

## Magnetic properties of single-crystal $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$ : Experimental evidence for a dimensional crossover

A. K. Pradhan, S. B. Roy, and P. Chaddah

*Low Temperature Physics Group, Centre for Advanced Technology, Indore 452 013, India*

C. Chen and B. M. Wanklyn

*Clarendon Laboratory, Department of Physics, Oxford University, Parks Road, Oxford, OX1 3PU, United Kingdom*

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Magnetization curves of high-quality superconducting  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  single crystals in fields oriented parallel to the  $c$  axis have been reported at various temperatures. The irreversibility line and magnetic relaxation of this single crystal have been measured in magnetic fields parallel to the  $c$  axis of the crystal. We have observed a low-field anomaly in the magnetization together with a sharp rise of the irreversibility line and a nonlogarithmic magnetic relaxation at 25 K. We argue that 25 K is a boundary temperature where the system undergoes a dimensional crossover from the three-dimensional (3D) to the 2D regime. The relaxation rate also predicts that although the flux creep above this crossover temperature follows the conventional Kim-Anderson thermally activated flux-creep model, below this crossover temperature it follows that predicted by the collective flux pinning or vortex-glass models.

### I. INTRODUCTION

Magnetic measurements have revealed much unusual behavior of high- $T_c$  superconductors (HTSC) in their mixed states. The magnetization measurements of HTSC as a function of magnetic field, temperature, magnetic history and time have provided immense information on vortex dynamics, flux pinning and dimensionality of the system. In HTSC, a very interesting anomalous feature in the isothermal hysteresis loops which show an enhancement of pinning in low-field region, has been observed.<sup>1-3</sup> It is established that oxygen vacancies are effective flux pinning centers<sup>4</sup> and that spatial variation of the oxygen ordering may well account for the anomalous "fishtail" magnetization feature observed in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$  (YBCO) crystal.<sup>1-3</sup> Very recently, such sharp features have been observed in  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  (BSCCO) crystals<sup>5,6</sup> below 40 K where the irreversibility field grows rapidly. In contrast to YBCO this anomaly in a BSCCO crystal was attributed to the pinning due to the matching of two-dimensional (2D) pancake vortices and microstructural 2D defects and also to a possible crossover from a 3D to a 2D pinning.

For a highly anisotropic layered superconductor such as BSCCO, the vortex line can be regarded as 2D pancake vortices confined to the  $\text{CuO}_2$  layers but weakly interacting between layers by Josephson and magnetic coupling. A decoupling transition called a "crossover" from 3D flux lattice to 2D pancake vortices may take place at high temperatures and high fields, when the energy due to thermal fluctuation becomes comparable with the Josephson and magnetic coupling energies between the vortices in different  $\text{CuO}_2$  layers.<sup>7</sup> This crossover transition is also explained in the framework of a vortex fluid to a vortex-glass transition<sup>8</sup> at the irreversibility line. However, these phenomena, no doubt, point to magnetic relaxation experiments to look for a possible crossover.

Another striking phenomenon in BSCCO is the observation of unusual time decay near the irreversibility temperature. It is interesting that nonlogarithmic time decay of magnetization has been reported<sup>9,10</sup> in many HTSC which cannot be easily described by the Anderson-Kim model. Several models have been proposed to describe these phenomena, which include the thermally activated flux flow (TAFF),<sup>11</sup> collective creep,<sup>12</sup> and self-organized criticality.<sup>13,14</sup> Fisher<sup>8,15</sup> considered long-range cooperative vortex interactions and discussed a vortex-glass model. The logarithmic decay with time for a metastable state is extrapolated to be in the form<sup>16</sup>

$$M(t) = M_0 / [1 + (\mu K_B T / U_0) \ln(t/t_0)]^{1/\mu}, \quad (1)$$

where  $M_0$  and  $M(t)$  magnetization at time  $t=0$  and at  $t$ ,  $K_B$  the Boltzmann constant,  $U_0$  the activation energy, and  $\mu$  is related to the glass exponent. Considering the above aspects it is clear that there are many controversies regarding the magnetic relaxation in HTSC especially for BSCCO. Moreover, correlation of all the magnetic studies in a systematic manner is lacking to reveal a 3D to 2D type of crossover in a BSCCO crystal.

