

## Metastability line in the phase diagram of vortices in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$

B. Sas

*Research Institute of Solid State Physics, Box 49, H-1525 Budapest, Hungary*

F. Portier

*Service de Physique de l'Etat Condensé, Commissariat à l'Energie Atomique, Saclay, F-91191 Gif-sur-Yvette, France*

K. Vad

*Institute of Nuclear Research, Box 51, H-4001 Debrecen, Hungary*

B. Keszei

*Research Institute for Materials Science, Box 49, H-1525 Budapest, Hungary*

L. F. Kiss

*Research Institute of Solid State Physics, Box 49, H-1525 Budapest, Hungary*

N. Hegman

*Institute of Nuclear Research, Box 51, H-4001 Debrecen, Hungary*

I. Puha

*Service de Physique de l'Etat Condensé, Commissariat à l'Energie Atomique, Saclay, F-91191 Gif-sur-Yvette, France*

S. Mészáros

*Institute of Nuclear Research, Box 51, H-4001 Debrecen, Hungary*

F. I. B. Williams

*Service de Physique de l'Etat Condensé, Commissariat à l'Energie Atomique, Saclay, F-91191 Gif-sur-Yvette, France*

(Received 28 July 1999)

Nonlinear transport in the low-temperature vortex glass state of single crystal  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  has been investigated with fast current pulses driven along the  $ab$  plane. Field cooled preparation shows a higher threshold current (marking a jump or break in slope in the voltage response) than the zero field cooled (ZFC) one, but it is found to be metastable and convertible to the ZFC response by a small field excursion. The metastability appears on the low temperature side of the peak in the temperature dependence of the ZFC threshold current. It is suggested that the onset of metastability signals a different thermodynamic ground state.

$\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  is the archetype of strongly type II anisotropic high temperature superconductors. It is composed of a weakly coupled stack of superconducting bilayers, described by the Lawrence-Doniach model of discretized Josephson-coupled planes.<sup>1</sup> The competition between the in-plane interaction between vortex segments, the interplane Josephson and magnetic coupling, and the thermal and static disorder fluctuations divides the  $(H, T)$  plane into vortex fluid and solid phases of two and three dimensional character as the magnetic field tunes the vortex density. Up to now vortex transport information has been obtained either from low current response at fixed  $(H, T)$ ,<sup>2,3</sup> or from a time and field averaged vortex mobility deduced from magnetization variations on cycling  $H$  or  $T$ .<sup>4-6</sup> The present experiments concern *nonlinear transport* ( $V$ - $I$  characteristic) *at fixed*  $(H, T)$  up to and beyond the threshold depinning force, as attempted in Ref.7. Although simple in principle, this could not be done reliably for the low temperature part of the phase diagram because of the problem of avoiding heating from high cur-

rent in the usual continuous methods, a difficulty which is circumvented here by limiting the energy input to short low duty cycle pulses.

Data were obtained for two single crystals each of size about  $1 \times 0.5 \times 0.003$  mm<sup>3</sup> fabricated by a melt cooling technique.<sup>8</sup> The critical temperatures were 83 K and 81 K with a transition width of  $\sim 2$  K at zero field. The anisotropy coefficients estimated from normal state resistivity were  $\gamma \approx 500$  with  $\rho_{ab} \approx 100$   $\mu\Omega$  cm at 90 K. Electrical contact was made by bonding 12  $\mu\text{m}$  gold wires with silver epoxy fired at 900 K. The two current contacts, of resistance  $\sim 3$   $\Omega$ , envelop the ends of the sample over the entire width and extend onto the two faces by  $\sim 150$   $\mu\text{m}$ , whereas the potential contacts of  $\sim 100$   $\mu\text{m}$  diameter are placed symmetrically near the edge of a face about 400  $\mu\text{m}$  apart. The sample was mounted flat against a 15 mm diameter 1 mm thick sapphire disk with silicone grease to ensure both thermal and mechanical anchoring together with a Cernox and a ruthenium oxide thermometer. The arrangement was fixed in

