# Vortex Lattice Melting in Untwinned and Twinned Single Crystals of $YBa_2Cu_3O_{7-\delta}$

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(Received 1 October 1992)

The melting transition in twinned and untwinned single crystals is measured resistively in fields up to 8 T as a function of the angle between the **c** axis and the **a-b** plane. The angular dependence follows the Lindemann criterion with $c_L=0.15$. The suppression of melting by strong pinning by twin boundaries is demonstrated.

PACS numbers: 74.60.Ge

The magnetic phase diagram of the high-$T_c$ superconductor $YBa_2Cu_3O_{7-\delta}$ has led to much debate over the variety of novel vortex states which have been proposed to exist [1]. It is proposed that at high fields and temperatures near $T_c$ where thermal fluctuations dominate, the usual sharp second-order phase transition at the upper critical field $H_{c2}$ is absent, replaced by a smooth crossover to the superconducting state. Below the mean-field $H_{c2}$ line, a vortex-liquid state persists down to an irreversibility line associated with a second-order transition to a vortex glass [2] or Bose glass state [3] or to a vortex melting line associated with a first-order transition into an Abrikosov vortex lattice [4–7], depending on the type and strength of the disorder. In highly disordered systems involving point defects, the vortex liquid is predicted to undergo a phase transition into a vortex glass state [2]. In systems with correlated defects like ion-induced columnar defects, or twin boundaries (TB), a transition of the vortex liquid into the Bose glass state is thought to occur [3]. However, in very clean systems, a first-order transition from a vortex liquid to an Abrikosov vortex lattice is predicted [5–7]. Recently, with picovolt sensitivity, Safar *et al.* [8] observed a hysteretic first-order phase transition interpreted as flux line lattice (FLL) melting. The phase diagram they obtained agreed well with the FLL melting curve obtained from previous equilibrium torque measurements by Farrell *et al.* [9].

In this Letter we present the detailed angular dependence of the "kink" feature observed in the magnetic-field-broadened resistive transition, which we associate with the melting temperature $T_m$, in high-quality untwinned and twinned crystals. We show that the angular dependence of $T_m(\theta)$ follows the effective-mass-dependent anisotropic scaling rule [10,11] expected for melting. In addition, we show for the first time a strong interaction between melting and pinning. By precise rotation of the magnetic-field direction away from the TB plane, we effectively adjust the pinning strength, and systematically suppress or reveal the melting transition.

The samples used in our study were grown by the self-flux method described elsewhere [12]. The untwinned crystal was produced by applying uniaxial pressure along one edge of the **a-b** plane [13]. Two twinned crystals were cleaved such that they contained only one ⟨110⟩ direction of TB's. For these crystals, the current contacts were oriented in the **a-b** plane either parallel (crystal $Y0$) or at 45° (crystal $Y45$) with respect to the TB's. A third crystal with both twin orientations ⟨110⟩ and $\langle 1\bar{1}0\rangle$ was also included in our study. Crystal dimensions are on the order of $1\times0.5\times0.05$ $mm^3$. ac resistivity was measured by the standard four-probe method with a measuring current density of $\sim1$ $A/cm^2$ at 17 Hz. Contacts were made to the crystals by first applying four strips of silver epoxy and sintering at 420 °C for 6 h. Gold wires were then attached to the silver strips with silver epoxy and cured at 120 °C for 1 h, resulting in contact resistance of $\sim1$ Ω. The high quality of the crystals is confirmed by their zero-field resistive transition of $R_{zero}>92.0$ K and $\Delta T_c(10\%–90\%)<200$ mK in all crystals. For magnetic-field measurements, the sample was placed in the bore of two superconducting magnets, a 1.5-T split coil transverse magnet which resides in the bore of an 8-T longitudinal solenoid magnet. The temperature dependence of the resistance $R(T)$ in magnetic fields up to 8 T was obtained by mounting the crystal with either the **c** axis or **a-b** plane parallel with the longitudinal magnetic field. In the latter geometry, the angular dependence of $R(T)$ for magnetic-field directions between the **c** axis and the **a-b** plane of the crystal may be obtained by rotating the sample about the longitudinal axis in the presence of a 1.5-T transverse field. Furthermore, the sample alignment with respect to the magnetic field can be checked to extremely high precision by energizing both magnets simultaneously and tilting the resultant magnetic field at small angles with respect to the crystallographic axes. This is important for accurate field alignments along the TB's where a depinning angle for TB pinning has been observed [14].