In this paper, we report our measurements of the isothermal magnetization at different temperatures and irreversibility line,  $H_{\text{irr}}(T)$  and  $T_{\text{irr}}(H)$  in a BSCCO single crystal. We have also measured the magnetic relaxation in a zero-field-cooled (ZFC) BSCCO crystal at different temperatures and calculated the relaxation rate and activation energy in a wide temperature region. The origin of the anomalous magnetization feature, irreversibility line, and nonlogarithmic time decay has been discussed in terms of dimensional crossover.

### II. EXPERIMENTAL DETAILS

The single crystal used in this study was grown with the flux method reported earlier.<sup>17,18</sup> The quality of the

crystal was examined by x-ray diffraction, resistivity, and magnetization measurements. The x-ray diffraction results showed a single phase  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  which was also confirmed by electronprobe microanalysis.<sup>17</sup> A sharp superconducting transition  $T_c$  at 89 K was observed in both FC and ZFC magnetization experiments in a field of 1 mT. The high quality as-grown crystal was used for the present studies and has a typical dimension  $1.7 \times 1 \times 0.3 \text{ mm}^3$  and weighs 2.3 mg.

The magnetization data were recorded with a superconducting quantum interference device (SQUID) magnetometer (Quantum Design MPMS5) over a wide range of temperature (5–100 K) and magnetic fields (1 mT–5 T). The magnetic field was always applied parallel to the  $c$  axis of the crystal. The isothermal magnetization loops were measured at different temperatures in a ZFC sample. The ZFC and FC magnetization was recorded as a function of temperature at different field values as well as a function of field at different temperatures to find the irreversibility line for each case separately.

For magnetic relaxation experiments, the crystal was first zero-field-cooled from above  $T_c$  to a desired temperature  $T$  and a magnetic field  $H > H_{II}$  (field for saturation remanence in the crystal at that temperature) was then applied, and the field was ramped down to zero after magnetizing the sample. As soon as the field was set zero, the remanent magnetization  $M_0$  of the sample was measured as a function of time,  $t$ . Using standard procedures with the commercial SQUID magnetometer, we cannot get relaxation information earlier than 100 seconds after the field is set zero. The travel length of the sample in each scan was 4 cm and we averaged three scans for all measurements. It should be noted that for our relaxation measurements in the remanent state the superconducting magnet is off (after setting the field to zero), hence inhomogeneities of its magnetic field are not relevant.

### III. RESULTS AND DISCUSSION

#### A. Hysteresis loops

Figure 1(a) shows the isothermal magnetization curves at various temperatures below 40 K. The field was increased to 5 T (which is much above the  $H_{II}$  of the crystal at that temperature) after the sample was zero-field cooled to the desired temperature, and the reverse magnetization curve was recorded. However, for  $T = 30 \text{ K}$ , we have recorded the complete hysteresis loop including the virgin magnetization as shown in Fig. 1. It may be noted that for all such measurements the magnet was swept in a hysteresis mode. The magnetization hysteresis curves did not show anomalous behavior at low fields in the temperature range 20–40 K as reported.<sup>5,6</sup> However, we noticed a blip in magnetization at 25 K. This is quite prominent in Fig. 1(b) where the magnetic field was increased to 5 T at 25 K and the reverse magnetization was recorded in the field region  $-5$  to  $5 \text{ T}$  [only a portion of the reverse magnetization, i.e.,  $-0.3$  to  $0.3 \text{ T}$  is shown in Fig. 1(b)]. It is noted that in this case the magnet was kept in persistent mode for each field setting to avoid any

noise or field instability. We noticed a more pronounced low-field feature at 25 K.

The anomalous double peak structure in YBCO has been explained in the framework of oxygen-deficient defects which act as pinning centers in a low-field region and turn normal at high fields.<sup>1</sup> The similar observation by Yang, Abell, and Gough<sup>5</sup> in a BSCCO crystal has been explained as a matching of 2D pancake vortices with a

