

## Transition from elastic to plastic vortex phase and its evolution with quenched disorder in $\text{YBa}_2\text{Cu}_3\text{O}_y$ single crystals

S. Kokkaliaris,\* A. A. Zhukov,† and P. A. J. de Groot

*Department of Physics and Astronomy, University of Southampton, Southampton, SO17 1BJ, United Kingdom*

R. Gagnon and L. Taillefer‡

*Department of Physics, McGill University, Montreal, Quebec, Canada H3A 2T8*

T. Wolf

*Forschungszentrum Karlsruhe, Institut für Technische Physik, Postfach 3640, D-76021 Karlsruhe, Germany*

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We report on a detailed investigation of history effects in the magnetic hysteresis of pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals with various types and densities of pinning sites. A partial magnetization loop technique is employed that enables us to detect the point where topological disorder first invades the vortex system. Accordingly, studies of detwinned single crystals with very low densities of point defects reveal a transition in the mixed state of the superconductor that separates a dislocation-free Bragg glass from a highly disordered vortex phase. The transition line is identified in the field-temperature phase diagram and found to lie in the vicinity of the onset of the second magnetization peak. The effect of decreasing oxygen stoichiometry in the studied region ( $6.550 \leq y \leq 6.999$ ) is the shift of this boundary to lower fields, and its final disappearance for high values of the oxygen deficiency in agreement with theoretical predictions. Above the transition metastable topological defects proliferate in the vortex lattice resulting in prominent history effects in the critical current. The last diminish at high enough fields for low oxygen concentrations near optimal doping, an effect not seen for samples close to the stoichiometric state. We also study the influence of extended defects and find that a low density of twin boundaries does not affect the Bragg glass significantly. However, a high concentration of twins as well as low densities of columnar defects are shown to suppress the transition and eliminate the memory effects.

### I. INTRODUCTION

In the traditional mean-field limit the field-temperature phase diagram of type-II superconductors comprises a Meissner-Ochsenfeld phase below the lower critical field  $H_{c1}$ , above which vortices penetrate the superconductor forming a homogeneous triangular lattice.<sup>1</sup> This constitutes the so-called Shubnikov or mixed phase that persists up to the upper critical field  $H_{c2}$  where superconductivity disappears.<sup>1</sup> In the case of the high-temperature superconductors (HTSC's), however, the significantly enhanced role of fluctuations due to the high operating temperatures, large anisotropies, and penetration depths has stimulated a re-examination of this simplified picture. Therefore it has been predicted that in these systems the vortex lattice melts well below  $H_{c2}$  thus defining a transition between a solid and a liquid vortex phase.<sup>2</sup> Indeed, such a melting transition has been observed in numerous experiments with a variety of techniques, and for clean samples has been established to be of the first order.<sup>3–10</sup>

The mixed state in the phase diagram of the HTSC becomes even more complicated if one considers the presence of quenched disorder and its interplay with thermal fluctuations. As shown long ago the Abrikosov lattice is unstable to point disorder beyond the Larkin length.<sup>11</sup> At larger scales it was argued that the vortex system (VS) forms a glassy phase characterized by zero linear resistivity even at finite temperatures.<sup>12–14</sup> This phase lacks translational order and

topological defects such as dislocations are expected to be favored.<sup>14</sup> The existence of the vortex glass has been postulated by several experiments on samples characterized by strong disorder, where a continuous transition from the vortex liquid to a phase with vanishing linear resistivity was observed.<sup>15,16</sup> Nevertheless, by taking into account the fact that at large scales the periodic structure of the lattice becomes significant, recent theoretical investigations have established that at low magnetic fields quasi-long-range order survives in the flux-line lattice in an elastic phase (termed the Bragg glass<sup>17</sup>) where dislocations are energetically unfavorable.<sup>17–22</sup> The Bragg glass should be experimentally discernible in pure samples and can disappear in two ways: with increasing temperature it melts into a liquid via a first-order phase transition, whereas by raising the field, effective disorder is enhanced leading to vortex entanglement and the proliferation of topological defects.<sup>17–19</sup> In the last case the VS forms a highly disordered phase that can be either a vortex glass or a strongly pinned liquid.<sup>17–19</sup> The existence of such a phase transition has been further supported by several numerical studies.<sup>23–25</sup> In the experimental level Lorentz microscopy on  $\text{Bi}_2\text{SrCa}_2\text{Cu}_2\text{O}_x$  and neutron scattering on untwinned  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  single crystals have demonstrated the existence at low fields of an almost ideal lattice.<sup>26</sup> However, concerning the existence of the theoretically predicted disorder induced transition, following earlier conjectures by various workers,<sup>27–31</sup> experimental evidence was provided only recently by magnetization studies of pure detwinned

$\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals and its boundary was mapped in the  $B$ - $T$  phase diagram.<sup>32</sup>

In the last investigation the detection of the transition was based on the fact that unlike a dislocation-free elastic lattice, a disordered vortex phase should display prominent thermomagnetic history effects similarly to other disordered systems like spin glasses.<sup>33</sup> Indeed as discussed in detail in Ref. 34, topological disorder present in the lattice manifests metastable behavior and consequently in the presence of dislocations the magnetotransport properties should depend on the past history of the superconductor. Such memory effects have been widely observed in the low- $T_c$  materials in the region of the conventional peak effect close to the  $H_{c2}(T)$  line.<sup>34–42</sup>

