## Separation of the Irreversibility and Melting Lines in Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> Crystals

D. Majer, <sup>1</sup> E. Zeldov, <sup>1</sup> and M. Konczykowski<sup>2</sup>

<sup>1</sup>Department of Condensed Matter Physics, The Weizmann Institute of Science, 76100 Rehovot, Israel

<sup>2</sup>Centre d'Etudes et de Recherches sur les Materieux, Laboratoire des Solides Irradies, Ecole Polytechnique, 91128

Palaiseau, France

(Received 3 April 1995)

 ${\rm Bi}_2{\rm Sr}_2{\rm CaCu}_2{\rm O}_8$  crystal was polished into a prism shape that eliminates the geometrical barrier and results in a *fully reversible* magnetization at  $T \gtrsim 76~{\rm K}$ . The vortex-lattice melting was determined independently by the magnetization step at the transition. At elevated temperatures the irreversibility line (IL) lies in the vortex-solid region significantly below the melting line, whereas at lower temperatures the IL is high within the vortex-liquid phase and is due to surface barriers. The results clearly demonstrate that the IL and melting stem from different and unrelated physical mechanisms.

PACS numbers: 74.60.Ec, 74.60.Ge, 74.60.Jg, 74.72.Hs

One of the most extensively studied features of hightemperature superconductors (HTSC) is the irreversibility line (IL). This line divides the H-T phase diagram into two parts; above the IL, magnetization of a sample is fully reversible, whereas below the line a hysteretic magnetization behavior is observed. The IL is measured by various experimental techniques which include dc magnetization [1-3], ac susceptibility [4,5], third harmonic detection [6], mechanical oscillator [7], vibrating reed [8,9], quasistatic magnetization [10], etc. The standard interpretation of the irreversibility is the onset of bulk pinning: At low temperatures the vortices are pinned and hence finite critical currents are present, whereas above the IL the vortices are unpinned and as a result reversible behavior is obtained [2-11]. It is further believed that the vortex solid, due to its finite shear modulus, is pinned much more efficiently by the material defects as compared to the vortex liquid [11]. Therefore, the IL is assumed to separate a pinned vortex solid from an unpinned vortex liquid and hence the IL is commonly identified with the vortex-lattice melting transition [7,10,12–16].

The above considerations do not take into account two important factors. First is the possibility that the vortex lattice may be unpinned. In very clean systems we expect a well ordered Abrikosov lattice which is not pinned. In addition, due to pinning-potential renormalization by thermal fluctuations [11,17], the vortex lattice may be unpinned at elevated temperatures even in the presence of weak disorder. In this case, the IL will follow the vortex-lattice depinning line which may lie significantly below the vortex-lattice melting line. The second factor is the presence of other hysteretic mechanisms which are not related to bulk vortex properties. These are the Bean-Livingston surface barrier [18-25] and the geometrical barrier [26-36]. Both mechanisms cause irreversible magnetization and an associated IL that is not related to the bulk vortex properties. Furthermore, in relatively clean systems these surface effects will be the dominant sources of irreversibility and hence the IL may lie either below or *above* the melting line in the region of vortex liquid.

The concept that identifies the IL with vortex-lattice melting is supported by two types of observations. One is that the temperature dependence of the measured IL compares favorably with the predictions of existing melting theories [10,12,16,31,37-39]. This observation, however, is not decisive since it was shown recently that the combination of geometrical and surface barriers results in a similar theoretical temperature dependence [25,26,40,41]. The second observation, which is much more substantial, is that in Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8</sub> (BSCCO) crystals the longrange order of the vortex lattice, as probed by neutron diffraction [13] and muon spin rotation [14], shows a pronounced change in the vicinity of the IL at low magnetic fields. In addition, a small step in magnetization, indicating a first-order melting transition, was observed in a BSCCO crystal close to the IL [15]. These observations are in apparent contradiction to the recent finding that the IL in BSCCO is determined by surface and geometrical barriers and not by bulk properties of the vortex lattice [40,41]. In this paper we resolve this controversy by independent measurement of both the irreversibility and vortex-lattice melting lines. The IL and the melting line in BSCCO stem from different physical origins. Nevertheless, in the vicinity of the critical temperature  $T_c$  these two lines may incidentally be close to each other. By changing the shape of the crystal from the natural platelet geometry to a prism shape we suppress the geometrical barrier, and hence shift the IL significantly, while the melting line remains unaffected. As a result, a clear separation of the irreversibility and melting lines is obtained.

