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Phase intergrowth effects on the magnetic and transport properties of $Bi_2Sr_2CaCu_2O_y$ thin films grown *in situ* by laser ablation

C. Maréchal
Laboratoire de Physique de la Matière Condensée, Université de Picardie-Jules Verne, Faculté des Sciences, 33 rue saint Leu, 80039 Amiens Cedex, France

R. M. Defourneau, I. Rosenman, and J. Perrière
Groupe de Physique des Solides, Universités Paris VI et Paris VII, URA 17, Tour 23, 2 Place Jussieu, 75251 Paris Cedex, France

Ch. Simon
Laboratoire de Cristallographie et Sciences des Matériaux, ISMRA, UMR 6508, 6 Bld Maréchal Juin, 14050 Caen Cedex, France

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$Bi_2Sr_2CaCu_2O_y$ (2212) films, grown *in situ* by laser ablation, show the presence of the intergrowth phenomenon: the random stacking of 2201 ($Bi_2Sr_2Cu_1O_x$) and 2212 cells. The precise fraction of the 2201 phase intergrowth depends on the growth conditions. We have measured the influence of such structural defects on electrical transport and magnetic properties. The main consequences of the intergrowth are a broadening of the superconducting transition and a decrease of the critical temperature. The irreversibility line of the films has been deduced from ac susceptibility measurements in magnetic fields H parallel to the c axis of the films using a superconducting quantum interference device magnetometer. A strong shift of the irreversibility line to low fields and temperatures is reported with increasing the percentage of 2201 stacking faults.
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INTRODUCTION

Numerous experiments probing the mixed state of cuprate superconductors have established the presence of a boundary in the magnetic phase diagram, which separates a magnetically irreversible, zero-resistance state from a reversible state with dissipative electrical transport properties.[1,2] This boundary has been suggested to be due to either depinning,[2–4] or to a vortex-glass formation,[5] or to flux-lattice melting.[6] The electronic and magnetic properties of $Bi_2Sr_2CaCu_2O_y$ in the superconducting state have proved to be rich in physics because of its large anisotropy. One of the subjects that still needs to be clarified is the dimensional crossover in the vortex system with the magnetic field parallel to the c axis. Theoretical studies[7–9] suggest that thermal fluctuations of the pancake vortices[10] lead to a thermally induced dimensional crossover, above which the superconducting layers are effectively decoupled and behave as a two-dimensional system. Such a dimensional crossover has been reported in some experiments on $Bi_2Sr_2CaCu_2O_y$ single crystals as the irreversibility line.[11,12] If the pinning in each layers is sufficiently strong, the correlation between pancake vortices in the c direction is considerably weakened above a crossover field B_{2D}, because the magnetic interaction between pancakes in the same layers becomes stronger than that between pancakes in adjacent layers.[11–14] B_{2D} has been reported to be around 50 mT.[11,12,14–16] An alternative explanation of the irreversibility line, based on geometrical effects, was recently suggested[17] where local vortex dynamics in $Bi_2Sr_2CaCu_2O_y$ single crystals was studied using microscopic GaAs/AlGaAs Hall-sensor arrays. The irreversibility line is found to exist in the absence of bulk pinning. At high temperatures the irreversibility line is due to geometrical barriers, whereas at intermediate temperatures, the irreversible behavior is determined by surface barriers.

In this paper we show that 2201 stacking faults modify the shape and the position of the irreversibility line. We also present resistance measurements and show how 2201 stacking faults reduce the critical temperature of our film compared to that of a pure 2212 film.

EXPERIMENT

Thin $Bi_2Sr_2CaCu_2O_y$ films were grown *in situ* on (001) oriented MgO substrates by pulsed-laser ablation. The details of the preparation process have been previously reported.[18] Briefly, a bulk superconducting target is irradiated with a frequency quadrupled pulsed Nd: YAG laser (B. M. Industries), which provides at 266 nm, 200 mJ pulses with a 7 ns duration at a 5 Hz repetition rate. The laser beam is focused through suprasil quartz window onto the target at a 45° oblique incidence, to give a power density in the 60 to 300 MW/cm^2 range. The emitted material is deposited onto MgO single crystals located 4 cm away. The deposition is carried out under a pure oxygen pressure (0.2 mbar) on heated substrates (700 °C).

