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An alternative method for determination of the lock-in angle in twinned superconductors

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An alternative method for determining the lock-in angle φ_L for pinning of the vortices on extended defects has been developed. This method does not require any preassumed criterion for defining φ_L. Highly twinned $Sm_{1+x}Ba_{2-x}Cu_3O_{6+y}$ single crystal was used for demonstrating the method. Appropriate scaling of the hysteresis loops measured for different angles between the field and twin planes in highly twinned SmBaCuO single crystal led to a clear discrimination between two vortex dynamics regimes. From this scaling, the lock-in angle was determined to be $6° \pm 0.1°$ for the single crystal investigated. This method significantly reduces the uncertainty in determining the lock-in angle when compared to all the other currently employed methods. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2171772]

I. INTRODUCTION

The investigation of flux pinning by twin planes has attracted considerable attention, since the naturally formed twin planes occur regularly in Y123 crystals.[1–3] One of the characteristic properties of vortex pinning by twin planes is a cusplike dependence of the irreversibility field (H_{irr}) on the angle φ between the twin plane and the magnetic field. The cusp appears around $\varphi=0°$, within the angular range φ_L.[4–6] The angle φ_L is called the lock-in angle. The measurement of the angular dependence of H_{irr} has been the principal method used to study the locking of magnetic vortices by twin planes and the determination of φ_L. However, the pinning by twin planes is effective only at high temperatures close to T_c.[7–9] Unfortunately, the determination of H_{irr} at these temperatures is subject to a high degree of uncertainty. Because of this, it is highly desirable to develop an alternative method for obtaining φ_L, which relies on the universal physical principles instead of arbitrary definitions. Such a method is presented in this work. It is based on the scaling of magnetic hysteresis loops, measured with different angles φ between magnetic field and the crystalline c axis. The scaled hysteresis loops fall into two distinct families, one for $\varphi < \varphi_L$ and the other for $\varphi > \varphi_L$, with a transition at sharply defined angle φ_L.

II. EXPERIMENT AND MICROSTRUCTURE

The preparation procedure for the present type of crystal with the chemical formula $Sm_{1+x}Ba_{2-x}Cu_3O_{6+y}$ ($x=0.04$) can be found elsewhere.[10] The crystal studied in the present work has a rectangular geometry and a size of $0.514 \times 1.773 \times 2.101$ mm^3. It is a highly twinned sample with the value of the twin spacing ranging from a few micrometers down to a few tens of nanometers, as obtained by both optical light microscopy techniques [Fig. 1(a)] and bright field transmission electron microscopy (TEM) [Fig. 1(b)]. The twinned interfaces can clearly be seen [arrowed in Fig. 1(b)] in the TEM image. Figure 2 is a selected area electron-diffraction pattern from this phase. Analysis of this pattern is consistent with it being the [001] zone axis of an orthorhombic crystal structure. The (110), (220), etc., reflections are marked. It can be seen that there is splitting of the reflections along this

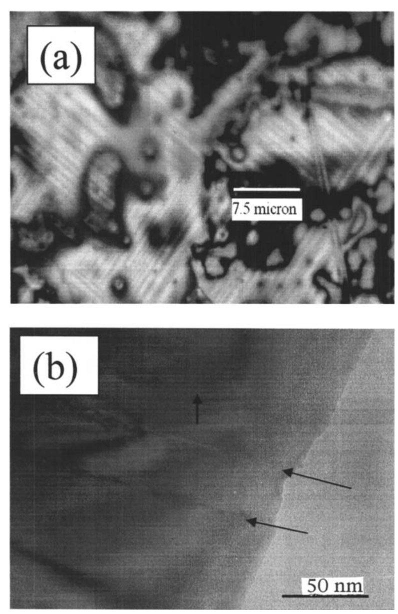

FIG. 1. (a) Optical micrograph of the Sm123 single crystal, which is highly microtwinned. The scale bar indicates 7.5 μm. (b) The presence of twins in the Sm123 sample was also confirmed by bright field transmission electron microscopy (marked by arrows).

