# Magnetic relaxation measurements on a heavily Pb-doped $Bi_2Sr_2CaCu_2O_{8+\delta}$ single crystal

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The magnetic relaxation measurements on a heavily Pb-doped Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8+δ</sub> single crystal were carried out in the vicinity of both the anomalous magnetization peak  $H_{2p}$  and its onset field  $H_{on}$  at different temperatures. The results show that both the normalized magnetic relaxation rate  $S(=-d \ln M/d \ln t)$  and the pinning potential  $U_0$  exhibit remarkable changes near  $H_{on}$  and  $H_{2p}$  in the temperature region where  $H_{on}(T)$  follows the relation of the disorder induced vortex lattice transition,  $H_{on}(T) = H_{on}(0) [1 - (T/T_c)^4]^{3/2}$ . For both  $H < H_{on}$ and  $H > H_{2p}$ , *S* increases and  $U_0$  decreases, respectively, with increasing magnetic fields. A negative power law of  $U_0(H) \propto H^{-\alpha}$  was observed for both  $H < H_{on}$  and for  $H > H_{2p}$ . There exists a minimum in the magnetic-field dependence of S(H) and a maximum in the pinning potential  $U_0(H)$  in the field range between  $H_{on}$  and  $H_{2p}$ . At lower temperature region where  $H_{on}(T)$  deviates from the above relation,  $U_0(H)$  behaves differently. The results are discussed in terms of both collective flux creep model and plastic flux creep model.

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### I. INTRODUCTION

The phenomenon of an anomalous increase of magnetization with increasing magnetic fields in the mixed state when an applied field parallel to the c axis of crystals, which is usually named as "anomalous magnetization peak (AMP)," "peak effect," "fishtail," or "arrow-head," etc., has been observed in almost all high-temperature superconductors (HTS).<sup>1-18</sup> Contrary to the peak effect observed in conventional low-temperature superconductors, which occurs in the vicinity of upper critical field  $H_{c2}$ , AMP of HTS occurs well below  $H_{c2}$ . The presence of AMP is regarded as a generic feature of anisotropic and relatively clean high-temperature superconducting crystals. For HTS, the origin of AMP has attracted much attention and many scenarios have been proposed for explaining the occurrence of AMP, including mainly inhomogeneities of  $T_c$ , <sup>1</sup> surface barriers,<sup>4</sup> a crossover from bulk pinning to surface barriers,<sup>5</sup> match effects,<sup>6</sup> dynamic effects,<sup>7</sup> a dimensional crossover from three dimensions (3D) to 2D in the vortex structure,<sup>8</sup> a disorder-induced transition from a relatively ordered vortex lattice (Bragg glass) at low fields to a highly disordered entangled vortex solid (vortex glass) at high fields.<sup>19-23</sup> For Bi-2212 superconductor, due to its rich and complicated H-T phase diagram originating from its strong anisotropy, short coherence length, and fierce competition among pinning energy  $E_{pin}$ , thermal energy  $E_{\rm th}$ , and elastic energy  $E_{\rm el}$  in the mixed state,<sup>24,25</sup> a lot of experiments have been focused on the study of AMP of Bi-2212 single crystals.<sup>4–10</sup> However, for optimum-doped ~90 K Bi-2212 superconducting single crystals, the characteristics of AMP lie in the narrow temperature range and the weak temperature dependences of its  $H_{2p}(T)$  and onset  $H_{on}(T)$ . At present, the origin of  $H_{2p}$  and  $H_{on}$  remains to be an open question.<sup>4-10</sup>

