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Interfacial effects and superconductivity in high-T_c materials

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A series of quasihomogeneous superconducting powders of $YBa_2Cu_3O_{7-x}$ have been prepared by sieving and characterized by electron microscopy and x-ray diffraction. Quantitative size-dependent and temperature-dependent magnetic levitation and susceptibility measurements of random powders and of field-oriented (at $T < T_c = 92$ K) small grains (< 20 μm) confirm previously observed symmetry-breaking effects associated with the twin boundary planes and suggest that the bulk anisotropic superconductivity is measurably perturbed by interfacial effects. Current models of interfacial superconductivity in high-T_c materials are evaluated in light of these and other results and are found to be lacking.

It is commonly assumed that $Yba_2Cu_3O_{7-x}$ is a *pure bulk* anisotropic superconductor[1,2] (BAS) whose properties are influenced by the (110) twin boundary planes (TP's) which are a natural consequence of its orthorhombic structure[3] and act as "weak links" between BAS regions. But the only theoretical model to date which quantitatively relates the bulk superconductivity to TP's is the superconducting glass model.[2] This model is controversial and the subject of serious challenge.[4]

Recently, Garcia *et al.*[5] suggested that the superconductivity in $Yba_2Cu_3O_{7-x}$ is a nonbulk TP effect. On the basis of scanning tunneling microscopy (STM), ac susceptibility, and qualitative levitation measurements they identified the TP's as "strong links" and attributed the superconductivity to a *purely interfacial* Allender-Bray-Bardeen (ABB) excitonic mechanism[6] which would function for all temperatures below T_c. While x-ray studies of oriented small-grained powders[7] showed symmetry-breaking supercurrent paths parallel to the TP's in addition to those parallel to the basal (*ab* CuO) planes, these studies were compatible with either a weak-link or strong-link origin. Subsequently, TP supercurrents have been associated with the hybrid phenomenon of twin-plane superconductivity[7] (TPS) and with oxygen vacancy ordering[8] (OVO) both of which are activated at a temperature $T_c' < T_c$ below which the BAS mechanism obtains. (Note that BAS is the underpinning of TPS and OVO neither of which can exist in pure form and both of which are strong-link models.) To date the TPS model has been employed to quantitatively account for the temperature dependence of the upper critical fields[9] and of the specific heat[10] of $Yba_2Cu_3O_{7-x}$. While the evidence for interfacial effects in the superconductivity of $Yba_2Cu_3O_{7-x}$ is suggestive it is not conclusive. Accordingly, we present in this article the first quantitative studies of the magnetic levitation and susceptibility of oriented powders of $Yba_2Cu_3O_{7-x}$. These studies are apparently incompatible with the assumption of *pure* BAS but support a hybrid mechanism which partially localizes the supercurrents to the neighborhood of the TP's.

Quasihomogeneous powders of $Yba_2Cu_3O_{7-x}$, $x \sim 0$, were synthesized by the usual method[11] and sieved into the following size distributions: $d < 20$ μm; $\delta - \epsilon < d < \delta + \epsilon$, $\epsilon = 5$, $\delta = 25$, 35, 45 μm; $\epsilon = 12.5$ μm, $\delta = 62.5$, 87.5 μm. Powder x-ray diffraction studies of these $Yba_2Cu_3O_{7-x}$ specimens revealed only those structural features associated with the 1:2:3 superconducting phase. We focus here on specimens with $d < 20$ μm the typical grains of which are shown in the scanning electron micrographs of Fig. 1. As can be seen from that figure, the typical grain is a disklike platelet with a basal (*ab*) surface and an aspect ratio [=(diameter)/(thickness)] of ~ 1.5. It is known[12] that for small grains of this type the TP's exhibit a unidirectional or at most bidirectional domain structure, the former of which is illustrated in Fig. 2.

Levitation studies were carried out using the apparatus[13] depicted in Fig. 2, with a transverse permanent magnet having $(H_y)_{max} \cong 5000$ G. The field $H_y(z)$, which was measured with a spatial resolution of 0.5 mm using a Hall probe, is shown in Fig. 3 together with a seven-parameter polynomial least-squares fit from which the gradient (also shown in Fig. 3) was obtained. Susceptibility studies and field-induced alignments were performed with a SHE variable-temperature susceptometer while x-ray studies were performed with a conventional Siemans powder diffractometer using a Cu $K\alpha$ source and a spinning sample holder.

