Overdoping effects in $Bi_2Sr_2CaCu_2O_{8+\delta}$: From electromagnetic to Josephson interlayer coupling

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We report a study of magnetic, structural, and transport properties of $Bi_2Sr_2CaCu_2O_{8+\delta}$ single crystals with different degrees of overdoping. The overdoping is achieved by high oxygen pressure (up to 190 bar) annealing. The parameters used to characterize the overdoping were the crystallographic *c*-axis lattice parameter and the superconducting transition temperature. The irreversibility line H_{il} and the characteristic field of the order-disorder transition of the vortex structure H_{sp} were obtained from dc magnetization with the applied magnetic field perpendicular to the CuO_2 planes. The large overdoped range has two well-defined regions: (i) close to the optimal doping the interlayer coupling is mainly electromagnetic and H_{sp} is given by $\Phi_0/\lambda_{ab}^2(0)$ [$\lambda_{ab}(0)$ is the in-plane penetration depth at $T=0$ K], and (ii) with further overdoping the Josephson interlayer coupling becomes relevant, so H_{sp} deviates from $\Phi_0/\lambda_{ab}^2(0)$ and is fixed by the effective-mass anisotropy. All the results indicate an increase in the coupling between CuO_2 planes with overdoping.

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I. INTRODUCTION

The layered superconductor $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi2212) has the weakest known CuO_2 planes coupling among the high-temperature superconductors (HTSC). In this material the hole carriers are generated in the BiO double layers by accommodating extra oxygens, and they are transferred to the CuO_2 planes.[1] The overdoping and underdoping of Bi2212 could be achieved by cationic substitutions[2,3] or by changes in the oxygen content.[4] The superconducting critical temperature T_c depicts a maximum at optimal doping.[5]

The effective-mass anisotropy $\gamma=\sqrt{m_c/m_{ab}}$ depends on the electronic nature of the material between CuO_2 layers and on the degree of charge transfer onto them. In the case of Bi2212, γ decreases monotonically with increasing doping[1] as has been observed in other HTSC.[6–9]

The *H-T* phase diagram of Bi2212, where *H* is the applied magnetic field parallel to the *c* axis, has several characteristic features strongly determined by the weak CuO_2 planes coupling: Bi2212 presents one of the largest vortex liquid regions among the HTSC, separated from a pinned vortex array by a characteristic line (H_{il}). At low fields and high temperatures a first-order phase transition takes place[10] where a sublimation of an ordered vortex structure occurs. The ordered vortex solid state is found only at low fields and temperatures. At higher fields, an order-disorder transition takes place at an almost temperature-independent field H_{sp}, where the magnetization shows a peak (called the second peak).[11,12] This order-disorder transition is believed to be a pinning-induced softening of the vortex structure.[13]

The oxygen doping in Bi2212 results in higher H_{sp} values as well as in a reduction of the vortex liquid region in the *H-T* phase diagram.[1,12]

In layered superconductors the coupling between superconducting planes is believed to occur via Josephson currents, tunneling, and electromagnetic interactions that link pancakelike[14,15] vortices. The Josephson coupling characteristic length is $s\gamma$, where *s* is the distance between CuO_2 planes. For electromagnetic coupling the in-plane penetration depth $\lambda_{ab}(0)$ is the typical length. When $s\gamma<\lambda_{ab}(0)$, the Josephson tunneling is relevant and the coupling is expected to be Josephson-like. In this case, at $s\gamma\approx a_0$ (where a_0, is the vortex lattice parameter), the shear modulus C_{66} overcomes the tilt modulus C_{44} and the vortex structure becomes two dimensional (2D).[13,15] Hence, the order-disorder transition characteristic field $B_{sp}=H_{sp}+4\pi(1-D)M$ (where *D* is the demagnetizing factor) should take the value $B_{2D}=\Phi_0/(s\gamma)^2$. On the other hand, when the anisotropy increases and $s\gamma>\lambda_{ab}(0)$, the electromagnetic coupling overcomes the Josephson coupling. When $\lambda_{ab}\approx a_0$, the vortices begin to overlap and C_{66} increases continuously with *B* although it changes its field dependence.[16] It is expected that pinning-induced short-wavelength tilt deformations of the vortex structure become increasingly relevant, and the system goes to a more 2D behavior due to this C_{44} softening. In this case, B_{sp} should be the signature of the characteristic field $B_{cr}=\Phi_0/\lambda_{ab}^2(0)$.[3,17]

