PHYSICAL REVIEW B VOLUME 55, NUMBER 14 1 APRIL 1997-II

Angular dependence of the first-order vortex-lattice phase transition in $Bi_2Sr_2CaCu_2O_8$

B. Schmidt and M. Konczykowski
CNRS, URA 1380, Laboratoire des Solides Irradiés, École Polytechnique, 91128 Palaiseau, France

N. Morozov and E. Zeldov
Department of Condensed Matter Physics, The Weizmann Institute of Science, 76100 Rehovot, Israel

(Received 20 November 1996)

The paramagnetic peak in the local ac susceptibility χ' is used to identify the first-order vortex-lattice phase transition in $Bi_2Sr_2CaCu_2O_8$ at various tilt angles θ of the dc magnetic field with respect to the c axis. The transition field H_m follows roughly the two-dimensional scaling function $H_{dc}\cos(\theta)$. The scaling fails for field orientations close to the ab plane. The amplitude of the paramagnetic peak, which is proportional to the jump in magnetization ΔB, does not depend on the tilt angle up to configurations very close to the ab plane ($\pm 1°$). From this we conclude that the entropy jump at the transition, Δs, is insensitive to the presence of the in-plane field. [S0163-1829(97)52314-9]

Phase transitions of the vortex lattice in high temperature superconductors (HTSC's) attract central attention not only because of the fundamental interest of such phenomena,[1–5] but also due to their implications for potential applications. The first-order transition identified by the jump in the equilibrium magnetization[6,7] is of particular interest as this thermodynamic observation provides a quantitative measurement of the relevant parameters like the entropy jump at the transition. The report on the local magnetization jump in $Bi_2Sr_2CaCu_2O_8$ (BSCCO) probed by a microscopic Hall sensor device[6] was followed by a series of papers.[8–16] Most of them confirmed the first-order nature of the transition. The existence of a finite shear modulus below the transition demonstrated by resistive measurements[17,18] and the effect of a low concentration of columnar defects[13] allows us to restrain the possible interpretation of the transition to a vortex-lattice melting[19–23] or simultaneous melting and decoupling transitions.[24] However, some crucial aspects of the transition are still not fully understood and remain under debate. The entropy jump Δs at the transition in $YBa_2Cu_3O_7$ is about $0.5k_B$ per pancake vortex as consistently derived from both magnetization and calorimetric measurements.[15] In BSCCO, on the other hand, Δs deduced from the Clausius-Clapeyron relation exhibits an anomalous temperature dependence and reaches excessively high values. Moreover, some measurements seemed to indicate that an in-plane field suppresses the magnetization step.[14] In order to check this observation and to get more insight into the phenomenology of the transition we have carried out a detailed study of the angular dependence of the first-order phase transition. An unusual technique was used to identify the transition and to determine the height of the magnetization jump.[25] This technique is making use of the paramagnetic peak that appears in the in-phase ac susceptibility as a result of the jump in the dc magnetization curve. The position and amplitude of this paramagnetic peak have been measured as a function of the tilt of the magnetic field with respect to the c axis. The angular dependence of the transition field H_m was traced at various temperatures.

A miniature ($80\times 80\times 80$ μm^3 active volume) Hall sensor made out of InSb, a narrow gap semiconductor,[26] was placed on top of a BSCCO sample of approximate dimensions $500\times 500\times 40$ μm^3. This sample has been cut out of the same larger crystal as the one investigated in a previous work.[6] The uniformity and high quality of this particular crystal was demonstrated by magneto-optical investigation of the flux penetration process.[27] The crystal with the Hall sensor were placed in the center of an excitation coil providing an ac magnetic field of 1 Oe at low frequency (7.75 Hz). The in- and out-of-phase components of the ac magnetic induction B'_{ac} and B''_{ac} were measured using NF Corporation lock-in amplifier (NF5810). An additional digital lock-in amplifier (Stanford Research SR850) was used to record the third harmonic component of B_{ac}. The high sensitivity (50 mΩ/G) and low resistance of the Hall probe allowed the resolution of the ac detection to be below 1 mG. The entire setup was mounted on a cold-finger, pumped nitrogen Dewar and placed in an electromagnet. In this setup the ac excitation field H_{ac} was always oriented along the c axis of the crystal while the external dc magnetic field H_{dc} provided by the electromagnet was rotated by a computer-controlled step motor, as illustrated in the inset of Fig. 1. Three types of experimental scenarios have been realized: temperature variation at constant dc field and fixed angle, rotation of the constant field at constant temperature, and field scans at fixed angle and constant temperature.