In the present article by means of magnetization measurements we study in detail memory effects in the hysteretic response of pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals, with various types and densities of pinning defects thus extending our earlier report.<sup>32</sup> A partial magnetization loop technique is employed,<sup>32</sup> which introduces specific protocols that permit the detection of the point where dislocations first invade the vortex system. Therefore by studying twin-free single crystals with low densities of point defects, we succeed in the observation of a transition in the mixed state of  $\text{YBa}_2\text{Cu}_3\text{O}_y$  that separates an elastic dislocation-free Bragg glass from a disordered vortex phase. The location of this transition is mapped in the  $B$ - $T$  phase diagram and found to be in proximity to the onset of the second magnetization peak. This line is found to depend on the density of oxygen vacancies as manifested by its shift towards lower fields with increasing oxygen deficiency in the region  $6.908 \leq y \leq 6.999$  and its suppression for the lowest oxygen contents under study ( $y = 6.550$  and  $6.760$ ). This behavior is in agreement with existing theoretical models.<sup>17–19</sup> Above the disorder induced transition, due to the proliferation of dislocations that exhibit metastability, the critical current density displays a strong dependence on the magnetic history of the superconductor and attains higher values for increasing the maximum field in which the VS is exposed. Nevertheless, for low oxygen concentrations near optimal doping the memory effects are shown to diminish for our highest fields due to saturation in the density of dislocations. This effect, however, is absent for the highest oxygen contents under investigation. The influence of extended defects like twin boundaries and columns is also investigated. We find that for a low density of twin planes the transition is almost unaffected. In contrast, a high density of twin planes as well as even low densities of columnar defects are shown to destroy the Bragg glass phase and lead to the elimination of the memory effects.

This study is mainly focused on the influence of quenched disorder on the Bragg glass-disordered phase transition reported in Ref. 32. However, for completeness and in order to demonstrate the universality of the effects under study we start by initially reviewing and at the same time extending some of the main observations briefly discussed in Ref. 32.

## II. EXPERIMENTAL DETAILS

### A. Samples

In this study we present results acquired on several pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals from two different sources. From

the first one we have investigated three detwinned ( $D1, D2, D3$ ), one naturally untwinned ( $U1$ ), and one twinned ( $T1$ ) sample. These crystals were grown by a conventional self-flux method, starting with powders of  $\text{Y}_2\text{O}_3$  (99.9999%)  $\text{BaCO}_3$  (99.999%), and  $\text{CuO}$  (99.9999%), mixed in a molar ratio  $\text{Y}:\text{Ba}:\text{Cu}$  of 1:18:45.<sup>43</sup> Ytria-stabilized zirconia crucibles were used for the growth, a procedure that gives crystals of excellent purity.<sup>29,32,44</sup> All samples were initially annealed at  $500^\circ\text{C}$  for 6 d in an oxygen atmosphere, a treatment that results in  $y = 6.934$ . Detwinning was achieved by applying a uniaxial pressure of  $\sim 50$  MPa at  $550^\circ\text{C}$  in air for 15 min. The detwinned samples were then reoxygenated for 1 d at  $500^\circ\text{C}$  in flowing  $\text{O}_2$ .  $D1$  and  $D3$  were subsequently annealed for a further 8 d at 525 and  $450^\circ\text{C}$ , respectively in 1 bar of oxygen, which should give oxygen contents of  $y = 6.908$  and  $6.970$ , respectively. After growth,  $U1$  was reannealed at  $750^\circ\text{C}$  at 1 bar for 24 h that results in  $y = 6.550$ . From the second source we have studied two naturally untwinned ( $ZY$  and  $ZX$ ), three densely twinned [here we discuss results for one of them ( $DN$ )], and two columnar irradiated ( $AH$  and  $AW$ )  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals with matching fields of 0.1 and 3 T, respectively. All samples were from the same batch and initially had an oxygen content of  $y = 6.970$ .  $ZY$  and  $ZX$ , that were the two parts of a larger crystal, were subsequently reannealed in oxygen in 370 bar at  $400^\circ\text{C}$  for 143 h and in 26.2 mbars at  $495^\circ\text{C}$  for 140 h, respectively, obtaining oxygen contents of  $y = 6.999$  and  $6.760$ , respectively. For more details concerning the growth and irradiation processes of these samples we refer the reader to Ref. 45. Oxygen contents were determined from Eq. (6) of Ref. 46 that relates the oxygen concentration with the annealing pressure and temperature. The oxygen content, dimensions, the superconducting transition temperature  $T_c$  (defined as the midpoint of the temperature range over which the in-phase component of the ac susceptibility in a field of 0.03 mT varies from 10 to 90% of its saturation value), as well as the defect status for all the samples can be found in Table I.

### B. Measuring procedure

Magnetic hysteresis measurements have been performed using an Oxford Instruments vibrating sample magnetometer for applied fields up to 12 T. A measuring procedure that involves partial magnetization loops (PL's) is employed and is realized in two different ways. In method I the sample is cooled in zero field that is subsequently swept up to a maximum value  $B_{\text{max}}$  before it is decreased back to zero. In a series of runs,  $B_{\text{max}}$  is gradually increased with a fine step (steps as small as 50 mT have been used) finally reaching values above the irreversibility field. In this case we obtain a complete magnetization loop (CL). In method II the previous procedure is inverted: after cooling the sample in zero field, the field is increased to a value above the irreversibility line (or up to our maximum accessible field of 12 T if the irreversibility field is larger than 12 T) and then after decreasing it down to a certain value  $B_{\text{min}}$  it is swept again to the same maximum field. This procedure is repeated for several values of  $B_{\text{min}}$ . In all cases, after completion of each run the sample is heated up into the normal state and then cooled again to

TABLE I. Oxygen content  $y$ , dimensions, transition temperature  $T_c$ , defect status, and the observation or not of history effects for the investigated samples.