When powders with $d < 20$ μm are dropped from a height z at which $H_y(z) = 0$ through liquid nitrogen (LN) into the inhomogeneous field of the magnet shown in Fig. 2, the monodispersed single-domain grains do not levitate but instead collect at the bottom of the confinement tube. If the temperature is reduced, these grains first levitate at 74 ± 2 K at $z = 4$ mm and continue to do so with decreasing temperature and/or increasing z until $z = 14$ mm for liquid-helium (LH) immersion. When small grains which are nonleviating in LN are lightly compacted (without sintering or chemical alteration) into a pellet, that pellet

FIG. 1. Scanning electron micrographs of the $d < 20$ μm powders of $YBa_2Cu_3O_{7-x}$ taken at magnifications of 1000 (lower panel) and 2000 (upper panel). The large flat faces of grains such as that centered in the upper panel are basal (ab) surfaces.

levitates in LN (LH) at $z = 8$ mm (17 mm). Grains with $d > 20$ μm also levitate in *both* LN and LH at these same heights. All of the loose and compact random powders are superconducting with typical Meissner fractions (measured according to methods established by Krusin-Elbaum, Malozemoff, and Yeshurun[14]) in excess of 50% as shown by the data of Fig. 4. The random powder data of Fig. 4 are typical of pure phase 1:2:3 material and show none of the features, e.g., kinks at $T < T_c$ which are normally associated with impurity phases and/or reduced oxygenation.[15]

Assume for the sake of analytic solutions that the $YBa_2Cu_3O_{7-x}$ particles are magnetically anisotropic pure BAS ellipsoids whose susceptibility and demagnetization tensors have coincident principal axes. A particle of volume V in an applied magnetic field $\mathbf{H}_0$ will adopt a configuration which minimizes U (Ref. 16) where

$$U = -\tfrac{1}{2}\int_V \mathbf{M}\cdot\mathbf{H}_0\,dv = -\tfrac{1}{2}\int_V \sum_{j=1}^{3} \frac{\chi_j}{1+N_j\chi_j} H_{0j}^2 \,. \qquad (1)$$

Here, $\mathbf{M}$ is the magnetization of the particle; N_j, χ_j, and H_{0j} are the demagnetizing factor, susceptibility, and projection of the field along the jth principal axis, respectively; and $\sum_j N_j = 4\pi$. The torque and force on the particle can be obtained from U, e.g., $\tau_\theta = \partial U/\partial\theta$, $F_z = \partial U/\partial z$, etc.

Equation (1) can be used to establish the z dependence of the levitation force when the free particle has oriented

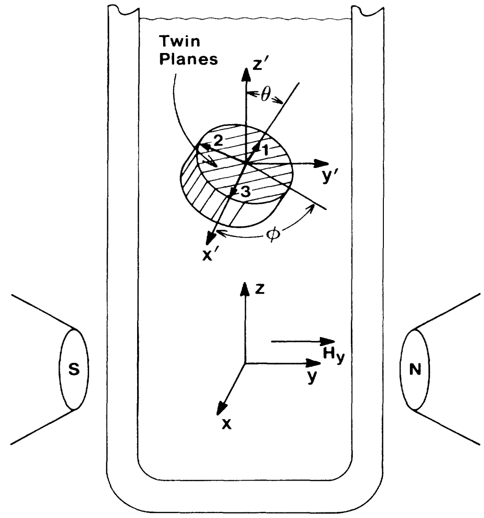

FIG. 2. A schematic diagram of the levitation experiment with axes and angles labeled for reference in the text. The principal axes of the demagnetization tensor are labeled 1, 2, 3 and are parallel, respectively, to the crystallagraphic axes c, a (or b), b (or a).