Typical results of the magnetic-field scans at various angles are presented in Fig. 1. The ac response B'_{ac} shows a gradual transition from a fully shielded state at low H_{dc} to a fully transparent state at higher dc fields. A very well defined paramagnetic peak in B'_{ac} appears at some dc field labeled $H_m(\theta)$, which increases with tilt angle θ from the c axis. It has been demonstrated previously that the appearance of such a paramagnetic peak indicates the existence of a first-order phase transition and reflects the jump in the equilibrium magnetization.[25] The height of the paramagnetic peak does not depend on the amplitude of the ac excitation and it allows a precise determination of the height of the magnetization jump. The width of the peak increases with increasing H_{ac}. A finite out-of-phase component B''_{ac} may occur at the

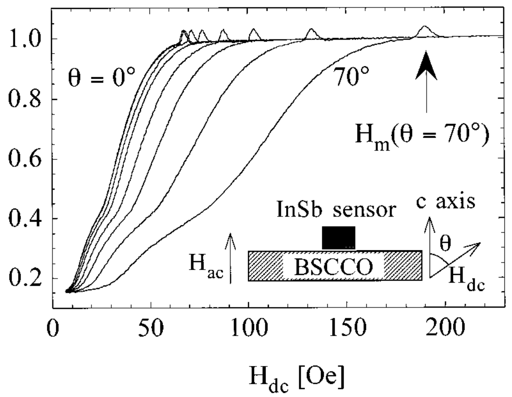

FIG. 1. Transmitted ac field as a function of applied dc field for different angles θ between c axis and dc field direction, $\theta = 0°$ to 70° in 10° intervals. $T = 80$ K, $H_{ac} = 1$ Oe, f = 7.75 Hz. The inset gives a schematic description of the setup and the orientations of the applied fields.

peak due to possible hysteresis at the transition.[25] In our measurements we were unable to detect any out-of-phase signal related to the peak at temperatures above 70 K. It can be shown that the height of the paramagnetic peak is given by $\Delta B\sqrt{2}/\pi$, where ΔB is the height of the magnetization step at the transition, while the width of the peak at half maximum is $\sqrt{6}H_{ac}$. This dependence was confirmed experimentally using two-dimensional electron-gas Hall sensors that measure the magnetic field at the surface of the sample.[25] However, the magnetic signal decreases rapidly with distance from the surface. Our InSb sensor is 80 μm thick and the measured signal thus corresponds to the average induction over the thickness. As a result the height of our paramagnetic peak is reduced by about a factor of 3 as compared to the value close to the surface.[25] The relatively large size of our Hall sensor resulted also in some smearing of the peak. In consequence no shrinking of the peak was found for ac amplitudes less than 1 Oe, whereas a broadening was observed for higher ac amplitudes in accordance with the calculation.

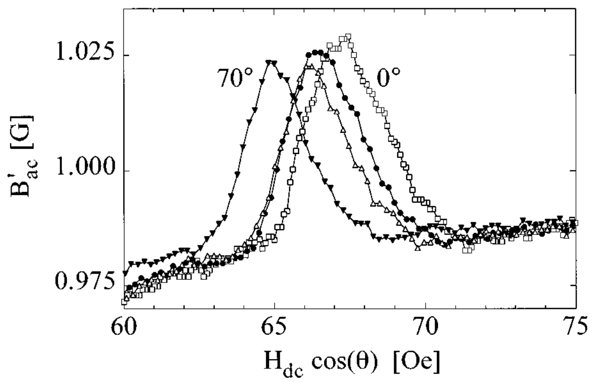

FIG. 2. Paramagnetic peaks of Fig. 1 as a function of the "c axis component" of the applied dc field [$H_{dc}\cos(\theta)$] for $\theta = 0°$, 30°, 60° and 70°. The peak shows a tendency to shift slightly to lower c axis fields at large angles.