The evolution of the main features of the (*H-T*) phase diagram of Bi2212 with oxygen content can be understood in terms of the different degrees of coupling between pancake-like vortices. However, there is no agreement as to whether the relevant coupling between them is electromagnetic or Josephson-like in the Bi2212 system when the oxygen content is varied. In fact, $s\gamma\sim\lambda_{ab}(0)\sim1500-2000$ Å for Bi2212, and both γ and $\lambda_{ab}(0)$ depend strongly on doping concentration. While γ shows a monotonic decrease with increasing doping,[1] $\lambda_{ab}(0)$ depicts the so-called *boomerang* path.[3,18–21] Starting from the underdoped region, $\lambda_{ab}(0)$ is a decreasing function of carrier concentration reaching a minimum in the slightly overdoped region, increasing again with a further carrier density increment. So it is expected that one can go from the Josephson coupling limit to the electromagnetic coupling limit by changing the carrier concentration (by cationic substitution or oxygen annealing).

In this work we report a systematic study of the influence of oxygen content on different properties of Bi2212 single crystals using several techniques such as x-ray diffraction (XRD), electric transport, dc magnetization, and ac suscepti-

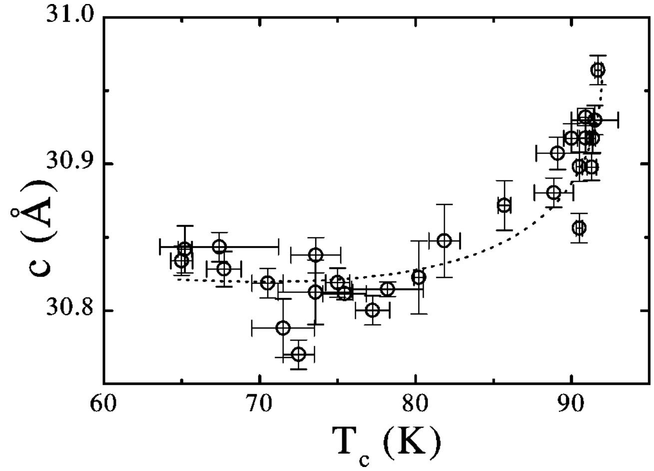

FIG. 1. The c-axis lattice parameter as a function of T_c. The line is a guide for the eye.

bility. We show that the overdoped range has two well-defined regions in this system. Close to the optimal doping the interlayer coupling is mainly electromagnetic. With further overdoping the coupling becomes Josephson-like. We show the exact boundary that separates both regions, removing the apparent controversy found in the literature on the kind of interlayer coupling in Bi2212.

II. RESULTS AND DISCUSSION

The crystals were grown in air using the self-flux technique. Nearly stoichiometric powder material $Bi_{2.4}Sr_2CaCu_2O_\delta$ was placed in Al_2O_3 boats in a cylindrical furnace. When the maximum temperature (970 °C) was reached the boat was moved to a furnace region with a large thermal gradient (25 °C/cm) and the temperature was then reduced to 825 °C at a rate of 0.02 °C/min. Single crystals of typical dimensions $1\times1\times(0.02–0.1)$ mm^3 and average composition $Bi_{2.07}Sr_{2.03}Ca_{0.93}Cu_{1.96}O_{8+\delta}$ were selected for this study. All the samples were annealed in Ar to achieve optimal doping before oxygen overdoping. The overdoping treatments were made in an oxygen high-pressure furnace at several fixed values of pure O_2 pressures (PO_2) ranging between 1–190 bar. The Ar and O_2 heat treatments were performed at 500 °C for 20 h followed by a fast cool down. The critical temperatures were determined by ac susceptibility.