The irreversibility line was investigated by ac susceptibility and dc magnetization measurements as already been done.[19,20] ac magnetic susceptibility measurements were performed in dc magnetic fields up to 20 kG parallel to the crystallographic c axis. Superconducting data are obtained using a Quantum Design MPMS2 superconducting quantum interference device magnetometer from 5 to 300 K and in the field cooled conditions. In Fig. 1, the temperature dependence of the real M' and the imaginary M'' parts of the ac

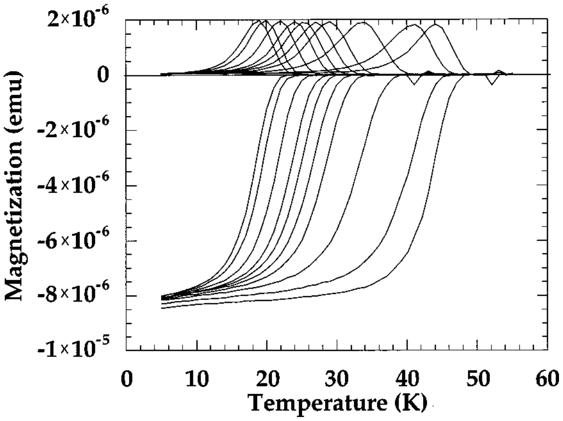

FIG. 1. Real (M') and imaginary (M'') parts of the ac magnetization ($H_{\mathrm{ac}}=0.01$ G, frequency $f=30$ Hz) of a 2212 thin film vs temperature in various dc fields: $H_{\mathrm{dc}}=2$ G, 50 G, 200 G, 500 G, 1 kG, 2 kG, 3 kG, 5 kG, 8 kG, and 10 kG (from right to left).

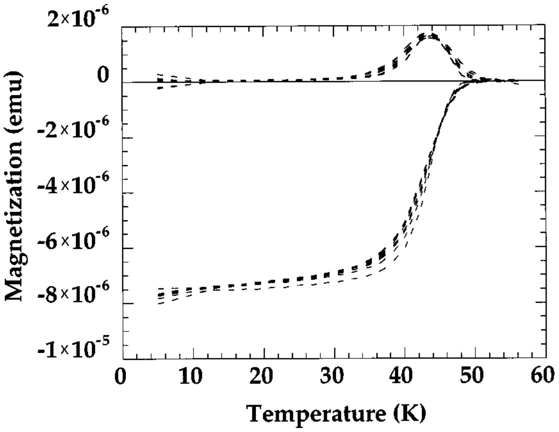

FIG. 2. Superposition of the magnetization curves obtained by a temperature shift $T(H)$ at low fields H_{dc} [see Eq. (2)].

magnetization is shown at different magnetic dc fields for a 2212 thin film. These curves can be superimposed by translating each by a temperature $\Delta T(H)$ that depends on the applied magnetic field:

$$M''[H,T-\Delta T(H)]=M''(H_0,T), \tag{1}$$

where $M(H_0,T)$ stand for the reference curve. The irreversibility field is defined as

$$T(H_{\mathrm{irr}})=T_{0\mathrm{irr}}-\Delta T(H_{\mathrm{irr}}), \tag{2}$$

where $T_{0\mathrm{irr}}$ is the temperature that defines the irreversibility field on the reference curve $M(H_0,T)$. Here, $T_{0\mathrm{irr}}$ is chosen to be the maximum of the imaginary part M'' of $M(H_0,T)$ for the lowest field $H_0\approx 2$ G. The function $\Delta T(H)$ has been evaluated from a mean least-squares fit of M'' around $T\approx T_0$:

$$\chi=\sum_i \{\alpha M''[H,T_i-\Delta T(H)]-M''(H_0,T_i)\}^2, \tag{3}$$

where the sum is over the measured points at H_0. The parameter α provides a better agreement and stays close to 1 within a few percent. The values of $M''[H,T_i-\Delta T(H)]$ are calculated from a cubic spline interpolation from the experimental data. The error on $\Delta T(H)$ is about 0.1 K and mainly depends on the number of measurements. Figure 2 shows the superposition of the ac magnetization at different magnetic dc field curves. At large magnetic fields ($H>5$ kG), there is no more exact superposition because of a broadening of the peak. Nevertheless, $\Delta T(H)$ is still evaluated by the same method.

