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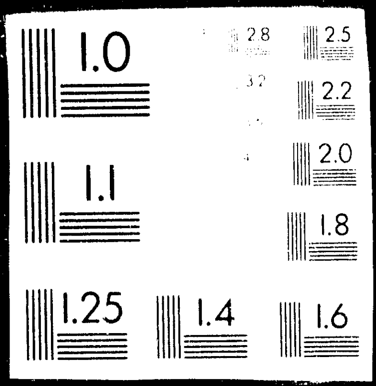

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A Comparison of Twin Boundary Pinning in Nearly Fully Stoichiometric and Oxygen Deficient $YBa_2Cu_3O_{7-\delta}$*

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A COMPARISON OF TWIN BOUNDARY PINNING IN NEARLY FULLY STOICHIOMETRIC AND OXYGEN DEFICIENT $YBa_2Cu_3O_{7-\delta}$

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Abstract--The angular dependence of the magnetoresistance was measured on the same single crystal of $YBa_2Cu_3O_{7-\delta}$ prepared initially with a superconducting transition temperature of 93.0K and then annealed to obtain a transition temperature of 56.9K. A second oxygen deficient sample was produced with a transition temperature 57.0K and nearly identical transport characteristics. Twin boundary pinning superimposed on flux flow behavior was observed in both the 56.9K and 93.0K states for magnetic field rotation in the **ab** plane. For magnetic field rotations off the **c** axis we observe twin boundary pinning only for the nearly stoichiometric state. We find the broadening of the resistive transition in magnetic field (H ll **c**) to occur over a much larger interval of reduced temperautre in the deoxygenated state. The enhanced broadening of the resistive transition in magnetic field and absence of twin boundary pinning may indicate that the vortex system is two dimensional, and in general, defects are rendered ineffective in pinning due to the absence of three dimensional collective elastic effects.

INTRODUCTION

An understanding of the effect of anisotropy on the pinning of flux lines seems crucial if the cuprate superconductors are to be fabricated such that they can carry lossless currents at high temperatures in reasonably large magnetic fields. An enhancement of the dissipative regime of the resistive transition in magnetic field of several different high temperature superconductors has been correlated with increasing electronic anisotropy. [1,2] Comparisons between different compounds can be complicated due to sample to sample variation of defects and varying number of CuO_2 planes. Ideally one would like to isolate the effect of increased anisotropy with a constant pinning contribution. The removal of oxygen from $YBa_2Cu_3O_{7-\delta}$ provides a means of increasing the anisotropy while maintaining the same parent crystal (for oxygn levels above the the tetragonal phase transition). Measurements of the reversible torque indicate that the anisotropy parameter $\gamma = m_c/m_{ab}$, where m_{ab} and m_c are the effective masses for motion parallel and perpendicular to the CuO_2 planes respectively, increases from $\gamma \sim 7$ for nearly stoichiometric samples ($\delta \sim 0.05$) to $\gamma \sim 20$ when $\delta \sim 0.35$. [3] The behavior of crystals with $\delta \sim 0.35$ and corresponding low superconducting transition temperatures $T_{co} \sim 55$-60K have been found to exhibit markedly different behavior from fully oxygenated crystals in high magnetic fields. The increased anisotropy in low T_{co} crystals manifests itself in the fluctuation diamagnetism which is found to obey a two dimensional scaling law in contrast to the three dimensional scaling form observed for fully oxygenated crystals. [4,5] The reversible region is found to extend over a larger fraction of the H-T phase diagram for reduced oxygen stoichiometries indicating a diminishing effect of pinning at high reduced temperatures and these oxygen contents. [5,6,7] We report results on the pinning properties of twin boundaries (TB's) on the same crystal for both reduced and nearly stoichiometric oxygen contents thus avoiding sample variation in preexisting pinning sites. A comparison between TB pinning when magnetic field (H) is oriented parallel to the **c** axis (H ll **c** ll TB) and when the field is aligned with the TB's and confined to the **ab** plane (H ll **ab** ll TB) yields information about the role of anistropy in pinning.