Two BSCCO crystals with  $T_c \approx 90$  K were studied using two-dimensional electron-gas (2DEG) Hall-sensor arrays. One of the crystals had a platelet geometry with dimensions of  $415 \times 160 \times 15~\mu \text{m}^3$ , whereas the second crystal was carefully polished into a prism shape  $660~\mu \text{m}$  long,  $270~\mu \text{m}$  wide, and  $70~\mu \text{m}$  high in the center. Each of the eleven sensors of the array has an

active area of  $10 \times 10 \ \mu m^2$ . The microscopic Hallsensor arrays allow very sensitive measurement of the magnetic field with a high spatial resolution. As a result, the melting transition of the vortex lattice can be observed directly by detecting the abrupt step in the local magnetization as described recently [42]. This field step is due to a thermodynamic change in the vortex density upon the transition from a solid to a liquid state that is analogous to the discontinuous change in the specific volume or density upon melting of a regular solid.

Figure 1(a) shows a "local magnetization" loop  $B - H_a$  of the platelet crystal at T = 80 K. A cross section of the experimental setup is shown schematically in the inset.

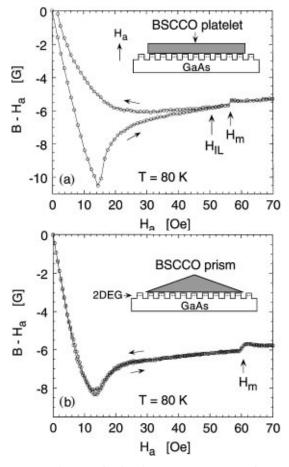


FIG. 1. Local magnetization loops  $B-H_a$  vs  $H_a$  in BSCCO crystals of platelet (a) and prism (b) shapes at  $T=80~\rm K$ . The platelet crystal shows hysteretic magnetization below the irreversibility field  $H_{\rm IL}$ . In the prism sample the geometrical barrier is eliminated and a fully reversible magnetization is obtained at temperatures above 76 K. The melting transition  $H_m$  is observed as a sharp thermodynamic step in the local magnetization. A cross section of the experimental setup is shown schematically in the insets (not to scale). The two-dimensional electron gas (2DEG) active layer of the sensors resides about 0.1  $\mu$ m below the surface. The BSCCO crystals are in contact with the GaAs surface and the local vertical component of the magnetic field B is measured directly. External field  $H_a$  is applied parallel to the crystalline c axis.

B is the local perpendicular magnetic field at the surface of the crystal as measured by one of the sensors, and  $H_a$  is the external magnetic field applied parallel to the c axis of the crystal and perpendicular to the surface. The magnetization loop shows a hysteretic behavior at applied fields below the irreversibility field  $H_{\rm IL}$  and reversible magnetization at higher fields. In addition, the vortexlattice melting transition is clearly observed at  $H_m$  by the magnetization step. At elevated temperatures  $H_{\rm IL}$ and  $H_m$  are close to each other as seen in Fig. 1(a), and it is very tempting to interpret these two features as arising from the same physical origin [13–15]; namely, vortices are pinned in the solid phase and the associated irreversible behavior disappears once the vortex lattice melts. However, by analyzing the spatial distribution of the magnetic field across the sample it was recently shown [26,31,40,41] that in clean BSCCO crystals vortex pinning is negligible, except at low temperatures, and the observed hysteretic behavior results from surface and geometrical barriers. Surface barriers govern the magnetization at intermediate temperatures, as discussed below, whereas at elevated temperatures the irreversibility is due to geometrical barriers. Yet the fact that  $H_{\rm IL}$  and  $H_m$  appear close to each other at high temperatures still leaves room for doubt.

