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Angular dependence of the irreversible magnetization of $YBa_2Cu_3O_7$ superconducting thin films

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$YBa_2Cu_3O_7$ superconducting thin films have been studied by magnetization measurements in oblique fields up to 0.8 T both at low temperature and at temperatures close to T_c. Both components of magnetization parallel and perpendicular to the applied field could be measured with two pairs of crossed detection coils. In all cases the magnetization is found to be perpendicular to the film plane. This is due to the self-field effects related to the thin-film geometry which force the critical currents to circulate along the film plane. At low temperature the properties are found to depend only on the component of the applied magnetic field perpendicular to this plane, as expected in the situation where demagnetizing field effects are dominant. However, at temperatures close to T_c the critical currents and therefore the pinning are found to increase when the applied field approaches the film plane. This is attributed to the effect of intrinsic pinning which may be related to the kink structure of the vortices in oblique fields. [S0163-1829(96)03845-3]

I. INTRODUCTION

Superconductivity at high temperature was discovered nearly ten years ago in copper-based oxides.[1] Since then, much effort has been put in the study of thin films, mainly of the compound $YBa_2Cu_3O_7$.[2] This was largely motivated by the high critical currents which could be obtained comparatively easily in the thin-film geometry and by the possibility of developing applications. Ten years later, the origin of these high critical currents and therefore of vortex pinning in thin films is still not well understood, even if several mechanisms such as the presence of twin planes or of screw dislocations have often been invoked.[3]

Most of the studies performed on thin films have been devoted to the transport properties with or without magnetic field and to the determination of critical currents using etching techniques in order to obtain narrow tracks. Few studies deal with the magnetic properties which however do not require the preparation of current tracks and probe the whole film. Magnetization measurements give easy access to the critical state induced by a magnetic field, therefore to the superconducting properties in the presence of currents close to the critical current J_c. The geometry is obviously different in the case of transport measurements since in this case the direction of the current is imposed by the geometry of the contacts.

One of the major problems related to the thin-film geometry is due to the self-fields (of the same origin as demagnetizing fields in magnetic materials) which induce strong shape anisotropy and make difficult the studies of intrinsic properties.[4,5] Since the irreversible magnetization is directly related to the critical current J_c, the evaluation of J_c is in this case comparatively easy, especially at low temperature when J_c is large. The situation is different at temperatures close to T_c where the magnetic moment and therefore the self-fields effect are much weaker. However in this case, since the shape anisotropy should be smaller, it may be possible to study intrinsic properties such as intrinsic pinning anisotropy. The aim of this article is to report results of the magnetic properties of $YBa_2Cu_3O_7$ thin films in oblique fields obtained both at low temperatures and at temperatures close to T_c.

In Sec. II, the experimental techniques including thin-film characterization and the description of the magnetization measurement apparatus will be described. In Sec. III, the theoretical background is summarized and some experimental results corroborating the theoretical models for the case of perpendicular fields will be reported. Section IV is devoted to the results obtained in oblique fields. They demonstrate that in the low-temperature case shape effects are always dominant, whatever the orientation of the applied field is. The results obtained at temperatures just below T_c show that the situation is different from the low-temperature one and that intrinsic effects may appear in this temperature range.

II. EXPERIMENTAL TECHNIQUES

Most of the results reported in this article have been obtained on thin films prepared by laser ablation at the University of Augsburg.[6] The films were deposited on substrates of $LaAlO_3$ and the thickness was 350 nm. The transition temperature was 91 K and the critical current of the order of 10^{11} A/m^2 at 4.2 K. Detailed crystallographic studies performed using grazing incidence x-ray diffraction[7] will be reported elsewhere.[8] The $LaAlO_3$ substrates have a rhombohedral (pseudocubic) symmetry and become twinned after the film deposition and the related heat treatment. They are cut in such a way that a pseudocubic [001] axis is perpendicular to the substrate plane. The lattice mismatch with respect to $YBa_2Cu_3O_7$ is 1.5%. The deposited films, oriented with the c axis perpendicular to the substrate, are found to be faceted as a consequence of the substrate twinning. The $\langle 110 \rangle$ crystallographic axes of the film and of the substrate are found to be aligned. The films are twinned with $\{110\}$ twin planes parallel to the $\{110\}$ planes of the substrate. In consequence, there are two equivalent orientations of the twin planes and four types of crystallites. The orthombicity of the film is found to be smaller than that of stoichiometric single crystals (b-a

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=0.055 Å to be compared to 0.065 Å in single crystals). This is probably due to the strain induced by the substrate. The in-plane mosaicity parameter (full width at half maximum of the (200) x-ray line) is found to be small, of the order of 0.5° (to be compared to 0.9° for films deposited on MgO substrates).