INTERGROWTH

Intergrowth of the different phases (2201, 2212, and 2223) of the Bi compound is a property of BiSrCaCuO films grown *in situ* by laser ablation, as shown by the shift and the broadening of the x-ray-diffraction peaks.[21] We have used an intergrowth model to simulate the diffraction patterns corresponding to the intergrowth of 2201 or 2223 phases in 2212 films. As shown by Hendricks and Teller and Ranno *et al.*,[21] x-ray-diffraction intensities can be written as

$$I(\theta,p)=|F(\theta)|^2\,\frac{2p(1-p)\{1-\cos[k(d_2-d_1)]\}}{1+p^2+(1-p)^2+2p(1-p)\cos[k(d_2-d_1)]-2p\,\cos(kd_1)-2(1-p)\cos(kd_2)}, \tag{4}$$

where p is the stacking faults percentage of 2201 or 2223 phases, d_1 is the distance between $\mathrm{Bi_2Sr_2O_y}$ subunits of the intergrown phase, and d_2 is the distance of the 2212 phase, $k=4\pi\,\sin\,\theta/\lambda_{\mathrm{Cu}}$ the scattering vector, θ is the Bragg angle, and $F(\theta)$ is the structure factor of the $\mathrm{Bi_2Sr_2O_y}$ subunit. We have fitted our data with Eq. (4), providing a value of p. As a more elaborate model including the intergrowth of the three phases does not improve the rms deviation,[21] we assume a two phase intergrowth model to be enough to explain the experimental data. The Bi/Cu concentration ratios are indeed in very good agreement with the atomic compositions deduced from Rutherford backscattering spectrometry analyses. All these measurements underline the absence of a well-defined stability limit between the different phases of the Bi family, since the intergrowth phenomenon allows one to change the structure continuously from one pure phase to another one. This continuous change is achieved by varying the oxygen pressure at a fixed substrate temperature. In the following we consider only 2201 intergrown films.

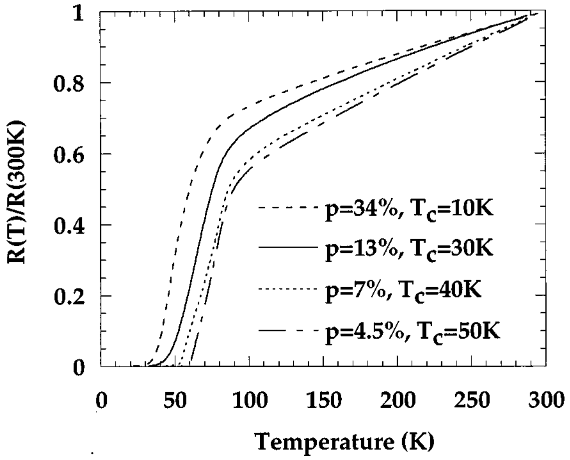

FIG. 3. Variation of the normalized resistance vs temperature for BiSrCaCuO thin films with different percentage p of 2201 stacking faults. The intergrowth broadens the superconducting transition and decreases the critical temperature. As p increases, the slope of the resistivity curve in the metallic regime decreases.

TRANSPORT PROPERTIES

The intergrowth phenomenon has an influence on transport properties. An important effect of the intergrowth appears on the resistivity curves as shown on the normalized resistivity R/R (300 K) of samples with different percentages p of 2201 stacking faults (Fig. 3). The transport resistivity was measured in a standard four-point geometry using gold contacts. The main consequences of the intergrowth are a broadening of the superconducting transition and a decrease of the critical temperature. As p increases, the slope of the resistivity curve in the metallic regime decreases. The decrease of T_c with the percentage of intergrowth is shown in Fig. 4. T_c decreases from 50 K for 4.5% of 2201 stacking faults down to 10 K for more than 30% of 2201 stacking faults.