itself to minimize U. Suppose a particle has descended into a region where its local field is less than the lowest critical field $H_{c1}^{\parallel}(77\text{ K})$ ($\parallel \rightarrow \mathbf{H}_0 \parallel ab$ planes). Then $\chi_j = -1/4\pi$, $j = 1,2,3$, and the term in Eq. (1) with minimum N_j minimizes U. The particle will align with its longest principal axis (axis 2 in Fig. 2) $\parallel \mathbf{H}_0$. If $N_2 \ll 4\pi$ or $H_{0y} \gg H_{c1}^{\parallel}$, the levitation condition is

$$F_z(z) = \tfrac{1}{2} V M_y (\partial H_y(z)/\partial z) = (\rho - \rho_{LN}) g V \,, \qquad (2)$$

where $M_y = H_y/4\pi$, $\rho = 6.38$ g/cm^3 is the mass density of $Yba_2Cu_3O_{7-x}$, $\rho_{LN} = 0.6$ g/cm^3 is the density of liquid nitrogen, and g is the gravitational acceleration.

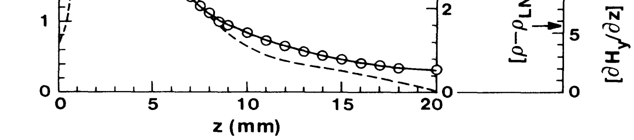

FIG. 3. The measured transverse field (circles) and a seven-parameter polynomial fit (solid line) (see text). The gradient (dashed line) was obtained analytically from the polynomial fit. The additional ordinate scale on the far right renormalizes the gradient to $\rho_{mag}^{\parallel}(z)(77\text{ K})$ the maximum value of which is indicated along with $\rho - \rho_{LN}$, the buoyancy-corrected gravitational mass density of $YBa_2Cu_3O_{7-x}$.

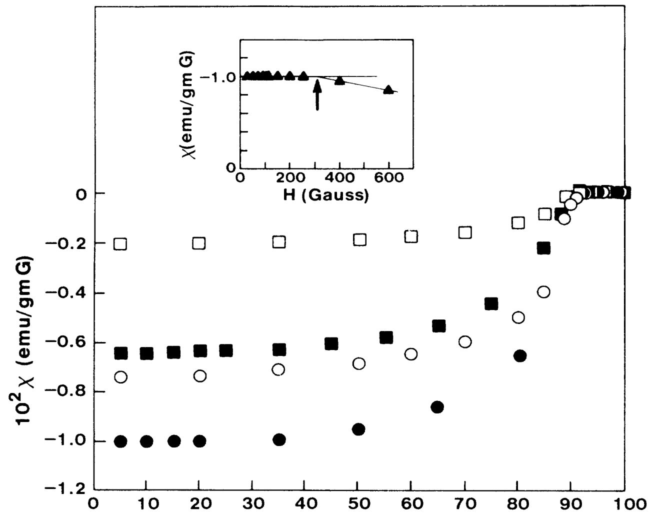

FIG. 4. The temperature dependence of the mass susceptibility of $YBa_2Cu_3O_{7-x}$ powder ($d < 20$ μm) in a random configuration (circles) and field-oriented (see text) configuration (squares). Open (solid) symbols represent field cooling at 18 G (zero-field cooling followed by field heating at 25 G). Inset, the field dependence of the 5-K susceptibility of random powder (▲) showing the extrapolated critical field $H_{c1}^{\parallel}(5\text{ K}) = 300 \pm 25$ G (vertical arrow).

Equivalently, if we define $\rho_{\mathrm{mag}}(z) = F_z(z)/Vg$ to be the magnetic mass density, the particle will levitate if $[\rho_{\mathrm{mag}}(z)]_{\mathrm{max}} \geqq \rho - \rho_{\mathrm{LN}}$.

When the local field exceeds the lower critical field H_{c1}^{j}, along one or more of the principal axes of the particle, it enters the vortex state. For $YBa_2Cu_3O_{7-x}$,

$$H_{c2}^{2} = H_{c2}^{3} = H_{c2}^{\parallel} \gg H_{c1}^{\parallel}, \quad H_{c2}^{1} = H_{c2}^{\perp} \gg H_{c1}^{\perp}$$

and

$$H_{c1}^{\parallel} < H_{c1}^{\perp} < H_{c2}^{\perp} < H_{c2}^{\parallel} .$$

(Magnetic anisotropy in the ab plane, although expected,[3] has been observed[7] but not yet measured.) In the range $H_{c1}^{j} < H_j \ll H_{c2}^{j}$, **M** is approximately linear in H in which case

