

# Thermodynamic observation of a vortex melting transition in the Fe-based superconductor $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$

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In cuprate high-temperature superconductors the small coherence lengths and high transition temperatures result in strong thermal fluctuations, which render the superconducting transition in applied magnetic fields into a wide continuous crossover. A state with zero resistance is found only below the vortex melting transition, which occurs well below the onset of superconducting correlations. Here we investigate the vortex phase diagram of the Fe-based superconductor in the form of a high-quality single crystal of  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$ , using three different experimental probes (specific heat, thermal expansion, and magnetization). We find clear thermodynamic signatures of a vortex melting transition, which shows that the thermal fluctuations in applied magnetic fields also have a considerable impact on the superconducting properties of iron-based superconductors.

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## I. INTRODUCTION

In classical type-II superconductors, typically two distinct superconducting phases are found: the Meissner state in low fields and the Abrikosov state with field-induced vortices or flux lines which form a regular long-range ordered periodic lattice of typically hexagonal arrangement. In the presence of disorder in the crystal structure, flux pinning effects can furthermore prevent the formation of the regular lattice structure and result in a rich variety of various amorphous glassy vortex phases.<sup>1–3</sup> In the layered cuprate high-temperature superconductors with their short coherence lengths and high transition temperatures, critical thermal fluctuations of the 3D-XY universality class have a significant impact on their superconducting properties in a large range around the superconducting transition temperature.<sup>4–9</sup> As a result, the solid vortex phases melt into a liquid vortex phase well below the superconducting transition temperature, which has been observed experimentally in the  $R\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  compounds with  $R = \text{Y, Eu, Dy, or Nd}$  (Refs. 10–21). Signatures of a vortex melting have been furthermore observed in some classical superconductors, but only in close proximity to the superconducting transition.<sup>22–25</sup>

The vortex melting transition represents the only sharp phase transition in a magnetic field which can be either of a first-order or a second-order nature, depending on whether the solid phase is crystalline or glassy.<sup>10–20,26–32</sup> It is of particular importance for technical applications of superconductors, as only the solid vortex phase can show true zero resistance. In the liquid state the vortices can move individually and freely, whereas in the solid phase the finite shear modulus causes collective pinning and thus can effectively anchor the vortices and prevent dissipation in presence of a current.<sup>3</sup> In this sense, the vortex melting transition represents the true superconducting transition in applied magnetic fields. Early approaches to describe this transition with a model of a phonon-induced melting<sup>3,33,34</sup> were based on a Lindeman criterion,<sup>35</sup> which states that when the amplitude of thermally induced vortex vibrations about their equilibrium position

reaches a critical value the solid lattice becomes unstable and melts. Later, experimental indications have been found that the vortex melting transition is closely linked to the main superconducting transition and may be rather induced by the increasing strength of critical fluctuations upon approaching the superconducting transition.<sup>7,23,36–40</sup>

With the recent discovery of a new class of high-temperature iron-based superconductors based on 2D Fe-As layers,<sup>41</sup> high-temperature superconductivity research has again become a primary focus of solid-state physics research. These superconductors show many similarities to the cuprate high- $T_c$ 's but also significant differences.<sup>42,43</sup> Thermal fluctuations have also been observed in the iron-based superconductors and appear to be stronger in the more two-dimensional 1111 compounds<sup>44</sup> than in the 122 compounds.<sup>45</sup> The overall strength of the fluctuations seems to be intermediate between the behavior in the cuprates and more classical superconductors like  $\text{Nb}_3\text{Sn}$ .<sup>23,24</sup> The reasons are certainly the lower transition temperatures and the weaker anisotropy and larger coherence lengths. Careful high-resolution specific-heat measurements nevertheless showed that thermal fluctuations and vortex melting can even be observed in the 3D bulk classical superconductor  $\text{Nb}_3\text{Sn}$ .<sup>23,24</sup> This raises the question of whether vortex melting also exists in the Fe-based superconductors. A perfect tool for investigating vortex melting is specific-heat measurement, as the heat capacity—in contrast to other thermodynamic probes—is typically unaffected by irreversible flux pinning effects.<sup>24</sup> These cause irreversible hysteresis effects, e.g., in the magnetization, which may mask the tiny thermodynamic vortex melting anomalies. The specific heat has been proven to be a powerful method to reveal the first-order character of the melting transition in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ,<sup>10,13,46</sup>  $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ,<sup>21</sup> and in  $\text{Nb}_3\text{Sn}$ .<sup>23</sup> On the other hand, thermal expansion measurements are particularly sensitive to pinning and thus serve as a sensitive probe of the vortex relaxation occurring around a vortex melting transition with some disorder.<sup>9</sup> In this paper we report high-resolution measurements of the specific heat in combination with dc magnetization and thermal expansion experiments on