For Bi-2212 superconductors, since Cai *et al.*<sup>9</sup> and Chong *et al.*<sup>26</sup> found that Pb doping has a remarkable effect on its

superconductivity and flux pinning, in particular, on its anisotropy, the investigation on AMP and the magnetic vortex phase diagram in the mixed state of Pb-doped Bi-2212 single crystals has attracted great attention.<sup>27</sup> Recently, a heavily Pb-doped (Bi,Pb)-2212 single crystal with pronouncedly reduced anisotropy was obtained. The lower anisotropy of (Bi,Pb)-2212 single crystals was verified by its remarkable decrease of out-of-plane resistivity ( $\rho_c$ ) in the normal state and the metallic behavior of  $\rho_c(T)$  above 122 K, which is considerably different from that of free-Pb Bi-2212 single crystal.<sup>28</sup> Furthermore, contrary to AMP observed in Pb-free Bi-2212 single crystal, both  $H_{2p}(T)$  and  $H_{on}(T)$  of the heavily Pb-doped Bi-2212 single crystal extracted from the detailed magnetization measurement have strong temperature dependence from low temperature ( $\sim 0.16T_c$ ) to the vicinity of  $T_c$  (~0.90 $T_c$ ).<sup>29</sup> Moreover, the temperature dependence of  $H_{on}(T)$  follows the relationship:  $H_{on}(T) = H_{on}(0) [1]$  $-(T/T_c)^4$ <sup>3/2</sup> as T>18 K, which characterizes the validity of disorder-induced vortex structure transition from a relatively ordered vortex lattice (or Bragg glass) to a vortex glass state.<sup>19–23</sup> The temperature dependence of  $H_{2p}(T)$  seems to support the decoupling transition mechanism with thermaldisorder induced vortex pancake decoupling.30

To understand further the origin of AMP of the heavily Pb-doped Bi-2212 single crystal, the vortex dynamics in the vicinity of both  $H_{on}$  and  $H_{2p}$  was probed in this work by magnetic relaxation measurements M(t) at different fields and temperatures.

### **II. EXPERIMENT**

(Bi,Pb)-2212 single crystals with nominal starting composition of Bi:Pb:Sr:Ca:Cu=1.8:0.6:2:1:2 were grown by the self-flux method using  $Bi_2O_3$  as flux. The mixture of highpurity  $Bi_2O_3$ , SrCO<sub>3</sub>, CaCO<sub>3</sub>, CuO, PbO powders was well ground and presintered two times at 800–820 °C for 48 h.



FIG. 1. Magnetic hysteresis loops M(H) for (Bi, Pb)-2212 single crystal at 16 and 25 K.

After prereaction, the mixture was well reground and put into in a sealed Al<sub>2</sub>O<sub>3</sub> crucible to avoid the volatilization of Pb during the growth of single crystals. The powder was melted at 1020 °C for 6 h and then cooled quickly to 900 °C. The temperature was then decreased very slowly at a rate of 0.6 °C/h to 830 °C. It was found that the crystal was formed during the slow cooling. Finally, temperature was cooled to room temperature by turning off the power. The as-grown crystals were black and shiny platelets with typical dimension of  $1 \times 0.5 \times 0.03$  mm<sup>3</sup>. The single crystals were postannealed at 500 °C in air for five days to ensure sample homogeneity. The actual composition of (Bi,Pb)-2212 single crystals determined by a large area energy-dispersive x-ray is Bi:Pb:Sr:Ca:Cu=1.8:0.8:2.1:1.1:2. It is (Bi,Pb)-2212 single crystals that have the highest amount of Pb content obtained so far, to the best of our knowledge. Magnetic susceptibility M(T), magnetization M(H), and magnetic relaxation measurements with applied fields H parallel to c axis of single crystals were performed using a quantum design  $\mu$ -metal shielded MPMS<sub>2</sub> (magnetic property measurement system-2) superconducting quantum interference device magnetometers. The time window of magnetic relaxation measurement was 35 s  $\leq M(t) \leq 10000$  s.

#### **III. RESULTS AND DISCUSSION**

X-ray diffraction measurements reveal that only the (00*l*) peaks with c = 3.0784 nm can be observed and no extra peaks from secondary phases can be found even though the diffraction intensity is plotted logarithmically, which implies that the studied single crystal is very clean. Sharp superconducting transition of  $T_c = 69$  K with a narrow transition width  $\Delta T_c \sim 1$  K also confirms the good quality of studied single crystal. Isothermal magnetization data were obtained with zero-field cooling from above  $T_c$  to a set temperature.

