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**Effect of Twin Boundary Density on the Vortex Melting Transition  
in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  Single Crystals\***

W.K. Kwok, J. Fendrich,<sup>a</sup> S. Fleshler,<sup>b</sup> U. Welp, and G.W. Crabtree

Materials Science Division and Science & Technology Center for Superconductivity  
Argonne National Laboratory, Argonne, Illinois 60439

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<sup>a</sup>Also at Department of Physics & Astronomy, Iowa State University, Ames, IA 50011.

<sup>b</sup>Present address: Los Alamos National Laboratory, MS K763, Los Alamos, NM 87545.

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# EFFECT OF TWIN BOUNDARY DENSITY ON THE VORTEX MELTING TRANSITION IN $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ SINGLE CRYSTALS

W. K. KWOK, J. FENDRICH<sup>a</sup>, S. FLESHLER<sup>b</sup>, U. WELP, AND G. W. CRABTREE  
Materials Science Division and Science and Technology Center for Superconductivity,  
Argonne National Laboratory, Argonne, IL 60439, U. S. A.

## ABSTRACT

We present magnetotransport  $\rho(H,T)$  measurements in clean untwinned and twinned crystals of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  in magnetic fields up to 8 Tesla for  $H \parallel c$ . In untwinned crystals, we show strong support for a vortex solid to liquid first order melting transition at a temperature  $T_m$ , much below the mean field transition temperature  $T_c(H)$ , characterized by an extremely sharp drop in resistivity  $\rho(T_m)$  and the appearance of hysteretic behavior at  $\rho(H_m)$ . In densely twinned crystals, the sharp transition is replaced with a continuous transition at a higher temperature  $T_{BG} > T_m$ . Furthermore, the cusp in  $T_{BG}(\theta)$  predicted by the Bose glass theory at fixed field is observed. In dilutely twinned crystals we demonstrate the competing effect of pinning and freezing at the phase transition. The vortex solid state at low fields is characterized by the appearance of a 'peak effect' just below the melting temperature.

## 1. Introduction

The vortex state in high temperature superconductors continues to yield novel results and a complex phase diagram. Recently, experimental support for a first order phase transition from the vortex liquid to the solid state has been demonstrated in clean untwinned  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  single crystals.<sup>1-4</sup> An interesting question is the effect of correlated defects on the first order phase transition. We investigate this effect using twin boundaries, which naturally occur during the growth process of this material. By varying the density and spacing between twin boundaries, we demonstrate that the vortex liquid to solid first order freezing transition can be suppressed in densely twinned crystals due to enhanced pinning in the vortex liquid state, leading to a continuous second order transition at a higher temperature. In dilutely twinned crystals where the spacing of the twin boundaries is about a hundred microns, we see a vortex competition between pinning and melting which reduces the size of the feature associated with the first order transition. Furthermore, in these crystals, we see a sharp 'peak effect'<sup>5</sup> in the critical current which occurs just below the first order melting transition, giving additional support to the first order vortex liquid to solid phase transition.

## 2. Sample Preparation and Experimental Set-up

Samples of high quality single crystals of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  with superconducting transition temperatures in excess of  $T_c > 92\text{K}$ , were grown at Argonne National Laboratory using the self-flux method<sup>6</sup>. The untwinned crystal was obtained by detwinning a crystal under uniaxial pressure at 430 C according to a method described elsewhere<sup>7</sup>. The densely twinned crystal was cleaved to obtain a single dense set of twin planes parallel to the long edge of the crystal, and the current direction was oriented

parallel to the twin boundaries so that the Lorentz force was directed perpendicular to the twin planes. The dilute twin boundary crystal consisted of only two twin planes at 45 degrees to the crystal edge and separated by a distance of about 140  $\mu\text{m}$  from each other. Four silver conductive pads were painted on the samples in the standard four probe geometry and sintered at 420 C. Gold wires were attached to the pads with silver epoxy and cured for one hour at 140 C. The resulting contact resistances were less than 1  $\Omega$ . For all samples, the current was directed in the ab-plane of the crystal with the magnetic field applied parallel to the crystallographic c-axis. The measuring currents ranged from 0.3 A/cm<sup>2</sup> to 40 A/cm<sup>2</sup> with an excitation frequency of 17 Hz. Voltage-current characteristics were obtained with DC current while alternating the direction at each value to minimize thermal voltages. The samples were placed in the bore of two concentric superconducting magnets<sup>8</sup>, an 8 Tesla solenoid and a 1.5 Tesla split-coil magnet which resides in the bore of the solenoid. The angle of the applied magnetic field with respect to the sample was changed by independent control of the current through the two magnets, with a resolution of approximately 0.005°.