Some results have been obtained on films deposited by coevaporation at the Defence Research Agency (Malvern U.K.).[9] The thickness was also 350 nm, the transition temperature 89 K, and the critical current in the range of 10^{11} A/m^2 at 4.2 K. As mentioned earlier, the film mosaicity was much larger than for $LaAlO_3$ substrates, in consequence of the larger lattice mismatch (8.6% between MgO and $YBa_2Cu_3O_7$).

Magnetic measurements have been performed with two vibrating sample magnetometers built in the laboratory. The first one, equipped with a superconducting coil providing a field up to 6 T and with a sensitivity of 10^{-8} A m^2 (unit of magnetic moment), was used for measurements in perpendicular fields between 4.2 K and T_c. In the second one, a magnetic field up to 0.8 T is produced by a rotating magnet. Measurements can be made for different angles θ between the film plane and the applied magnetic field between 20 K and T_c. The angle accuracy is 0.1° and the sensitivity 10^{-7} A m^2. Two pairs of detection coils with perpendicular axes allow measurements of the components of the magnetization parallel and perpendicular to the applied field. The rate of change of the magnetic field is kept constant equal to 1.5 mT/s for all experiments.

III. THEORETICAL BACKGROUND

The problem of the relative importance of intrinsic anisotropy and of shape anisotropy for the irreversible magnetization of flat superconducting samples has become important with the discovery of the high-T_c superconducting oxides. These materials show indeed both an intrinsic anisotropy due to their layered crystal structure and shape anisotropy due to the platelet shape of the available single crystals or to the thin-film geometry. In this context, it was shown five years ago by Hellman *et al.* that the irreversible magnetization of superconducting Nb samples is oriented perpendicular to the platelet (or film) plane when the aspect ratio is larger than 10/1.[10]

However, it is only recently that the problem of flux penetration in thin films in the presence of magnetic fields perpendicular to the film plane has been solved analytically, assuming a field-independent critical current.[11,12] The deformation of the flux lines in the vicinity of the sample induces a curvature of the vortices inside the film. The radial field at the surface is of the order of the applied field in low applied fields. The critical current J_c is then related to the field gradient across the thickness and therefore to the vortex curvature. It is of the order of H_a/d, where H_a is the applied field and d the film thickness. This is different from the result of the classical Bean model for parallel geometry where J_c is related to the radial field gradient. Further calculations have been performed by Brandt for the case of time-dependent magnetic fields and in the presence of transport currents.[13] A very complete theory of magnetization and transport currents in thin superconducting films has also been developed more recently by Zeldov *et al.*[14] All these calculations agree with the results of Refs. 11 and 12 corresponding to the more simple case of only dc transverse fields.

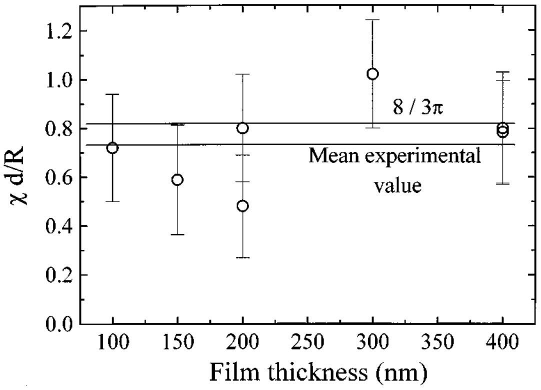

FIG. 1. Initial susceptibility at $T=4.2$ K multiplied by the geometrical factor d/R as a function of the film thickness d. R is the average film radius. Films elaborated by laser ablation on $SrTiO_3$ substrates. (From Refs. 12 and 13).

According to these calculations, the initial susceptibility is found to depend only on a geometrical factor, the ratio of the film radii to the film thickness ($\chi=8R/3\pi d$). Complete flux penetration is obtained when the applied field reaches a value H^*, related to the critical current J_c and to the film thickness ($H^*=J_c d$). The value M_r^{sat} of the saturated remanent magnetization obtained after applying a high field is the same as in the classical Bean model for parallel geometry ($M_r^{\text{sat}}=J_c R/3$).