the mixing chamber of a dilution refrigerator placed in the bore of a superconducting magnet which applied the field along the  $c$  axis. For temperatures above 4 K, exchange gas replaced the dilution mixture and the temperature was electronically regulated. The  $V$ - $I$  characteristics were measured by a pulsed four-point technique, the standard excitation being a unipolar isosceles triangle current pulse of maximum amplitude  $10 \text{ mA} < I_M < 400 \text{ mA}$ , total duration  $25 \mu\text{s} < \tau_D < 1 \text{ ms}$  and repetition time  $0.1 \text{ s} < \tau_R < 100 \text{ s}$ . The current feed was symmetrized to minimize the common mode and the differential voltage was read with a 4 MHz bandwidth, low noise ( $2.5 \text{ nV Hz}^{-1/2}$ ) differential preamplifier, the output of which was digitized by a 20 MHz, 8-bit signal averaging oscilloscope to alleviate the increase in noise introduced by the large bandwidth. Averaging over  $10^2$  shots leaves a noise level  $\sim 300 \text{ nV}$ , while the residual common mode contribution to the measured resistance was less than  $2 \mu\Omega$ .

Most of the heat is generated in the contacts; integrating the instantaneous power dissipation over the pulse gives about  $40 \text{ nJ}$  for a typical  $100 \mu\text{s}$  pulse of  $I_M = 20 \text{ mA}$ . A similar estimate for the energy dissipated in the bulk, assuming uniform dissipation along the length of the sample and overestimating in taking no account of the finite threshold, gives  $40 \text{ pJ}$ . These values should be compared with the total phonon heat capacity of  $\sim 2(T/\Theta_D)^3 \text{ mJ K}^{-1} \approx 4 \text{ nJ K}^{-1}$  at  $5 \text{ K}$  ( $\Theta_D \approx 275 \text{ K}$  in Ref.9). Experimentally there is no sign of heating during the pulse.  $V$ - $I$  characteristics for successive temperatures show no tendency to merge with increasing current or pulse duration for pulses shorter than a few hundred microseconds. The onset of heating effects was estimated by applying a square pulse of amplitude just below the threshold and observing with what delay a voltage appears. The delay of about  $250 \mu\text{s}$  (at  $T = 15 \text{ K}$ ) is attributed to thermal diffusion from the contacts to the region under test. The diffusion constant so estimated,  $D \sim 1 \text{ cm}^2 \text{ s}^{-1}$ , corresponds to a phonon mean free path  $l \sim 1 \mu\text{m}$ . The absence of earlier heating indicates either that the vortices themselves do not take up the energy of dissipation or that they relax to the phonons on a time scale shorter than the microsecond resolution of the experiment.

Data on the two samples were qualitatively similar and for brevity data on one sample only are presented here. Typical time resolved raw data and  $V$ - $I$  characteristics are shown at  $T = 4.2 \text{ K}$  in Fig. 1. The  $V$ - $I$  curves show a prethreshold region with some nonlinear continuous creep terminated by either a clear jump or break in slope which is the criterion used for defining the threshold current  $I_{th}$ . The dynamic resistance in the post-threshold region is typically of the order of  $1 \text{ m}\Omega$  ( $\sim 1.2 \text{ m}\Omega$  in Fig. 1). The voltage wave form almost always shows hysteresis in current and usually terminates in a step, if one exists at the threshold current upon increasing current. Our convention is to take the current value corresponding to the upward going feature as the depinning threshold.

Of all the potential information that could be extracted from the  $V$ - $I$  curves, the focus here is on the threshold current. It is nonetheless interesting to note that the dynamic resistance for  $I > I_{th}$  is of the order of what one might expect from the Bardeen-Stephen model<sup>10</sup> for free flux flow if the current is assumed to be distributed in the depth of several