$$\chi_j = -(1/4\pi)(H_{c1}^{j}/H_j) , \tag{3}$$

$$H_j = H_{0j} + (N_j/4\pi)H_{c1}^{j} , \tag{4}$$

$$\chi_j/(1+N_j\chi_j) = (-1/4\pi)(H_{c1}^{j}/H_{0j}) . \tag{5}$$

Then the levitation condition is given by Eq. (2) but with the term $H_y(z)$ replaced by H_{c1}^{j}. For $H_{0j} \gg H_{c1}^{j}$, demagnetizing effects are negligible and the particle will align in a direction which minimizes flux expulsion, i.e., along the axis with the minimum value of H_{c1}^{j}. In the language of anisotropic Ginzburg-Landau theory[17] this is the direction corresponding to the smallest effective mass and thus the highest H_{c2}^{j}. But the product of H_{c1}^{j} and H_{c2}^{j} is roughly constant, i.e., $(H_{c1}^{j}H_{c2}^{j})^{1/2} \cong H_c$ where H_c is the thermodynamic critical field. So the direction with maximal H_{c2}^{j} also has minimal H_{c1}^{j} as asserted above.

We have used Eqs. (1)–(5) to determine the levitation force on and preferred orientation of a BAS particle. The results are summarized in Table I. The values of $(\rho_{\mathrm{mag}}^{\parallel})_{\mathrm{max}}$ cited in that table were deduced from Fig. 2 using $H_{c1}^{\parallel}(77\text{ K}) = 90 \pm 8$ G. The latter was computed[18] from the lower critical field, $H_{c1}^{\parallel}(5\text{ K}) = 300 \pm 25$ G, at which $\chi(H, T = 5\text{ K})$ of a random powder linearly departs from constancy (see inset, Fig. 4). This departure commences when $H_0 \cong H_{c1}^{\parallel}$ for particles in the powder which happen to be aligned with their ab planes $\parallel H_0$. Although the departure is in principle quadratic in applied field for single-crystal specimens or fully aligned widely spaced grains, its analytic form, which depends strongly on uncalculable local-field corrections, is not known for powder specimens. The assumption of a linear departure which we used to compute $H_{c1}^{\parallel}(5\text{ K})$ from the inset data of Fig. 4 is thus reasonable and uncertainties which this assumption introduce are reflected in the large error bars on our measured value of $H_{c1}^{\parallel}$. Moreover, the above specified value of $H_{c1}^{\parallel}(5\text{ K})$ is consistent with our measurements of oriented powders and lies at the low end of the range of reported values[1,19] for that parameter.

Notice from Table I that a pure BAS analysis predicts that the particle will not levitate in the Meissner state because field limitations constrain $\rho_{\mathrm{mag}}^{\parallel}$ to values $\ll \rho$. How-

TABLE I. Orientation and levitation of a bulk anisotropic superconducting particle in a transverse field, $H_y(z)$ at 77 K. The symbols m, v, and n denote the Meissner, vortex, and normal states, respectively, and $S = \frac{1}{2} M_y V(\partial H_y(z)/\partial z)$, where V is the particle volume and M_y is the induced moment. Axis labels refer to Fig. 2.

State	Applied field	Axis orientation	F_z (dyn)	$(\rho_{\mathrm{mag}}^{\parallel})_{\mathrm{max}}$ g/cm^3
m	$H_y < H_{c1}^{\parallel}\left(1 - \dfrac{N_2}{4\pi}\right)$	(2) $\parallel H_y$	$SH_y(z)$	0.73 $\pm$ 0.05
v	$H_{c1}^{\parallel}\left(1 - \dfrac{N_2}{4\pi}\right) < H_y < H_{c2}^{\perp}$	(2) $\parallel H_y$	$SH_{c1}^{\parallel}$	26.3 $\pm$ 3.5
n	$H_y > H_{c2}^{\perp}$	(1) $\parallel H_y$	0	0
n	$H_y > H_{c2}^{\parallel}$	None	0	0