a high-quality single crystal of  $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$  with  $x = 0.5$  in magnetic fields up to 14 T. All three experimental probes show clear signatures of a sharp transition, which we associate with an underlying vortex melting transition in the presence of weak pinning.

## II. EXPERIMENTAL

The  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  single crystals were grown from self-flux using an  $\text{Al}_2\text{O}_3$  crucible. Ba and K (3:2) were mixed with prereacted FeAs in a ratio of 1:5, filled into the crucible, and enclosed and sealed in a steel container. After heating to  $1151^\circ\text{C}$ , the crucible was cooled down very slowly to  $1051^\circ\text{C}$  at a cooling rate of  $0.20^\circ\text{C/h}$ . At the end of the growth the crucible was tilted to decant the remaining flux and then was slowly pulled out of the furnace.

The magnetization, specific heat, and thermal expansion measurements have all been performed on pieces taken from the same single crystal. The specific-heat experiments on a tiny crystal with a mass of  $60\ \mu\text{g}$  were performed under field-cooled (FC) conditions with a homemade high-resolution microcalorimeter which is perfectly adapted to measurements of samples with masses down to a few  $\mu\text{g}$ . It can be used with either a dc “long-relaxation” technique for measurements with precision up to 1% or a modulated-temperature ac technique. The latter is less accurate, but provides high resolutions of  $\Delta C/C$  of  $10^{-5}$  at a high density of data points. The data presented in this paper were measured with the ac technique, but calibrated with data taken by the relaxation technique. The magnetization was measured with a commercial Quantum Design Vibrating-Sample SQUID magnetometer under zero-field-cooled (ZFC) and FC conditions, which can provide extremely high resolutions (better than  $10^{-8}$  emu). A capacitive dilatometer was used for the measurements of the thermal expansion. As this technique requires a certain sample length of a few mm in order to obtain sufficient resolution, the experiments were done on a second larger piece of the single crystal with mass of 2.3 mg. For the specific heat and thermal expansion all data have been measured under FC conditions. In all the experiments the sample was oriented with the  $c$  axis parallel to the applied field.

## III. RESULTS

Figure 1 shows the total heat capacity in zero field and various magnetic fields up to 14 T. The zero-field superconducting transition is seen as a slightly broadened jump centered at 34.4 K. Application of a magnetic field reduced the transition temperature and additionally broadens the transition. In order to look for the vortex melting transition, the zero-field data have been subtracted as a background in Fig. 1(b). The large downturn towards higher temperature is caused by the superconducting transition anomaly at  $T_c$  in the subtracted zero-field data. Below this downturn, tiny upwards steplike anomalies can be resolved, which move towards lower temperature upon increasing field [arrows in Fig. 1(b)]. The anomalies resemble those observed in the specific heat of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ <sup>10,13,46</sup> and  $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ,<sup>21</sup> where the transition appears to be second order due to residual pinning. For fields higher than 6 T, there appears to be a