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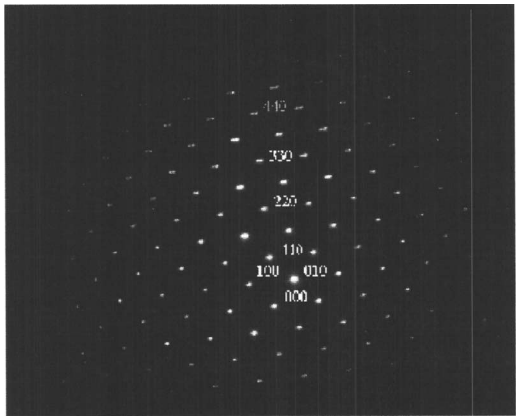

FIG. 2. [001] selected area electron-diffraction pattern. The (110), (220), etc., reflections are marked. Splitting of these reflections is consistent with the presence of twinning in this phase.

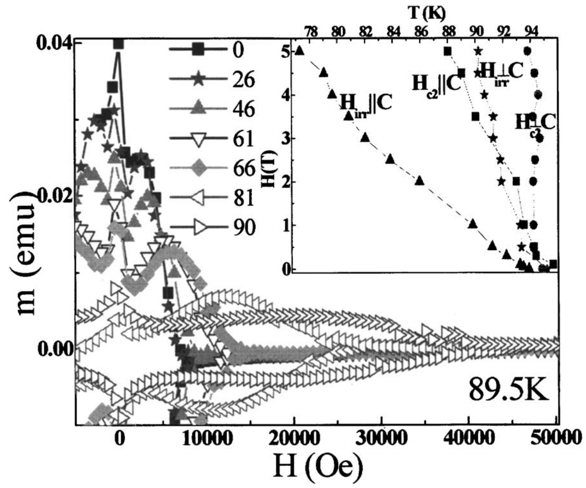

FIG. 3. Angular dependence of hysteresis loops at 89.5 K. The angle between H and the c axis was varied between 0° and 90°. The inset shows the temperature dependence of the upper critical field and the irreversibility line for the two principal directions of the field.

row due to twinning. This implies that the twin planes are not only all parallel to the crystal c axis but also that they have (hk0) orientation.

X-ray-diffraction analysis of the sample gave a single-phase spectrum. The strong and narrow peaks correspond to Miller indices $(00l)$, with $l=1$, 3, 5, 6, and 7, which is an indication of a high degree of preferred orientation of the crystallographic planes perpendicular to the c axis. From the energy dispersive x-ray spectroscopy (EDS) map, the oxygen distribution was found to be uniform. Analysis of the diffraction data of a sample grain was again consistent with the presence of an orthorhombic phase.

III. MAGNETIC MEASUREMENTS

The magnetic measurements were performed with an MPMS-5T superconducting quantum interference device (SQUID) magnetometer. The critical temperature of the crystal was obtained from measurements of the magnetic moment with an applied field of 20 Oe. Its value was obtained to be $T_c=95$ K, with transition width $\Delta T<2$ K. Since extended defects are directional pinning centers, the dominant defect structure can be determined by changing the angle between the applied field and the extended defects.[11] Figure 3 shows the magnetic hysteresis (m-H) loops recorded at 89.5 K, with the applied field (H) inclined at various angles φ relative to the crystal c axis. This was the lowest temperature for which closure of the loop for $\varphi=90°$ at the highest accessible field, 5 T, was possible. It can be seen that the m-H loops display the secondary peak effect over the entire angular range of $0° < \varphi \leqslant 90°$. The maximum of the secondary peak is at the field H_{peak}, and H_{peak} increases with φ. The inset of the figure shows the temperature dependence of the upper critical field and the irreversibility line for the two principal directions of the field. After the sample was zero field cooled the upper critical field was determined by scanning the temperature from far below T_c to above T_c at different fields, and the criterion of $\Delta m=10^{-5}$ emu was selected to mark the onset of deviation from the horizontal axis. The irreversibility line was determined from the merging point of the field-cooled (FC) and zero-field-cooled (ZFC) curves with the same criterion as the upper critical field. The ratio of the two upper critical fields was obtained to be about 10, which is of the same order as the anisotropy parameter for yttrium barium copper oxide (YBCO).[12,13]