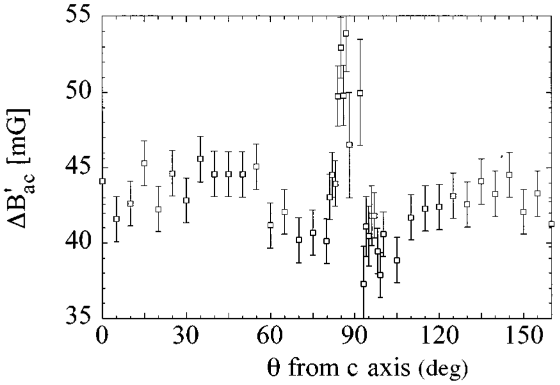

FIG. 3. Paramagnetic peak height $\Delta B'_{ac}$ as a function of the applied field angle θ with respect to the c axis at $T = 80$ K.

Figure 2 shows a closer view of the paramagnetic peak. The dc field in this figure has been scaled according to $H_{dc}\cos(\theta)$ in order to trace the measured B_{ac} as a function of the c component of the applied field. It can clearly be seen that the height of the peak remains practically the same as the field is turned away from the c axis. Figure 3 shows the measured peak height as a function of angle for $T = 80$ K. Measurements at 70 and 88 K give similar results. Within our experimental resolution the peak height has a constant value up to tilt angles of at least 85° from the c axis. Up to 89° the peak still remains observable, but the determination of its height becomes more imprecise. It is remarkable, however, that even at such a high tilt angle the peak is still observed, because this angle corresponds to the presence of an in-plane field of about 3 kOe for the measurement at

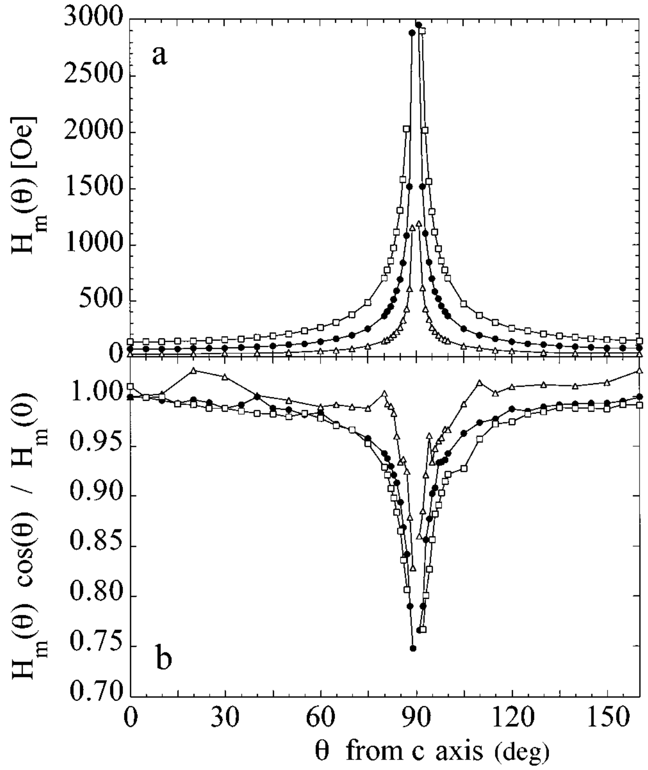

FIG. 4. (a) Angular dependence of the transition field $H_m(\theta)$ recorded at temperatures of 88 K (△), 80 K (●), and 70 K (□). (b) The same data represented in terms of the normalized c axis component of the field at the transition $H_m(\theta)\cos(\theta)/H_m(0)$.