Sample	$y$	$l \times w \times t$ (mm <sup>3</sup> )	$T_c$ (K)	Defect status	History effects
<i>U1</i>	6.550	0.98×0.72×0.07	62.8	untwinned	no
<i>ZX</i>	6.760	2.43×1.59×0.24	71.7	untwinned	no
<i>D1</i>	6.908	1.07×0.86×0.08	92.7	detwinned	yes
<i>D2</i>	6.934	1.69×1.08×0.09	92.8	detwinned	yes
<i>D3</i>	6.970	1.29×1.25×0.08	91.7	detwinned	yes
<i>ZY</i>	6.999	2.16×0.67×0.24	88.8	untwinned	yes
<i>T1</i>	6.934	0.78×0.59×0.09	92.6	sparsely twinned	yes
<i>DN</i>	6.970	1.37×0.85×0.03	89.6	densely twinned	no
<i>AW</i>	6.970	1.40×0.75×0.02	90.4	columns ( $B_\phi = 0.1$ T)	no
<i>AH</i>	6.970	1.26×0.57×0.02	90.4	columns ( $B_\phi = 3$ T)	no

the required temperature in zero field. The sweep rate of the magnetic field is varied in the range of 2–20 mT/sec.

### III. RESULTS

We have performed extensive magnetization measurements on all detwinned samples. All of them have demonstrated history effects similar to the ones reported in Ref. 32 for *D2*. However, the variation in the oxygen stoichiometry has revealed some interesting features. In order to outline them, we mainly concentrate on a comparison of the results for *D1* and *D3*, whose oxygen contents differ significantly. Figure 1(a) shows several PL's together with the CL for crystal *D1* at 78 K obtained by method I for the whole field range up to the irreversibility field. As can be seen, in the intermediate field region after reversing the field from  $B_{\max}$  the descending legs of the PL's do not follow the CL but rather the magnetization attains lower values. Nevertheless, with further decreasing field the PL's finally merge with the CL; the field reduction  $\Delta B_{\text{sup}}$  below  $B_{\max}$  required for this can be as high as 1 T. We should note that  $\Delta B_{\text{sup}}$  is rather large for the deviation of the PL's from the CL to be attributed to a possible influence of an incomplete reversal of the field profile in the superconductor. Indeed as has been predicted for thin disks in an axial magnetic field, the field of full penetration should be approximately equal to  $\mu_0(J_c t/2)\ln(4a/t)$ , where  $a$  is the radius and  $t$  the thickness of the disk.<sup>47</sup> For a rectangular sample of large aspect ratio (like *D1*)  $a$  can be replaced by an effective radius given by  $a_{\text{eff}} = (3w/4)(1-w/3l)$ ,<sup>48</sup> with  $l$  and  $w$  being the length and width of the crystal, respectively. Using this method and the critical current density values, as calculated from the magnetization width using the Bean formula,<sup>48</sup> we find that for *D1* at  $T=78$  K this field is less than 13 mT, a value much too small to account for the results displayed in Fig. 1(a). This behavior of the PL's continues until  $B_{\max}$  reaches a characteristic field  $B_{\text{sat}} = 2.25$  T (see Ref. 32 for a detailed description of the way that the values for this field are determined) above which the PL's coincide with each other as can be clearly seen from Fig. 1(a). PL's obtained by method II for the same sample and temperature are displayed in Fig. 1(b). As can be seen in this case the effect is opposite: increasing

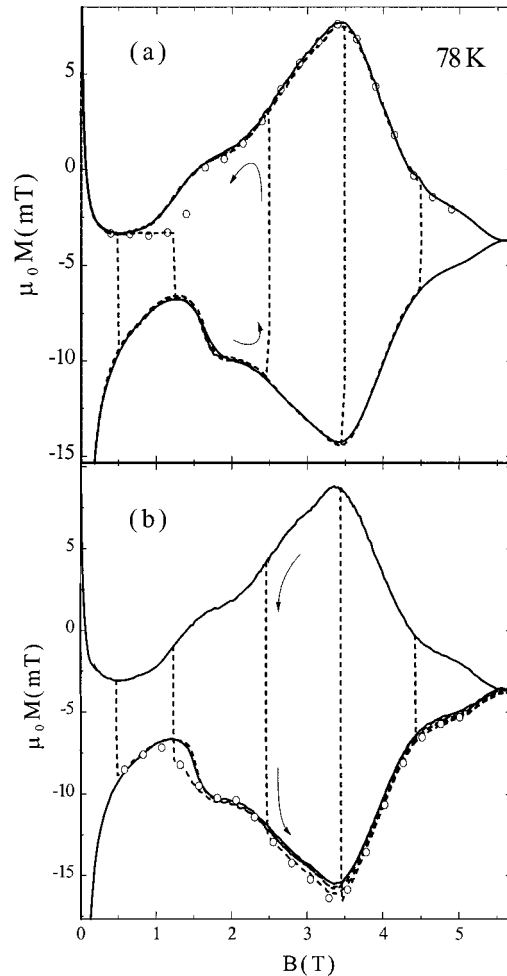


FIG. 1. (a) Magnetization loops for sample *D1* ( $y = 6.908$ ) at 78 K. The solid line represents the CL whereas with the dashed lines we show PL's obtained by method I for several  $B_{\max}$ . The arrows show the direction of the field sweep. The open circles indicate magnetization values in the descending branches of various method I PL's at 0.1 T below  $B_{\max}$ . (b) The CL (solid line) together with several PL's obtained by method II (dashed lines) for *D1* ( $y = 6.908$ ) at 78 K. Arrows indicate the direction of the field sweep. The open circles represent magnetization values in the ascending branches of various method II PL's at 0.1 T above  $B_{\min}$ .

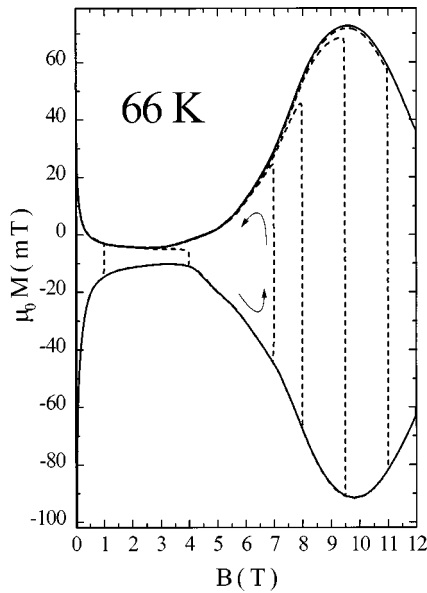


FIG. 2. Magnetic hysteresis curves for sample *D3* ( $y=6.970$ ) at  $T=66$  K. The solid line represents the CL whereas the dashed lines indicate PL's (method I) at various  $B_{\max}$ . The arrows show the direction of the field sweep.

the field above  $B_{\min}$  results in enhanced hysteresis. No merging with the CL is observed and a small increase in the magnetization of the PL's persists up to the irreversibility field.