In order to compare the commonly used method of determining lock-in angle via appearance of the cusplike feature in the irreversibility field and the method presented in this paper, the irreversibility field was determined from the merging point of the upper and lower branches of the hysteresis loop at each angle, using the same numeric criteria as for FC-ZFC curves. Figure 4 is the result of this measurement, which was carried out at 89.5 K. For angles φ close to 0° and in the range dominated by the twin planes, the hysteresis loop measurements were performed with smaller angle increments to extract any cusplike dependence of H_{irr} on φ. Despite the presence of a high density of twin planes (Fig. 1) no cusplike feature could be observed due to small signal-to-noise ratio close to the irreversibility field, as can be better seen in the inset. In what follows, it is shown that the lock-in angle can be extracted from the same data with-

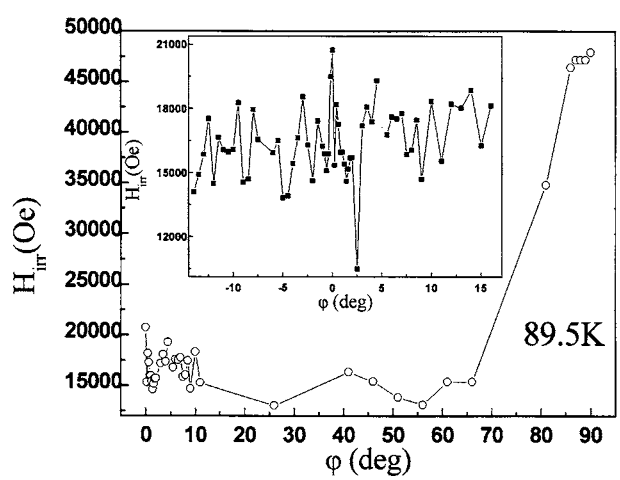

FIG. 4. Angular dependence of the irreversibility field at 89.5 K. No cusplike dependence of the irreversibility field at $\varphi=0°$ could be detected. The inset shows the angles close to $\varphi=0°$ on a magnified scale.

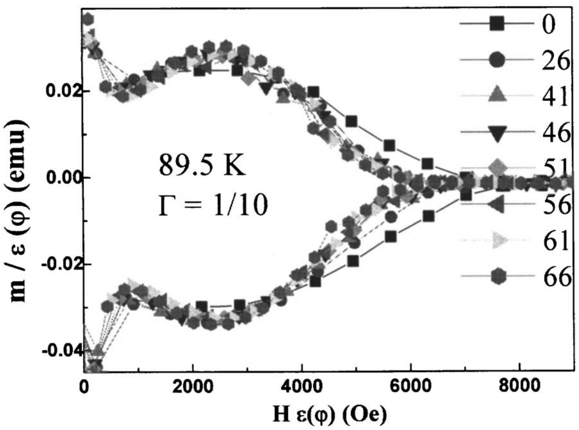

FIG. 5. The scaling of the data in Fig. 3 from 0° to 66° revealed that the loops for angles very close to 0° cannot be scaled with the other loops.

out conducting any extra measurements, using a different approach in the data analysis. In order that the intrinsic anisotropic properties of the superconductor, which are related to its layered structure, are not confused with the effects of twin boundaries, the anisotropic effects must be taken into account in this analysis. According to Blatter *et al.*,[14] an anisotropic superconductor can be mapped onto an isotropic superconductor using a scaling method. In this approach, the measured hysteresis loop must be scaled using $H\varepsilon(\varphi)$ and $m/\varepsilon(\varphi)$, where $\varepsilon^2(\varphi)=\cos^2\varphi+\Gamma^2\sin^2\varphi$. Γ is the anisotropy parameter. Taking $\Gamma^{-1}=10$, the hysteresis loops are scaled using the approach of Blatter *et al.*, as shown in Fig. 5. The results of the scaling for angles from 0° to 90° showed that all loops except for the ones close to $\varphi=0°$ and $\varphi=90°$ are well scaled. (The loops for angles close to 90° have not been shown, for clarity.)

It has already been mentioned that for twinned samples there is a characteristic angle between the field and the crystal c axis called the lock-in angle φ_L. The vortex structure for angles above φ_L assumes a zigzag-shaped pattern (kinked vortices).[15] Oussena *et al.* showed that for $\varphi<\varphi_L$, vortices are trapped on the twin planes and the vortex system forms a Bose glass phase. With increasing φ, the vortices are liberated from the twin planes and point disorder would be the dominant type of pinning, implying that a transition to the vortex glass regime occurs.[16] The overlap of the scaled hysteresis loops (Fig. 5) for $0°<\varphi<90°$ (but not close to the extreme angles) shows that the isotropic point disorder has the dominant role in governing the vortex dynamics in this angular range. On the other hand, the twins and Cu–O planes determine the vortex dynamics for the extreme cases of $\varphi\approx0°$ and $\varphi\approx90°$, respectively.