Figure 1 shows the temperature dependence of the resistance for the detwinned crystal in magnetic fields up

 

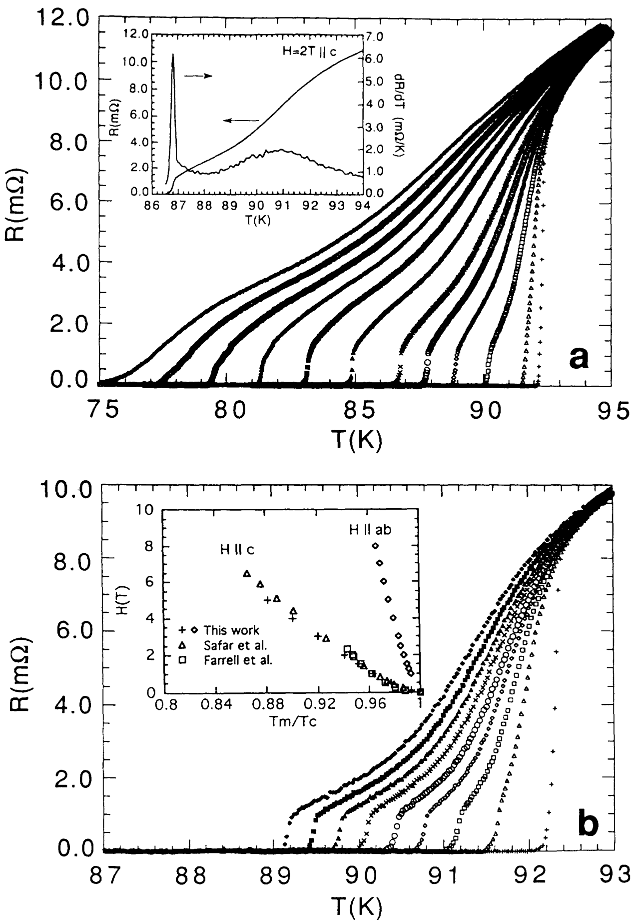


FIG. 1. (a) Resistive transition in magnetic fields of 0, 0.1, 0.5, 1, 1.5, 2, 3, 4, 5, 6, 7, and 8 T for **H**||**c** in an untwinned $YBa_2Cu_3O_{7-\delta}$ crystal. Inset: Determination of $T_m$ from the inflection peak of $dR/dT$ for $H=2$ T. (b) Resistive transition in magnetic fields of 0, 1, 2, 3, 4, 5, 6, 7, and 8 T for **H**||(**a**,**b**). Inset: Phase diagram of the melting transition for **H**||**c** and $H$||(**a**,**b**).

to 8 T for **H**||**c** and **H**||(**a**,**b**). The zero-field transition in this crystal is extremely sharp, with $T_c=92.33$ K and $\Delta T_c(10\%\text{-}90\%)\leq 100$ mK. The sharp drop in resistivity or "kink" observed near $\rho/\rho_n \sim 10\%$ for **H**||**c** has been attributed to a first-order melting transition of the vortex lattice [8]. We find linear resistivity in the entire region above this kink and *non-Ohmic* behavior below it. At higher fields, $H>5$ T, this kink is gradually replaced with a smooth resistive tail. The absence of a distinct kink for $H>5$ T may be attributed to enhanced pinning at high fields due to collective pinning effects [15]. In less disordered untwinned crystals, we see the sharp kink up to $H>8$ T. For $H<5$ T, the resistive height of the kink remains virtually constant at $\rho/\rho_n \sim 10\%$. This kink is distinct from the smooth shoulder observed in twinned crystals near $\rho/\rho_n \sim 20\%$ which represents the onset of TB pinning [14]. A similar kink in the resistivity, also occurring at the same resistance $\rho/\rho_n \sim 10\%$ but at higher temperatures, is observed for **H**||(**a**,**b**)⊥**J** as shown in Fig. 1(b). For this orientation, the kink remains sharp up to the highest applied field of $H=8$ T.

We associate the kink with FLL melting, and in order to quantify melting, we identify $T_m(H)$ as the peak in the derivative of the resistive transition as shown in the inset of Fig. 1(a) for a field of 2 T. This inflection point describes the temperature at which the slope in the resistive transition is a maximum. The width of the peak visually demonstrates the sharpness of the melting transition which occurs in a temperature range of $\sim 100$ mK at 2 T, comparable to the width of the superconducting transition in zero field. The magnetic phase diagram obtained in this fashion for **H**||**c** and $H<5$ T is shown in the inset to Fig. 1(b) and agrees quite well with previous measurements by Safar *et al.* [8] and Farrell *et al.* [9] on untwinned crystals. We obtain a fit of $H(T)=103(1-T/T_c)^{1.41}$ and $H(T)=842(1-T/T_c)^{1.37}$ for **H**||**c** and **H**||(**a**,**b**), respectively, using a $T_c$ value of 92.33 K. A fit to Safar's published results [8] gives $H(T)\sim 107(1-T/T_c)^{1.39}$ with $T_c=92.3$ K. Farrell *et al.* [9] report a value of $H(T)=0.774(1-T/T_c)^2$ with $T_c=90.2$ K. Near $T_c$, and at low fields, theory [7,16,17] predicts an exponent $n\sim 2$ for the melting transition. However, at high fields, this exponent is expected to become closer to unity [7] over a wide range of temperatures, a trend we observe in our high-field data.