In order to know the electronic nature of the 2201 layers in 2212 thin films, we have grown pure 2201 thin films by laser ablation, under the same growth conditions as for 2212 films but using a 2201 target. Figure 5 displays the variations of the normalized resistance with the temperature for a pure 2201 thin film and shows an insulating behavior. Thus we expect the 2201 layer in 2212 films to have an insulating behavior. Oxygen deficiency of 2201 layers may change the hole carrier density of 2212 films, lowering the critical temperature. Superconducting (2212)/insulating (2201) superlattices, as (2212)/(2201) or $Y_1Ba_2Cu_3O_7/Pr_1Ba_2Cu_3O_7$, can be considered as films with stacking faults, and a broadening of the superconducting transition and a decrease of the critical temperature were also found in this case.[22–24]

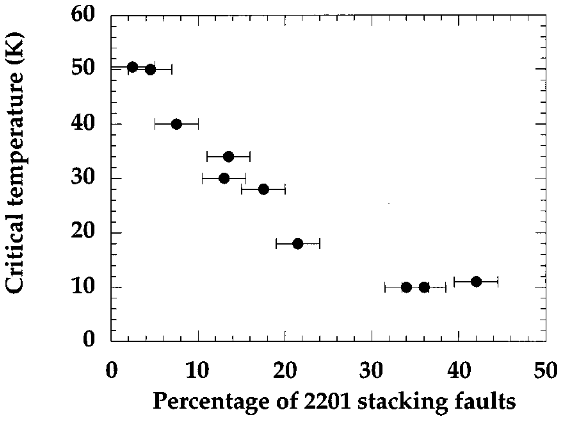

FIG. 4. Decrease of the critical temperature with the percentage of 2201 stacking faults.

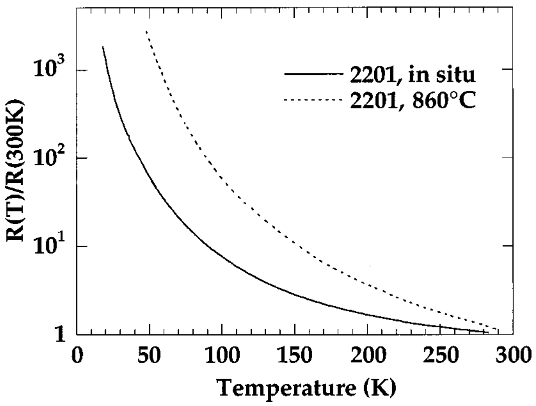

FIG. 5. Variation of the normalized resistance vs temperature for a pure 2201 thin film and the same film after annealing. The curves show an insulating behavior.

MAGNETIC PROPERTIES

The intergrowth phenomenon has a drastic effect on the magnetic transition. As seen in Fig. 6, intergrowth broadens and shifts the magnetic transition to the low temperatures; the critical temperature decreases. ac magnetic susceptibility measurements are performed in dc magnetic fields up to 20 kG parallel to the c axis and lead to the irreversibility line. Each irreversibility line drawn in Fig. 7, in a semilogarithmic plot, corresponds to a precise percentage of 2201 stacking faults. We show two distinct regimes and it appears difficult to fit the curves by the same law over the whole range of temperature. Nevertheless, the temperature dependence of the irreversibility line behavior in the low-field region (<250 G) near T_c can be accurately fitted by power laws. Two

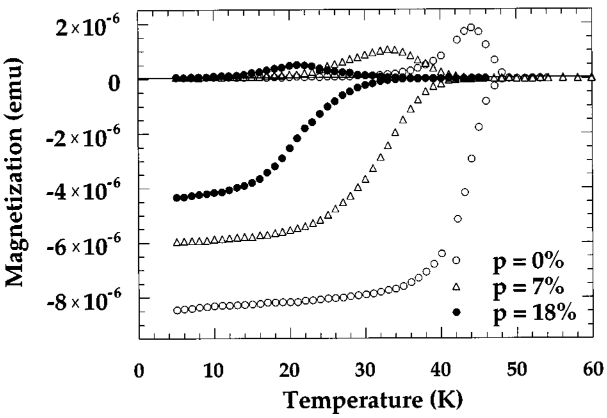

FIG. 6. Real and imaginary part of the ac magnetization (H_{ac}=0.01 G and f=30 Hz) of BiSrCaCuO thin films with different percentage of 2201 stacking faults vs temperature in a 2 G dc field. Intergrowth broadens and shifts the magnetic transition to the low temperatures; the critical temperature decreases.