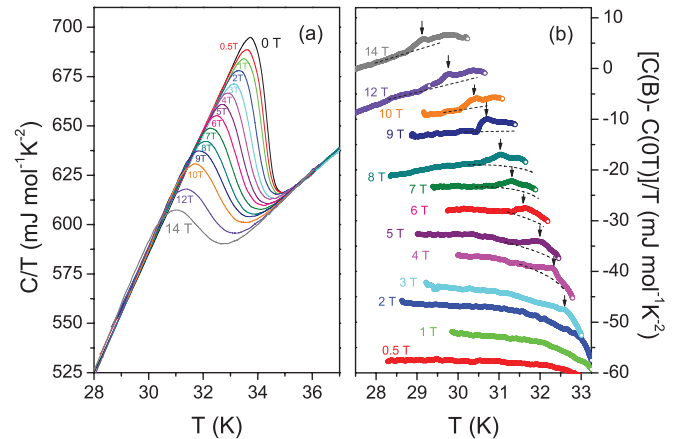


FIG. 1. (Color online) (a) Specific-heat of  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  in magnetic fields up to 14 T. (b) The same data as in (a) but with the zero-field data subtracted as a background. The downturn in smaller fields on the right-hand side is due to the subtracted zero-field superconducting transition anomaly. Except for the 14-T data, all data have been shifted vertically relatively to each other for clarity. Small steps, as marked by the arrows, indicate the vortex melting transition anomalies ( $T_m$ ). For clarity, only data in the temperature range of  $T_m$  are presented. The dotted lines serve as guides for the eye.

very small first-order peak superimposed on top of this step; however, the effect is close to the resolution limit of our experiment. The lack of a prominent first-order peak, as we discuss later, is attributed to flux pinning in our sample. No significant difference was found when the data have been taken upon heating or cooling the sample.

Figure 2 shows data of the magnetization under ZFC and FC conditions in various magnetic fields. The two branches split at the temperature which is usually defined as the irreversibility temperature  $T_{\text{irr}}$ , below which flux pinning sets in. Above  $T_{\text{irr}}$ , a large reversible range occurs within the superconducting state, which is attributed to the liquid vortex phase. A sharp

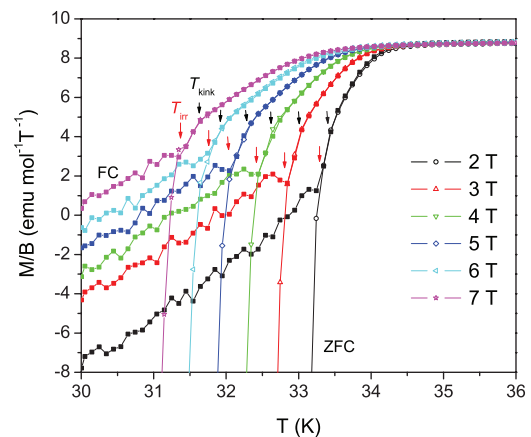


FIG. 2. (Color online) dc magnetization data of  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  measured with a VSM SQUID magnetometer upon continuous temperature sweeps upon heating under ZFC and FC conditions. The red arrows mark the irreversibility line ( $T_{\text{irr}}$ ) and the black arrows additional kinks ( $T_{\text{kink}}$ ) in the magnetization (see text for details). Note that the positive background originates from the normal-state magnetic properties of the material.

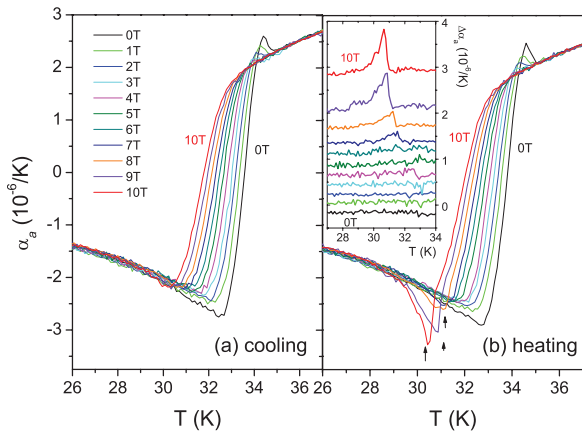


FIG. 3. (Color online) Linear thermal expansion data of  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  along the crystallographic  $a$  axis upon cooling (a) and subsequent heating (b). Large irreversible peaks ( $T_{\text{peak}}$ ) appear upon heating in the vicinity of the vortex melting transition, as marked by the arrows. (Inset) Difference  $\Delta\alpha$  of the data measured upon cooling and heating to show the peaks more clearly. The data has been shifted vertically relatively to each other for clarity.