EXPERIMENT

The AC resistivty of a densely twinned single crystal was measured with a standard four probe technique. A measuring current of 0.1 mA at 17 Hz was applied in the **ab** plane. The crystal had dimensions 0.8mm x 2.0mm x 0.04mm with the shortest length along the **c** axis. Electrical contacts with resistances on the order of an ohm were established by sintering strips made with silver epoxy at 430°C in flowing oxygen for several hours in the nearly stoichimetric case. Gold wires were subsequently attached to the contact strips with silver epoxy and cured at 100°C in flowing oxygen.

The crystal was then placed in the bore of two orthogonal magnets, an 8 Tesla longitudinal magnet and a 1.5 Tesla transverse magnet. These two magnets were swept simultaneously to produce a resultant magnet field which could be rotated relative to the crystal. In addition, the sample was placed on a stage which could be rotated about the longitudinal field axis.

After measuring the angular dependent magnetoresistance in the oxygen rich state the wires were removed from the sample and the crystal was reannealed with the original contact strips. The reduced oxygen content was obtained by heating the sample in 15 Torr of oxygen at 530°C for 10 days and quenching to room temperature in the same oxygen environment. The sample quality before and after deoxygenation was characterized by the zero field resistive transition and the low field diamagnetic transition as measured by DC magnetization in an applied magnetic field of 0.5 Oersted parallel to the **c** axis. Prior to the reduced oxygen anneal the crystal had an onset transition temperature of $T_{co} \sim 93$K with a transition width of approximately 0.4K, and subsequent to the anneal the crystal had an onset temperature of $T_{co} \sim 56.9$K with a transition width of ~0.5K, as determined from the DC magnetization measurements. After deoxygenation, wires were reattached to the contact pads with silver paste and cured in air at 100°C for 20 minutes. Disorder on the oxygen sublattice created by the low temperature curing stage was allowed to relax for two weeks at room temperature before the sample was remounted in the cryostat. Photographs under polarized light of two samples with a lower density of TB's undergoing the same heat treatment revealed that the TB patterns were not altered dramatically by the anneal and a one-to-one correspondence between twin domains in these crystals was identified prior to and after deoxygenation. A second crystal was produced with a low field transiton T_{CO}~57.0K and a transition width of ~0.4K to check reproducibility of our results. We find identical results for both oxygen deficient crystals and shall not distinguish between them presently.

RESULTS

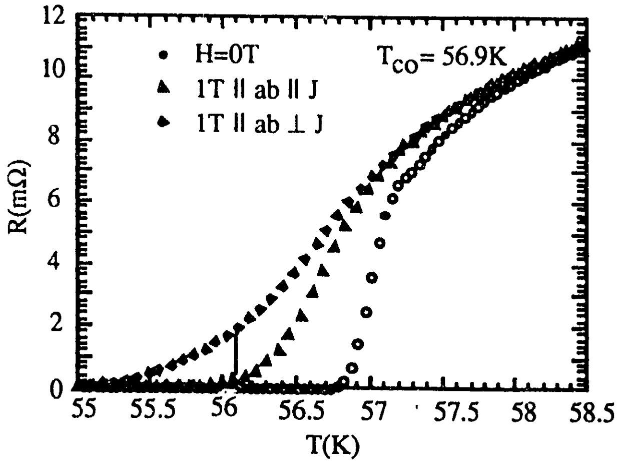

Fig. 1 The resistive transition for magnetic field oriented in the **ab** plane both parallel and perpendicular to the current. The line at 56.1K indicates the excess Lorentz force induced dissipation observed in the angular scan presented in figure 2.