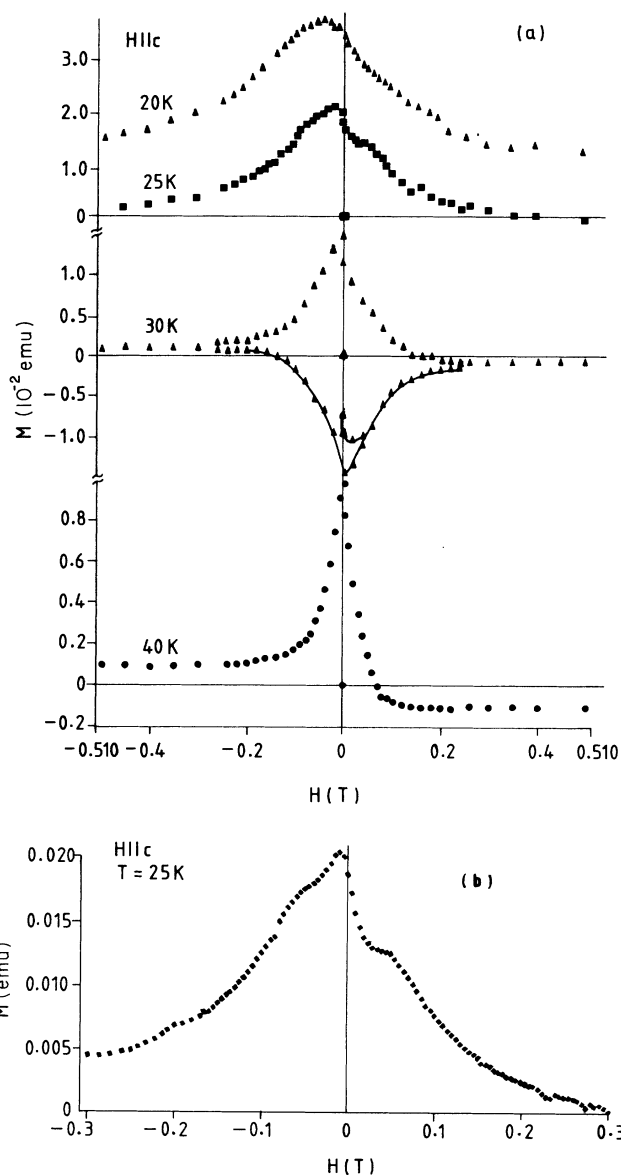


FIG. 1. (a) Isothermal magnetization hysteresis curves at  $T = 40, 30, 25$ , and  $20 \text{ K}$  of  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  crystal. The magnetic field in each case ramped above  $H_{II}$  of the crystal and reverse magnetization curves are taken. The hysteresis at  $T = 30 \text{ K}$  has been shown including virgin magnetization. All the curves are shown for ZFC crystal. (b) Reverse magnetization curve at  $T = 25 \text{ K}$  is shown which is recorded with a very small field interval after magnetizing a ZFC crystal above  $H_{II}$ . The curve is recorded taking each point in persistent mode of the magnet.

2D defect structure and Tamegai *et al.*<sup>6</sup> discussed this feature in terms of dimensional crossover. The anomaly we noticed at 25 K may have several origins. It is also reasonable to think that whether this phenomenon is signaling the onset of a significant cooperative phenomenon leading to a vortex-glass state. Our results did not show a drastic pinning as reported by Yang, Abell, and Gough.<sup>5</sup> However, if it is really a crossover from 3D to 2D state at such temperature, other experimental probes such as irreversibility of magnetization, magnetic relaxation should reveal this phenomenon because they exhibit significant characteristics at such transitions.

We may mention that a fall in magnetization, as the field is reversed to zero, can arise because local internal fields fall below  $H_{c1}$ .<sup>19</sup> Tamegai *et al.*<sup>6,20</sup> have pursued this idea and observed anomalies in local internal fields close to zero applied fields. We do not believe that the anomaly we observe at 25 K has an origin related to  $H_{c1}$ , because our observed anomaly disappears below 20 K.