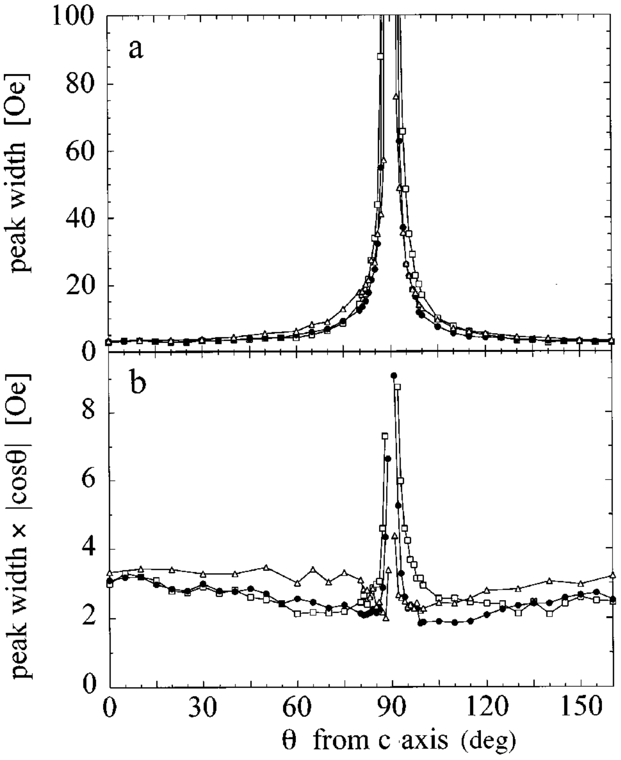

FIG. 5. (a) The width of the paramagnetic peak as a function of angle for temperatures of 88 K (△), 80 K (●), and 70 K (□). (b) Peak width times $|\cos(\theta)|$ as a function of angle.

80 K. The position of the phase transition accurately scales with $H_{\mathrm{dc}}\cos(\theta)$ at low tilting angles, as expected in the case of a nearly two-dimensional superconductor. For tilting angles beyond 70° however, this 2D scaling fails to reproduce the exact angular dependence of H_m.

A representation of the angular dependence of the transition field H_m at fixed temperatures of 70, 80, and 88 K is given in Fig. 4(a). The same data, rescaled to the c axis component of the field at which the paramagnetic peak is observed, are shown in Fig. 4(b). In the case of a perfect 2D behavior the value $H_m(\theta)\cos(\theta)$ should be independent of angle. As can be seen, this 2D scaling fails for field orientations close to the ab plane. At the highest temperature (88 K) the 2D scaling seems to describe the data well up to a few degrees from the ab plane. At 70 K the deviations start at about 70°. A more complete scaling function $[H_m\sqrt{\cos^2(\theta)+\epsilon^2\sin^2(\theta)}]$, which takes into account a finite anisotropy ϵ, was also tested with no significant improvement of the fit. A similar scaling might also be expected for the width of the peak. Figure 5(a) shows the full width at half maximum of the peak as a function of angle. The same data multiplied by $\cos(\theta)$ are shown in Fig. 5(b). As can be seen from this figure the peak width also follows a cosine law up to 10° or less from the ab plane. Close to the ab plane the width grows faster than $1/\cos(\theta)$.

In addition, an alternative procedure to study the angular dependence of the phase transition was used. This procedure consisted in rotating a constant dc magnetic field at a given temperature. The rotation changes the projection of H_{dc} on the c axis and apparently this is similar to a field sweep at a given temperature. Typical results of such measurements are shown in Fig. 6. For $\theta=0°$ the applied field of 138 Oe exceeds the irreversibility field for the c direction, no ac shielding is observed and $B'_{\mathrm{ac}}=H_{\mathrm{ac}}=$ 1 Oe. With increasing tilt angle the c component of the dc field diminishes and screening of the ac field becomes effective. Finally, for $H_{\mathrm{dc}}\parallel ab$ the ac field is completely screened. Also with this procedure a paramagnetic peak is observed at the proper temperature-dependent angle. The amplitude of the peak increases slightly with increasing temperature, in agreement with the previous measurements for $H_{\mathrm{dc}}\parallel c$ axis.[25]

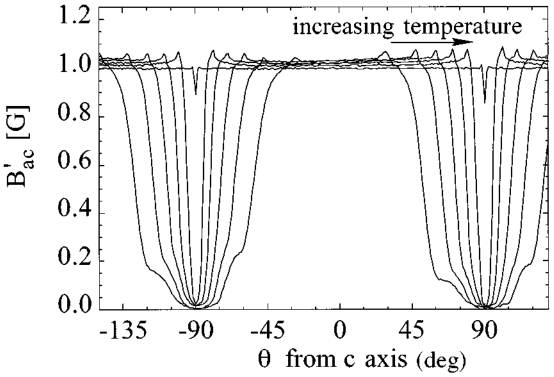

FIG. 6. Angular variation of the measured ac field for several temperatures (T= 72 K to 92 K in steps of 4 K). With increasing temperature the peak moves away from the c axis towards the ab plane ($\theta=90°$). The field value H_{dc} was chosen such that $H_{\mathrm{dc}}=H_m(0°)$ at $T=70$ K.