Figure 1 shows magnetic hysteresis loops at 16 and 25 K. It shows that AMP  $H_{2p}$ =4000 G and the onset field  $H_{on}$ = 800 G, respectively, at 16 K. At 25 K,  $H_{2p}$ =1600 G and  $H_{on}$ =600 G. The obvious temperature dependence of  $H_{2p}(T)$  and the onset field  $H_{on}(T)$  with extended temperature widows can be found in Ref. 29. Moreover, magnetic



FIG. 2. Semilogarithmic plot of the normalized magnetic moment M(t) vs time for (Bi, Pb)-2212 single crystal at T=25 K, with H=300-1000 G (a) and 1200-2000 G (b).

hysteresis loop of 25 K has an obvious asymmetry caused by surface barrier.<sup>7</sup>

The magnetic relaxation behavior at T=25 K in the vicinity of both  $H_{on}$  and  $H_{2p}$  is shown semilogarithmically in Fig. 2(a) and Fig. 2(b). Figure 2(a) reveals that the slope of M(t) vs. t curves begins to vary at H=600 G and a pronounced crossover is observed at H=700 G. This feature is clearly demonstrated from the curves at H=700, 800, and 1000 G, whose slopes are smaller than those of curves for H < 600 G. At H=1400 G, a similar variation of slopes of M(t) curves is also observed as shown in Fig. 2(b).

The magnetic relaxation results are analyzed according to a linear barrier  $U(j) \propto (1-j/j_{c0})$ , i.e., the Anderson-Kim thermally activated flux creep model,<sup>31,32</sup> a logarithmic barrier  $U(j) \propto \ln(j_{c0}/j)$ ,<sup>33</sup> i.e., Maley's method<sup>34,35</sup> and a negative power-law barrier  $U(j) \propto j^{-\mu}$ , i.e., collective creep theory.<sup>36</sup> As  $H < H_{on}$ , it is found that M(t) can be well described by formula<sup>34,35</sup>

$$M(t) = M_0 \exp[-(k_B T/U_0) \ln(t/t_0)], \qquad (1)$$

where  $U_0$  is the pinning potential,  $t_0$  is some attempt time. Based on Eq. (1), the normalized relaxation rate  $S = -(1/M)dM/d \ln t = -d \ln M(t)/d \ln t$  can be expressed as



FIG. 3. The normalized relaxation rate S as a function of H for (Bi, Pb)-2212 single crystal at T=25 K.

$$S = k_B T / U_0. \tag{2}$$

 $U_0$  and S at different fields can be obtained by fitting curves of  $M(t)/M_0$  vs t according to Eq. (1).

As  $H \ge H_{on}$ , it is found that M(t) can be well described using the so-called "interpolation formula," <sup>36</sup>

$$M(t) = M_0 [1 + (\mu k_B T/U_0) \ln(t/t_0)]^{-1/\mu}, \qquad (3)$$

where  $U_0$  and  $t_0$  have the same meaning as Eq. (1),  $\mu$  is the exponent that governs the growth of the potential barriers with decreasing current *j*,  $U(j) \propto j^{-\mu}$ . Both  $U_0$  and  $\mu$  at different fields can be obtained by fitting curves of  $M(t)/M_0$  vs *t* according to Eq. (3).

Based on Eq. (3), the normalized relaxation rate S =  $-d \ln M(t)/d \ln t$  can be expressed as

$$S = k_B T / [U_0 + \mu k_B T \ln(t/t_0)].$$
(4)

According to the fitting values of  $U_0$  and  $\mu$ , taking t = 100 s and the attempt time  $t_0 = 10^{-6}$ ,<sup>24</sup> the corresponding normalized relaxation rate *S* can be calculated based on Eq. (4).

The magnetic field dependence of *S* is shown in Fig. 3. It indicates that *S* increases slightly with increasing fields for  $H < H_{on}(600 \text{ G})$ , while above  $H_{on} S$  decreases sharply with increasing fields. The decrease of *S* indicates the improvement of the effectiveness of pinning. It can be explained in terms of the proliferation of topological defects in the ordered flux line lattice,<sup>37,38</sup> which results in the entanglement of vortices and increases the effectiveness of pinning.<sup>19–23,39</sup> The minimum *S* occurs at  $H \sim 1200 \text{ G}$ , which is smaller than  $H_{2p} = 1600 \text{ G}$ . That is to say, the slowest magnetic relaxation does not occur exactly at  $H_{2p}$  but does prior to  $H_{2p}$ . The similar phenomenon was also observed by Pissas *et al.*<sup>40</sup> For H > 1200 G, *S* increases with increasing fields again.