### B. Irreversibility line

Figure 2 shows the  $H$ - $T$  phase diagram of a BSCCO crystal where  $H_{irr}(T)$  and  $T_{irr}(H)$  were plotted. The rapid decrease in ZFC magnetization is caused by a very fast drop of the critical current density at temperatures around 20–30 K. The point of coincidence between the FC and ZFC curves at a fixed field,  $T_{irr}(H)$ , is a signature of the transition into the reversible regime in the  $H$ - $T$  plane.  $H_{irr}(T)$ , determined from the point of field of the merger of the upward and downward legs of the hysteresis curve at a fixed temperature, is also the same signature of the transition into the reversible regime at different temperatures. Both methods yield similar results as shown in Fig. 2 and are close to those obtained by others;<sup>21–23</sup> however, Grover *et al.*<sup>24</sup> and Suenaga *et al.*<sup>25</sup> found that the  $H_{irr}(T)$  line lies above the  $T_{irr}(H)$  line in Nb and Nb<sub>3</sub>Sn, respectively. Inset in Fig. 2 shows how  $T_{irr}(H)$  is drastically suppressed even with a very small magnetic field applied parallel to the  $c$  axis of the

BSCCO crystal.

Understanding the physical origin of the irreversibility line is of considerable practical importance, since dissipationless supercurrents will not flow above it. It is still the subject of controversy whether the irreversibility line in HTSC is related to melting in a pure system. The irreversibility of BSCCO at  $H \approx 1$  T is only around 30 K, even though the temperature corresponding to the upper critical field is around 80 K. The extreme anisotropy of BSCCO leads to a vortex lattice which becomes increasingly 2D at high fields and at high temperatures. The apparent collapse of the critical region near 2 T may be related to the 3D to 2D crossover fields (see Fig. 2). This was also observed by Pradhan *et al.*<sup>26</sup> in their voltage-current characteristic measurements in a similar BSCCO crystal. Fisher<sup>27</sup> has discussed that the system crosses over to 3D with power law diverging conductivity temperature of order 25 K in a field of 1 to 2 T. Safar *et al.*<sup>22</sup> reported that around 26 to 28 K, the crossover from thermal activation to the low-temperature vortex-glass critical behavior occurs in magnetic fields from 2 to 6 T. In Fig. 2, if we extrapolate the irreversibility line to the temperature axis, we get  $T_M \approx 26$  K. Thermal fluctuations of the flux line lattice give rise to a considerable reduction of the pinning strength and thereby a sharp fall of the critical current density at a temperature above a certain depinning temperature was observed. It seems reasonable to expect a change in flux-creep behavior in this temperature range and look for whether there is a melting transition or simply a thermally activated depinning of the vortex lattice.

It is well known that the BSCCO sample has a large anisotropy ( $\gamma \approx 0.02$ ),<sup>28</sup> a very small coherence length, and weak interlayer coupling. The 2D pancake vortices are created spontaneously by thermal fluctuations even in a zero-magnetic field giving rise to a Kosterlitz-Thouless type transition.<sup>8,26,29</sup> Even a small magnetic field perpendicular to the  $c$  axis enhances the dissociation of bound vortex-antivortex pairs in BSCCO. For a field parallel to the  $c$  axis, the formation of these vortices is extremely enhanced, causing a large dissipation and suppressing the

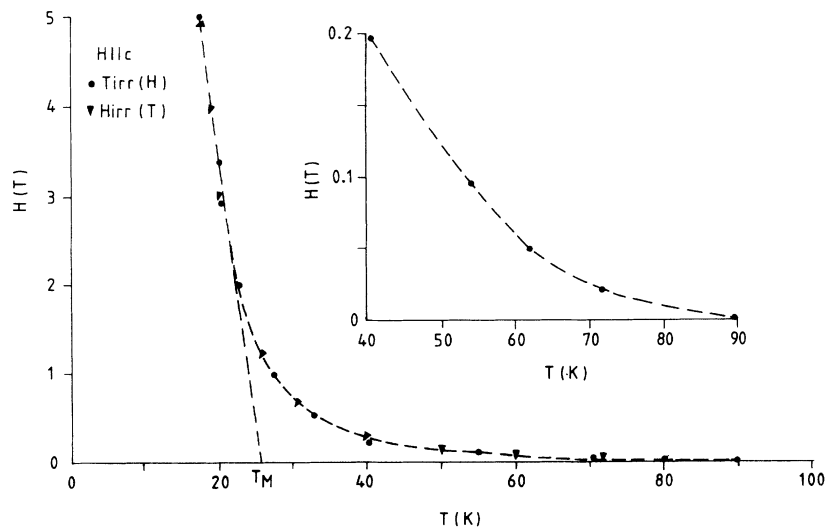


FIG. 2. Irreversibility line,  $T_{irr}(H)$  and  $H_{irr}(T)$  of single crystal  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  as a function of temperature. The extrapolation of the linear portion of the line to the temperature axis gives  $T_M$  of the system. The inset shows irreversibility line at high-temperature region where the line is more suppressed.