From the above results it becomes clear that for pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals, the irreversible magnetization and thus the critical current density is strongly dependent on the magnetic history of the sample in correspondence with observations in the low-temperature superconductors (LTS's).<sup>34–42</sup> Hence proper field cycling of the superconductor can lead to enhanced critical currents.<sup>32</sup> Additionally the path dependent hysteresis has two very important implications. First, it makes evident that description by a simple Bean model, where the critical current density is supposed to be a single valued function of the field, should not apply in this case. Second, it suggests the existence of plastic deformations, since for an elastically distorted vortex lattice no such memory effects should be realized.

Careful inspection of the shape of the CL shown in Fig. 1 reveals another interesting feature: in the range of  $0.5 < B < 2$  T the ascending and descending branches of the complete magnetization loop are very asymmetric. Such an asymmetry is characteristic of pure single crystals and as demonstrated in Ref. 32, it stems from the history dependence of the irreversible magnetization. Indeed, as shown by the open circles in Figs. 1(a) or (b), that represent the magnetization values in the upper or lower branches of the PL's obtained by methods I or II at 0.1 T below  $B_{\max}$  or above  $B_{\min}$ , respectively, the asymmetry disappears and the ascending or descending branch of the full loop is almost perfectly reproduced.

Figure 2 shows PL's for crystal *D3* at 66 K. For brevity we show only the ones obtained by method I. For this low oxygen deficiency ( $y=6.970$ ) the memory effects are still present and become even more prominent as compared to the single crystals with lower oxygen concentrations. Indeed for

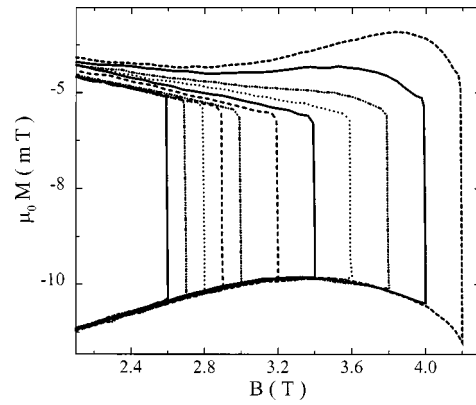


FIG. 3. PL's for *D3* ( $y=6.970$ ) as obtained by method I at 66 K in the region  $2.6 \leq B_{\max} \leq 4.2$  T with  $B_{\max}$  increasing with a step of 0.1 for  $2.6 \leq B_{\max} \leq 3$  T and 0.2 T for  $3 \leq B_{\max} \leq 4.2$  T.

*D3*, the obtained  $R = (\Delta M_{CL} - \Delta M_{PL}) / \Delta M_{CL}$  values<sup>32</sup> ( $\Delta M$  denotes the magnetization width) can be as high as 0.5, i.e., field cycling can lead to an increase in the critical current density by up to 100% (for *D1* and *D2*, we find  $R \leq 0.3$ ). In addition, for this oxygen content no suppression of the memory effects at a field  $B_{\text{sat}}$  is seen, i.e., successive PL's do not merge with each other even for the highest accessible fields of our experiments (see Fig. 2), a behavior that is distinctly different from the one shown in Fig. 1(a) for *D1*. The same result was obtained for *ZY*.

Figure 3 displays in detail several PL's for *D3* at 66 K as obtained by method I and for  $2.6 \leq B_{\max} \leq 4.2$  T. As can be clearly seen for field excursions up to a characteristic field  $B_{\text{pl}} = 3$  T ( $B_{\text{pl}}$  is defined as the  $B_{\max}$  of the lower of two successive method I PL's, for which we observe the onset of a finite difference in their magnetization widths, as described in detail in Ref. 32), the PL's follow the same curve, whereas the irreversible magnetization and thus the critical current density is independent of the magnetic history of the sample. Since the absence of path dependent hysteresis is characteristic of the elastic regime, this observation suggests that in this field range the vortex lattice is elastically distorted. For  $B_{\max}$  above  $B_{\text{pl}}$  however, the PL's start to deviate from the universal line and the magnetization shows a strong dependence on  $B_{\max}$  attaining higher values and approaching the CL with increasing  $B_{\max}$ . Figure 3 also provides us with useful information regarding the development of the departure of the PL's from the common curve. Indeed as can be clearly seen, for the fine step of 0.1 T shown here the deviation of the PL's is rather gradual. The same result is obtained for the smallest step of 50 mT used in the present study.