We expect the melting transition to obey the Lindemann criterion for FLL melting which is given by the following expression [7]:

$$c_L^4 \sim \frac{[B_m/H_{c2}(T_m)]}{(1-T_m/T_c)}\frac{G_i\gamma^2}{5.6}, \tag{1}$$

where $B_m$ is the melting field, $H_{c2}$ is the upper critical field, $\gamma^2=59$ (Ref. [18]) is the mass anisotropy ($m_c/m_{ab}$), and $G_i$ is the Ginzburg parameter $16\pi^3\kappa^4(k_BT_c)^2/\Phi_0^3H_{c2}(0)\sim 5\times 10^{-5}$ for $YBa_2Cu_3O_{7-\delta}$ with $\kappa=55$. Using our experimental values of $B_m=2$ T, $T_m=86.82$ K, $T_c=92.33$ K, and $H_{c2}^c=9.9$ T (using $dH_{c2}^c/dT\sim -1.8$ T/K), we obtain a physically reasonable value of $c_L \sim 0.2$.

The Lindemann melting criterion can be obtained also from the angular dependence of the melting temperature $T_m(\theta)$. According to the scaling rules for anisotropic superconductors [10,11], $T_m(\theta)$ shows the following angular dependence:

$$k_BT_m=\frac{\Phi_0^{5/2}c_L^2}{4\pi^2\lambda^2(T_m)B^{1/2}\varepsilon^{1/4}(\theta)}, \tag{2}$$

where $\Phi_0$ is the flux quantum, $\varepsilon=\sin^2(\theta)+\gamma^2\cos^2(\theta)$, and $\lambda(T)$ is the temperature-dependent penetration depth which we estimate using the two-fluid model.

Figure 2(a) shows the kink in $R(T)$ in a field of 1.5 T for a series of field directions between the **c** axis and the (**a**,**b**) plane and normal to the current. Figure 2(b) shows $T_m(\theta)$. Using values of $\gamma=7.7$ (Ref. [18]) and $\lambda_{ab}(0)=1400$ Å for $YBa_2Cu_3O_{7-\delta}$, the best fit is obtained with $T_c=92.7$ K, leading to a Lindemann criterion

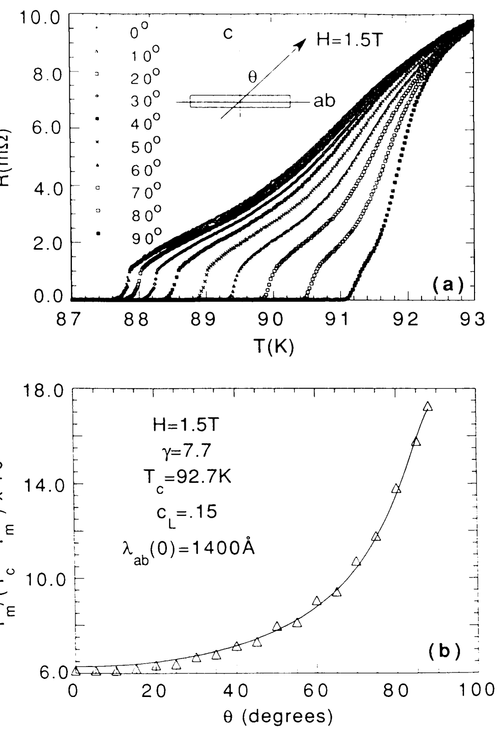


FIG. 2. (a) Resistance vs temperature in a magnetic field of 1.5 T at various field orientations from H‖c to H‖(a,b) of an untwinned crystal. (b) Angular dependence of the vortex lattice melting temperature $T_m(\theta)$ of an untwinned crystal.