irreversibility line. The anisotropy is small enough that 2D effects in BSCCO dominate over a wide region of the phase diagram. In low fields, the system will be three dimensional, but when  $\gamma(\phi_0/B)^{1/2} \ll d$ , where  $\phi_0$  is the flux quantum,  $B$  the applied field and  $d$  the interlayer spacing, the layers become only weakly coupled. Therefore, irreversibility line separates two phases: below this line a 3D vortex lattice and above is a 2D vortex liquid phase. A recent report<sup>30</sup> shows that the irreversibility line in BSCCO can be described from the concept of melting using the Lindemann melting criterion for vortex-lattice melting and predicts for a crossover from a 3D to 2D regime. Although the method of determination of irreversibility line in Ref. 30 is different from ours, we get essentially the same result from our experiments.

### C. Magnetic relaxation

We have measured magnetization vs  $\ln t$  data at different temperatures from 5 to 80 K. To avoid the controversy of the incomplete field penetration, we have performed the time decay experiments only above the saturation remanence field,  $H_{II}$ , of the crystal. As the field for saturation magnetization (and  $J_c$ ) rises with falling temperature, we experimentally detected that  $H_{II}$  of the sample at 5 K is below 4.5 T. This field value of 4.5 T was used for every temperature to ensure that the maximum field used to magnetize the sample is always above the  $H_{II}$  of the crystal at that temperature. The magnetization vs  $\ln t$  plots roughly give quite good straight lines (except for a temperature region 20 to 30 K), indicating the logarithmic behavior of the time decay. The decay curves at 20 and 25 K (see Fig. 3) deviate drastically from the logarithmic behavior exhibiting nonlinearity in the slope. This deviation starts around 30 K, peaks at 25 K,

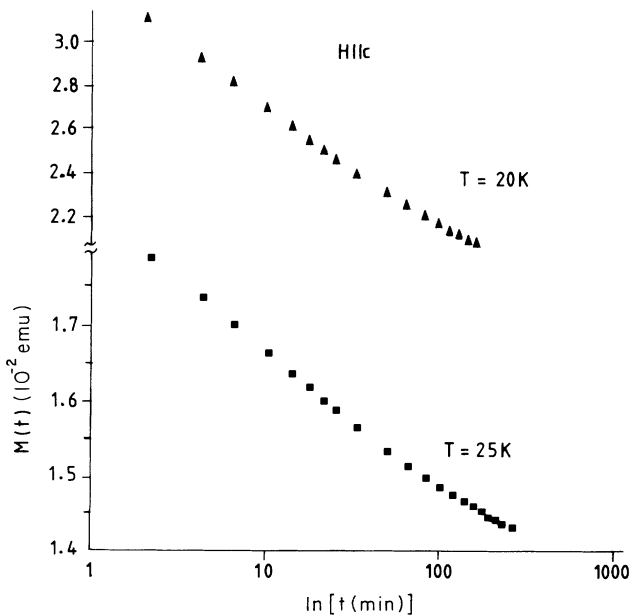


FIG. 3. Magnetization vs  $\ln t$  of a ZFC BSCCO crystal at  $T = 20$  and 25 K showing nonlogarithmic behavior.

and again disappears at 18 K. We have also measured the decay curve of the same ZFC BSCCO crystal at 20 and 25 K in a magnetic field of 0.2 T. The nonlogarithmic decay is again very prominent at 25 K. Figure 4 shows the rate of relaxation,  $S = -1/M_0(dM/d \ln t)$  vs temperatures. As can be seen in Fig. 4 the magnetization exhibits a small relaxation rate at lower temperatures (5–10 K), gradually increases with increasing temperature, reaching a maximum at 20 K, and then decreases at higher temperatures. However, for the first time we found a second peak in relaxation rate near 60 K in a BSCCO crystal. This peak, although small, is prominent. The inset in Fig. 4 shows remanent magnetization vs temperature where a sharp increase in  $M_{rem}$  was found around 20 to 25 K. The activation energies  $U_0$ 's were calculated and are shown in Fig. 5 as a function of temperature. We noticed some sort of plateau between 35 to 55 K.  $U_0$  increases very slowly up to 25 K (inset in Fig. 5) after which it sharply increases. We notice two sharp steps of increases in  $U_0$ , one from 25 to 35 K and another from 60 to 80 K.

