Vortex-glass phase transition and superconductivity in an underdoped (Ba,K)Fe₂As₂ single crystal

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Measurements of magnetotransport and current-voltage (I-V) characteristics up to 9 T were used to investigate the vortex phase diagram of an underdoped (Ba,K)Fe₂As₂ single crystal with T_c =26.2 K. It is found that the anisotropy ratio of the upper critical field H_{c2} decreases from 4 to 2.8 with decreasing temperature from T_c to 24.8 K. Consistent with the vortex-glass theory, the I-V curves measured at H=9 T can be well scaled with the vortex-glass transition temperature of T_g =20.7 K and critical exponents z=4.1 and v=1. Analyses in different magnetic fields produced almost identical critical exponent values, with some variation in T_g , corroborating the existence of the vortex-glass transition in this underdoped (Ba,K)Fe₂As₂ single crystal up to 9 T. A vortex phase diagram is presented, based on the evolution of T_g and H_{c2} with magnetic field.

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I. INTRODUCTION

The recent discoveries of several families of FeAs-based superconductors such as AFeAsO_{0.89}O_{0.11} (A=La, Nd, Sm, and Pr) (FeAs-1111) (Refs. 1-7) and oxygen-free Ba_{1-r}K_rFe₂As₂ (FeAs-122) (Ref. 8) have triggered a surge of research activities for understanding the basic superconducting mechanism as well as for finding practical applications. These families of compounds have shown remarkably high superconducting transition temperatures; the highest T_c in the FeAs-1111 system is currently 54 K (Ref. 1) and in the FeAs-122 system is 37.5 K. In a LaFeAsO_{0.89}F_{0.11} polycrystalline specimen, the zero-temperature upper critical field $H_{c2}(0)$ was found to be as high as 65 T.⁹ Furthermore, the anisotropy ratio of the upper critical field, i.e., $\gamma = H_{c2}^{\perp c}/H_{c2}^{\parallel c}$, measured in a single crystal of NbFeAsO_{1-x}F_x was reported to be about 4 near T_c .¹⁰ However, the superconducting gap nature of the FeAs-1111 compounds seems to be yet controversial. 9-11 Moreover, the investigation of the vortex state is still lacking in the FeAs-1111 series, possibly due to the mostly polycrystalline nature of the samples prepared.

On the other hand, the FeAs-122 compounds exist in the single crystalline form and their transition temperature can be controlled by replacing the alkaline elements (Ba, Ca, and Sr) with K and Na. 12-15 Therefore, it is expected that one can investigate anisotropic superconducting properties and vortex phase diagram with variation in T_c . In the case of an optimally doped Ba_{0.6}K_{0.4}Fe₂As₂ single crystal with T_c \approx 38 K, $H_{c2}(0)$ along the c axis was estimated to be 130 T by applying the Werthamer-Helfand-Hohenberg (WHH) formula 16 to the H_{c2} curve measured up to magnetic field H=9 T, ¹⁷ and the anisotropy ratio γ was found to be 3–4 near T_c 17-19 The vortex solid-liquid phase boundary was suggested from the temperature-dependent irreversibility line in the magnetization curves.²⁰ However, these studies have been restricted to the optimally doped samples so far. In addition, the vortex phase boundary based on the irreversibility line needs further investigation of its microscopic transition nature, including the type of phase transition and the critical exponents.

Historically, in order to understand the nature of the vortex phase transition in cuprate superconductors, Fisher *et*

al. ²¹ proposed a theory of the vortex-glass phase, in which the current-voltage (*I-V*) characteristics follow a universal scaling function near the vortex-glass transition temperature T_g . According to this vortex-glass (VG) theory, ²¹ the vortex-glass correlation length (ξ) diverges near T_g , described by $\xi \sim |T-T_g|^{-\nu}$ and a correlation time scale ξ^z , where ν is a static exponent and z is a dynamic exponent. The experimental evidence for a vortex-glass phase has been identified for various superconductors such as YBa₂Cu₃O₇, ²² YNi₂B₂C, ²³ and MgB₂ (Ref. 24) through the scaling exponents ν and z in the range of $\nu \sim 1-2$ and z > 4, as predicted by the VG theory. ²¹ However, to date, the vortex-glass phase transition has not been investigated in detail in the FeAs-based superconductors.