In order to investigate the lock-in transition for twin boundaries in the sample, hysteresis loops for angles φ between 0° and 16° were measured with a 0.5° increment [Fig. 6(a)]. From Fig. 6(b), which is an enlargement of the marked area, two opposite trends on the two sides of the converging point around 0.18 T are observed: with the angle φ decreasing from 16°, the peak effect is gradually suppressed and the minimum in the magnetic moment at low fields disappears. This trend has also been reported by other groups (e.g., see Fig. 6 in Ref. 17) and seems to be a general behavior for twinned superconductors at high temperatures.[18] Despite the observations of this trend, no attention has been paid to the possible scaling of loops. In some reports,[16,19] the anisotropy factor of the superconductor has been disregarded and the scaling factor $\varepsilon(\varphi)=\cos\varphi$ has been employed, which is only suitable for highly anisotropic superconductors such as Bi2212. Our data analysis showed that $\varepsilon(\varphi)=(\cos^2\varphi+\Gamma^2\sin^2\varphi)^{1/2}$ can be used for less anisotropic superconductors, such as 123 systems.

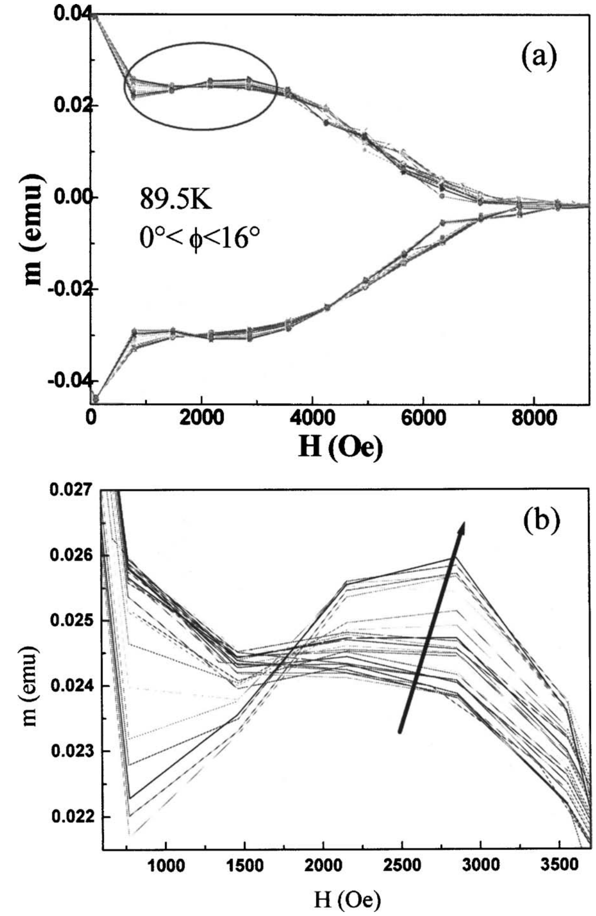

FIG. 6. (a) Angular dependence of hysteresis loops for angles $0°<\varphi<16°$ with 0.5° angular increment. (b) The enlarged marked area shows that with decreasing angle from 16°, the peak effect and the minimum in m at low fields are smoothly depressed. The arrow shows the direction of angular increase.

Figure 7 (which is one of the major results of this work) shows the results of the scaling of the data of Blatter *et al.* for $0°<\varphi<16°$, presented in Fig. 6(a). This scaling reveals two groups of hysteresis loops, those for φ between 0° and 6° and those for φ above 6°. Whereas the minimum at low fields and the maximum (i.e., the second peak in magnetic hysteresis loop) are clearly seen for the latter group, the former group does not exhibit a pronounced second peak. Because the scaling divides the hysteresis loops into two distinct groups and the scaled loops are angle independent within each of the groups, two separate pinning mechanisms are responsible for the observed behavior.