kink occurs in the reversible part of the curve ( $T_{\text{kink}}$ ) slightly above the irreversibility line. Similar kinks have been observed previously for cuprate high- $T_c$  superconductors and have been interpreted as artifacts from the measurement routine of conventional SQUID magnetometers in close vicinity of the irreversibility line.<sup>47</sup> It is therefore unlikely that the kinks represent the thermodynamic signature of the vortex melting transition. However, as both  $T_{\text{kink}}$  and  $T_{\text{irr}}$  follow  $T_m$  as obtained from the specific-heat data, both anomalies are likely related to the vanishing of irreversibility at the vortex melting transition.

Figure 3 shows linear thermal expansion coefficient  $\alpha(T) = 1/L_0 \times dL/dT$  measured along the crystallographic  $a$  axis upon field cooling (a) and subsequent heating (b). The thermal expansion coefficient  $\alpha(T)$  is closely related to the specific heat and phase transition anomalies appear with the same shape but, in contrast to the specific heat, can have either a positive or a negative signature. The negative signature of the large jump at the superconducting transition indicates, according to the thermodynamic Ehrenfest relation,<sup>48</sup> a negative uniaxial pressure dependence of the transition temperature for pressure along the  $a$  axis, in contrast to the positive pressure dependence reported for Co-doped Ba122.<sup>49,50</sup>

Apart from the large jump at  $T_c$ , additional smaller anomalies are visible in the heating curves near the proposed vortex melting, as well as a small spikelike anomaly above  $T_c$  in both heating and cooling curves. The latter anomaly is, we believe, an artifact, possibly due to a small misaligned crystal region with a slightly higher  $T_c$  and it disappears above 3 T. Other crystals from the same batch did not show this feature, and here we concentrate on the anomalies associated with vortex melting, which are observed only upon heating for fields greater than 6 T [see Fig. 6(b)]. In the inset we plot the difference between heating and cooling curves, in which the anomalies at  $T_{\text{peak}}$  are made more prominent. The absence of these anomalies in the cooling data indicates an irreversible nonthermodynamic origin. The maxima of the peaks coincide rather well with  $T_m$  as obtained from the specific heat

(see Fig. 1). Similar peaks have been observed at the vortex melting transition of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ <sup>51</sup> in samples with weak but finite flux pinning, which were attributed to the decay of some nonequilibrium screening currents near a second-order vortex melting transition.<sup>9</sup> Due to the close similarity of the present effects observed in the thermal expansion with those in YBCO, we also attribute these to an underlying vortex melting transition.

#### IV. DISCUSSION

Signatures of an underlying vortex melting transition are observed with all three of our experimental probes; the origin of these signatures is, however, slightly different for each probe, as explained in detail below. The specific heat as a purely thermodynamic quantity is usually insensitive to the effect of nonequilibrium screening currents, which originate from irreversible flux pinning effects,<sup>51</sup> and therefore provides a purely thermodynamic signature of the vortex melting transition. The shape of the specific heat transition anomalies shows the characteristic upward step indicative for the additional degrees of freedom in the high-temperature liquid vortex phase. Additionally, in larger fields, a tiny peak, indicative of the latent heat of a first-order melting transition, appears superimposed on this step. In comparison with specific heat on reversible  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ <sup>10,13,46</sup> and  $\text{NdBa}_2\text{Cu}_3\text{O}_{7-\delta}$ <sup>21</sup> samples, the peaks are, however, much smaller. This suggests that the pinning in our sample is stronger and the crystallization of the flux line lattice is incomplete, presumably related to the formation of amorphous vortex-glass phases.<sup>1-3</sup>