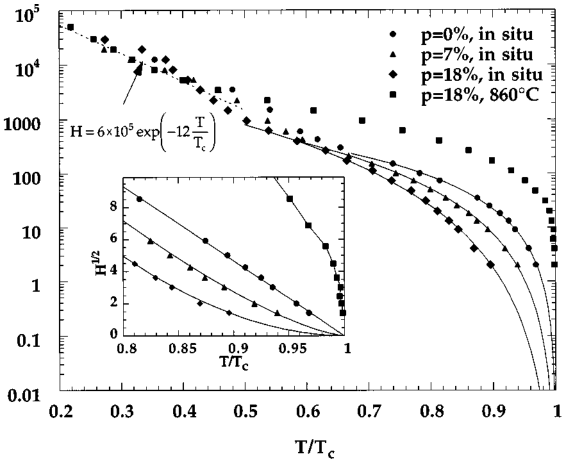

FIG. 7. Irreversibility lines of BiSrCaCuO thin films with different percentage p of 2201 stacking faults and an annealed film (860 °C, under 1 bar of oxygen). Solid lines are the power law fit (6) of the experimental data near T_c, except for the annealed thin film that was fitted by a linear law. The insert displays an enlargement of the low-field region. In this regime ($H<4$ kG), the irreversibility line is shifted to low field and temperature by increasing p and is shifted to much higher field after annealing. All the irreversibility lines merge in the 4–20 kG field range and the dashed line is fited by an exponential law (7).

power laws describe the experimental data (equivalent close to T_c):

$$H = H_0\left(\frac{T_c}{T} - 1\right)^n, \tag{5}$$

$$H = H_0\left(1 - \frac{T}{T_c}\right)^\alpha, \tag{6}$$

where n, α, H_0, and T_c depend on the percentage of 2201 stacking faults. The law (5) describes our results (Table I except for $p = 18\%$. A parabolic dependence [(5), with $n = 2$)] has been proposed for the irreversibility line of Bi-based superconductors.[11,26] This temperature dependence can be explained in the framework of a flux creep model, as has been shown by Yamasaki *et al.*[26] Schilling and co-workers propose that the parabolic dependence is attributable to flux-line melting in 2212 single crystals due to the thermal fluctuations of vortices at low magnetic fields, in the temperature range near T_c. The second power law (6) fits the experimental data of all our samples. α increases with p, while T_c decreases with p as it has been shown before. The power-law fits provide accurate evaluation of the critical temperature of each sample. The results are presented in Table II and in Fig. 7. Without stacking faults the power value $\alpha=2$ is the largest value observed for all high T_c superconductors. It is larger than the value $\alpha=1.5$ predicted by the standard flux creep model, but equal to the value given by the vortex lattice melting model.[2] Zeldov *et al.* have found $\alpha=1$ for $Bi_2Sr_2CaCu_2O_y$ single crystals, consistent with vortex penetration through geometrical barrier.[27] Our exponent is apparently in contradiction with such effects, even if they may lead to a different exponent on thin films.

TABLE I. Fit of the irreversibility line by the law $H = H_0(T_c/T - 1)^n$ (Eq. 5), near T_c, for different percentage of stacking faults.

Percentage of 2201 cells	T_c (K)	n	H_0 (G)
0%	45	1.6	814
7%	33	2.1	966

TABLE II. Fit of the irreversibility line by the law $H = H_0(1 - T/T_c)^\alpha$ (Eq. 6), near T_c, for different percentage of stacking faults.