The best parameter to characterize the effects of the overdoping procedure on the crystals is the oxygen concentration or the charge-carrier concentration. Although these variables are not known, the oxygen annealing pressure PO_2 is a fairly good parameter correlated to those variables[22] since all the annealings were done at the same fixed temperature. Kaul and Nieva[22] showed that T_c decreases with PO_2. However, there is a lack of precision of the actual oxygen partial pressure in the Ar-annealed samples and a pronounced change in T_c for low values of PO_2. Thus, we will use T_c as the parameter to evaluate the charge-carrier density.

In Fig. 1, the c-axis lattice parameter (extracted from XRD measurements) is shown as a function of T_c. A systematic decrease of c with decreasing T_c is observed. How-

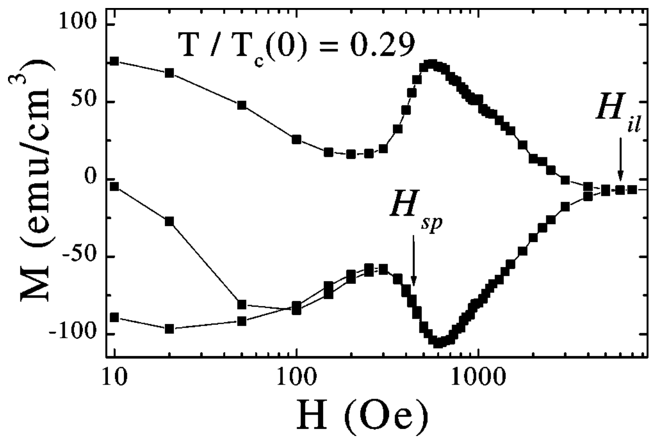

FIG. 2. A typical magnetization loop. It shows the irreversibility field H_{il} and the second peak field H_{sp}.

ever, this behavior saturates for T_c below 78 K. The peak width of the rocking curves shows no systematic change after overdoping. Planar defects in Bi2212 are known to influence the c-axis alignment preferentially in planes perpendicular to those of the defects.[23] Since our rocking curve measurements were done at random directions, the above-mentioned effect could have overcome any systematic change of the peak width with doping.

H_{il} and H_{sp} were extracted from dc magnetization loop measurements performed with a Quantum Design superconducting quantum interference device magnetometer. A typical magnetization loop for $T=25$ K is shown in Fig. 2. H_{il} was calculated as the onset of the irreversible behavior and H_{sp} as the midpoint between the valley and the peak, averaging the values obtained from both branches of the loop. With our sample geometry, $H\approx B$ provided that the demagnetizing factor is very close to 1 (0.9–0.99).[24]

The irreversibility line H_{il} versus T_c is shown in Fig. 3. The main panel depicts the data taken at a reduced temperature of 0.35. The enhancement of H_{il} with overdoping depends on temperature. In the inset of Fig. 3 we plotted the maximum span of H_{il} [i.e., H_{il}(190 bar) and H_{il}(0 bar) vs

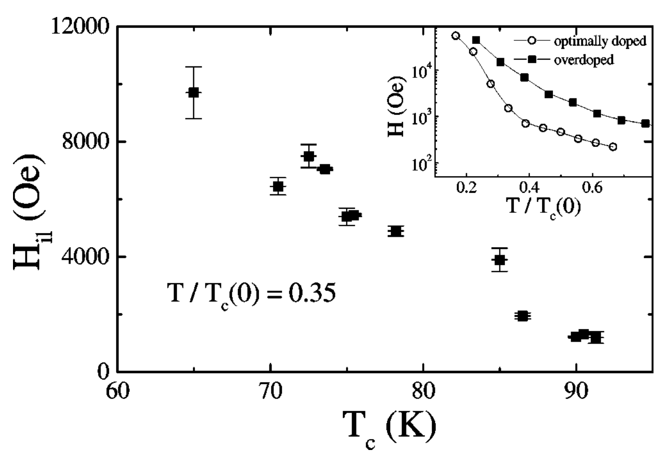

FIG. 3. The irreversibility line as a function of T_c. Inset: H_{il} of an optimally doped and a maximum overdoped (190 bar) sample versus reduced temperature.