It has been predicted previously that a small additional ac field superimposed onto the applied dc field can help the vortices to overcome the pinning forces and relax into the crystalline state.<sup>52,53</sup> With help of specific-heat data it has been demonstrated, e.g., for  $\text{Nb}_3\text{Sn}$  that a vortex melting peak occurs if an ac field of a few Oe in the kHz range is applied parallel to the dc field.<sup>23</sup> In this orientation, the ac field will modulate the density of the vortices and cause vortex vibrations, which help the vortices to overcome pinning forces. To see if the first-order character of the vortex melting transition can be enhanced by such an ac field, we added a small solenoid around our microcalorimeter and applied weak ac fields to the sample. The result is shown for the 10 T in Fig. 4. The ac field causes a strong increase of the specific heat in the vortex melting range and also below. This may be indicative for additional degrees of freedom related to unpinning vortices, which can absorb additional heat. The best results have been obtained for ac fields of  $\sim 10$  Oe amplitudes at frequencies in the kHz range. At lower frequencies and amplitudes the effect vanished abruptly, whereas larger fields or frequencies had little effect. The peak at the melting transition becomes clearly more pronounced. Dissipative heating effects from the ac field can be ruled out as the released heat would cause a negative contribution instead of the increase observed here. The effect was fully reversible and when the ac field was removed during a measurement, the specific heat relaxed back to the original value within a few seconds. Although the exact effect of the additional ac field on the superconductor and the calorimetric technique is hard to predict, this experiment can be regarded as a further hint that the weak pinning does play a significant role in the solid vortex

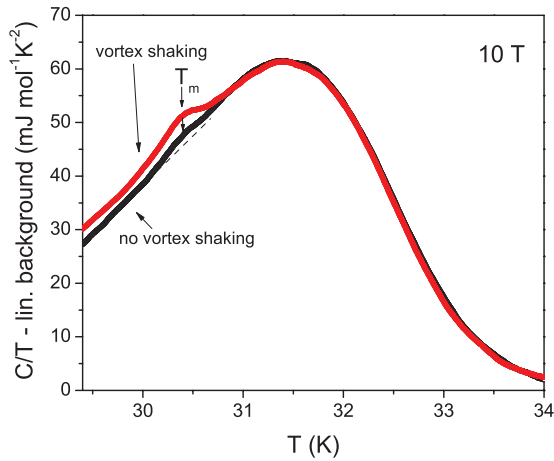


FIG. 4. (Color online) Specific-heat data in 10 T with the same linear background fitted above  $T_c$  subtracted for clarity. The lower data (“no vortex shaking”) are identical to the data shown in Fig. 1. During the measurement of the upper data (“vortex shaking”), an additional weak ac magnetic field ( $\sim 10$  Oe, 1 kHz) has been superimposed onto the ac field in order to help the vortices to reach thermodynamic equilibrium.

phase and confirms that the solid phase is slightly disordered and most likely represented by the Bragg glass phase predicted by Giarmarchi and Le Doussal.<sup>2</sup>

Magnetization and thermal expansion are probes, which are sensitive to both the reversible thermodynamic contribution and irreversible contributions due to flux pinning. The magnetization data in Fig. 2 show a large thermodynamic reversible range above  $T_{irr}$ , which represents the liquid vortex phase. The magnetization is, however, also directly sensitive to induced nonequilibrium screening currents, which appear below  $T_{irr}$ , as visible in the difference between the ZFC and FC branches. These screening currents may furthermore influence the thermal expansion via the magnetic interaction of the screening currents and the applied field. The applied field applies a “pressure” on the currents which is transferred to the crystal structure via the flux pinning centers. The observed comparatively large spikelike irreversible anomalies in the thermal expansion in the vicinity of the thermodynamic vortex melting transition are therefore likely related to the decay of such screening currents in the vicinity of the vortex melting transition. The similar anomalies which have been observed previously in  $YBa_2Cu_3O_{7-\delta}$  samples in the presence of weak flux pinning<sup>36</sup> have been demonstrated to occur at a second-order vortex melting transition. These anomalies showed behavior comparable to a kinetic glass transition and are thus most likely related to some glassy vortex phases in the presence of weak collective pinning. Upon approaching the vortex melting transition upon subsequent heating, the currents rapidly decay so that a large peak appears in  $\alpha(T)$ .