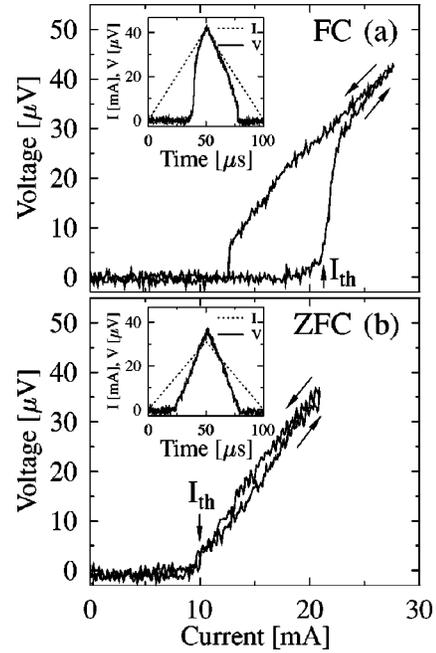


FIG. 1.  $V$ - $I$  characteristics at  $4.2 \text{ K}$  and  $H = 15 \text{ kOe}$  for (a) field cooled (FC) and (b) zero field cooled (ZFC) prepared samples. The insets show the imposed excitation current (dotted line) and the measured voltage (solid line) as a function of time.

microns. It does not, however, show the predicted field dependence. The conclusions of the present article are, however, independent of the detailed analysis of the form of the  $V$ - $I$  response and closer analysis is reserved for a future article on current distribution and its influence on the nonlinear response.

Figure 2 shows the threshold depinning current  $I_{th}$  for field cooled (FC) and zero field cooled (ZFC) preparations as a function of temperature for fixed fields. When the sample is prepared by cooling in zero field the threshold current at fixed  $H$  increases approximately linearly with the temperature up to a maximum at  $T = T_p$  which is about double the zero temperature value, after which it turns around to diminish and eventually vanish. If, however, the sample is prepared by first setting the field at  $T > T_p$  and then cooling to  $T < T_p$ , the value of  $I_{th}$  for the same temperature is higher by a factor that depends on the temperature vs time profile of cooling, but attained a value of 3 at the lowest temperature.

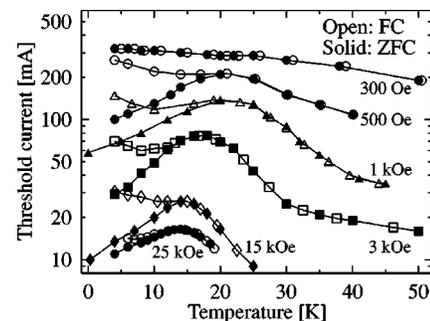


FIG. 2. Temperature dependence of threshold current at fixed fields for FC and ZFC prepared samples, measured on increasing the temperature above the initial preparation temperature.

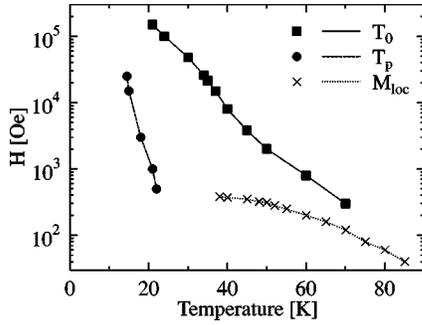


FIG. 3. Features of the phase diagram. Filled symbols represent the present work. The boundary line defined by vanishing threshold current is denoted by  $T_0$ .  $T_p$  is the locus of the maximum in the threshold current for ZFC state and the limit of metastability of FC state.  $M_{loc}$  is the first order transition line measured by the local magnetization step (Ref. 12).

Unlike the ZFC case, the FC threshold diminishes upon raising the temperature above the preparation point. The exact form of the FC curve depends on the cooling rate; it always coincides with the ZFC curve at and beyond the peak in the latter but occasionally merges at lower temperature. The irreversibility in temperature is also delimited by the peak. The different preparations are also reflected in the form of the  $V$ - $I$  characteristic; the low temperature FC  $V$ - $I$  shows much stronger hysteresis than the ZFC curve; the FC hysteresis diminishes with temperature while the ZFC hysteresis increases until at  $T_p$  the two coincide. Both the FC and ZFC threshold currents are irreversible in temperature: they remain practically constant on reducing the temperature again as long as  $T < T_p$ . Both preparations give identical, reversible results for  $T > T_p$  and for  $H < 300$  Oe, regardless of the preparation temperature. No values are found below the (unique) curve defined by increasing the temperature from any ZFC preparation.