The two models can be readily tested as follows. The irreversibility due to the geometrical barrier arises from the fact that in platelet geometry the equilibrium Meissner shielding current that extends over the entire width of the sample exerts a Lorentz force on penetrating vortices and drives them towards the center of the sample. This effect results in hysteretic magnetization and in a well defined IL [26,27]. In a sample with an elliptical cross section the vortex length and the associated vortex potential are position dependent, which results in a force that tends to drive vortices out of the sample due to the potential gradient. For an ellipse this force is precisely balanced by the Lorentz force and hence an elliptical sample will show a completely reversible magnetization curve in the absence of bulk pinning. In contrast, if the irreversibility is due to bulk pinning, the position of the IL should be practically unaffected by the sample geometry. Therefore one can test the origin of the IL by using an elliptical or spherical crystal. However, such a geometry cannot be easily realized with the small typical sizes of HTSC crystals. On the other hand, the elliptical cross section is in fact the marginal case: A "flatter" geometry will behave hysterically, whereas a more "tapered" cross section will generally result in reversible magnetization. It should be emphasized that the geometrical barrier is not just an effect of the shape of the sample edges. A platelet crystal with rounded edges will still display a hysteretic magnetization loop similar to that of a rectangular sample. The geometry of a flat crystal has to be modified over its entire width in order to eliminate the geometrical barrier. We have therefore chosen a prism as one of the

shapes that should have no geometrical barrier and yet can be relatively easily achieved by careful polishing of the crystal.

Figure 1(b) shows the measured local magnetization loop of the BSCCO prism at 80 K. The astonishing result is that the magnetization is *fully reversible* at temperatures down to about 76 K. To the best of our knowledge a fully reversible magnetization in HTSC was never observed previously at such relatively low temperatures of more than 10 K below  $T_c$ . Yet the melting transition  $H_m$  is still clearly observed as indicated by the arrow. The result in Fig. 1(b) clearly demonstrates three major conclusions: (i) In clean BSCCO crystals at elevated temperatures the vortex lattice is unpinned; (ii) the magnetic hysteresis and the irreversibility line are due to geometrical barriers; and (iii) the vortex-lattice melting transition, as manifested by the magnetization step, is not related to the IL.

At lower temperatures Bean-Livingston surface barriers were shown to govern the magnetic hysteresis in BSCCO [22,40,41]. Figure 2 is a magnified view of a magnetization loop in the vicinity of the melting transition at T = 46 K. The melting transition with the corresponding magnetization step at  $H_m$  is indicated by the arrow. The IL at this temperature lies at a significantly higher field and the hysteretic magnetization behavior is present both below and above  $H_m$ . Clearly the irreversibility and melting are due to different mechanisms at this temperature as well. Moreover, vortex pinning cannot be the source of the hysteresis here since it persists also in the vortexliquid phase above  $H_m$ . In addition, it was recently argued [29,31,33] that the Bean-Livingston barrier is of no significance in HTSC and that any surface hysteresis effects should disappear above the vortex-lattice phase transition. Figure 2 clearly demonstrates that this is not the case and a significant magnetic hysteresis may be present also above  $H_m$ .

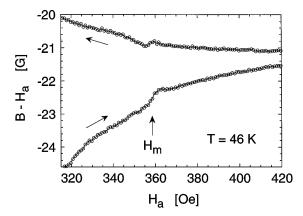


FIG. 2. Expanded view of the local magnetization loop in the prism crystal in the vicinity of the melting transition  $H_m$  at 46 K. The irreversibility field due to surface barriers is significantly higher than  $H_m$  at this temperature.

Following the above procedure we have measured the temperature dependence of  $H_m(T)$  and  $H_{IL}(T)$ . Figure 3 shows the result for BSCCO crystal of the prism shape. The irreversibility and the melting lines are well separated. At  $T \gtrsim 76$  K the IL is completely suppressed and the magnetization is fully reversible. At lower temperatures the IL is present due to the Bean-Livingston surface barrier. Note that at elevated temperatures a vortex solid is present both above and below the irreversibility line, whereas at  $T \lesssim 53$  K a vortex-liquid phase is present both below and above the IL. So, clearly, the IL in clean BSCCO crystal does not indicate the state of the vortex matter and does not reflect a phase transition of the vortex lattice. It should be emphasized that in more disordered HTSC samples the vortex lattice could be pinned more efficiently and the surface effects could be reduced, in which case the IL would indeed follow the melting line. However, at least in clean BSCCO crystals this is not the case. A more detailed analysis of the observed first-order vortex-matter phase transition and the possible underlying mechanisms are discussed in Ref. [42].