The field dependence of  $\mu$  extracted by fitting M(t) based on Eq. (3) is plotted in Fig. 4 for  $H \ge H_{\text{on}}$ . It indicates that  $\mu \approx 0.5$  at  $H = H_{\text{on}}$  and it then increases to about 2 at a field range of  $\sim 1200-1400$  G. When H > 1400 G,  $\mu$  drops sharply with the increase of fields. At H = 4000 G,  $\mu \approx 0.18$ .



FIG. 4. The field dependence of the exponent  $\mu$  for (Bi, Pb)-2212 single crystal at T=25 K. The dashed line is a guide to the eye only.

For the three-dimensional case, collective creep theory predicts that  $\mu = 1/7$  is for single-vortex creep ( $L_c < a_0$ ) at low fields and high current,  $\mu = 3/2$  is for the creep of small vortex bundles  $(a_0 < R_{\perp}, R_{\parallel} \approx L_c < \lambda)$  at intermediate current,  $\mu = 1$  is for the creep of intermediate vortex bundles ( $a_0$  $< R_{\perp} < \lambda < R_{\parallel} \approx L_c$ ), and  $\mu = 7/9$  is for the creep of large vortex bundles  $(\lambda < R_{\perp}, R_{\parallel} \approx L_c)$  at low current and high fields. Here,  $R_{\perp}$  and  $R_{\parallel}$  denote the dimensions of the vortex bundles transverse and parallel to the flux motion, respectively;  $L_c$  is the longitudinal dimension of the vortex bundles along the field;  $a_0$  is the vortex spacing and  $\lambda$  is penetration depth.<sup>24,25</sup> Based on the  $\mu$  values shown in Fig. 4, it indicates that the magnetic relaxation below 1200 G can be described by the collective creep theory. The variation of  $\mu$  values with fields implies the change of the size of vortex bundles with fields. However, as H > 1400 G, the sharp drop of  $\mu$ , and in particular, low  $\mu$  values below 0.2 at high fields imply that there exists a crossover from collective creep to a single vortex creep according to the collective creep theory. In fact, this seems to be impossible because the condition of a single vortex creep cannot be met due to  $L_c > a_0$  at high fields. This means that the  $\mu$  values at high fields are inconsistent with the collective creep theory, and that the magnetic relaxation at high fields may be controlled by other creep mechanism. This can be also further demonstrated by the field dependence of pining potential as shown below.

Figure 5 is the plot of the field dependence of pinning potential  $U_0(H)$  extracted by fitting M(t) based on Eq. (1) for  $H < H_{on}$  and based on Eq. (3) for  $H \ge H_{on}$  at 25 K. It shows that  $U_0$  decreases with increasing field for  $H < H_{on}$ .  $U_0(H)$  follows a negative power law  $U_0 \propto H^{-0.1}$ . As  $H \ge H_{on}$ ,  $U_0$  increases with increasing fields up to a maximum value at  $H^* \sim 1200$  G, below  $H_{2p}$ , then decreases with further increasing fields. As  $H > H_{2p}$ ,  $U_0(H)$  can be fit approximately by the negative power law:  $U_0 \propto H^{-0.7}$ . Based on the collective flux creep mechanism,  $U_0$  increases with increasing fields,<sup>24</sup> the negative power law of  $U_0(H)$  observed for  $H > H^*$  is inconsistent with this theory.<sup>41-43</sup> Similar  $U_0(H)$ relation was also observed in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> and



FIG. 5. The pinning potential  $U_0$  as a function of field H for (Bi, Pb)-2212 single crystal at T=25 K. The solid lines are fitting curves (see text).