Comparison of the results for *D3* ( $B_{\text{pl}} = 3$  T at 66 K) and *D1* ( $B_{\text{pl}} = 0.5$  T at 66 K) also demonstrates that following the behavior of the magnetization peak, with increasing oxygen content the characteristic field  $B_{\text{pl}}$  is shifted to higher fields. In the same manner  $\Delta B_{\text{sup}}$  increases as well attaining values that can be as high as 2 T. On the other hand for the strongly underdoped *U1* ( $y=6.550$ ) and *ZX* ( $y=6.750$ ) the history effects disappear completely, and the magnetization loop now displays the conventional “fishtail” behavior indicating enhanced disorder. This result is illustrated in Fig. 4 where we show for *U1* the CL together with several PL's as obtained by method I at 45 K. In addition resistivity studies



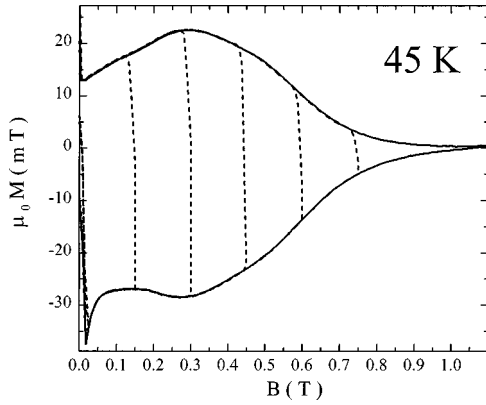


FIG. 4. The magnetization field dependence for  $U1$  ( $y = 6.550$ ) at 45 K for several values of  $B_{\max}$  (dashed lines). The CL is also shown (solid line).

reveal that for this low oxygen content the melting transition becomes of the second order in the whole field range.<sup>49</sup>

Finally we have studied the influence on the memory effects of extended defects such as twin planes and columns. Figure 5(a) shows PL's for sample  $T1$  for fields applied along the  $c$  axis. As can be seen, the magnetization history dependence is not significantly affected by the presence of the twin planes for the density of twin boundaries in this sample and the results are similar to the detwinned crystals. In contrast, as depicted in Fig. 5(b) for  $DN$  the history effects are almost completely eliminated (a small remanant effect with  $R \sim 0.02$  can still be seen at fields above 4.5 T). The same result was obtained for the columnar irradiated samples as shown in Fig. 5(c) for  $AW$  ( $B_{\Phi} = 0.1$  T). From Figs. 5(b) and (c) one can also discern the significant influence of both a high density of twin planes and the columnar defects on the magnetic hysteresis loop. Indeed, in these samples the characteristic “neck” observed in the  $M(B)$  curves below the peak for all the detwinned crystals as well as for  $T1$  disappears, and the hysteresis width in this field region becomes comparable to the one seen at the peak. The columnar irradiated crystals also show a pronounced increase in pinning in the whole field range. For example for  $AW$  at 70 K, we observe an enhancement in the critical current density by as much as 3.5 times as compared to the initial state of the sample.

#### IV. DISCUSSION

##### A. History effects

As discussed above, several recent theoretical studies and numerical simulations have predicted the existence of a disorder induced transition in the mixed state of type-II superconductors from a Bragg glass to a highly disordered phase.<sup>17–25</sup> In the former, that is stable at low fields, elasticity survives and the flux-line lattice maintains quasi-long-range translational order. Upon raising the field above the region of its stability, however, topological defects such as dislocations become energetically favorable and invade the vortex lattice. Consequently, long-range order in the lattice either diminishes or is completely lost and the VS is transformed into the high-field disordered phase.<sup>17–19</sup> Being of different nature these two phases are characterized by differ-

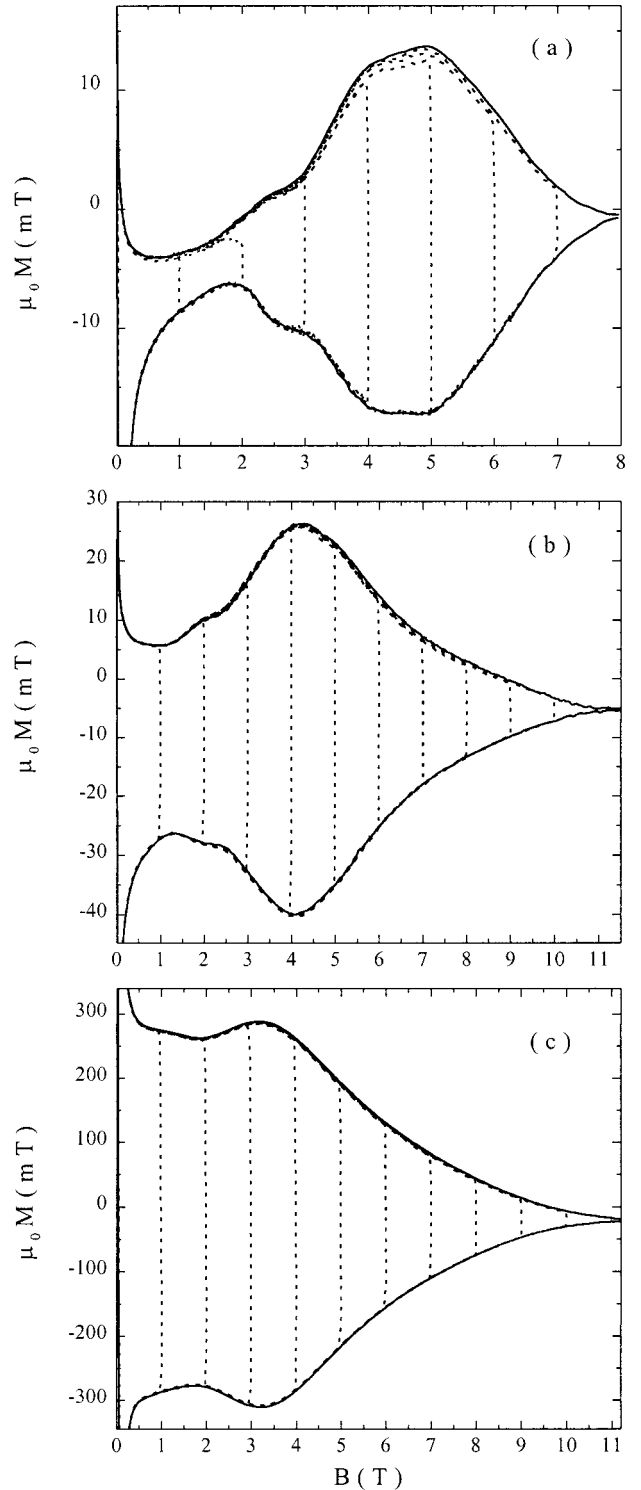


FIG. 5. The CL (solid line) together with several PL's obtained by method I (dashed lines) at  $T/T_c = 0.8$  are displayed for samples (a)  $T1$ , (b)  $DN$ , and (c)  $AW$ .