### 3. First Order Vortex Melting Transition

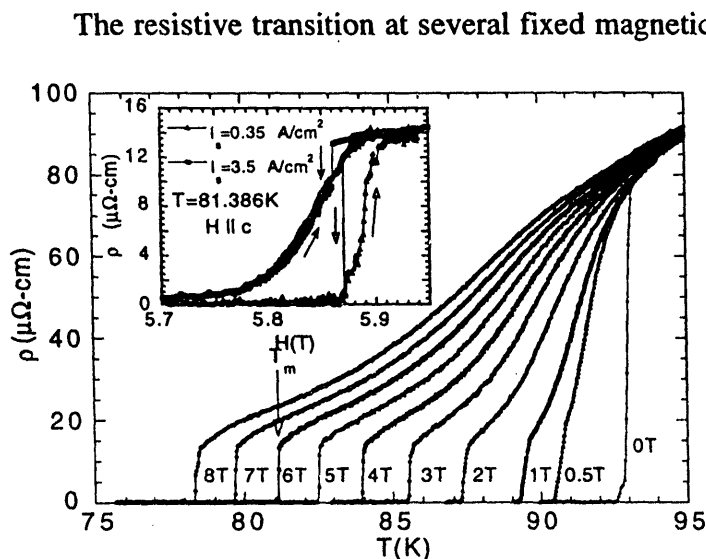


Figure 1 Resistivity versus temperature for an untwinned single crystal of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub> for H || c. (Inset) Resistivity versus field at T=81.386K for two different measuring currents.

both increasing and decreasing magnetic field sweeps. At the lower measuring current, the hysteresis is quite robust,  $\Delta H \sim 200$  Oe. For decreasing magnetic field, the transition at the freezing field  $H_m \sim 5.87$  T is extremely sharp,  $\Delta H_m < 10$  Oe, whereas for increasing magnetic field the transition is much broader. The different width of the freezing and melting transition field demonstrates the different initial pinning states of the vortices. The vortex state above  $H_m$  is characterized by weak pinning due to the high quality of the crystal and the liquid nature of the vortices. Thus upon decreasing the magnetic field, the vortex-vortex interaction dominates over the vortex-pin site interaction and the

The resistive transition at several fixed magnetic fields is shown in Fig. 1 below for a high quality untwinned single crystal of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-δ</sub>. The sharp 'kink' in the resistivity near the tail of the transition has been previously associated with a first order phase transition of the vortex liquid to the vortex solid state<sup>2-4</sup>. The temperature width of the kink' is about  $\Delta T_m(10-90\%) < 60$  mK at fields between 3 T and 7 T, much less than the zero field transition width of about  $\Delta T_c(10-90\%) < 200$  mK. Hysteresis as a function of applied magnetic field is shown in the inset of Fig. 1 for two different measuring currents of  $I_s = 0.35$  A/cm<sup>2</sup> and 3.5 A/cm<sup>2</sup> and for

vortices can arrange themselves in the lattice configuration with little interference from pinning. Nucleation of the vortex solid state at  $H_m$  occurs throughout the entire crystal within a very narrow field range. Below  $H_m$ , a finite shear modulus  $C_{66}$  appears with the vortex lattice, greatly enhancing the pinning effectiveness of each defect. Upon increasing the magnetic field from this state, the vortex lattice melts inhomogeneously at fields dictated by the local defect density. In addition, depinning of the vortex lattice may also occur concurrently, leading to the broader transition.