HgBa<sub>2</sub>CuO<sub>4+δ</sub> single crystals by Abulafia *et al.*<sup>44</sup> and Pissas *et al.*,<sup>40</sup> respectively. Abulafia *et al.* proposed that the negative power law of  $U_0 \propto H^{-0.7}$  can be well explained by a dislocation mediated plastic flux creep mechanism based on the proliferation of dislocations in the entangled vortex structure phase, which is analogous to the diffusion of dislocations in atomic solids.<sup>45</sup> This proliferation should influence the flux creep mechanism in the system. This means that the dislocation mediated plastic flux creep is expected to occur at high fields of  $H > H_{2p}$ . One of the main characteristics of the dislocation mediated plastic flux creep mechanism is the decrease of pinning potential  $U_0$  with increasing fields.<sup>46</sup> This is contrary to the collective (elastic) flux creep mechanism. Based on the assumption that a dislocation semiloop is formed between two valleys separated by a distance  $a_0$ , the zero-current activation energy for the motion of a dislocation in the vortex lattice is given by<sup>45,46</sup>

$$U_{\rm pl}^0 = \varepsilon \varepsilon_0 a_0 \propto H^{-1/2},\tag{5}$$

where  $\varepsilon_0 = (\Phi_0/4\pi\lambda)^2$  is the vortex line tension,<sup>24</sup>  $a_0 \approx (\Phi_0/H)^{1/2}$  is the mean intervortex distance,  $\Phi_0$  is the flux quantum, and  $\varepsilon = (m_{\rm al}/m_c)^{1/2}$  is the mass anisotropy parameter. Equation (5) shows that the activation energy  $U_{\rm pl}^0$  of plastic flux creep decreases with increasing fields. Therefore, our results indicates that the vortex motion mechanism for  $H > H_{2p}$  is mainly dominated by the dislocation mediated plastic flux creep. For  $H_{on} < H < H_{2p}$ , there is a mixed flux creep mechanism, i.e., including the collective flux creep and the plastic flux creep simultaneously. The competition between them results in the appearance of  $U_0$  maximum at  $H^*$ below  $H_{2p}$ . However, for  $H_{on} < H < H^*$ , the collective flux creep controls the flux dynamics though there also exists the plastic flux creep component, which gives rise to the increase of U with increasing field. For  $H > H^*$ , the plastic flux creep governs the flux dynamics though there also exists the collective flux creep component, which gives rise to the decrease of U with increasing field. This means that the flux creep mechanism in the field range between  $H_{on}$  and  $H_{2p}$  is



FIG. 6. The pinning potential  $U_0$  as a function of field *H* for (Bi, Pb)-2212 single crystal at T = 16 K. Inset is the plot of *S* as a function of fields at 16 K. The dashed lines are guides to the eyes only.

complex, and that the flux creep process is governed by the smaller activation energy among the collective flux creep activation energy  $U_{\rm el}$  and the plastic flux creep activation energy  $U_{\rm pl}$ .<sup>44</sup> In addition, our results also reveal that the plastic flux creep does not occur exactly at  $H_{2p}$  but does at  $H^*$  prior to  $H_{2p}$  because U begins to decrease as  $H > H^*$  as shown in Fig. 5. Similar flux dynamic phenomena described above are also observed at T=33 K (not shown here).

Why does the field dependence of  $U_0$  extracted by fitting M(t) based on Eq. (3) at different fields not agree with the collective creep theory? This seems to be illogical. This may be explained in terms of the plastic vortex creep theory developed recently by Kierfeld et al.47 This theory shows that the current dependence of plastic creep activation energy  $U_{\rm pl}(j)$  also follows an inverse power law, i.e.,  $U_{\rm pl}(j) \propto j^{-\mu}$ . This means that both the collective flux creep and the plastic vortex creep theories are of the same current dependence of activation energy. Now that two theories have the same U(j)relationship, the magnetization relaxation law predicated by them should also be identified because  $M(t) \propto j(t)$  and the time evolution of the screening current density j(t) can be determined from the assumed U(j) relationship and the general form of  $U(j) = k_B T \ln(t/t_0)$ .<sup>48</sup> This means that the extracted  $U_0$  by fitting M(t) according to Eq. (3) has two kinds of possibilities, i.e., it corresponds to the collective creep activation energy  $U_{\rm el}^0$  or does to the plastic creep activation energy  $U_{\rm pl}^0$ . The actual creep mechanism occurring in the sample at different field regimes can be only distinguished from the different field dependence of the activation energy predicated by two theories. Namely, the characteristic of the collective creep mechanism is the increase of  $U_{el}^0$  with increasing field and the characteristic of the plastic creep mechanism is the decrease of  $U_{\rm pl}^0$  with increasing field.