Previously obtained experimental results[15,16] are well accounted for by these recent calculations. Figure 1 shows the ratio $\chi d/R$ as a function of thickness for films of different thicknesses obtained by laser ablation. The average value of this ratio is clearly close to the theoretical value. Another confirmation of these models is given by the experimental dependence of the remanent magnetization M_r as a function of the maximum applied field H_a^m during an hysteresis loop, as shown in Fig. 2. The curve of the ratio M_r/M_r^{sat} vs H_a^m/H^* is perfectly fitted by the formula calculated from the results of Zhu *et al.*[12]

$$\frac{M_r(H_a^m)}{M_r^{\text{sat}}}=\frac{4}{\pi}\left[-\frac{1}{2}S\left(2\frac{H_a^m}{H^*}\right)+S\left(\frac{H_a^m}{H^*}\right)\right],$$

where

$$S(x)=\arccos\left[\frac{1}{\cosh(x)}\right]+\frac{\sinh|x|}{\cosh^2(x)}.$$

This formula takes into account a more realistic current distribution for decreasing fields than the model of Mikheenko and Kuzovlev.[11]

The situation is much more complicated in oblique fields in the case of thin films since both the intrinsic anisotropy related to the layered structure of high-T_c superconductors and the self-field effect should be taken into account. For bulk layered superconductors, theory predicts in oblique fields the existence of pancake vortices in the CuO planes

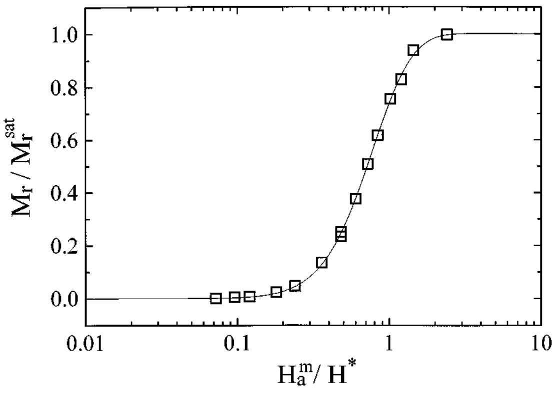

FIG. 2. Normalized remanent magnetization M_r/M_r^{sat}, as a function of the normalized maximum applied field H_a^m/H^* obtained at the temperature of 10 K. M_r^{sat} is the saturated remanent magnetization obtained after applying a maximum field of 6 T and H^* is the full penetration field. (From Refs. 12 and 13). The squares are experimental data and the line the results of the model of Zhu *et al.* (Ref. 12).

connected by Josephson strings running along the *ab* plane.[17,3,4] When the angle θ between the *ab* plane and the magnetic field is close to 0, the flux lines therefore form kinks. If pinning is not too strong, they may switch into the *ab* plane for a critical value θ_c. This corresponds to the so-called lock-in transition. θ_c has been predicted to be 4° for $YBa_2Cu_3O_7$.[17]

In the thin-film geometry, demagnetizing fields become important. Brandt predicts that the perpendicular component of the applied field will penetrate the film for angles θ larger than $(1-N)\ B_{c1}^{z}/B_{a\perp}$.[4] The demagnetizing coefficient N of thin films is typically of the order of $1-\epsilon$, with $\epsilon=10^{-4}$ and $B_{c1}^{z}=0.17$ T for $YBa_2Cu_3O_7$. This leads in any case to limiting values of θ very close to 0 (typically 10^{-4} degree) and indicates that the perpendicular component of B will practically always penetrate a thin film. However no detailed theory is available for the complicated situation of a layered superconductor in the thin-film geometry in the presence of oblique fields. There is only one assumption that can be made for the critical state: since the critical current has to circulate along the *ab* plane because of shape effect, in all cases the magnetization will be perpendicular to the film plane, as demonstrated by Hellman *et al.* in the case of Nb thin films.[10] Possible currents with other geometries would in any case give negligible (nonmeasurable) contributions to the magnetization.