Percentage of 2201 cells	T_c (K)	α	H_0 (G)
0%	45,2	2	2172
7%	33	2,8	4681
18%	23,7	3,8	11149

In the higher-field region (>3 kG), the irreversibility line can be fitted by an exponential law

$$H = H_0 \exp\left(a\frac{T}{T_c}\right). \tag{7}$$

The results are presented in Fig. 7. An exponential temperature dependence is consistent with vortex penetration through a Bean-Livingston surface barrier by a thermally activated process.[28] The exponential behavior can be also interpreted by the existence of the breakdown field in which a proximity effect induced superconductivity is destroyed.[29] The structure may be seen as a superlattice composed of laminae alternatively normal and superconducting. The normal laminae could be the SrO-BiO-BiO-SrO layers, the superconducting ones being the CuO_2 layers. On the other hand, in the high-field region, Schilling *et al.* find that the exponential behavior is a good approximation to describe the melting of the vortex ensemble in $Bi_2Sr_2CaCu_2O_y$.

As seen in Fig. 7, in the low-fields region ($H<4$ kG), the temperature dependence of the irreversible magnetic field is clearly modified with the proportion of 2201 stacking faults. The irreversibility line is strongly shifted to low fields and temperatures by increasing the proportion of 2201 stacking faults. This effect is stronger near T_c: at $T/T_c = 0.9$, the irreversibility field is 15 times stronger for $p = 0\%$ than for $p = 18\%$. In the high-fields region ($H>4$ kG), the irreversibility line does not depend significantly on the percentage of 2201 stacking faults.

To understand, in the low-fields region, the shift of the irreversibility line to higher fields with the decreasing of 2201 stacking faults, we propose two explanations. Oxygen deficiency of 2201 layers could reduce the hole carrier density and would result in critical temperature reduction. When the 2201 stacking faults increase, the London penetration depth λ of shielding currents in the *ab* direction is expected to increase because the critical temperature is decreased. At the same time the anisotropy parameter γ increases with decreasing carrier density.[14,30] As the irreversibility line depends both on λ and γ, it should be affected by the variation of the carrier density. In this sense, the exponent α of the power law (6) decreases by increasing carrier density. By increasing the carrier doping, experiments have shown that the irreversibility line of the $Bi_2Sr_2CaCu_2O_y$ single crystal is shifted to higher fields over the whole range in temperature.[31] In our case, in the high-fields region ($H > 4$ kG) the irreversibility line does not depend on the carrier density. It should be noted that intergrowth is a random stacking of 2201 and 2212 layers and one can expect that the carrier density is heterogeneous, while for a single crystal, the doping states are homogeneous.

On the other hand, in the low-fields region ($H < 4$ kG), the observed shift of the irreversibility line to low fields and temperatures could be due to the influence of stacking faults on the Josephson coupling between pancakes vortices belonging to adjacent layers. A random stacking of 2201 insulating layers between 2212 layers could weaken the coupling between pancakes. At low inductions, the vortices are expected to form an ensemble of pancake strings with 3D-like fluctuations. Therefore, an increase in p could lead to an enhanced two-dimensional behavior of the system by breaking these strings. Thus the exponent α of the power law (6) increases with the anisotropy of the structure enhanced by the intergrowth phenomenon. A three-dimensional vortex behavior in $Bi_2Sr_2CaCu_2O_y$ thin films at 77 K has been reported.[32] This finding is in line with the theory written by Glazman and Koshelev,[7] where at magnetic fields below B_{2D}, the vortex system in $Bi_2Sr_2CaCu_2O_y$ consists of well-correlated vortex lines rather than layers of decoupled vortex pancakes. Similar recent results on $Bi_2Sr_2CaCu_2O_y$ single crystals show that this behavior is intrinsic to $Bi_2Sr_2CaCu_2O_y$ materials.[11,33,34] At low inductions near T_c, we find a parabolic temperature dependence, while for larger inductions, H decreases exponentially as it has been reported for magnetic fields H parallel to the c axis for $Bi_2Sr_2CaCu_2O_y$ single crystals.[11] The two regimes reflect the three and the quasi-two-dimensional character of the respective vortex fluctuations.[11] For high applied magnetic field, the interaction between vortex objects within one layer is stronger than the coupling between these 2D objects belonging to adjacent layers. This leads to a quasi-2D behavior of the thermal vortex fluctuations. In the high-fields region ($H > 4$ kG), we find that the irreversibility line does not depend on the percentage of 2201 stacking faults. Indeed, insulating 2201 layers could not weaken further the coupling between pancakes along the c axis.