At higher measuring currents, the sharp freezing transition near  $H_m$  is still observable, however, the hysteresis collapses below this transition. Apparently, the depinning critical current has been exceeded for the solid, leading to a flux flow behavior of the vortex solid below  $H_m$ . The extremely narrow transition width of  $\Delta T_m \sim 60 \text{ mK}$  and  $\Delta H_m < 10 \text{ Oe}$ , compared with one part in  $10^4$  of the ambient temperature and field, and the pronounced hysteresis in  $\rho(H)$  strongly suggest a first order vortex liquid to solid phase transition.

#### 4. Densely Twinned Crystal

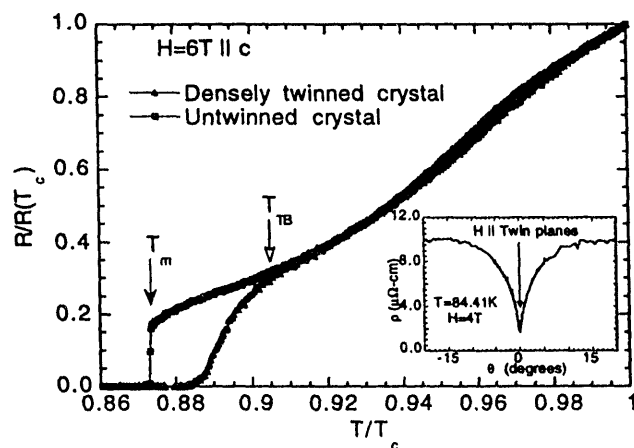


Figure 2. Comparison of the resistive transition of a densely twinned and untwinned crystal. Inset show dependence of twin boundary pinning.

observed instead of the 'kink' in the twinned crystal. Whereas the 'kink' separates an ohmic regime above  $T_m$  from non-ohmic behavior below<sup>10</sup>, the resistivity is ohmic both above and below the 'shoulder' at  $T_{TB}$  in the densely twinned sample. Thus the first order phase transition observed in untwinned crystals is absent in densely twinned samples. The 'shoulder' in twinned samples reflects the onset of twin boundary pinning in the vortex liquid state<sup>9</sup>. Since twin boundaries are planar

Figure 2 compares the resistive transition in a field of 6 Tesla of an untwinned crystal and a densely twinned crystal where the measuring current and magnetic field induce the maximum Lorentz force perpendicular to the twin planes. This geometry produces the largest vortex pinning effect with respect to the twin boundaries<sup>9</sup>. The resistivity and the temperature have been normalized to their respective values at  $T_{c0}$  to facilitate a direct comparison. The salient difference between the two curves is that a pronounced 'shoulder' is

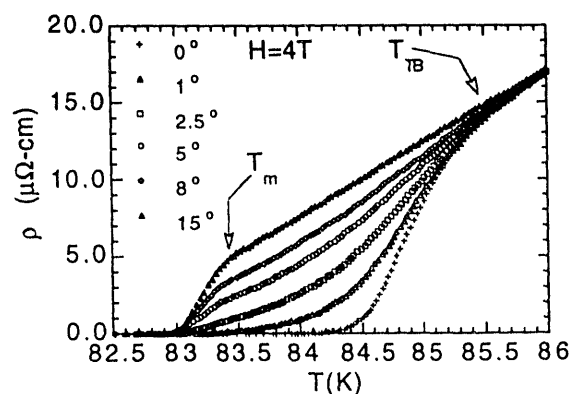


Figure 3. Resistivity versus temperature for different angles of the magnetic field from the c-axis.

objects, one expects vortex pinning to be maximized when the vortices are aligned parallel to the twin planes, and reduced when the vortices are tilted away from the twin planes.