In summary, by modifying the crystal shape we have proved that the IL in BSCCO is not related to the vortex-lattice melting transition, in contrast to the prevailing belief. At high temperatures the irreversibility is due to geometrical barriers, and at intermediate temperatures, surface barriers determine the IL. In addition, the vortex lattice is unpinned at elevated temperatures in the entire field range. As a result, a fully reversible magnetization is obtained once the geometrical barrier is removed by a proper shaping of the crystals. The vortex-lattice melting transition is directly observed through a magnetization

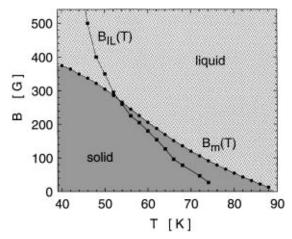


FIG. 3. The vortex-lattice melting line  $B_m(T)$  (dots) and the irreversibility line  $B_{\rm IL}(T)$  (squares) in BSCCO crystal of prism shape. The magnetization is fully reversible above 76 K due to the absence of geometrical barrier and bulk pinning. At lower temperatures the IL is governed by surface barriers. Below 53 K the IL resides in the vortex-liquid phase, whereas at higher temperatures it crosses through the vortex-solid region. The irreversibility and melting lines in BSCCO stem from two different and unrelated mechanisms.

step and the melting line is shown to be uncorrelated with the IL.

We thank H. Motohira for providing the BSCCO crystals and H. Shtrikman for growing the GaAs heterostructures. Discussions with V.M. Vinokur and V.B. Geshkenbein are gratefully acknowledged. This work was supported by the Israeli Ministry of Science and the Arts, by Minerva Foundation Munich, Germany, and by France-Israel cooperation program AFIRST.

- K. A. Muller, M. Takashige, and J. G. Bednorz, Phys. Rev. Lett. 58, 1143 (1987).
- [2] Y. Yeshurun and A. P. Malozemoff, Phys. Rev. Lett. 60, 2202 (1988).
- [3] For a recent review, see Y. Yeshurun, A.P. Malozemoff, and A. Shaulov, Rev. Mod. Phys. (to be published).
- [4] A.P. Malozemoff, T.K. Worthington, Y. Yeshurun, F. Holtzberg, and P.H. Kes, Phys. Rev. B 38, 7203 (1988).
- [5] L. Krusin-Elbaum, L. Civale, F. Holtzberg, A. P. Malozemoff, and C. Feild, Phys. Rev. Lett. **67**, 3156 (1991).
- [6] A. Shaulov and D. Dorman, Appl. Phys. Lett. 53, 2680 (1988).
- [7] P. L. Gammel, L. F. Schneemeyer, J. V. Waszczak, and D. J. Bishop, Phys. Rev. Lett. 61, 1666 (1988).
- [8] A. Gupta, P. Esquinazi, H. F. Braun, and H. W. Neumuller, Phys. Rev. Lett. 63, 1869 (1989).
- [9] For a recent review, see M. Ziese, P. Esquinazi, and H. F. Braun, Supercond. Sci. Technol. 7, 869 (1994).
- [10] A. Schilling, R. Jin, J.D. Guo, and H.R. Ott, Phys. Rev. Lett. 71, 1899 (1993).
- [11] For a recent review, see G. Blatter, M. V. Feigelman, V. B. Geshkenbein, A. I. Larkin, and V. M. Vinokur, Rev. Mod. Phys. **66**, 1125 (1994).
- [12] A. Houghton, R. A. Pelcovits, and A. Sudbo, Phys. Rev. B 40, 6763 (1989).
- [13] R. Cubitt et al., Nature (London) 365, 407 (1993).
- [14] S.L. Lee et al., Phys. Rev. Lett. 71, 3862 (1993).
- [15] H. Pastoriza, M. F. Goffman, A. Arribere, and F. de la Cruz, Phys. Rev. Lett. 72, 2951 (1994).
- [16] G. Blatter and B. I. Ivlev, Phys. Rev. B 50, 10272 (1994).
- [17] L. Burlachkov and V. M. Vinokur, Physica 194–196B, 1819 (1994).
- [18] C. P. Bean and J. D. Livingston, Phys. Rev. Lett. 12, 14 (1964); P. G. de Gennes, Superconductivity of Metals and Alloys (W. A. Benjamin, New York, 1966), p. 76.
- [19] J.R. Clem, in *Proceedings of the 13th Conference on Low Temperature Physics*, edited by K.D. Timmerhaus *et al.* (Plenum, New York, 1974), Vol. 3, p. 102.
- [20] L. Burlachkov, Phys. Rev. B 47, 8056 (1993).
- [21] M. Konczykowski, L. Burlachkov, Y. Yeshurun, and F. Holtzberg, Phys. Rev. B 43, 13707 (1991); Physica (Amsterdam) 194C, 155 (1992).