Therefore, the present work reports on the vortex phase diagram and related vortex physics in an underdoped $(Ba,K)Fe_2As_2$ single crystal with T_c =26.2 K, with measurements of magnetotransport, magnetization, and I-V characteristics. We obtained a good scaling of the I-V curves with the critical exponents z=4.1 and ν =1, uncovering the vortex-liquid (VL) to vortex-glass transition. Using the scaling analyses of the I-V curves and H_{c2} values, we determined the vortex phase diagram of the underdoped $(Ba,K)Fe_2As_2$ single crystal up to 9 T.

II. EXPERIMENTS

The single crystals were grown using the Sn flux method. ¹⁸ The elements Ba, K, Fe, As, and Sn with the molar ratio of 0.6:0.8:2:2:20 were put into an alumina crucible and then sealed inside a quartz ampoule under a partial Ar atmosphere. The ampoule was heated up to 850 °C and cooled at 4 °C/h. The resultant crystals had a thin platelike shape. X-ray θ -2 θ diffraction patterns showed that the c axis is parallel to the plate surface. The stoichiometry of the sample was analyzed by using the electron probe microanalysis (EPMA) method, revealing Ba:K=0.64:0.36. Compared with the optimally doped sample Ba_{0.55}K_{0.45}Fe₂As₂ with T_c ~38 K,8 our crystals correspond to an underdoped composition.

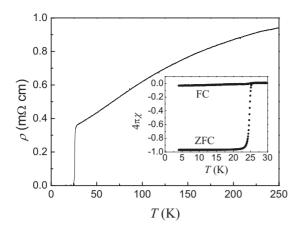


FIG. 1. The temperature dependence of resistivity at zero field for the $(Ba,K)Fe_2As_2$ single crystal. The transition temperature is about 26.2 K and the transition width is 1.2 K. Inset shows a temperature-dependent magnetization curve measured at H =15 Oe applied along the c axis.

Two pieces of single crystals were selected with dimen- $0.056 \times 0.7 \times 0.7 \text{ mm}^3$ and 0.025×2.06 ×1.68 mm³ for the investigation of transport properties and magnetization, respectively. Electrical contact was made using silver epoxy (Epotek H20E) and gold wires. We have kept the epoxy curing temperature below 200 °C as the samples were found to degrade above this temperature. Temperature-dependent resistivity was measured up to H=9 T, both parallel and perpendicular to the c axis, respectively, by using a physical property measurement system (PPMSTM, Quantum Design). The I-V characteristics were also investigated for various H parallel to the c axis by using the PPMS. The magnetization curves were measured up to H=9 T using a vibrating sample magnetometer operating inside the PPMS.

III. RESULTS AND DISCUSSION

Figure 1 shows the resistivity (ρ) curve of the underdoped (Ba, K)Fe₂As₂ single crystal at zero field. The transition temperature of T_c =26.2 K was obtained by choosing the temperature where a linear extrapolation line from the normalstate resistivity meets with another line extrapolating the steeply increasing ρ region at the superconducting transition. A superconducting transition width of $\Delta T_c \sim 1.2$ K was determined from the temperature difference between 90% and 10% dropoff of $\rho(30 \text{ K})$. The residual resistivity at 30 K of $0.4~\text{m}\Omega$ cm was similar to that in Ref. 18, but ten times higher than that in Ref. 20. The residual resistivity ratio is found to be $\rho(300 \text{ K})/\rho(30 \text{ K})=2.7$, which is lower than that (7.4) of the optimal-doped sample, ¹⁷ but is similar to the ratio observed in underdoped material.²⁵ The inset of Fig. 1 shows the magnetization curve for both zero-field-cooled (ZFC) and field-cooled (FC) conditions. Assuming the ZFC and FC curves deviate from each other at the superconducting transition temperature, T_c is estimated to be 25.6 K. This value is close to the value of 26.2 K determined by the resistivity. We also confirmed that the T_c values of several