The resistive transition in 1 Tesla aligned in the **ab** plane and parallel (H ‖ **ab** ‖ J) and perpendicular (H ‖ **ab** ⊥ J) to the current is shown in figure 1. The zero field resistive transition is also shown for reference. We observe the anomalous Lorentz force independent contribution to the resistivty for H ‖ **ab** ‖ J and an additional dissipation that depends on the Lorentz force similar to results previously reported for stoichiometric samples. [8] Kwok et al. showed that the additional dissipation for magnetic fields oriented in the **ab** plane at finite angle (θ) with respect to the curretnt obeyed a $\sin^2\theta$ relation indicative of conventional Lorentz force induced dissipation. The angular dependence of the resistivity for rotation of a 1 Tesla magnetic field in the **ab** plane at a fixed temperature 56.16K is displayed in figure 2. Superimposed on what appears to be flux flow behavior are sharp drops in the resistivity at 90° intervals which we associate with strong pinning by the TB's. A fit to the data with $\sin^2\theta$ for those angles at which TB pinning does not occur is represented by the line on figure 2. We observe good agreement with the Lorentz force induced dissipation formula, and thus, we attribute the excess resistivity between the orientations H ‖ **ab** ‖ J and H ‖ **ab** ⊥ J as indicated by the line in figure 2 to conventional flux flow. Note that the TB pinning occurs at the approximate angles 25° and 115° with respect to the current as opposed to the standard orientations θ=45° and θ=135°. This is due to the fact that the crystal was cleaved at an oblique angle relative to the original edges in order to obtain a large "clean" section of the crystal. Misalignment of the magnetic field was checked in both cases and found to be less than 2°.

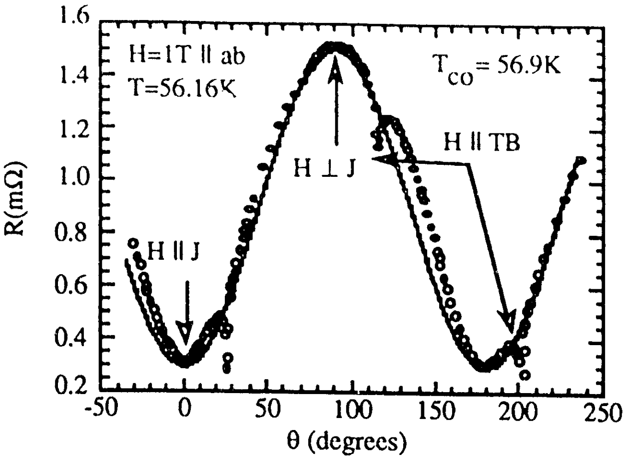

Fig. 2 The angular depencence of the resistivity for a 1 T magnetic field rotated in the **ab** plane exhibits sharp drops due to pinning by twin boundaries superimposed on a $\sin^2\theta$ (line) Lorentz force induced dissipation.

Figures 3 and 4 exhibit the resistive transition for the T_{CO}~93K and T_{CO}~56.9K crystals, respectively, for magnetic fields of 1,2,4,8 Tesla oriented normal to the CuO_2 planes (H ‖ **c**) plotted in reduced temperature. The resistive transitions for the two stoichiometries have two outstanding differences. First, the transition in magnetic field extends over a much larger range of reduced temperature for the oxygen deficient case. For example, in 8 Tesla for the fully oxygenated sample the zero resistivity point occurs at a reduced temperature T/T_{CO}~ 0.82 (76K), whereas in the oxygen deficient case the zero resistivity temperature is T/T_{CO}~ 0.23 (13K). Similar results have been previously reported and were correlated with the increased electronic anisotropy of the system. [1,2] The second readily distinguishable difference between the transitions is the presence of the downturn to zero resistivity or shoulder (indicated by arrows) present for all fields shown for the 93K state in contrast to the absence of such a feature with the possible exception of the 8 Tesla curve in the deoxygenated state.