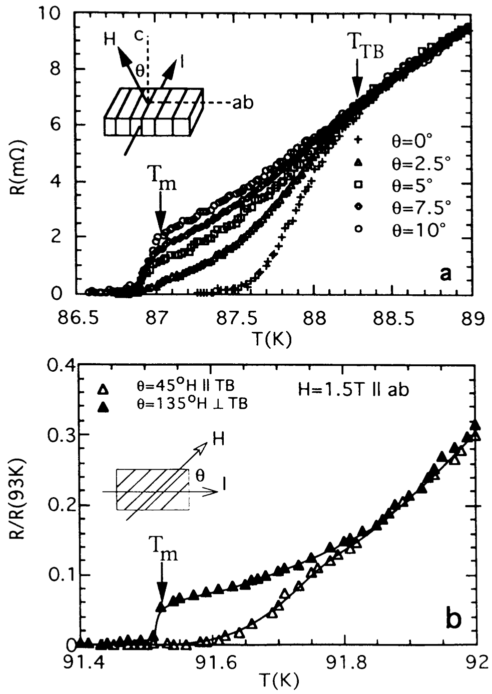


FIG. 3. (a) Resistance vs temperature for crystal $Y0$ for $H=2$ T rotated about the TB plane in the c axis. (b) Resistance vs temperature for crystal $Y45$ for $H=1.5$ T rotated through the TB plane in the (a,b) plane. The lines are a guide to the eye.

parameter of $c_L=0.15$. This value is in excellent agreement with the value of 0.16 determined from low-frequency torsional-oscillator measurements [11] and in good agreement with the value obtained using the field dependence of $T_m$ for **H**‖**c**.

In twinned crystals, no melting transition has been identified for **H**‖**c**. Instead, we have previously shown [14] that a smooth shoulder displaying *Ohmic* behavior appears at $\rho/\rho_n \sim 20\%$ signifying the onset of TB pinning for **H**‖**c**, and a sharp kink displaying *non-Ohmic* behavior appears for **H**‖(**a**,**b**) [19]. We now report that a small misalignment of the field from the **c** axis reveals a sharp kink at the expected $\rho/\rho_n \sim 10\%$, and that the same interference of TB pinning with melting occurs for field directions aligned and misaligned with TB's in the **a**-**b** plane. The intentional misalignment of the field with the TB provides a precise, continuous adjustment of the TB pinning strength, and allows the interplay of melting and pinning to be clearly observed.

Figure 3(a) shows the temperature dependence near the tail region of the resistive transition in a magnetic field of 2 T for several small angle misalignments of the field and the **c** axis for crystal $Y0$. For **H**‖**c**, the top arrow points to the smooth shoulder in $R(T)$, represented by $T_{\rm TB}$ and characterized by *Ohmic* behavior. In this configuration, the flux lines are pinned by the TB planes and zero resistance is reached at ~87.5 K. With a misalignment angle of 2.5°, a substantial finite resistance appears at this temperature because TB pinning is weaker. Upon further misalignment, a small kink characterized by *non-Ohmic* behavior appears at the foot of the resistive transition, the shoulder virtually disappears, and the lower resistive kink develops to a value of approximately $\rho/\rho_n \sim 10\%$. We associate this kink with $T_m$. Analogous behavior is seen in Fig. 3(b) for crystal $Y45$, for which the magnetic field is oriented parallel to the **a**-**b** plane of the crystal. When the magnetic field is oriented parallel to the TB's ($\theta=45°$), the kink is suppressed. This can be compared with the curve for $\theta=135°$ where the magnitude of the Lorentz force is identical to $\theta=45°$, but the direction of the magnetic field is now perpendicular to the twin planes. For this situation, TB pinning is substantially decreased, and a small kink associated with vortex melting is observed at low temperatures. This is in agreement with our expectation that the confinement of a substantial fraction of vortices by TB's should inhibit melting of the vortex lattice. These measurements clearly demonstrate the effect of TB's in suppressing the FLL melting transition.

Figure 4(a) shows the temperature dependence of the resistive transition in a magnetic field of 1.5 T at several angles between the **c** axis and the **a**-**b** plane of a twinned crystal with both twin orientations ⟨110⟩ and ⟨1$\bar{1}$0⟩. The inset to Fig. 4(a) shows the resistive shoulder characteris-