ent magnetotransport properties.<sup>17</sup> A direct consequence of the introduction of topological disorder should be the increase in the critical current density since the lattice has additional effective degrees of freedom and can adapt to the pinning potential more easily.<sup>17</sup>

Dislocations appear in the VS due to the influence of both quenched disorder and thermal fluctuations, which induce transverse wandering of vortices from their equilibrium po-

sitions resulting in an entangled vortex configuration.<sup>17–19</sup> In this sense the magnetic field plays an important role as it tunes the effective disorder by changing the mean intervortex distance, thus stimulating the injection of dislocations.<sup>17–19</sup> Therefore with increasing field the elastic structure becomes unstable and a transformation from an elastically to a plastically distorted lattice is expected, a process accompanied by the invasion of topological defects at short length scales.<sup>17</sup> We believe that our observations can be understood within this framework. During an isothermal magnetization loop, when the field increases beyond the elastic regime, dislocations appear and the VS is forced into an increasingly disordered state. By decreasing the field from this state the amorphous structure of the lattice becomes energetically unfavorable. However, as discussed in detail in Ref. 34, topological disorder exhibits metastable behavior due to trapping of dislocations in local energy minima. Thus the density of topological defects does not vary reversibly with field and one should expect a path dependent critical current. This is indeed the case shown in Fig. 1: with decreasing field the VS in the return leg of the CL tends to retain topological defects from the high-field state leading to higher currents than the ones corresponding to the ascending branch. This is demonstrated by the observed reduced or enhanced irreversible magnetization values for the PL's obtained by methods I or II, respectively, as compared to the full loop. Only when the field attains low enough values, does the remanent dislocation network completely heal out of the VS. Hence any memory of the lattice formation history is erased, as illustrated by the coincidence of the PL's with the CL at low fields.

It is stressed that although for our pure crystals, that demonstrate effects very sensitive to sample homogeneity,<sup>44</sup> it is difficult to suppose the existence of granularity, nevertheless, even if present, the latter could not explain the observed behavior. In a granular sample the standard relation between hysteresis width and remagnetization field breaks due to the existence of two types of currents: intergranular and intragranular.<sup>50</sup> As the latter can attain very high values, the self-field of the grains can lead to significant differences between the internal and applied fields.<sup>50</sup> This difference is positive or negative for decreasing or increasing fields, respectively. Consequently, in the region where the current increases with field (like below the peak) one should expect enhanced or decreased magnetization values for the PL's similarly to the results depicted in Figs. 1(a) and 1(b). Above the peak, however, where the current decreases with field, this model requires the opposite behavior that is clearly not seen in our experiments and therefore it should be rejected.

Surface effects cannot account for our observations either. Indeed, the Bean-Livingston barrier<sup>51</sup> should be ruled out since although it could cause an asymmetry in the  $M(B)$  loops, it cannot account for the history effects. Also the influence of the geometrical barrier<sup>52</sup> for the field range of interest ( $B \geq 1$  T) should be excluded, since as shown in Ref. 53 this barrier becomes irrelevant for applied fields above  $\sim H_{c1}/2$  that for  $T=78$  K is of the order of only some mT, far below the region under investigation. Finally, the irrelevance of surface barriers is also supported by recent transport measurements,<sup>54</sup> which have clearly disproved their importance for pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$ .

## B. Phase diagram

The introduction of the partial loop technique is very important since it allows the detection of the point where topological disorder invades the vortex system for the first time. Indeed as long as the maximum field excursion is not high enough to drive the VS out of the region where elasticity dominates, then due to the reversibility characterizing the structure of an elastically deformed lattice no history effects should be observed.<sup>1</sup> Consequently within this range of  $B_{\max}$  the PL's must follow the same universal curve. Such behavior is indeed seen below the characteristic field  $B_{\text{pl}}$  as depicted in Fig. 3. When  $B_{\max}$ , however, is raised above this value the PL's start to deviate significantly from the common line. From this point the critical current displays a strong dependence on the magnetic history of the superconductor (see Fig. 3), which demonstrates the presence of metastable disorder and plasticity in the VS. In this region with increasing field the density of dislocations rises. At oxygen contents below 6.970 this increase continues only until  $B_{\text{sat}}$  is reached. From this point the history effects become negligible and the PL's coincide with each other. As we have already suggested in Ref. 32, this occurs due to the saturation in the density of dislocations that remains almost unaffected by further increasing field. However, as mentioned above, such saturation is not seen for the highly oxygenated *D3* and *ZY* up to our maximum field of 12 T. We attribute this effect to the reduced effective disorder in these samples due to the much lower density of pinning centers as compared to the crystals with higher oxygen deficiency, which requires fields beyond our maximum accessible values for the saturation in the density of dislocations to occur.

Our results strongly indicate that below  $B_{\text{pl}}$  the VS is elastically deformed and that topological defects are absent. These characteristics are identical to the Bragg glass phase.<sup>17–25</sup> Therefore as we have already proposed in Ref. 32,  $B_{\text{pl}}$  features the transition from the Bragg glass to a disordered vortex phase where plasticity plays a dominant role. The loci of the  $B_{\text{pl}}$  values for the various oxygen contents under study are shown in the  $B$ - $T$  phase diagrams of Fig. 6.<sup>55</sup> In these plots we have also included the lines corresponding to the saturation field  $B_{\text{sat}}$ , the magnetization peak  $B_p$ , and the peak onset  $B_{\text{on}}$ .<sup>56</sup> The melting lines that were obtained from resistivity measurements on identical crystals from the same batch<sup>10</sup> are also shown.