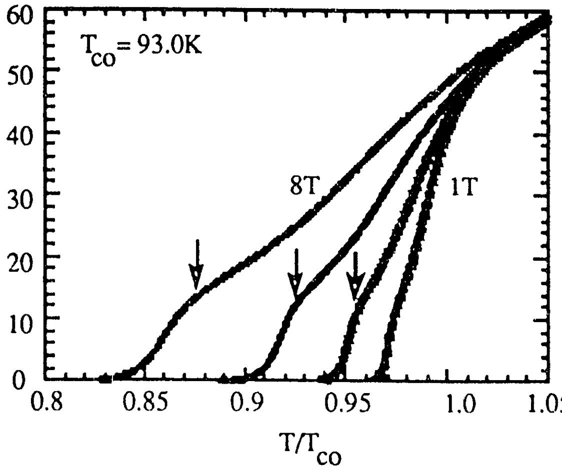

Fig. 3 The resistivity of the nearly stoichiometric sample for H=1,2,4,8T ‖ **c** is plotted versus reduced temperature. The TB pinning is experimentally observed to occur at temperatures below the shoulder which is marked by arrows.

The angular dependence of the resistivity of the 93K state for magnetic field rotation of 1T and 4T in the plane containing the c axis and bisecting the two distinct twin planes (see the inset of the same figure) for temperatures below the shoulder are shown in the top panel of figure 5. The resistivity is found to increase initially as the field is rotated off the c axis and reach a local maximum at some characteristic angle (θ~17° for 1 Tesla) and then decrease as dictated by the anisotropy of the crystal. The drop in the resistivity for magnetic fields oriented within the characteristic angle of the c axis is interpreted as an effect of TB pinning. The angular range over which the TB's affect the resistivity is considerably larger for this sense of rotation then for rotations in the **ab** plane. In the framework of anisotropic Ginzburg Landau theory the vortex tilt modulus is larger for magnetic field rotations in the **ab** plane. One expects that the vortices cannot bend as readily to take advantage of the pinning energy of the TB's for H ll **ab**, and thus, the characteristic angle is smaller for this direction. Similar measurements in the oxygen deficient state is displayed in the bottom panel. The resistivity is found to fall off monotonically as the magnetic field is rotated toward the **ab** plane and no pinning feature associated with the TB's is observed. This measurement was repeated for several temperatures but TB pinning was not observed.

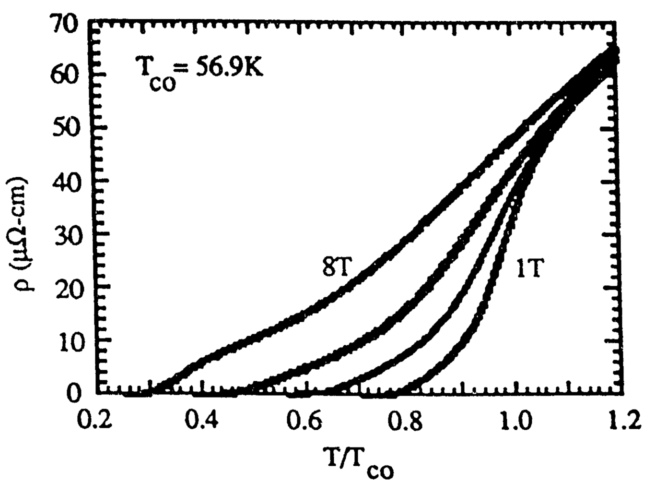

Fig. 4 The resistivity of the oxygen deficient sample plotted versus reduced temperature for H=1,2,4,8T ll **c**. Dissipation occurs over a much larger range of reduced temperature than for the oxygen rich sample. The very noticeable shoulder in the transitions of the previous figure are not present for this reduced stoichiometry with the possible exception of the 8 Tesla curve.

DISCUSSION

A study of the temperature dependence of the angular dependent magnetoresistance reveals that the onset temperature for twin boundary, the temperature below which the TB's are effective in pinning, coincides with the shoulder in the resistive transition for H ll **c**. [9,10] As the temperature is increased from values below the shoulder the drop in resistivity due to TB pinning becomes less pronounced until it finally disappears when the temperature corresponding to the shoulder is reached. Thus, the absence of a shoulder in the resistive transition and the lack of TB pinning for rotation off the c axis are consistent. The large reduced temperature regime over which dissipation occurs indicates that in general the pinning in oxygen deficient materials is supressed relative to the fully stoichiometric compound. This suppression of pinning is most likely due to the two dimensional nature of the flux lines in highly anisotropic superconductors. As oxygen is removed from the parent structure $YBa_2Cu_3O_{7-\delta}$ becomes more anisotropic and the tilt modulus of the flux lines for H ll c decreases. In the extreme case of a very large anisotropy weakly coupled pancake-like vortices are expected to form in the CuO_2 layers. [11] Specifically, our results imply that planar defects which provide strong pinnng sites for the three dimensional flux lines in the oxygen rich state are ineffective in retarding the flux motion of quasi-two dimensional or pancake vortices. [12]