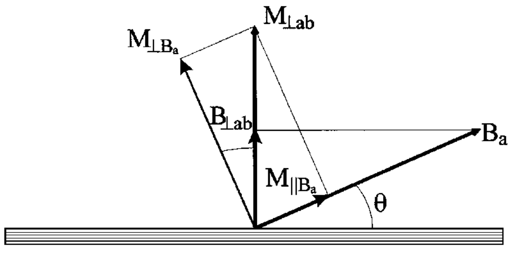

FIG. 3. Scheme of the field and magnetization orientations. B_a is the applied field. The magnetization, $M_{\perp ab}$, is assumed to be oriented along the perpendicular to the film plane which is also the *c*-crystallographic axis.

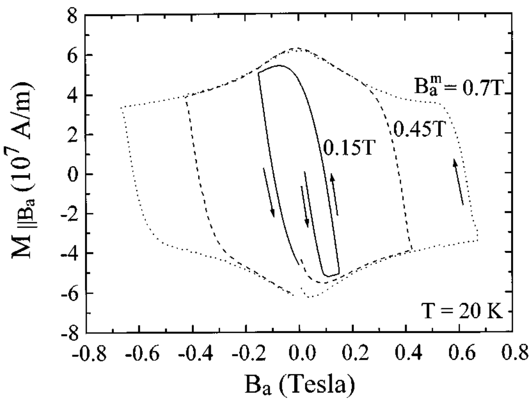

FIG. 4. Hysteresis loops obtained at 20 K with perpendicular applied fields of maximum amplitude B_a^m. Film *A* prepared by coevaporation on a MgO substrate (see text and Ref. 9).

IV. EXPERIMENTAL RESULTS AND DISCUSSION

A. Magnetization in oblique fields at low temperatures ($T \ll T_c$)

All data reported here have been obtained at 20 K after cooling the sample in zero field. As shown in Fig. 3, both the component of the magnetization parallel to the applied field, $M_{\parallel B_a}$, and the perpendicular one, $M_{\perp B_a}$, can be measured. First, we give as reference typical hysteresis loops obtained in the simple perpendicular geometry ($B_a \perp$film plane) for several values of the maximum applied field B_a^m (Fig. 4). One notes that in large fields, the increasing (or decreasing) branches of the loops are superimposed for different values of B_a^m (except for the region where the field is reversed). Figure 5 shows for the same sample hysteresis loops for various angles θ, obtained with the values of the component of the magnetization parallel to the applied field, $M_{\parallel B_a}$, vs B_a. Typical results for the hysteresis loops obtained by plotting now $M_{\perp B_a}$ vs B_a are shown in Fig. 6(a) for different angles θ. Since we assume that the magnetization is perpendicular to the film plane and since it is likely that due to shape effects only the component of B_a perpendicular to this plane is effective, one may plot $M_{\perp ab}=M_{\perp B_a}/\cos\theta$ vs $B_{\perp ab}$, as shown in Fig. 6(b). The corresponding loops are very similar to those of Fig. 4. One should note that the initial

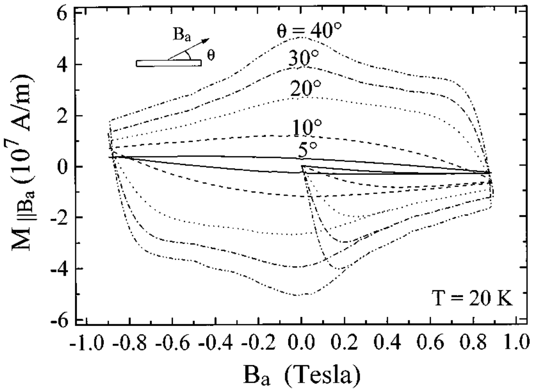

FIG. 5. Hysteresis loops obtained at 20 K by plotting the component of the magnetization along the applied field B_a, $M_{\parallel B_a}$, as a function of B_a, for different angles θ between B_a and the film plane (Film *A*).