In order to test this scenario and to investigate how these screening currents build up and decay under the FC field-heating conditions of our dilatometry measurements, we performed magnetization measurements under similar conditions (Fig. 5). We applied a field of 7 T above the zero-field  $T_c$  and then first continuously field-cooled the sample at 0.1 K/min and afterwards heated at the same rate.

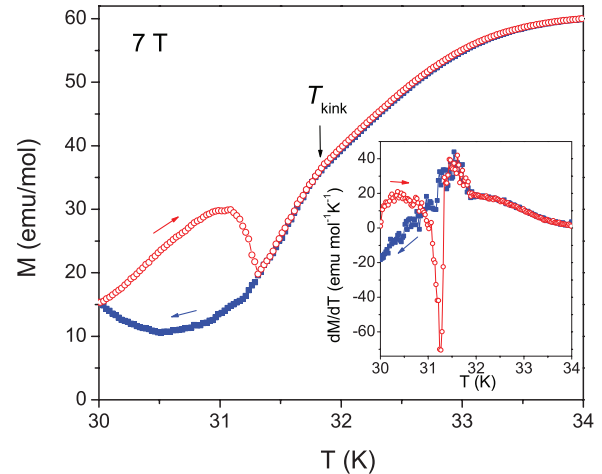


FIG. 5. (Color online) FC magnetization data of  $Ba_{0.5}K_{0.5}Fe_2As_2$  in a field of 7 T upon cooling the sample with 0.1 K/min and subsequent heating with 0.1 K/min. A nonequilibrium screening current builds up at low temperature, which decays upon approaching  $T_{irr}$ . (Inset) The corresponding derivative  $dM/dT$  of the magnetization. The decay of the induced screening current forms a sharp peak below  $T_{irr}$  of identical shape to the peaks occurring in the thermal expansion.

The two branches are separated below  $T_{irr}$  with a small positive contribution appearing in the heating curve, in a similar manner as described by theory in Ref. 54. This difference originates from some nonequilibrium currents (presumably due to temperature gradients in the sample), which are gradually formed when the temperature is changed below  $T_{irr}$ . In the inset of Fig. 5 we show a plot of the corresponding temperature derivative  $dM/dT$  of the magnetization. It shows a sharp peak in the heating curve, related to the decay of the screening current when  $T_{irr}$  is approached, of very similar shape as in  $\alpha(T)$ . This confirms that the origin of the sharp peaks in the thermal expansion at the vortex melting transition is due to the decay of screening current due to the abrupt breakdown of collective pinning in the vicinity of the vortex melting transition.

The presence of these peaks further illustrates that our samples show some irreversible effects related to flux pinning. Thermal expansion data taken on an extremely clean sample of  $YBa_2Cu_3O_{7-\delta}$  without any noticeable effect of flux pinning<sup>36</sup> showed the same reversible thermodynamic signature of the vortex melting transition as the specific heat,<sup>46</sup> which is masked in the present heating data by the additional larger irreversible anomalies. The flux pinning, which causes these screening currents, may furthermore explain the small latent heat observed in the specific-heat experiments.

In Fig. 6 we summarize our results in a magnetic field vs temperature phase diagram of  $Ba_{0.5}K_{0.5}Fe_2As_2$ . The position of the spikelike anomalies in the thermal expansion coincides well with the position of the thermodynamic signature of the vortex melting transition in the specific heat. Furthermore, the position of the irreversibility line agrees well with the vortex melting transition. The line where the sharp kink occurs in the reversible magnetization runs closely spaced in parallel to the vortex melting transition as extracted from the specific heat. As the steplike anomaly in the specific heat is not perfectly