The HTSC exhibits strong logarithmic magnetic relaxation with time and this is explained by a thermally activated flux creep<sup>2,31</sup> proposed by Anderson. Using this model, for  $t \gg \tau$  (where  $\tau \sim 10^{-6} - 10^{-12}$  s is the characteristic relaxation time of a flux bundle), one obtains

$$U_0 = -(K_B T M_0) / (dM/d \ln t), \quad (2)$$

which is extensively used to determine the value of  $U_0$ . Yeshurun and Malozemoff<sup>9</sup> invoked the critical state model to explain the details of magnetization decay in HTSC. The Bean's model can be combined with an expression for the time decay of persistent current

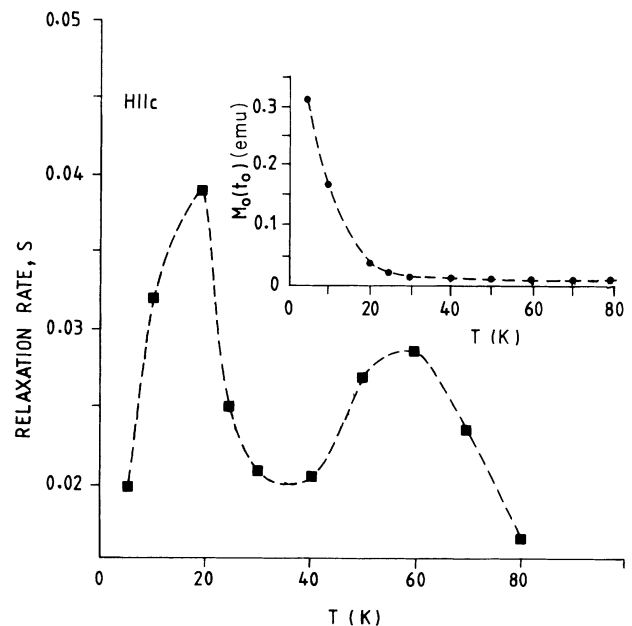


FIG. 4. Magnetization decay rate  $S$  vs temperature. The inset shows remanent magnetization vs temperature which sharply rises at around 25 K.

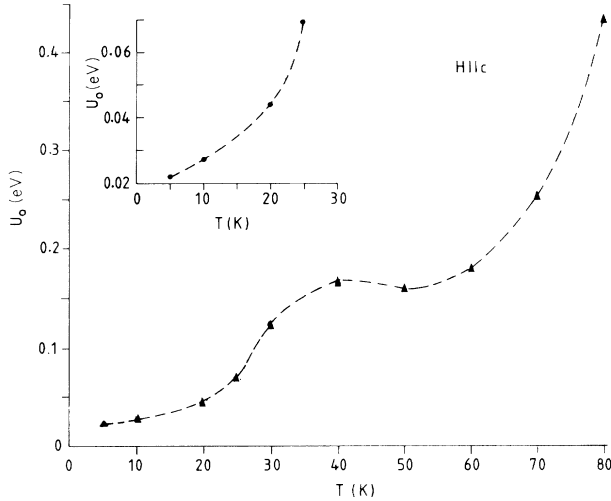


FIG. 5. Activation energy vs temperature showing three distinct regions. The inset shows the activation energy in a low-temperature region.

$$J_c(t) = J_{co} [1 + (K_\beta T / U_0) \ln(t/\tau)] , \quad (3)$$

where  $J_{co}$  is the critical current density when no thermal disturbance is present. This equation basically explains that there are constant  $B$  shells in samples, where the shielding current at each shell decays because of flux creep. Chaddah and Bhagwat<sup>32</sup> modified this equation introducing an extra term  $A(H)$  which depends on the ratio  $H/H_I$  and accordingly  $U_0$  can correctly be determined. ( $H_I$  is the field where virgin leg merges with the return hysteresis leg.)