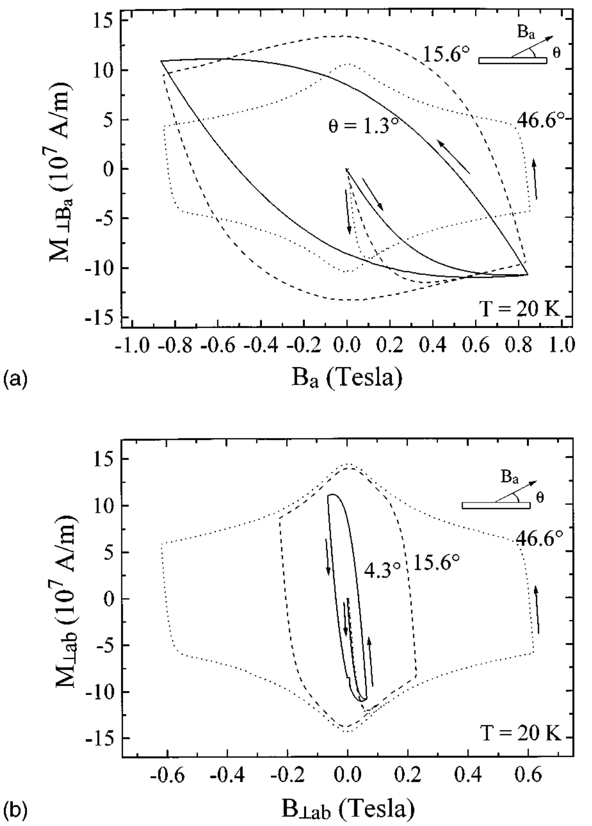

FIG. 6. (a) Hysteresis loops obtained at 20 K by plotting the component of the magnetization perpendicular to the applied field B_a, $M_{\perp B_a}$, as a function of B_a, for different angles θ between B_a and the film plane. (Film B obtained by laser ablation on a $LaAlO_3$ substrate, see text and Ref. 6). (b) Same as (a) obtained by plotting the full magnetization, $M_{\perp ab}=M_{\perp B_a}/\cos\theta$ vs $B_{\perp ab}$, the component of the applied field perpendicular to the film plane.

susceptibility $M_{\perp ab}/B_{\perp ab}$ is found equal to the value obtained in perpendicular field, $\chi=8R/3\pi d$. The increasing and decreasing branches of the loops are also superimposed, when fields larger than the full penetration field have been applied. This is a confirmation of our hypothesis.

One may also check that the remanent magnetization is always perpendicular to the film plane. For that, one records a hysteresis loop with an applied field making a given angle θ with the film plane and then one decreases the applied field to zero. The component of the remanent magnetization along the pickup coils axis is then measured as a function of the angle ϕ between the axis of these rotating coils and a reference direction. This is shown in Fig. 7 for previous maximum applied fields of 0.8 T. Obviously, the component of the remanent magnetization along this axis $M_r^{\parallel}$ follows a law of the type cos ϕ. It is saturated for angles θ larger than 10°. The cos ϕ law is valid for all angles θ and establishes that in all cases the remanent magnetization is aligned along a direction perpendicular to the film plane independent of the angle θ. Similar results had been obtained in Ref. 18.

The previous results corroborate that at low temperature, the magnetization is always perpendicular to the film plane, even for angles between the applied field and this plane as small as 1°. They also demonstrate that M and therefore the critical state depends only on the component of the magnetic field perpendicular to the film plane. This is a consequence of the strong demagnetizing field effects which dominate the properties at low temperatures. However these results do not allow us to predict the structure of the vortices in oblique fields. One may only suggest that, since the properties at low temperature do not seem to be affected by the component of the applied field parallel to the film plane, the vortex properties may depend only on the perpendicular component, as a consequence of these strong shape effects.

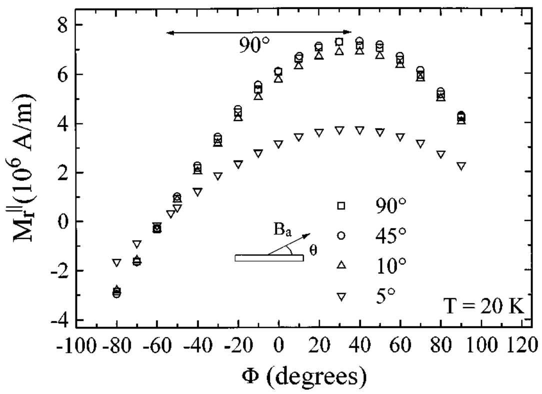

FIG. 7. Component of the remanent magnetization, $M_r^{\parallel}$, along the axis of the rotating pickup measurement coils, as a function of the angle ϕ between this axis and a reference direction. Curves obtained at 20 K for different angles θ between the applied field B_a, which has previously induced this remanent magnetization, and the film plane (Film B).