Most significantly, the higher threshold FC state is *metastable*: by perturbing the field by a few hundred Oe up or down and returning to the starting value, the  $V$ - $I$  characteristic is converted to be very close to that of the ZFC prepared state. Once the perturbation is effected, the conversion takes place continuously over a few minutes. The locus  $T_p(H)$  of the peak in the ZFC threshold value separates a low temperature region, where metastable states exist, from a high temperature region where a unique, preparation independent, and apparently stable  $V$ - $I$  characteristic is found, presumably reflecting a unique, stable state for the vortices.

The locus  $T_0(H)$  of points on the  $(H, T)$  plane at which the threshold current vanishes and the locus  $T_p(H)$  of the peak that corresponds to the onset of metastability for the FC preparation are shown in Fig. 3. We also measured the magnetic moment hysteresis loops at fixed temperatures on the same sample by a superconducting quantum interference device (SQUID). The points at which the magnetic moment becomes reversible coincide well with the points of the ‘‘magnetization irreversibility’’ line measured by Schilling *et al.*<sup>11</sup> Also shown in Fig. 3 is the first order transition line seen in local magnetization measurements.<sup>12</sup>

We interpret the vanishing of  $I_{th}$  [the  $T_0(H)$  line] to indicate that the elastic shear modulus vanishes. The locus is

similar to, but to the right of, that of the magnetization irreversibility, which has traditionally been associated with vanishing of the threshold force.<sup>11</sup> Clearly the trend is the same, but the vanishing of our  $V$ - $I$  threshold occurs at distinctly higher temperatures. The difference might be understood as arising from prethreshold vortex mobility (creep), which is sufficient to ensure reversibility of the magnetization on the long time scale SQUID measurements even for  $I_{th} \neq 0$ . The Bean critical state profile is a short time configuration that evolves toward a uniform equilibrium distribution for long times<sup>13</sup>; e.g., if the prethreshold response were linear and characterized by volume resistivity  $\rho$ , the evolution would be described by a diffusion constant  $D \sim \rho/\mu_0$ . Magnetization would become homogeneous over 1 mm on a time scale of 10 s for  $D \sim 10^{-1} \text{ mm}^2 \text{ s}^{-1}$  corresponding to  $\rho = 10 \text{ p}\Omega \text{ cm}$ , indicating a sample resistance of 30 n $\Omega$ . Our measurements show prethreshold resistivities much higher than this on the low temperature side of the point where the threshold vanishes.

The onset of metastability  $T_p(H)$  is a valuable indicator for resolving the phase diagram. It may seem at first sight surprising that it is the FC and not the ZFC preparation that is associated with metastability. The critical state Bean model displays metastability for ZFC preparation, but this concerns the density profile of vortices, which has little influence on local order as long as the length scale of the density fluctuation is longer than the correlation length. A similar FC metastability converting toward ZFC behavior is found in the NbSe<sub>2</sub> vortex system.<sup>14</sup> Remembering that the  $V$ - $I$  characteristics no longer show a distinction between FC and ZFC preparations for fields below 300 Oe, the picture also is consistent with the step in local magnetization with temperature that occurs for low fields  $\leq 500$  Oe (Fig. 3), which was argued to be a thermodynamic signature of a first order phase transition, probably from a liquid directly into a three dimensional (3D) glass.<sup>12</sup>

We suggest, then, that on cooling from a vortex liquid in a field above the decoupling field of  $\sim 500$  Oe, order can set in for each plane independently to create a 2D solid with an in-plane correlation length ultimately limited by pinning to the disorder field. As thermal fluctuations weaken, the higher entropy of the independent 2D correlated planes of vortex segments loses out to the lower energy of a state possessing also plane to plane correlation. However, there may be no easily accessible path by which to attain equilibrium if each plane has been randomly prepinned on field cooling, and it is only by annealing that the transformation can take place. The vortex density change associated with a change in flux could precipitate the conversion by reorienting the 2D crystallites to best accommodate the random pins; during this process they can lock together. Whether lower threshold current should be associated with a 3D or a 2D state depends on whether the in-plane or the out of plane characteristic dominates.<sup>15</sup> We believe the former to be the case where the 3D state may be supposed to have a lower depinning threshold as fewer pins would be operative.<sup>16</sup> As there can be some partial thermal annealing upon cooling through the 2D to 3D glass transition, there can be a mixture of phases. The whole system must, however, move together so the threshold force takes on intermediate values, but never a value lower than

that of the pure 3D phase. In this interpretation,  $T_p(H)$  corresponds to a first order phase transition, possibly a continuation of the transition line indicated by the local magnetization.<sup>12,17</sup>