Disappearance of the low-field minimum for $\varphi<6°$, in Fig. 6(a), may be a confirmation of the twin planes as effective pinning centers at low fields, when the twin spacing

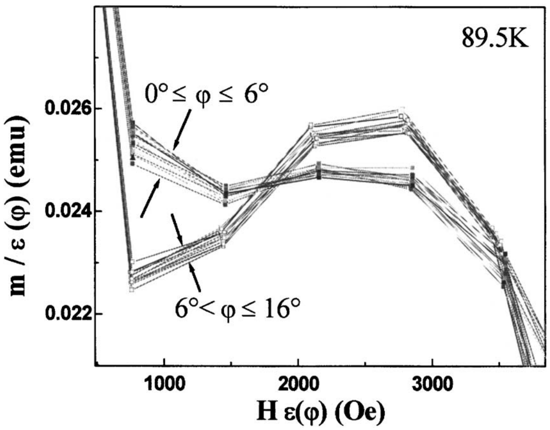

FIG. 7. After scaling, the loops in Fig. 6 were naturally divided into two groups: from 0° to 6° and from 6° to 16° and above.

matches the mean vortex distance (matching effect).[20] The vortex lattice parameter can be calculated via $a \approx (\phi_0/B)^{1/2}$, where ϕ_0 is the magnetic-flux quantum with a value of 2.07×10^{-7} Oe cm^{-2}. For fields between 700 and 1800 Oe, the vortex lattice parameter is obtained to be $\leqslant 17\ \mu$m, which corresponds to the average measured twin plane spacing. This is supported by the work of Pradhan *et al.*,[20,21] where the low-field peak in the double peak structure of a defect-free twinned $Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$ single crystal is ascribed to the matching effect by twin planes. The suppression of the second peak in the hysteresis loop for $\varphi < 6°$ is believed to be a result of channeling or shearing of vortices along the twin walls.[18,22]

In this way, the scaling of the hysteresis loops measured at different values of φ gave a value of the lock-in angle of 6°. For $\varphi > 6°$, the pinning is dominated by the isotropic point disorder, which forms a vortex glass structure. The main advantage of this method over the conventional method of measuring the cusp in H_{irr} vs φ is that it does not rely on an arbitrary definition of H_{irr} and it can give a more accurate value of φ_L, due to the sharp transition between the two groups of the scaled hysteresis loops.

The above method for obtaining φ_L was performed with a scaling based on the anisotropy factor (Γ), which was determined by the ratio of the two upper critical fields. These fields are themselves defined by an arbitrary criterion. However, the splitting of the scaled hysteresis loops into two groups occurred at the same value of φ_L, regardless of the criterion employed for H_{c2}. It should be noted that the twin boundaries are effective pinning centers only at high temperatures,[7–9] where it is difficult to measure H_{irr} reliably. The presented method for obtaining φ_L does not suffer from the lack of sensitivity at these temperatures and its accuracy is only limited by the accuracy of measuring φ.

We expect that the method developed in this work should work well for other twinned RE123 systems, too. Namely, the pinning mechanism on twin planes is the same for all these systems.[5,16–18,23] The only difference between them will be the temperature range in which the pinning on twin boundaries dominates over other types of pinning. They will all have a well-defined lock-in angle below which the vortices are pinned strongly by twin boundaries, for which our method will give two distinct families of the scaled hysteresis loops at a well-defined angle. This is confirmed by the investigation of Oussena *et al.* for Y123 system, which shows that the shape of hysteresis loops significantly depends on the number of twin domains in the sample.[18] It seems to be possible to scale magnetic hysteresis loop width ΔM at a particular field with the angle φ for Y123, at temperatures above 60 K.[13] There is a clear dip in the value of ΔM at the lock-in angle. This is again consistent with what is presented in this report. The dip in ΔM can also be used for obtaining the lock-in angle.[13,15] However, this method can only be used for fields and RE123 systems where channeling of magnetic vortices along the twin planes occurs, causing a decrease of the effective vortex pinning by the twin planes. The method presented in this manuscript is a more general one, requiring only the locking of the vortices on the twin planes.

IV. CONCLUSION

In conclusion, an alternative method for determining the lock-in angle for vortices pinned by twin planes was found, employing anisotropic scaling of magnetic hysteresis loops for fields at different angles from the crystal *c* axis. This method was employed at 89.5 K for a highly twinned SmBaCO single crystal. The uncertainty in the determination of the lock-in angle by this method is considerably lower than with the conventional method using the cusplike dependence of the irreversibility field on the angle between the applied field and the *c* axis.

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