Though the relaxation in our experiment on a BSCCO crystal satisfies well the conventional Anderson's flux-creep picture above 30 K, the nonlogarithmic relaxation found in the temperature range 20–30 K demonstrates the breakdown of this picture. However, the nonlogarithmic or some sort of quasiexponential behavior observed in BSCCO favors the predictions of the collective flux creep<sup>12,33</sup> or vortex-glass model.<sup>15</sup> In our analysis using Eq. (1) we get the values of exponent  $\mu$  in the range of 1 considering  $U_{eff}$  in the quasiexponential region separately for  $T=20$  and 25 K. However, the values of  $\mu$  below 20 K are found to be less than 1 and in good agreement with Sun *et al.*<sup>34</sup> Furthermore, the peak at 20 K in decay rate predicts also some sort of crossover followed by a very sharp increase in  $M_{rem}$  in that region. Our results, therefore, strongly support the concept of collective flux pinning which may also be taken as evidence for a vortex-glass state produced by random pinning and large thermal fluctuations. In BSCCO crystals, these random pinnings are oxygen vacancies which play a major role in pinning in a low-temperature regime.

The plateau in the relaxation rate above 30 K is very exciting and generates a variety of competing models involving distributions of pinning barrier heights,<sup>35</sup> critical current densities,<sup>36</sup> and also can be described in the con-

text of vortex-glass or collective pinning models.<sup>16,37</sup> The second peak in the decay rate is the reflection of the softening of the flux lattice on approaching the melting line as discussed by Vinokur, Feigelman, and Geshkenbein.<sup>38</sup> A very similar peak has also been observed<sup>39</sup> in YBCO at 85 K. However, the cause of the origin of the second peak is not very clear. Similarly, the sharp increase in activation energy at 25 K clearly predicts a crossover and the observation of plateau is clear evidence of a cooperative phenomenon leading to collective flux creep or vortex-glass state. Using the magnetic relaxation data, a consistent interpretation within the collective pinning or vortex-glass model is found.

#### D. 3D-2D crossover

As discussed earlier, for a very strongly anisotropic layered superconductor like BSCCO, flux vortices which are regarded as 2D pancakes on adjacent CuO planes interact weakly by Josephson and magnetic couplings. At low temperatures, the vortex-vortex interaction between layers is stronger than the thermal fluctuation energies and as a result the 3D flux lattice penetrates the whole sample. Above the irreversibility line, the coupling between layers becomes comparable to the thermal fluctuation energy and magnetic decoupling occurs giving rise to 2D vortices. This is what exactly happens in a BSCCO crystal. Around 25 K such decoupling process starts giving rise to an anomaly in magnetization and this is the temperature where  $T_M$  for the irreversibility line is identified, critical current density sharply increased, and deviation from logarithmic behavior in time decay occurred. Once the vortices are decoupled between the layers, within any single layer the pancake vortices can readjust themselves to take advantage of the maximum available pinning energy leading to a sudden increase in critical current. Therefore, below 25 K, flux pinning becomes very strong which results in a strong interaction between pancakes in different layers and 3D flux line pinning. It can be argued that 25 K is the boundary temperature between coupling and decoupling of 2D pancake vortices. Therefore, above this temperature the pancake vortices undergo a 3D to 2D transition and obey thermally activated flux-creep behavior. Below this crossover temperature collective flux-creep or vortex-glass model is valid in magnetic relaxation.

#### IV. CONCLUSION

Magnetic hysteresis measurements signal the onset of a 3D to 2D crossover at 25 K. On raising the temperature above 25 K, thermal fluctuations result in a vortex structure transition from 3D to 2D pancake vortices on the individual CuO<sub>2</sub> layers with weak correlation between layers. From the temperature dependence of the irreversibility line, the crossover from a 3D to 2D regime manifests itself in a distinct sharp rise of fields towards lower temperatures. The magnetic relaxation experiments showed two distinct regions of pinning separated by a plateau. The pinning at lower temperatures is due to the vortex-glass or collective pinning resulting from a cross-

over indicated by a plateau in activation energy. The high-temperature peak gives the evidence of softening of flux lattice. The interesting phenomenon is the observation of nonlogarithmic time decay which is very prom-

inent at 25 K. We describe this as a crossover from a 3D to 2D regime, which can be explained more effectively in the framework of either a vortex-glass or a collective flux pinning model.

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