We take pleasure in acknowledging discussion with Nicole Bontemps, Thierry Giamarchi, Pierre Le Doussal, and

István Tüttő. B. Sas thanks the Direction des Relations Internationales of the Atomic Energy Commission for financial support to visit the Saclay laboratory. Research in Hungary has been supported by Grant No. OTKA-T029877. We are grateful also for the support of the ‘‘BALATON’’ collaboration program of the French and Hungarian Foreign Affairs Ministries.

- 
- <sup>1</sup>W.E. Lawrence and S. Doniach, in *Proceedings of the XIIIth International Conference on Low Temperatures*, edited by E. Kanda (Keigaku, Tokyo, 1971), p. 361.
- <sup>2</sup>T.T.M. Palstra, B. Batlogg, R.B. van Dover, L.F. Schneemeyer, and J.V. Waszczak, *Phys. Rev. B* **41**, 6621 (1990).
- <sup>3</sup>H. Safar, E. Rodriguez, F. de la Cruz, P.L. Gammel, L.F. Schneemeyer, and D.J. Bishop, *Phys. Rev. B* **46**, 14 238 (1992).
- <sup>4</sup>Y. Yeshurun, N. Bontemps, L. Burlachkov, and A. Kapitulnik, *Phys. Rev. B* **49**, 1548 (1994).
- <sup>5</sup>F. Iga, A.K. Grover, Y. Yamaguchi, Y. Nishihara, N. Goyal, and S.V. Bhat, *Phys. Rev. B* **51**, 8521 (1995).
- <sup>6</sup>H. Pastoriza, F. de la Cruz, D.B. Mitzi, and A. Kapitulnik, *Phys. Rev. B* **46**, 9278 (1992).
- <sup>7</sup>M.P. Maley, J.H. Cho, S. Fleshler, and L.N. Bulaevski, *Appl. Supercond.* **2**, 667 (1994).
- <sup>8</sup>B. Keszei, Gy. Szabó, J. Vandlik, L. Pogány, and G. Oszlányi, *J. Less-Common Met.* **155**, 229 (1989); J.R. Cooper, L. Forró, and B. Keszei, *Nature (London)* **343**, 444 (1990).
- <sup>9</sup>B. Revaz, A. Junod, A. Mirmelstein, A. Erb, J.-Y. Genoud, and G. Triscone, in *Proceedings of the XXth Conference on Low Temperature Physics*, edited by S. Danis, V. Gregor, and K. Zaveta [*Czech. J. Phys.* **46**, 1205 (1996)].
- <sup>10</sup>J. Bardeen and M. Stephen, *Phys. Rev.* **140**, 1197 (1965).
- <sup>11</sup>A. Schilling, R. Jin, J.D. Guo, and H.R. Ott, *Phys. Rev. Lett.* **71**, 1899 (1993).
- <sup>12</sup>E. Zeldov, D. Majer, M. Konczykowski, V.B. Geshkenbein, V.M. Vinokur, and H. Shtrikman, *Nature (London)* **375**, 373 (1995).
- <sup>13</sup>S. Berry, M. Konczykowski, P.H. Kes, and E. Zeldov, *Physica C* **282-287**, 2259 (1997).
- <sup>14</sup>W. Henderson, E.Y. Andrei, M.J. Higgins, and S. Bhattacharya, *Phys. Rev. Lett.* **77**, 2077 (1996).
- <sup>15</sup>F. de la Cruz, E. Rodriguez, H. Pastoriza, A. Arribier, and M.F. Goffman, *Physica B* **197**, 596 (1994).
- <sup>16</sup>A.E. Koshelev and P.H. Kes, *Phys. Rev. B* **48**, 6539 (1993).
- <sup>17</sup>B. Khaykovich, M. Konczykowski, E. Zeldov, R.A. Doyle, D. Majer, P.H. Kes, and T.W. Li, *Phys. Rev. B* **56**, R517 (1997).