Two interesting features characterize the  $B_{\text{pl}}(T)$  dependence. First, although at low  $T$  this line has a plateau, with increasing temperature an upturn is seen; second, it shifts towards higher fields with decreasing oxygen deficiency. Both trends are in agreement with the theoretically predicted transition line separating the Bragg glass from the high-field disordered phase<sup>17–19</sup> and can be understood as follows. The structure of the vortex solid is determined in general by the competition between the pinning and elastic energies. When the pinning energy increases beyond the elastic one topological defects are introduced.<sup>19</sup> At elevated temperatures, however, due to thermally activated motion of vortices pinning by point disorder is reduced. Consequently, dislocations in the VS will proliferate only when higher fields (as compared to the lower temperatures) are reached resulting into the upward curvature of the  $B_{\text{pl}}$  line.<sup>17–19</sup> With raising  $\delta$  in the

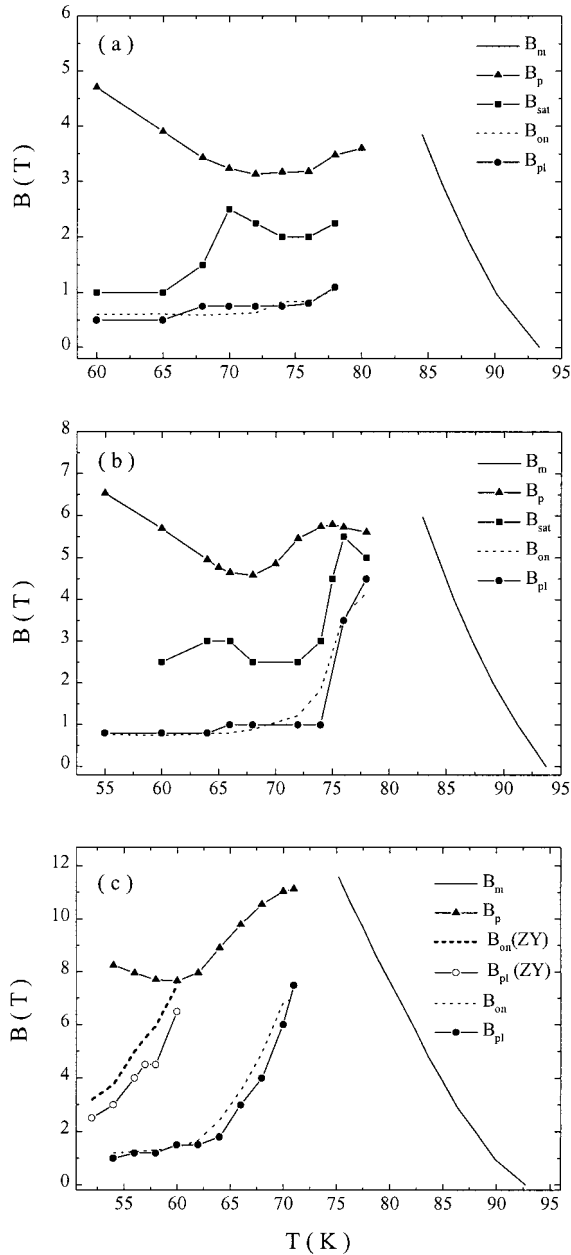


FIG. 6. Position of the characteristic fields  $B_p$ ,  $B_{pl}$ ,  $B_{on}$ , and  $B_{sat}$  in the  $B$ - $T$  phase diagram for D1 ( $y=6.908$ ) (a), D2 ( $y=6.934$ ) (b), and D3 ( $y=6.970$ ) (c). The corresponding melting lines are also displayed. The open circles and thick dashed line in Fig. 6(c) illustrate the temperature dependence of  $B_{pl}$  and  $B_{on}$ , respectively, for crystal ZY ( $y=6.999$ ). The position of the peak for this sample is shifted beyond our maximum field of 12 T.

region near optimal doping due to the increase in the density of oxygen vacancies, pinning disorder is enhanced whereas at the same time the elastic energy remains almost unchanged.<sup>10</sup> Consequently, as predicted in Refs. 17–19, a shift of the location of the  $B_{pl}(T)$  line to lower field values should take place in agreement with our observations. For the same reason for larger  $\delta$ , the  $B_{pl}$  plateau is expected to extend to higher temperatures in accordance with the results depicted in Fig. 6. However, for rather high values of the oxygen deficiency (like in the underdoped samples U1 and ZX) the pinning energy always dominates over the elastic one leading to the destruction of the Bragg glass and the

formation of a disordered phase in the whole  $B$ - $T$  plane as demonstrated by the absence of the history effects and the suppression of the first-order melting transition.

An interesting and still not elucidated issue is whether the magnetization peak seen in  $\text{YBa}_2\text{Cu}_3\text{O}_y$ , originates from the same underlying physical mechanism as in weakly anisotropic or isotropic LTS's like for, e.g., the widely studied  $2H\text{-NbSe}_2$ . Experiments show that there are distinct differences between the peak effect in these materials. Specifically in  $2H\text{-NbSe}_2$  the peak is narrow and close to the upper critical field.<sup>37–40</sup> Accordingly following the behavior of  $H_{c2}$  the position of the peak shifts monotonously to lower fields with increasing temperature.<sup>37–40</sup> In contrast, in  $\text{YBa}_2\text{Cu}_3\text{O}_y$  the magnetization peak is rather broad, located far below both the irreversibility and upper critical field lines and is characterized by a nonmonotonic temperature dependence.<sup>29–32,57</sup> In Ref. 32 we have demonstrated that the peak is located into a region where the flux-line lattice exists in a disordered plastic state in agreement with earlier suggestions.<sup>29–31,57</sup> This behavior is similar to the one seen in the LTS's.<sup>34,37–41</sup> On the other hand, as shown in Ref. 37 for  $2H\text{-NbSe}_2$  single crystals, the peak effect separates two regimes with radically different behavior: one below the peak where thermomagnetic history effects are prominent and one above where these effects disappear completely. This was attributed to a transition to a completely amorphous vortex state above the peak,<sup>37</sup> which has also been recently supported by neutron-scattering experiments on Nb single crystals.<sup>58</sup> In contrast, our results on  $\text{YBa}_2\text{Cu}_3\text{O}_y$  do not reveal such a behavior: for the samples with low oxygen content the memory effects cease well below the peak whereas for high oxygen concentrations persist even above, illustrating a distinct behavior of the vortex lattice around the peak in this compound. Thus one can conclude that the manifestation of the peak effect in these materials is different. Whether this is due to a different basic mechanism or it simply reflects the significantly enhanced role of thermal fluctuations in  $\text{YBa}_2\text{Cu}_3\text{O}_y$  is not as yet clear. Further theoretical and experimental work is required to clarify this point.