The presented angular dependence of the transition field is consistent with what is expected for very anisotropic superconductors, where only the c axis component of the field is relevant.[1] The slight departure from 2D scaling for fields very close to the ab plane may be related to the underlying mechanism of the transition which is not entirely clear at the present. A recent model for the low field phase diagram in layered superconductors[24] points out that both Josephson and magnetic interactions between pancake vortices are relevant for the determination of the transition fields. One may expect that for the large tilt angles explored in our experiments the pancake vortices in adjacent layers forming a flux line are slightly shifted with respect to each other leading to the suppression of the magnetic interaction and to a deviation from the anisotropic scaling.

We conclude that our susceptibility measurements show that the first-order transition in the vortex lattice persists in the presence of the large in-plane field contrary to the recent reports based on torque magnetometry.[14] In a wide range of angles, only the c axis component determines the position of the transition. The independence of the amplitude of the paramagnetic peak on the direction of the dc magnetic field implies that the entropy jump at the transition is insensitive to the presence of an in-plane field and is determined only by the density of the pancake vortices.

We thank V. B. Geshkenbein for helpful discussions and are grateful to H. Motohira for providing the BSCCO crystals. This work was supported by the Israeli Ministry of Science and the Arts and the French Ministry of Research and Technology (AFIRST).

[1] For a recent review, see G. Blatter *et al.*, Rev. Mod. Phys. **66**, 1125 (1994).

[2] For a recent review, see E. H. Brandt, Rep. Prog. Phys. **58**, 1465 (1995).

[3] D. R. Nelson, Nature (London) **375**, 356 (1995).

[4] E. Brézin, D. R. Nelson, and A. Thiaville, Phys. Rev. B **31**, 7124 (1985).

[5] D. R. Nelson, Phys. Rev. Lett. **60**, 1973 (1988).

[6] E. Zeldov *et al.*, Nature (London) **375**, 373 (1995).

[7] H. Pastoriza, M. F. Goffman, A. Arribére, and F. de la Cruz, Phys. Rev. Lett. **72**, 2951 (1994).

[8] T. Hanaguri *et al.*, Physica C **256**, 111 (1996).

[9] R. Liang, D. A. Bonn, and W. N. Hardy, Phys. Rev. Lett. **76**, 835 (1996).

[10] B. Revaz *et al.*, Europhys. Lett. **33**, 701 (1996).

[11] U. Welp *et al.*, Phys. Rev. Lett. **76**, 4809 (1996).

[12] B. Khaykovich *et al.*, Phys. Rev. Lett. **76**, 2555 (1996).

[13] B. Khaykovich *et al.* (unpublished).

[14] D. E. Farrell *et al.*, Phys. Rev. B **53**, 11 807 (1996).

[15] A. Schilling *et al.*, Nature (London) **382**, 791 (1996).

[16] D. T. Fuchs *et al.*, Phys. Rev. B **54**, R796 (1996).

[17] H. Pastoriza and P. H. Kes, Phys. Rev. Lett. **75**, 3525 (1995).

[18] D. T. Fuchs *et al.*, Phys. Rev. B **55**, R6156 (1997).

[19] A. K. Nguyen, A. Sudbø, and E. Hetzel, Phys. Rev. Lett. **77**, 1592 (1996).

[20] A. Houghton, R. A. Pelcovits, and A. Sudbø, Phys. Rev. B **40**, 6763 (1989).

[21] E. H. Brandt, Phys. Rev. Lett. **63**, 1106 (1989).

[22] R. Šášik and D. Stroud, Phys. Rev. Lett. **75**, 2582 (1995).

[23] G. Blatter and B. I. Ivlev, Phys. Rev. B **50**, 10 272 (1994).

[24] G. Blatter, V. B. Geshkenbein, A. I. Larkin, and H. Nordborg, Phys. Rev. B **54**, 72 (1996).

[25] N. Morozov, E. Zeldov, D. Majer, and M. Konczykowski, Phys. Rev. B **54**, R3784 (1996).

[26] M. Konczykowski, F. Holtzberg, and P. Lejay, Supercond. Sci. Technol. **4**, S331 (1991).

[27] M. V. Indenbom *et al.* (unpublished).