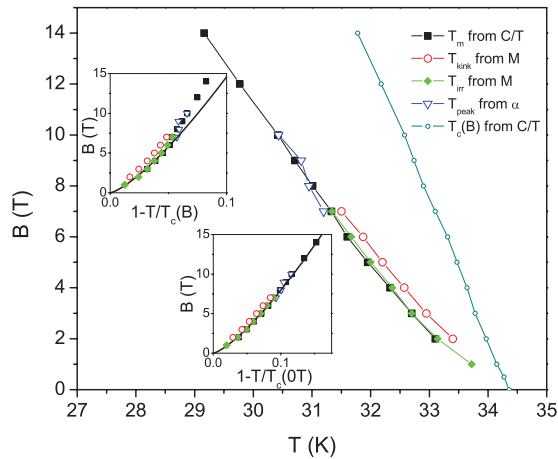


FIG. 6. (Color online) Magnetic field versus temperature phase diagram of  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  with the vortex melting transition ( $T_m$ ) as obtained from  $C/T$ , the irreversibility line ( $T_{\text{irr}}$ ), the line where the kink appears in the reversible magnetization ( $T_{\text{kink}}$ ), the temperature where the large irreversible peaks appear in the thermal expansivity, as well as the midpoint of the main superconducting transition [ $T_c(B)$ ] in the specific heat. The insets show fits to these data according to the theoretical prediction of a vortex melting transition caused by 3D-XY critical fluctuations plotted as function over relative temperature scales  $1-T/T_c(B)$  and  $1-T/T_c(OT)$  (see text for details).

sharp, the kink in the magnetization most likely coincides with the upper onset of the vortex melting transition and may be associated with the first signature of collective flux pinning in the sample, which then gets far more pronounced below  $T_{\text{irr}}$  when the Bragg glass phase is fully established. Especially in lower fields all the lines show a typical power-law behavior. In  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ <sup>13,38</sup> it has been found that the vortex melting line can be described by a power law dependence of the form  $B_m \sim [1-T_m/T_c(OT)]^{2\nu}$ , where  $\nu \approx 0.67$  is a critical exponent of the 3D-XY universality class describing the divergence of the coherence length in the vicinity of the critical point at the zero-field  $T_c$ . This has been discussed as evidence that the vortex melting is caused by critical fluctuation effects of the 3D-XY universality class.<sup>7,37-40</sup> In the insets of Fig. 6 we plot the data vs  $1-T/T_c$ . The fact that  $T_c$  shows a clear field dependence complicates the analysis, as in the pure 3D-XY model  $T_c$  is not field dependent but only gets broadened by fluctuations. We tried fitting the data as a function of both

$1-T/T_c(B)$  and  $1-T/T_c(OT)$ . When the field-dependent  $T_c$  is chosen, the fit fails in the high-field regime. Surprisingly, we can fit the data perfectly by using the zero-field  $T_c$  instead, which is expected in the 3D-XY model. It is, however, not clear what the implications of these results are, since critical fluctuations in  $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$  are certainly expected to be much weaker than in the cuprates. Nevertheless, the fact that the vortex melting line in  $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$  follows the same power-law dependence as  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  indicates that fluctuation effects are of considerable importance for the vortex dynamics in finite magnetic fields of several teslas.

## V. CONCLUSION

To conclude, we have observed clear signatures of a phase transition in the vortex matter of the iron arsenide superconductor  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$ . The specific heat data are consistent with a second-order-type transition, which possibly crossed over to a very weak first-order transition in higher fields. This thermodynamic signature is accompanied by the appearance of irreversible effects in the magnetization when collective pinning sets in. The linear thermal expansion coefficient shows large spikelike anomalies only upon FC heating, which originate from the rapid decay of nonequilibrium screening currents at the underlying vortex melting transition. They build up continuously upon the preceding field cooling of the sample. The small latent heat at the vortex melting transition in combination with the irreversible effects observed in the magnetization and thermal expansion suggest that the crystallization of the flux line phase is incomplete and the solid phase is mostly likely associated with a weakly disordered topologically ordered Bragg glass phase.<sup>2</sup> The existence of vortex melting in  $\text{Ba}_{0.5}\text{K}_{0.5}\text{Fe}_2\text{As}_2$  demonstrates that the effect of thermally induced superconducting fluctuations cannot be neglected in iron arsenide superconductors.

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