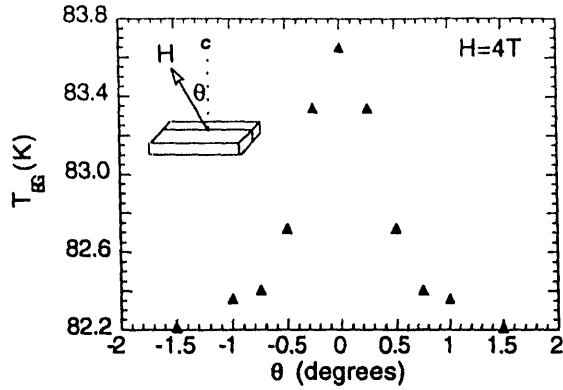


Figure 4. Angular dependence of the Bose glass transition temperature showing a cusp like behavior near  $H \parallel$  twin boundaries. Data for negative angles are a reflection of the data from the positive angles.

from the vortex liquid to a Bose glass state<sup>11</sup> at  $T_{BG}$  is predicted with resistivity scaling as  $\rho(T) \sim (T - T_{BG})^s$ , and  $s=4 \sim 6$  for columnar defects. A fit of our data near the tail of the transition for  $1T < H < 5T$  yields  $s \sim 4.87 \pm 0.8$ . Furthermore, the angular dependence of the Bose glass transition temperature  $T_{BG}(\theta)$  obtained from fits to the data of Fig. 3 yields a 'cusp' near  $\theta=0^\circ$ , as predicted for the Bose glass, due to the spatial anisotropy of the pinning defect, as shown in Fig. 4.

## 5. Dilute Twin Boundary Pinning

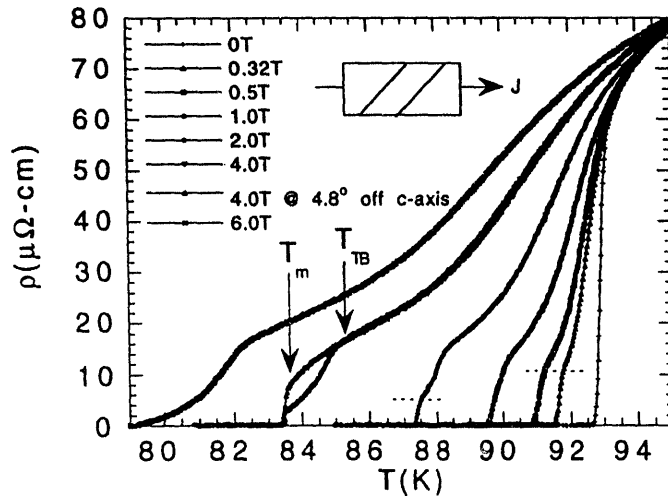


Figure 5. Resistivity versus temperature of a crystal with only two twin boundaries for  $H \parallel c$ . The horizontal dashed lines depict the decreasing height of the 'kink' with increasing magnetic field.

This is clearly demonstrated in Fig. 3, where the resistive transition near the 'shoulder' is shown for various magnetic field angles tilted away from the twin planes. With increasing tilt angle, the effect of twin boundary pinning decreases as seen by the rise in the resistivity value for the same temperature. Furthermore, a small 'kink' in the resistivity at the tail of the transition develops with decreasing twin boundary pinning. The 'kink' develops to its full constant height at the tilt angle where twin boundary pinning disappears as shown in the inset to Fig. 2. Comparison with Fig. 2 shows that the first order vortex liquid to solid phase transition is recovered.

For vortex pinning by twin boundaries, a second order phase transition at  $T_{BG}$  is predicted with resistivity scaling

Fig. 5 shows the resistive transition of a crystal with only two twin boundaries for  $H \parallel c$ . At magnetic fields between 0.5T and 6 Tesla, both the 'shoulder' depicting the onset of twin boundary pinning and the 'kink' associated with the first order vortex liquid to solid phase transition are observed. At lower fields, only the sharp 'kink' is present, and at higher fields, only the 'shoulder' is observed. With increasing field beyond 0.5 Tesla, the 'shoulder' develops and reduces the resistive height of the 'kink', showing the

competing effect of pinning in the vortex liquid state which tends to suppress the first order phase transition at  $T_m$ . For  $H=4T$ , we show that the full height of the 'kink' can be recovered by tilting the magnetic field away from the two twin planes, similar to the behavior shown in Fig. 3 above.