- [22] N. Chikumoto, M. Konczykowski, N. Motohira, and A. P. Malozemoff, Phys. Rev. Lett. 69, 1260 (1992).
- [23] M. Xu, D. K. Finnemore, G. W. Crabtree, V. M. Vinokur, B. Dabrowski, D. G. Hinks, and K. Zhang, Phys. Rev. B 48, 10630 (1993).
- [24] V. N. Kopylov, A. E. Koshelev, I. F. Schegolev, and T. G. Togonidze, Physica (Amsterdam) 170C, 291 (1990).
- [25] L. Burlachkov, V.B. Geshkenbein, A.E. Koshelev, A.I. Larkin, and V.M. Vinokur, Phys. Rev. B 50, 16770 (1994).
- [26] E. Zeldov, A. I. Larkin, V. B. Geshkenbein, M. Konczykowski, D. Majer, B. Khaykovich, V. M. Vinokur, and H. Shtrikman, Phys. Rev. Lett. 73, 1428 (1994).
- [27] E. Zeldov, A. I. Larkin, M. Konczykowski, B. Khaykovich, D. Majer, V. B. Geshkenbein, and V. M. Vinokur, Physica (Amsterdam) 235–240C, 2761 (1994).
- [28] Th. Schuster, M. V. Indenbom, H. Kuhn, E. H. Brandt, and M. Konczykowski, Phys. Rev. Lett. 73, 1424 (1994).
- [29] Th. Schuster, H. Kuhn, E. H. Brandt, M. Indenbom, M. R. Koblischka, and M. Konczykowski, Phys. Rev. B 50, 16684 (1994).
- [30] B. Khaykovich, E. Zeldov, M. Konczykowski, D. Majer, A. I. Larkin, and J. R. Clem, Physica (Amsterdam) 235– 240C, 2757 (1994).
- [31] M. V. Indenbom, G. D'anna, M.-O. Andre, W. Benoit, H. Kronmuller, T. W. Li, and P. H. Kes, in *Proceedings* of the 7th International Workshop on Critical Currents in Superconductors, edited by H. W. Weber (World Scientific, Singapore, 1994), p. 327.
- [32] M. V. Indenbom, H. Kronmuller, T. W. Li, P. H. Kes, and A. A. Menovsky, Physica (Amsterdam) 222C, 203 (1994).
- [33] M. V. Indenbom, G. D'anna, M.-O. Andre, V. V. Kabanov, and W. Benoit, Physica (Amsterdam) 235–240C, 201 (1994).
- [34] T. B. Doyle and R. Labusch (to be published).
- [35] A. Fortini, A. Hairie, and E. Paumier, Phys. Rev. B 21, 5065 (1980), and references therein.
- [36] J. R. Clem, R. P. Huebener, and D. E. Gallus, J. Low Temp. Phys. 12, 449 (1973).
- [37] E. H. Brandt, Phys. Rev. Lett. 63, 1106 (1989).
- [38] L. I. Glazman and A. E. Koshelev, Phys. Rev. B 43, 2835 (1991).
- [39] S. Hikami, A. Fujita, and A. I. Larkin, Phys. Rev. B 44, 10400 (1991).
- [40] D. Majer, E. Zeldov, M. Konczykowski, V. B. Geshkenbein, A. I. Larkin, L. Burlachkov, V. M. Vinokur, and N. Chikumoto, Physica (Amsterdam) 235-240C, 2765 (1994)
- [41] E. Zeldov, D. Majer, M. Konczykowski, A. I. Larkin, V. M. Vinokur, V. B. Geshkenbein, N. Chikumoto, and H. Shtrikman, Europhys. Lett. 30, 367 (1995).
- [42] E. Zeldov, D. Majer, M. Konczykowski, V. B. Geshkenbein, V. M. Vinokur, and H. Shtrikman, Nature (London) 375, 373 (1995).