 K occurs within the shaded region. From the discussion above, we conclude that the pairing amplitude is nonzero when the oscillations onset. Above H_{m2} , the oscillations exist in the vortex liquid state where the magnetization is manifestly diamagnetic. The coexistence of the dHvA oscillations with diamagnetic response was previously observed in ref. 15.

Magnetic Phase Diagram and Discussion

The combined experiments uncover a rather rich magnetic phase diagram that allows us to relate the charge-ordering field scales obtained by NMR and X-ray diffraction to the vortex states in underdoped YBCO. In Fig. 8 the melting field H_m , which defines the stability region of the vortex solid, increases monotonically as

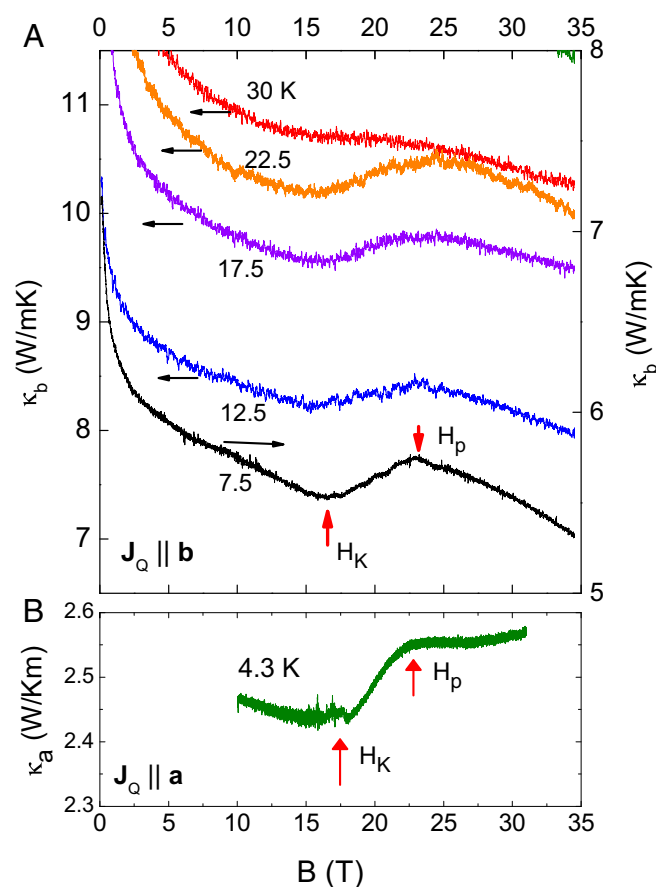


Fig. 6. Expanded views of curves of the thermal conductivity at selected T . (A) Traces of κ_b measured in sample 4 measured in a field $H \parallel c$ with the heat current density $J_0 \parallel b$ (chain axis). The vertical scale for 7.5 K is on the right axes. At the fields H_K and H_p (arrows), κ_b exhibits a distinct changes of slope. (B) κ_a measured at 4.3 K in sample 5 with $H \parallel c$ and $J_0 \parallel a$.

correlations survives well above 23 T. A similar inference based on the X-ray peak intensity at 28 T was reported by Gerber et al. (9).

The different experimental probes provide a picture that is richer and more interesting than the identification (7) of a mean-field Bardeen–Cooper–Schrieffer-type transition at which the pair condensate simply vanishes (at 23 T). In a *d*-wave superconductor, the electronic thermal conductivity κ^e (we suppress the subscript *xx*) is overwhelmingly dominated by the quasi-particle (qp) population at the nodes (at $T \ll T_c$). Hence changes to κ^e predominantly reflect changes to the Fermi surface (FS) at or close to the node (example, closing of the gap along an FS arc or creation of a small pocket). Moreover, the curves in Fig. 6 show that raising *T* above 25 K suppresses all traces of the step change in κ_{xx} (5% at 4.3 K). Thermal broadening of the broad peak at H_K in χ_d is equally rapid (Fig. 3). This provides direct evidence that the FS changes at the node involve energy scales of ~ 1 –2 meV, far smaller than the gap amplitude Δ_0 at the antinodes. In Fig. 8, the convergence of the lower melting curve H_{m1} (which separates the two vortex solids) and the cross-over curve H_p to values close to 23 T as $T \rightarrow 0$ suggests to us that, as the 3D CDW amplitude grows, the pair condensate undergoes an abrupt transition at 23–24 T to accommodate the competing CDW (this strongly affects the vortex solid shear modulus as discussed below). We propose that the peak in κ^e reflects changes (at energy scales of 1–2 meV) to the nodal states caused by the CDW transition, rather than suppression of the gap amplitude Δ_0 (40–80 meV).

In ref. 7, a mean-field H_{c2} curve extending from 23 T at 2 K to zero at T_c is drawn without reference to experiment. The magnetization curves in Fig. 1 are incompatible with such a mean-field curve (M_d varies smoothly through the proposed H_{c2} curve; see arrow labeled “MF”). (We extended measurements of both κ_{xx} and the thermal Hall conductivity κ_{xy} to lower *T*. For the doping level in our crystal, we observe at 0.5 K a sharp cusp in κ_{xy} at 18 T, followed by the onset of a large negative thermal Hall contribution. The critical field 18 T is lower than 23 T, but

consistent... with the dashed line linking solid triangles in Fig. 8. As 18 T lies below H_{m1} , the system is well within the vortex solid 1 phase when the transition affecting the nodal qp states occurs.)

An interesting issue raised by the phase diagram is the fate of both the vortex solid and liquid in the quantum limit $T \rightarrow 0$ (21). As remarked above (Fig. 7), the rapid expansion of the vortex solid 2 phase (between H_{m1} and H_{m2}) suggests that the pair condensate may modify its pairing pattern to accommodate the static CDW. Several groups have proposed (22–27) either the pair-density-wave formation or the related Fulde–Ferrell–Larkin–Ovchinnikov state (28). It is intriguing that when $T \rightarrow 0$, the transition at $H_{m1} \sim 24$ T also occurs close to the field H_p . The present experiments show that this allows the vortex solid to survive to much higher *H* at the cost of a substantial decrease in its shear modulus.

The question whether the vortex liquid exists as a stable quantum phase at $T=0$ is resolvable by extending the torque measurements below 0.1 K to fields above 41 T. The properties of the quantum vortex liquid (in an ultraclean superconductor) are largely unknown but may become accessible to torque magnetometry at mK temperatures in the near future. The existence of the vortex liquid to fields substantially higher than 41 T implies that the Cooper pair amplitude in underdoped YBCO is too strong to be suppressed at these field scales.

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