ever, the particle *should* levitate in the vortex state for which $(\rho^{\parallel}_{\mathrm{mag}})_{\max} > 4(\rho - \rho_{\mathrm{LN}})$. If flux is more effectively expelled from the TP's (of width t with spacing L) than from the intervening regions (of width $L-t$) as would be the case in a strong-link hybrid model, $(\rho^{\parallel}_{\mathrm{mag}})_{\max}$ will be *reduced* by approximately t/L. Then under the condition $t/L < (\rho - \rho_{\mathrm{LN}})/(\rho^{\parallel}_{\mathrm{mag}})_{\max} = 0.22$ small particles will not levitate at 77 K, as is observed. Nonlevitation is thus likely since the spread in reported values of t (Refs. 4 and 20) and L (Ref. 21) limit their ratio to $\frac{5}{2000} < t/L < \frac{80}{200}$. If T_L is the temperature at which small particles just begin to levitate at maximum gradient,

$$[(1/8\pi g)(t/L)H^{\parallel}_{c1}(T_L)[\partial H_y(z)/\partial z]_{\max} = \rho - \rho_{\mathrm{LN}} ,$$

where

$$H^{\parallel}_{c1}(T_L) \cong H^{\parallel}_{c1}(5\ \mathrm{K})[1-(T_L/92)^2] ,$$

an empirical relation[18] which yields the expected linear variation[1,8] near $T_c = 92$ K. Using the fact that for $T_L = 74 \pm 2$ K small particles just levitate at $z = 3.5 \pm 0.5$ mm, the height of maximum gradient (see Fig. 3), we find $t/L = 0.19 \pm 0.04$. For the case of $t \sim 80$ Å (Ref. 4) [5 Å (Ref. 20)], $L = 400 \pm 100$ Å (25 ± 6 Å), well within (outside) the accepted range.[21]

The TP correction factor does not apply to random powders, multigrained particles, or to large particles with multidirectional TP domains. These morphologies cannot uniquely align and the tilted (nonaligned) TP's screen flux from the intervening regions. It is impossible to analytically calculate the levitation force for such agglomerates because the nonlinear susceptibility in the vortex state leads to nonuniform local-field corrections. However, if we treat a compacted pellet empirically as a disk with an *isotropic* χ, the levitation force it will experience is given by the force expression for the vortex state (row 2, column 4 of Table I). Using our measured value of $H^{\parallel}_{c1}(77\ \mathrm{K})$ we find that the pellet should levitate when $\partial H_y(z)/\partial z = 2242 \pm 187$ G/cm. This value obtains at $z = 7.5 \pm 0.8$ mm (see Fig. 3). A corresponding analysis for $T = 5$ K yields a levitation height of 13 ± 2 mm. Both of these results agree well with observation.

We now address our susceptibility measurements of random and oriented powders of small $YBa_2Cu_3O_{7-x}$ particles. Figure 4 shows $\chi(T)$ for random $d \cong 20$ μm powder (solid and open circles) and of the same powder after orientation in a field of 5 kG at $t = 5$ K (solid and open squares). These data have been used to compute[18] Meissner fractions, F, of 54% and 22% at 5 K, respectively. The applied fields were well below the lowest critical field of $YBa_2Cu_3O_{7-x}$ and in the same direction as the orienting field.

The dramatic drop in the Meissner fraction of the oriented specimen is not likely to be due to flux penetration since $\lambda^{\parallel}(5\ \mathrm{K}) \le 0.8$ μm (Refs. 1 and 19) which is considerably less than the ~ 10 μm thickness of a typical aligned particle (see further discussion below). Moreover, this drop does not derive from field-induced decoupling of weak links since such links between loosely packed single grains will be too weak to sustain supercurrents in either random or oriented powders. The drop in F could result from anisotropic flux trapping in pure BAS particles.[14] Alternatively, if the supercurrents are partially localized in the TP's that align with the orienting field,[7] flux could penetrate between these planes resulting in a drop in F. Our levitation results point towards the latter explanation.