Results obtained on annealed (860 °C, under one bar of oxygen) thin films could suggest that the observed shift of the irreversibility line is related to the carrier density, rather than to the coupling between pancakes. As seen in Fig. 7, in the low-fields region ($H < 4$ kG), the irreversibility line of an annealed thin film is strongly shifted to higher fields. The annealed film contains 18% of 2201 stacking faults as before annealing (x-ray-diffraction measurements). To know the electronic nature of 2201 stacking faults in the annealed film, we anneal (860 °C, under one bar of oxygen) pure 2201 thin films grown by laser ablation, under the same growth conditions as for 2212 thin films, but using a 2201 target. Figure 5 displays the variations of the normalized resistance with the temperature for a pure 2201 annealed thin film and shows again an insulating behavior. Despite 2201 insulating stacking faults, the irreversibility line of the annealed film is shifted to higher fields. Thus, this shift is not related to the coupling between pancakes but to the carrier density that increases during the annealing at 860 °C under 1 bar of oxygen. Oxygen deficiency of the 2212 layers could be compensated by forming superconducting 2201 layers as has been reported by Li *et al.*,[22] or by annealing thin films. As seen in Fig. 8, the critical temperature of the thin film increases after annealing and reaches 76 K. This value is deduced from a linear extrapolation of the irreversibility line. We note that in the 55 to 75 K temperature range, the irreversibility line shows a power-law dependency (6), with an exponent α decreasing from 3.8 before to 1.85 after annealing. In this sense, as seen before, the exponent decreases by increasing carrier density. At lower temperature (18 to 28 K range), the irreversibility line is described by an exponential law (7). As seen in Fig. 7, all the irreversibility lines merge in the 4 to 20 kG field range. In the high-fields region, the thermal vortex fluctuations present a 2D behavior[11,12] where the anisotropy no longer plays a role. The anisotropy that varies with the carrier density,[14,30] controls the irreversibility line. Thus, in the high-field region, the irreversibility line should not shift either by intergrowth or by annealing.

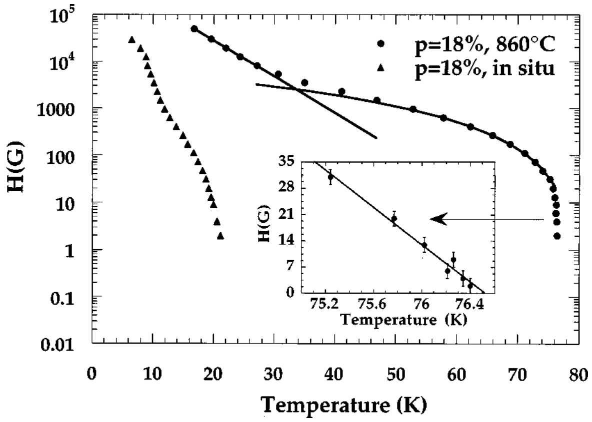

FIG. 8. Irreversibility line before and after annealing. One sees the dramatic increase of the critical field after annealing. The insert shows the linear dependence of the line at low field near the critical temperature.

CONCLUSION

The intergrowth phenomenon reduces the superconducting properties of 2212 films. The random stacking of 2201 insulating layers and 2212 layers broadens the magnetic transition and the resistivity curves. By increasing the percentage of 2201 layers, the critical temperature decreases. Intergrowth shifts the irreversibility line to low fields and low temperatures because it would reduce the hole carrier density of the film. As the irreversibility line is strongly shifted to the higher fields and the critical temperature increases for an annealed thin film, the transport properties appear to be better in annealed thin films than in *in situ* grown films.

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