### C. Extended defects

The effect of twin planes on the vortex dynamics is a complicated and still not fully resolved topic. Twins have been shown to lead to increased pinning at high temperatures due to the reduction of thermal fluctuations,<sup>57,59</sup> whereas they become channels for easy flux penetration at lower temperatures.<sup>60</sup> Our results on twinned samples demonstrate clearly that the presence or not of the disordered induced transition depends on the density of twin boundaries. Specifically as can be seen from Fig. 5(a), T1, that is characterized by a low twin density (mean twin distance of the order of  $\sim 5 \mu\text{m}$ ), shows behavior almost identical to the detwinned crystals. Nevertheless, for the microtwinned DN (average twin separation of  $\sim 100 \text{ nm}$ ) the transition is suppressed [see Fig. 5(b)]. It is natural to attribute this effect to the much higher density of twin planes in this sample. Indeed at a typical field of for example 1 T, where according to the results for the detwinned crystals the Bragg glass phase should be present, the space between successive twin boundaries for T1 and DN contains on average 110 and 2 lattice parameters, respec-

tively. Therefore for  $T1$  the distortion in the lattice caused by the twins should not be important and accordingly the Bragg glass is preserved. For  $DN$ , however, the role of twin boundaries as pinning centers is expected to be significantly enhanced deteriorating the periodicity of the vortex lattice and leading to the destruction of the Bragg glass. The weak memory effects seen for  $DN$  at higher fields ( $B > 4.5$  T) can be possibly attributed to the contribution of the regions between twins that becomes important at these fields thus reproducing the behavior of the detwinned samples. These observations are also in correspondence with recent calorimetric measurements on high-purity twinned  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals.<sup>61</sup> For these samples it was found that for as long as the field is high enough for the intervortex distance to be much less than the average twin boundary separation, with increasing temperature the VS melts to a vortex liquid via a first-order transition demonstrating the existence of an undistorted vortex lattice.<sup>61</sup> This transition, however, becomes of second order at low enough fields<sup>61</sup> where the vortices are predominantly pinned by twin boundaries probably forming a Bose glass phase.<sup>62,63</sup>

As we have shown in Sec. III the history effects are not present in the columnar irradiated samples. Within our interpretation this behavior can be understood as follows. Columnar defects due to their extended dimensions localize vortices and consequently as has been predicted theoretically<sup>62</sup> and shown in several experiments<sup>64,65</sup> are very efficient pinning centers. Such strong pinning is expected to eliminate long-range order in the vortex lattice.<sup>1,62,63</sup> As has been predicted by Vinokur *et al.*<sup>19</sup> the Bragg glass cannot survive for matching fields  $B_\Phi = n\Phi_0 > c_L^2 B_{pl}$ , where  $n$  is the areal density of columns and  $c_L$  the Lindemann number. For  $c_L \approx 0.16$  (Ref. 19) and the  $B_{pl}$  values of  $\sim 1$  T observed in the detwinned samples, the minimum  $B_\Phi$  required for the suppression of the disorder induced transition is found to be of the order of  $10^{-2}$  T. Consequently, since the matching fields for both  $AW$  and  $AH$  are much larger than this value, the memory effects are expected to disappear in these samples in agreement with our findings.

## V. CONCLUSIONS

In summary, in this work we have presented a detailed study of history effects in the irreversible magnetization of

pure  $\text{YBa}_2\text{Cu}_3\text{O}_y$  single crystals with several types and densities of pinning centers. By using a partial magnetization loop technique we have been able to detect a transition in the vortex system of the superconductor between a low-field quasicrystalline Bragg glass and a high-field disordered phase. The locus of this transition has been identified in the field-temperature phase diagram and found to be in proximity to the onset of the second magnetization peak. The influence of changes in the density of pointlike defects on this transition has also been investigated by varying the concentration of oxygen vacancies in the region  $6.550 \leq y \leq 6.999$ . We have found that its location shifts to lower fields with decreasing oxygen content and disappears for the highest oxygen deficiencies under study ( $y = 6.550, 6.750$ ) in agreement with theoretical predictions. Above the transition metastable topological disorder invades the VS leading to a pronounced dependence of the critical current density on the maximum field to which the superconductor is exposed and thus on the formation history of the vortex lattice. For low oxygen contents close to optimal doping the history effects diminish for the highest fields of our experiment due to the saturation in the density of topological defects. Such saturation, however, is not observed for the highest oxygen concentrations, which we have attributed to the reduction in pinning by point pinning centers. Finally, studying the influence of extended defects has revealed that a low density of twin boundaries leaves the Bragg glass-disordered phase transition unaffected. However, in densely twinned as well as in columnar irradiated samples the transition is suppressed and the history effects are eliminated.

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\*Author to whom correspondence should be addressed. Electronic address: sk@phys.soton.ac.uk

<sup>†</sup>On leave from the Chemistry Department, Moscow State University, Moscow 117234, Russia.

<sup>‡</sup>Present address: Department of Physics, University of Toronto, Toronto, Ontario, Canada M5S 1A7.

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