Implicit in the above analyses of levitation and susceptibility is our assertion that the effects which we have observed are not merely a manifestation of ordinary flux penetration which would reduce both of these observables. To further explore this point we note that for a thin slab aligned with its face parallel to the applied field, the susceptibility and the levitation force should both be multiplied by the factor $k = \{1 - [\tanh(a/\lambda)]/(a/\lambda)\}$ where λ is the penetration depth and $2a$ is the width of the slab.[22] If we attribute nonlevitation to this factor then $k = (\rho - \rho_{\mathrm{LN}})/(\rho^{\parallel}_{\mathrm{mag}})_{\max} = 0.22$ and $a/\lambda = 0.94$. If we use the empirical relation

$$\lambda(T) = \lambda(T_0)[1-(T_0/T_c)^4]/[1-(T/T_c)^4]$$

with $T_0 = 5$ K and $\lambda(5\ \mathrm{K}) = 0.8$ μm,[19] the maximum reported value, then $\lambda(77\ \mathrm{K}) = 1.92$ μm and $a/\lambda \gtrsim 5.2$. This value is more than a factor of 5 larger than the value required for nonlevitation. Furthermore, if we make the conservative but clearly unfounded assumption that the oriented powder used for the susceptibility measurements was *fully* aligned, a reduction factor of only 0.22 cannot account for the approximate factor of 2 drop in the Meissner fraction evidenced in Fig. 4. Thus flux penetration cannot rescue the pure BAS model.

Although we have demonstrated certain deficiencies in pure BAS models of $YBa_2Cu_3O_{7-x}$, we cannot identify which, if any, of the currently proposed TP perturbative mechanisms is applicable. The ABB model[5,6] and other purely interfacial models of this type such as those based on surface plasmons require metal-semimetal/semiconductor interfaces and consequently significant volume fractions of bulk semimetal/semiconductor interfaces. Surface-sensitive STM experiments[5] show large regions of semiconductor but bulk NMR measurements[23] do not. The TPS hybrid model[8,24] produces measurable effects only when the TP spacing is less than the Ginzburg-Landau correlation length of ~ 34 Å. There is no evidence to date of such small TP spacings in $YBa_2Cu_3O_{7-x}$. Indeed, spacings of this magnitude would result in a large volume fraction of TP's ($t/L \cong 1$). This situation is not supported by our levitation results. Finally, the OVO (Ref. 9) hybrid model predicts the onset of BAS at the unreasonably low temperatures of $T_c' \sim 40$ K, a condition that is, to our knowledge unsupported by any measurement to date.

While it is clear that TP's modify the superconducting nature of $YBa_2Cu_3O_{7-x}$, the detailed mechanism which induces this modification remains to be identified and/or confirmed. In particular, although our own data and those cited in Refs. 8 and 10 favor the strong-link hypothesis we recognize that there are also many measurements which favor the weak-link picture. A definitive answer to this dilemma may lie with experiments on vortex decoration in the region of the TP's. Vortices should be attracted to (repelled from) the TP boundaries if the TP's are weak (sufficiently strong) links.

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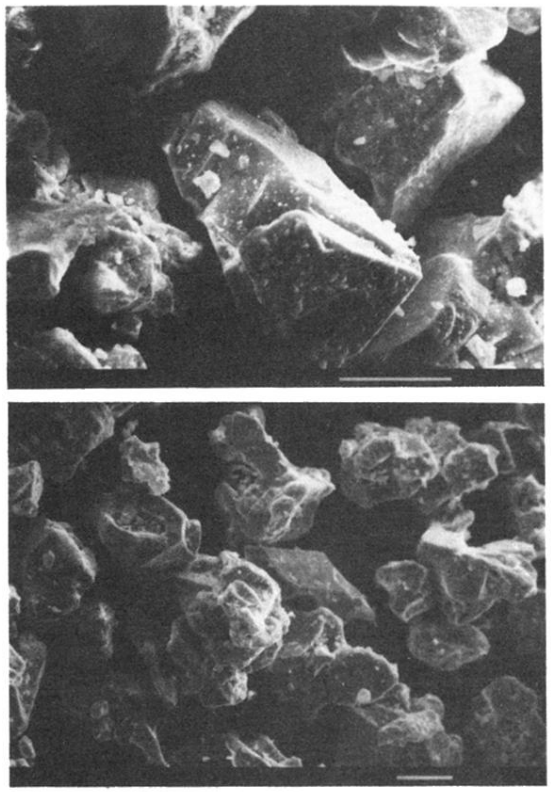

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