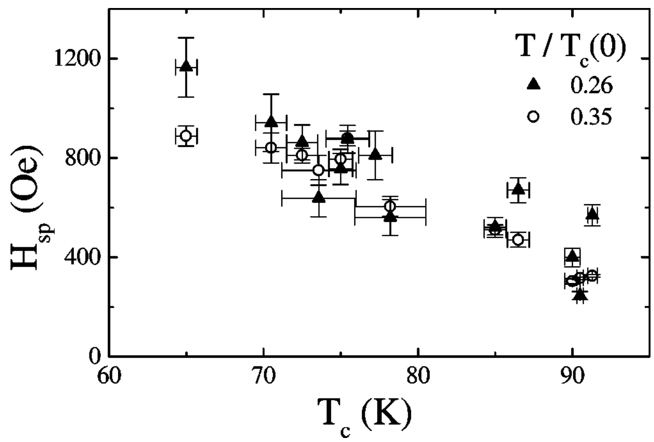

FIG. 4. Characteristic second peak field versus T_c for two different temperatures. It shows that H_{sp} is independent of temperature.

the reduced temperature]. The normalized difference between both curves seems to have a maximum at around $T/T_c = 0.4$.

In Fig. 4 we show the dependence of H_{sp} on T_c for two different temperatures, extracted from dc magnetization loop measurements. H_{sp} is temperature independent and increases monotonically with decreasing T_c. This behavior makes evident that the transition boundary H_{sp} moves toward higher fields in the H-T phase diagram when the oxygen concentration increases. This is expected if the interlayer coupling enhances. Unlike the c-axis lattice parameter, neither H_{il} nor H_{sp} exhibit a saturation at low T_c (highest overdopings).

For several samples involved in this study we have determined the in-plane penetration depth $\lambda_{ab}(0)$. We extracted $\lambda_{ab}(0)$ from the reversible part of the dc magnetization loops supposing a two-fluids model, i.e., $\lambda_{ab}(T) = \lambda_{ab}(0)\sqrt{1/[1-(T/T_c)^4]}$. We found a nonmonotonic T_c dependence of $\lambda_{ab}(0)$ in agreement with other authors.[3,19,25] This effect was ascribed to a pair breaking, provided that $n_s/m_{ab} \propto 1/\lambda_{ab}^2(0)$ (n_s is the superconducting pair density) decreases, although the normal-state carrier concentration increases in the overdoped region.[19,25]

Transport measurements were performed in a sample with optimal doping and in the same sample with the maximum overdoping (i.e., annealed at 190 bar). In-plane and out-of-plane resistances were determined using a multiterminal (eight-contact) configuration.[26] With this technique, described in detail in Ref. 27, homogeneous as well as inhomogeneous current distribution could be established in the samples. This allows not only the determination of ρ_c and ρ_{ab} but also to check for sample defects that could lead to a erroneous determination of ρ_c. From these measurements we have estimated the electronic effective-mass anisotropy parameter γ as the ratio $\gamma^2 = \rho_c/\rho_{ab}$ taken at $T = 100$ K.