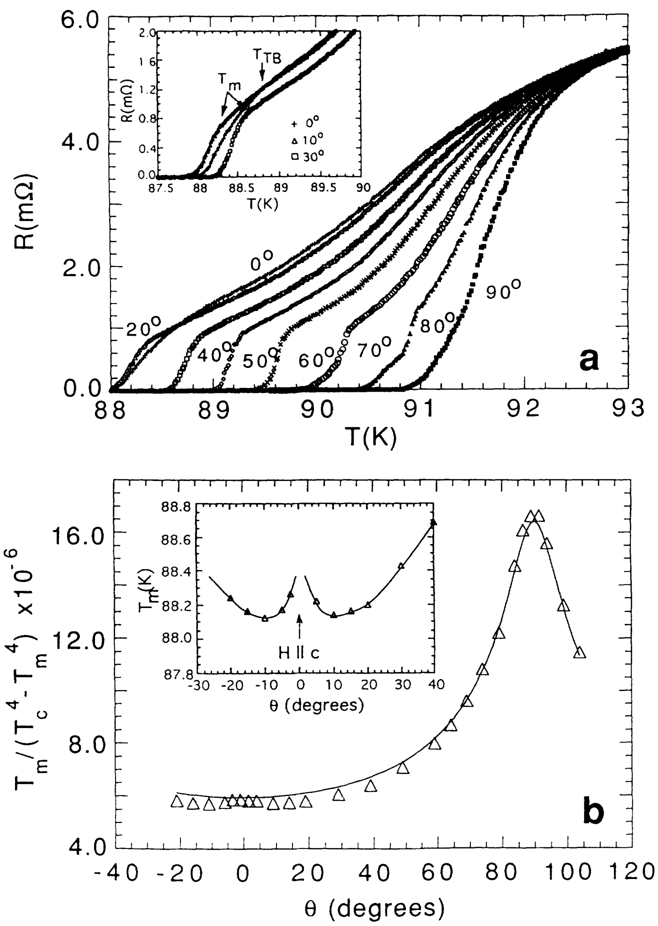


FIG. 4. (a) Resistance vs temperature in a magnetic field of 1.5 T at various field orientations from **H**||**c** to **H**||(**a**,**b**) of a twinned crystal. Inset: The region near **H**||**c**. (b) Angular dependence of the vortex lattice melting temperature $T_m(\theta)$ of a twinned crystal. Inset: Plot of $T_m$ vs angle displaying a cusp near **H**||**c**. The line is a guide to the eye.

tic of $T_{TB}$ for **H**||**c** and the resistive kink characteristic of $T_m$ for field rotated with respect to the **c** axis by 10° and 30°. For this crystal the zero-resistance temperature initially decreases, as shown in the resistance curve with a 10° field misalignment from the **c** axis. Eventually, as the magnetic field is rotated beyond 20° from the **c** axis, the angular range over which substantial TB pinning occurs, the superconducting anisotropy dominates the angular dependence of the resistivity and shifts the zero-resistance point to higher temperatures. This TB pinning behavior leads to a shallow cusp in $T_m(\theta)$ near **H**||**c** [see inset to Fig. 4(b)]. Such behavior may be a vestige of the Bose glass transition due to correlated disorder from TB's whose correlated volume diverges with two anisotropic correlation lengths [3]. Using values of $\gamma = 7.7$ and $\lambda_{ab}(0) = 1400$ Å as before, we obtain the best fit with a slightly higher $T_c = 93.3$ K and a Lindemann criterion of $c_L = 0.15$, in good agreement with our results obtained for the untwinned crystal. Similar angular-dependent measurements at $H = 6$ T lead to a larger deviation of the fit, i.e., a larger fitting $T_c$, and may be related to the larger deviation from the low-field value of $n = 2$, implicit in the scaling of $T_m(\theta)$.

In summary, we have shown for the first time that the sharp "kink" observed in the magnetic-field-broadened resistive transition in a high-quality untwinned crystal obeys the angular dependence expected from the Lindemann criterion of FLL melting. We obtain physically reasonable values of $c_L = 0.15$ consistent with torque measurements. In addition, we have shown directly that the melting transition occurs in twinned crystals for the off-c-axis magnetic-field geometry, and that confinement of the vortices by TB's inhibits the melting transition. For **H**||**c**, the TB's act as regions of correlated disorder which may lead to a Bose glass transition as seen by the cusp in $T_m(\theta)$. The Lindemann criterion parameter obtained for the twinned crystal is in good agreement with the value obtained for the untwinned crystal. Angular-dependent transport measurements near **H**||**c** in high-quality twinned crystals serve as a convenient method to vary the strength of pinning experienced by the vortices, and as a means to study the interplay among the vortex glass transition, the Bose glass transition, and the FLL melting transition.

This work was supported by the U.S. Department of Energy, BES-Materials Science under Contract No. W-31-109-ENG-38 (W.K.K., S.F., V.M.V., J.D., G.W.C.) and the NSF-Office of Science and Technology Centers under Contract No. STC8809854 (U.W.), Science and Technology Center for Superconductivity and the Office of Naval Research and DARPA (M.M.M.).

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