One should now compare these results with data obtained on bulk materials. LaSrCuO single crystals have been recently studied at low temperature with a similar technique.[19] In the case of single crystals, secondary peaks are observed on the hysteresis loops (see also Ref. 20, and references therein). These peaks are obviously absent in the case of thin films. Another difference concerns the low-field behavior: in single crystals, the magnetization is not parallel to the c axis for applied fields away from the c axis. This is attributed to the penetration of vortices along the ab plane as predicted theoretically for layered superconductors.[20] Such an effect is absent in thin films at low temperatures as a consequence of large demagnetizing field effects.

B. Magnetization in oblique fields at high temperatures ($T \leq T_c$)

At temperatures close to T_c the critical currents and therefore the magnetization are weak. The shape effect may become negligible and one may expect to detect intrinsic properties of the material. The experimental studies are made difficult by the weakness of the magnetic moment to be measured. Since it is likely that in all cases the component of the magnetization parallel to the film plane is zero within the experimental accuracy, one has to measure only the magnetization along the c axis, whatever the angle θ is. This is made possible with a magnetometer having two pairs of crossed pickup coils. The results of such a study are given for values of θ between 0.3° and 9° at 80 K in Fig. 8. One should note that for θ larger than approximately 1°, the remanent magnetization is saturated, which shows that the film is fully penetrated by vortices in the remanent state. To compare the properties with the low-temperature ones, one

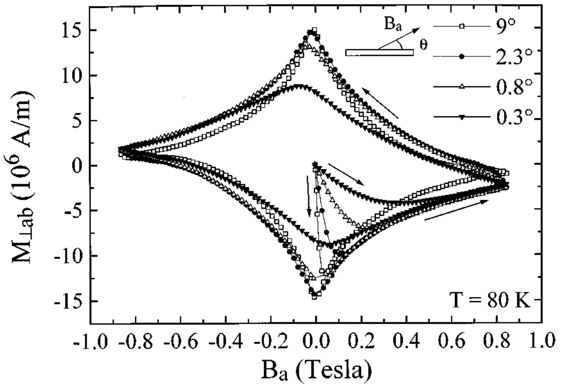

FIG. 8. Hysteresis loops obtained by plotting the magnetization (perpendicular to the film plane), $M_{\perp ab}$, as a function of B_a, for angles θ between 0.3° and 9° between B_a and the film plane, at the temperature of 80 K (Film *A*).

should now plot $M_{\perp ab}$ vs the component of B perpendicular to the film plane $B_{\perp ab}$. This is shown in Fig. 9 for small values of θ. The curves are superimposed in low fields, which indicates that the initial flux penetration in low fields is controlled only by $B_{\perp ab}$, as in the low-temperature case. On the contrary, in high fields, the curves are not superimposed: this shows that in this case the critical state depends on the value of B_a, which is different from the situation at low temperature. To corroborate this result, one has measured hysteresis loops, $M_{\perp ab}$ vs $B_{\perp ab}$ for $\theta=0.5°$ and 90°, with the same values of $B^m_{\perp ab}$ (8×10^{-3} T) and the same rate of change $\partial B_{\perp ab}/\partial t$ of the field (Fig. 10). The two loops are superimposed only for the initial part at low fields, which means that the mechanism of penetration of vortices is the same at both angles and that it depends only on $B_{\perp ab}$, just as at low temperatures. The other parts of the loop are completely different and this indicates that the value and orientation of the applied field may now be important, at variance with the low-temperature situation. From such data, one can extract a field $B^p_{\perp ab}$ corresponding to the maximum of the initial magnetization $M_{\perp ab}$. This field is close to the full penetration field. $B^p_{\perp ab}$ is plotted as a function of θ at different temperatures in Fig. 11(a). It shows an increase vs θ at intermediate and high temperatures. If one assumes that the predictions of the perpendicular model are still valid in this low-field regime, $B^p_{\perp ab}$ should be proportional to the critical current at $B=B^p$ ($B^p_{\perp ab}=\mu_0 J_c d$). The θ dependence of $B^p_{\perp ab}$ would then be correlated to the field dependence of J_c. The curve of $B^p_{\perp ab}$ vs B^p would correspond to the curve of