Aegerter *et al.*[17] have shown a linear dependence of H_{sp} with $1/\lambda_{ab}^2(0)$ in the optimally doped and slightly overdoped region for Bi2212, and this behavior was also corroborated for other HTSC.[17] However, there are reports (in other HTSC or different doping regions) in which H_{sp} is not determined only by $\lambda_{ab}(0)$ but also γ must be taken into

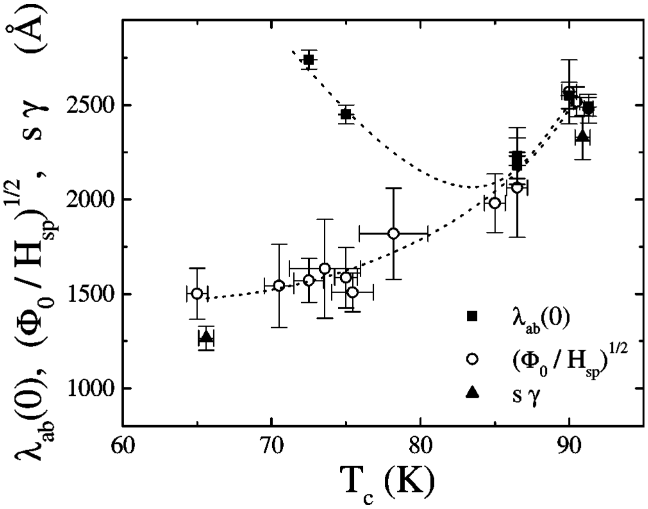

FIG. 5. London penetration depth (full squares), $\sqrt{\Phi_0/H_{sp}}$ (open circles), and electronic anisotropy multiplied by the interlayer distance s (full triangles) as a function of T_c.

account.[3,9] In particular, it was shown[1,28] that in Bi2212 there is a correlation between H_{sp} and the electronic anisotropy parameter.

In Fig. 5 is shown the dependence of $\lambda_{ab}(0)$, $s\gamma$, and $\sqrt{\Phi_0/H_{sp}}$ with T_c. We found that a linear dependence of H_{sp} with $1/\lambda_{ab}^2(0)$ is valid only in the region close to the optimal doping. For higher degrees of doping, H_{sp} is related, in turn, to $1/(s\gamma)^2$. The characteristics of our transport experiments and the fact that these measurements were done on the same sample before and after oxygen annealing, ensure that the only point of γ in the overdoped region is a very good estimation.

The fact that $\sqrt{\Phi_0/H_{sp}}$ follows the $\lambda_{ab}(0)$ dependence close to the optimal doping region means that the electromagnetic limit is obeyed in this region. For higher doping levels $\sqrt{\Phi_0/H_{sp}}$ deviates from $\lambda_{ab}(0)$ indicating that the trend is to a Josephson limit and H_{sp} should be progressively fixed by γ. See the excellent agreement between $s\gamma$ and $\sqrt{\Phi_0/H_{sp}}$ in Fig. 5. The crossover from electromagnetic to Josephson coupling in this system coincides with the minimum value measured for $\lambda_{ab}(0) \sim 2200$ Å and therefore values of $s\gamma$ much larger than this will not scale with H_{sp}. In particular the underdoped samples of this material with larger values of $s\gamma$ would be in the electromagnetic coupling regime, in agreement with the reports of Villard *et al.*[3]

III. CONCLUSIONS

We have shown that the superconducting critical temperature is an excellent parameter to characterize the effects of oxygen annealing due to the fact that T_c is highly correlated with carrier concentration. The c-axis lattice parameter has a slight but monotonous reduction as the oxygen concentration increases. This reduction should enhance the interlayer coupling (and therefore reduce the anisotropy) as the increase in the number of carriers does. However, we are not able to evaluate both contributions separately.

The irreversibility line and the order-disorder transition move toward higher fields just as it was expected from the coupling enhancement in the overdoped region. The larger enhancement of the pinned vortex array region (limited by H_{il}) between the optimally doped and the maximum overdoped sample corresponds to $T/T_c \sim 0.4$.

H_{sp} follows the $\Phi_0/\lambda_{ab}^2(0)$ expression in the optimally doped and slightly overdoped region indicating that the pancake vortices are coupled by electromagnetic interactions. However, for higher doping levels H_{sp} deviates from $\Phi_0/\lambda_{ab}^2(0)$ meaning that the coupling becomes progressively Josephson-like. The crossover from one type of coupling to the other was found to be at $\lambda_{ab}(0) \sim s\gamma \sim 2200$ Å.

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