Fig. 6a shows an expanded view of the resistive transition at different measuring current densities  $I_s$ , just below the melting temperature  $T_m \sim 91.1K$  for  $H=0.5T \parallel c$ . At  $I_s=6.7\sim 40A/cm^2$ , the resistivity goes to zero below  $T \sim 90.76K$ . With increasing measuring current above  $I_s=6.7 A/cm^2$ , we observe a distinct increase in the resistivity below the zero resistance temperature. A similar behavior is observed in Fig. 6b where we have fixed the measuring current at  $I_s=40 A/cm^2$  and measured the resistive transition below  $T_m$  at different tilt angles of the magnetic field with respect to the twin planes. Comparison between the two graphs shows that tilting the magnetic field from the twin planes and thereby reducing twin boundary pinning, has the same effect as increasing the measuring current. The inset to Fig. 6b shows the critical current density as a function of temperature for various tilt angle of the magnetic field with respect to the twin planes. A dramatic 'peak effect'<sup>5</sup> is observed just below the melting temperature  $T_m$  at  $T_p=90.60K$ . The fact that this peak in the critical current disappears with increasing tilt angle is evidence that the 'peak effect' in  $YBa_2Cu_3O_{7-\delta}$  is strictly due to twin boundary pinning.

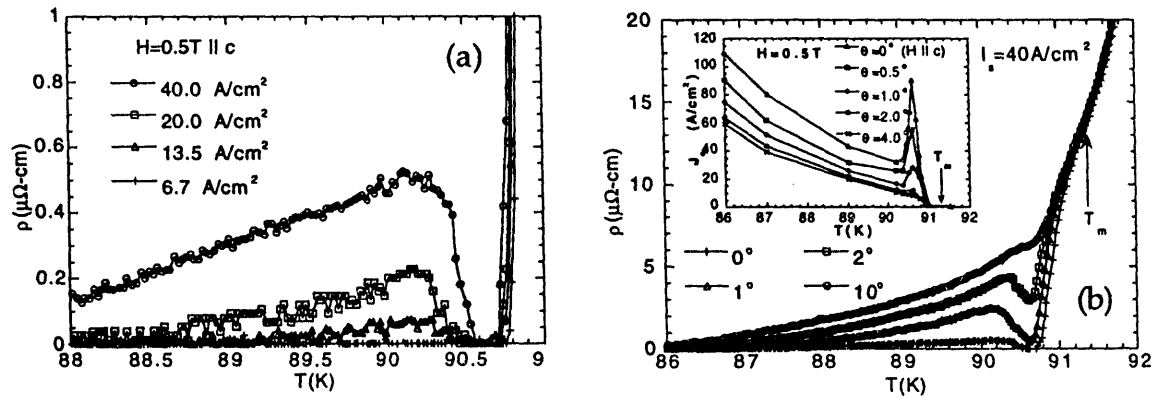


Figure 6 (a) Resistivity versus temperature below the vortex freezing transition  $T_m$  at  $H=0.5T \parallel c$  at various measuring currents. (b) Resistivity versus temperature below  $T_m$  at various angles of the magnetic field with respect to the twin planes at a fixed measuring current. (inset) Peak effect in the critical current below  $T_m$  for various tilt angles of the magnetic field with respect to the twin planes.

## 6. Conclusion

We have shown that in clean untwinned crystals, the 'kink' in the magnetoresistive transition is associated with a first order vortex liquid to solid phase transition through the observation of a pronounced hysteresis in  $\rho(H)$  and by the extreme sharpness of the transition. In densely twinned crystals, we showed that the first order phase transition is replaced with a continuous second order transition into a Bose glass state. A cusp in the angular dependence of  $T_{BG}(\theta)$  as predicted by the Bose glass theory was observed. In dilutely twinned crystals we demonstrated the competition of the pinned vortex liquid state with the first order freezing transition. In addition, we showed the existence of a



peak effect in the critical current just below the vortex freezing transition and demonstrated that the peak effect is a direct result of twin boundary pinning.

## 7. Acknowledgements

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## 8. References

- a Also at Dept. of Physics and Astronomy, Iowa State University, Ames, Iowa 50011, U. S. A.
- b Present address at Los Alamos National Laboratory, Mail Stop K763, Los Alamos, New Mexico 87545, U. S. A.

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