As to the negative power law of  $U_0(H)$  for  $H < H_{on}$ , it may be related to the surface barrier. It can be seen that there exists an obvious contribution from the surface barrier based on the asymmetry of M(H) hysteresis loop, i.e., the magnetization M is remarkably less when the field decreases than that when the field increases, in particular, in the field range of  $H < H_{on}$  as shown in Fig. 1, Therefore, the creep mechanism for  $H < H_{on}$  may be very complicated due to the contribution of the surface barrier, which may result in the negative power law of  $U_0(H)$  at  $H < H_{on}$ . This is confirmed by the result obtained at 16 K as shown below, in which the contribution of the surface barrier can be negligible based on the almost symmetric M(H) hysteresis loop due to the strong bulk pinning property at low temperatures.

In the lower temperature region of T < 18 K where  $H_{on}(T)$  deviates from the relation of the disorder induced vortex lattice transition:<sup>29</sup>  $H_{on}(T) = H_{on}(0)[1]$  $-(T/T_c)^4$ ]<sup>3/2</sup>, the magnetic relaxation in the vicinity of both  $H_{on}$  and  $H_{2n}$  behaves differently compared to the behavior of T > 18 K. Based on the magnetic relaxation measurement at T = 16 K, the pinning potential  $U_0$  as a function of field H is plotted in Fig. 6. The magnetic-field dependence of S shown in the inset of Fig. 6 also behaves differently from S(H) at T=25 K. Figure 6 indicates that  $U_0(H)$  increases with increasing field for  $H \le H_{on}$ , which is different from  $U_0(H)$  at T=25 and 33 K. As  $H>H_{2p}$ , the negative power law of  $U_0(H)$  described above is also absent and an almost constant  $U_0(H)$  is observed as  $H > H_{2p}$ . In the collective vortex pinning and single vortex pinning regime  $U_0 \sim \text{constant}$  is expected as T is below the depinning temperature  $T_{dp}$ .<sup>39</sup> The physical reason for the change of the creep mechanism at T = 16 K is not clear at present, because exact reason of low temperature  $H_{on}(T)$  behavior deviating from the predication by the disorder induced vortex lattice transition theory is also unclear.<sup>29</sup> It may be related to the specific vortex structure at low temperatures.

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## **IV. CONCLUSION**

In summary, we have performed magnetic relaxation measurements on a heavily Pb-doped  $Bi_2Sr_2CaCu_2O_{8+\delta}$  single crystal in the vicinity of both  $H_{2p}$  and  $H_{on}$  at 16, 25, and 33 K. The results show that  $H_{2p}$  and  $H_{on}$  are related to the crossover in the behavior of the pinning potential as a function of fields  $U_0(H)$  for T = 25 K, 33 K. Since the crossover of  $U_0(H)$  occurs at  $H_{on}$ , it may be related with the contribution of surface barrier and does not originate from the variation of the creep mechanism. In the field region of  $H_{on} < H < H_{2p}$ , flux creep mechanism is complicated, and it includes the competition between the collective flux creep and the plastic flux creep, resulting in the appearance of  $U_0$ maximum at  $H^*$  prior to  $H_{2p}$ . For  $H > H_{2p}$ , the negative power law of  $U_0(H) \propto H^{-0.7}$  stems from the plastic flux creep. At lower temperature, 16 K, both S(H) and  $U_0(H)$ demonstrate different behaviors compared with those at 25 and 33 K. The vortex dynamics variation in the vicinity of  $H_{2p}(T)$  implies that AMP may be of the dynamics origin and that it may be related to the underlying change of the vortex structure in essence.

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