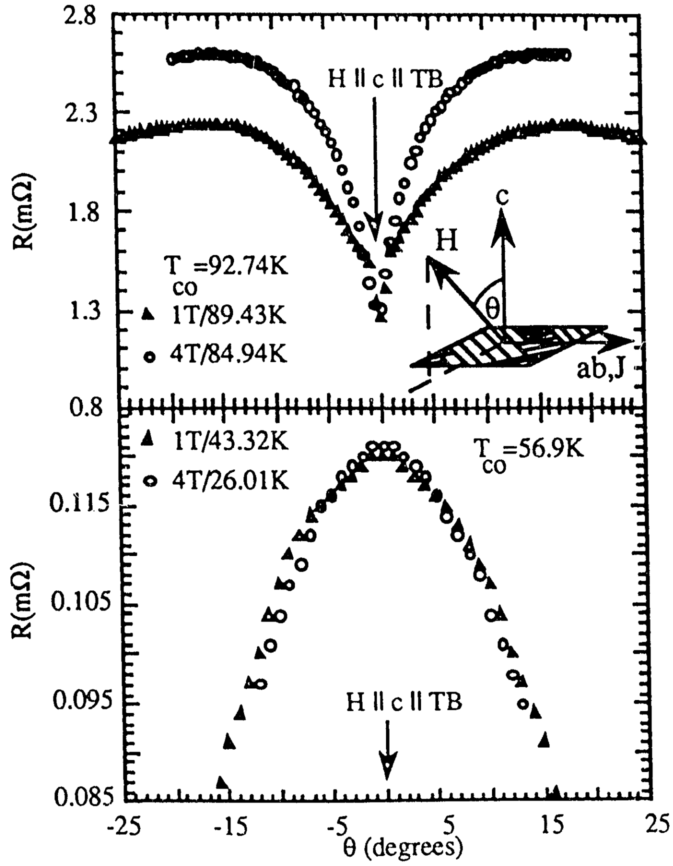

Fig. 5 The angular dependent magnetoresistance for the T_{co}=93K oxygen state exhibits sharp drops associated with TB pinning (top) whereas no such feature is found for the deoxygenated case (bottom).

In conclusion, we have observed TB pinning and Lorentz force induced dissipation in oxygen deficient $YBa_2Cu_3O_{7-\delta}$ for H ll **ab**. The magnetic field broadening is not substantially larger for this case than for fully oxygenated samples.The size of the magnetic field broadening of the resitive transition of the oxygen deficient sample for the two orientations with H ll **ab** appears to be similar to that for fully stoichiometric samples. However, the kink usually observed for H ll **ab** ⊥ J in 90K samples is not present in the corresponding resistive transition of the oxygen deficient sample. [8] Thus, with the exception of the absence of a kink in the resistive transition for H || **ab** ⊥ J, the oxygen deficient samples display similar behavior to the oxygen rich sample for H || **ab**. For H || **c** two dramatic differences between the transport characteristices for the two stoichiometries were observed. For the oxygen deficient samples the resistive transition covered a much larger regime of reduced temperature and the shoulder in the transition did not appear up to at least 6 Tesla. Twin boundary pinning was not observed in any angular scans of the magnetoresistance for the T_{co}=56.9K sample. The data for H || **c** reveal that all defects including TB's are rendered ineffective in pinning over a large region of the H-T plane in these highly anisotropic materials resulting in broader resitive transitions, and consistent with, a shift of the irreversibitily line to lower reduced temperature.

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