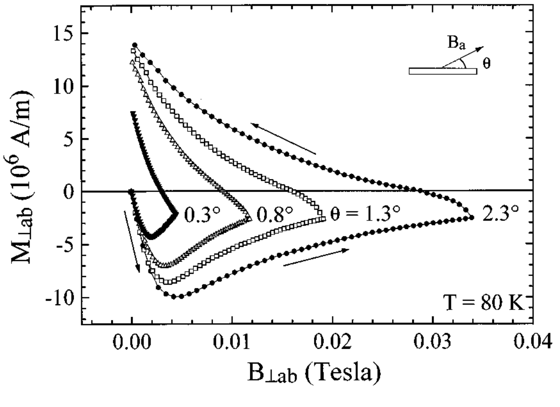

FIG. 9. Half-hysteresis loops obtained at 80 K by plotting the magnetization (perpendicular to the film plane), $M_{\perp ab}$, as a function of $B_{\perp ab}$, for angles θ of 0.3° to 2.3° between B_a and the film plane (Film *B*).

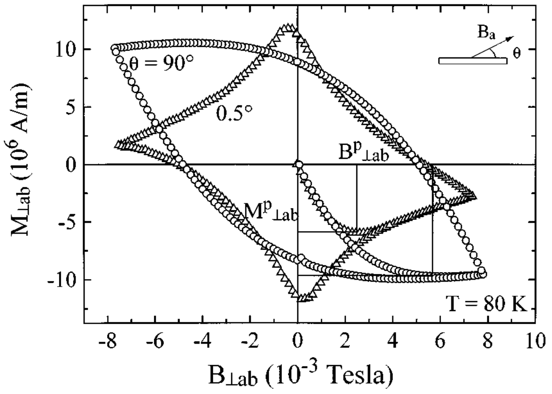

FIG. 10. Hysteresis loops obtained at 80 K by plotting the magnetization (perpendicular to the film plane), $M_{\perp ab}$, as a function of $B_{\perp ab}$, for angles θ of 0.5° and 90° for the same maximum value of $B_{\perp ab}$ (Film *B*).

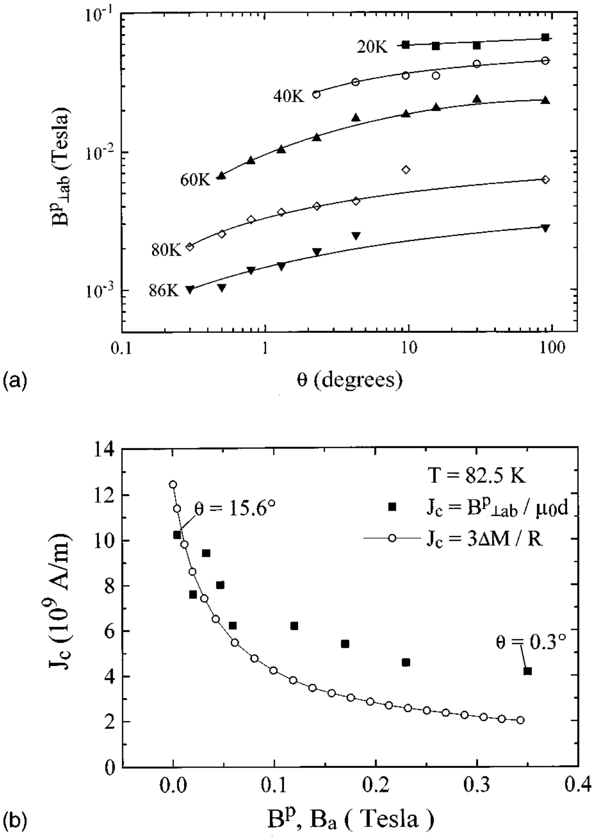

FIG. 11. (a) Value of the component of the applied field perpendicular to the film plane $B^p_{\perp ab}$ corresponding to the maximum of the initial magnetization, as a function of the angle θ. $T=80$ K (Film *B*). (b) Critical current evaluated from the values of the field $B^p_{\perp ab}$ (full squares) vs B^p (see text) and from the irreversible magnetization (open circles) vs the applied field B_a.

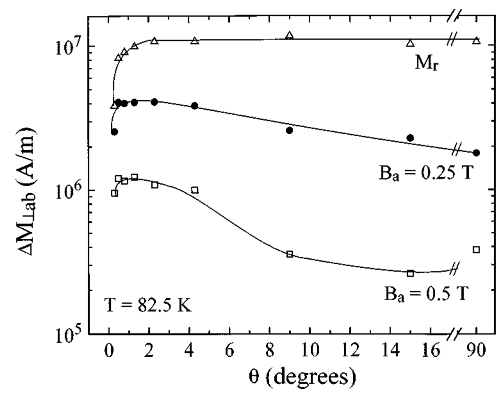

FIG. 12. Irreversible magnetization, $\Delta M_{\perp ab}$, as a function of the angle θ for several values of the applied field B_a. T=80 K. The upper curve shows the remanent magnetization M_r vs θ (Film B).

J_c vs B. Figure 11(b) shows the critical current evaluated from $B^p_{\perp ab}$ vs B^p and the values obtained from the irreversible magnetization vs the applied field. The two sets of data are consistent for low values of field, therefore for large values of θ. This indicates that the perpendicular field model applies indeed only when the perpendicular component of the applied field is large enough.

One may also obtain from these data the initial susceptibility

$$\chi = \mu_0 M_{\perp ab} / B_{\perp ab},$$

in the limit of low fields, at temperatures between 20 and 87 K and for θ between 0.3° and 90°. χ is found to be approximately independent of the temperature and of the angle and, within the experimental accuracy, close to the expected value of $8R/3\pi d$ (2280 for the measured sample) in the framework of the perpendicular field model.

Figures 9 and 10 show clearly that, at temperatures close to T_c, the curves of $M_{\perp ab}$ vs $B_{\perp ab}$ depend on the angle θ except in the initial magnetization regime. One may therefore assume that, in this regime, the critical state depends strongly on the applied field B_a and not only on $B_{\perp ab}$. It is therefore tempting to plot the irreversible magnetization $\Delta M_{\perp ab}$ (half height of the hysteresis loop) vs the angle θ for different values of the applied field B_a, as shown in Fig. 12 for B_a=0.25 and 0.50 T at T=82.5 K. The remanent magnetization (case B_a=0) is also shown for comparison. One notes first that there is in all cases a decrease of the irreversible magnetization at angles smaller than approximately 1°. At higher angles, while the remanent saturated magnetization is angle independent, $\Delta M_{\perp ab}$ clearly decreases with increasing value of θ for nonzero values of the applied field.

We will first discuss the small angle regime. Looking first at the remanent magnetization and at Fig. 9, it is obvious that the decrease of M_r at small angles is due to the fact that the perpendicular component of the applied field was not large enough to saturate the remanent magnetization. The same argument applies for the cases B_a=0.25 and 0.50 T. The situation is different at larger angles: we suggest that the increase of $\Delta M_{\perp ab}$ and therefore of the critical current at angles smaller than approximately 8° may be due to the effect of intrinsic pinning when the applied field approaches a parallel to the CuO layer ab plane. This effect is very likely competing with the demagnetizing shape effects. Therefore it appears only at intermediate fields when the critical current and then the magnetization are weak enough to make the demagnetizing field negligible. At higher fields, the sensitivity of the apparatus is not high enough to detect the magnetization at that temperature.

One should note that the above result is probably based on the same mechanism as the anisotropy of the transport critical current reported by Roas, Schultz, and Saemann-Ischenko.[22] We suggest that it is possibly due to the kink structure of the vortices in the presence of oblique applied fields. When the angle θ is approaching zero, the length of the portion of the vortices of the Josephson type, parallel to the layers, is increasing and therefore the effect of the intrinsic pinning would be getting stronger.[17,21]

V. CONCLUSION

This article is focused on the vortex properties of high-T_c $YBa_2Cu_3O_7$ thin films studied by magnetization measurements in oblique fields. We have verified that at all temperatures, the magnetization is perpendicular to the film plane (ab-crystallographic plane), as expected since the critical currents are forced to circulate along this plane as a consequence of the shape anisotropy. At low temperatures, the properties are dominated by large self-field effects and the magnetization depends only on the perpendicular component of the applied field.

The situation is different at temperatures just below T_c. In this case, when the applied field is oriented close to the film plane, the demagnetizing field effects compete with the intrinsic properties of the material: we have demonstrated that the critical current and therefore the vortex pinning are increasing when the angle θ between the film plane and the external applied field is smaller than approximately 8°. This is attributed to the kink structure of the vortices and to the increase of the length of the Josephson portions which are pinned between the CuO layers.

Further work involves $YBa_2Cu_3O_7$-$PrBa_2Cu_3O_7$ multilayers. Preliminary results indicate that the above properties may be enhanced in multilayers and may depend on the coupling between the superconducting layers.

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