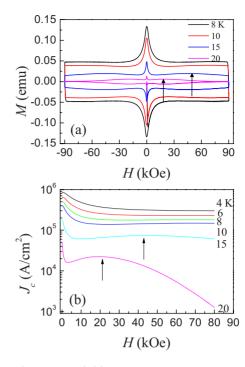


FIG. 2. (Color online) (a) The magnetic hysteresis curves of the $(Ba,K)Fe_2As_2$ single crystal with H||c axis at T=8, 10, 15, and 20 K. (b) The critical current density between 4 and 20 K estimated from magnetization curves by using the critical state model of Bean (Ref. 26). Peaks in both M and J_c vs H are marked with arrows.

single crystals from the same grown batch were consistent within $0.5\ \mathrm{K}.$

Figure 2(a) shows the magnetic hysteresis curves measured at temperatures from 8 to 20 K in the field range of -9 T $\leq H\leq 9$ T. Even at 20 K, the highest magnetic field in our experiments (9 T) was not sufficient to achieve closure of the magnetic hysteresis curve. Extrapolating these 20 K data, the closure field would be about 10 T, giving at least one value for the irreversibility line (to be discussed below with the phase diagram). Figure 2(b) shows the critical current density (J_c) , estimated from the magnetization curves at various temperatures. The J_c was calculated by using the critical state model of Bean²⁶ $(J_c=20\Delta M/[w(1-w/3l)])$, where ΔM , l, and w are the height of magnetization loops, the length, and the width of the sample (w < l), respectively. Under a self-field, the value of J_c is found to be \sim 0.85 MA/cm² at 4 K. This value is comparable to the results of the FeAs-122 with T_c =36.2 K.²⁰ When H is increased, the magnetization decreases overall as expected in a typical type-II superconductor above the lower critical field. However, in the data above T=10 K, the magnetization curves develop a second peak (H_{sp}) as marked with arrows in Fig. 2(a); magnetization increases with field below $H_{\rm sp}$ and decreases above $H_{\rm sp}$. The calculated J_c curve, which is proportional to ΔM , naturally shows a similar peak effect [marked with arrows in Fig. 2(b)]. It is interesting to recall that similar phenomena have been also observed in the high- T_c cuprate superconductors. With extensive studies, the origin of the second peak in the cuprates seems to be relatively well understood; for YBa₂Cu₃O₇, the second peak effect was attributed to the twin-pinning center,²⁷ while for the

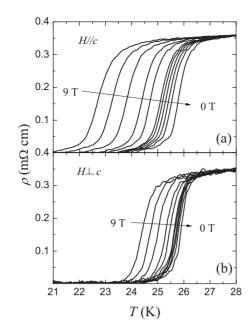


FIG. 3. The temperature dependence of resistivity along (a) $H \parallel c$ axis and (b) $H \perp c$ axis in H=0, 0.2, 0.4, 0.6, 0.8, 1, 2, 3, 5, 7, and 9 T (from left to right) for the (Ba, K)Fe₂As₂ single crystal.

Bi-based superconductors, it was due to the change in the vortex type from a three-dimensional (3D) vortex lattice to a two-dimensional (2D) droplet. 28,29

On the other hand, the origin of the second peak in the FeAs system is not clearly understood. The occurrence of the high-temperature second peak does not seem to be dependent on the material system as it was also observed in the optimaldoped FeAs-122 (Ref. 20) and SmFeAsO_{0.89} polycrystalline samples.³⁰ In the former study, the authors suggested that the second peak effect might be due to the vortices pinned by the small-sized normal cores.²⁰ We note that the optimal-doped FeAs-122 in the former study has been grown with the selfflux FeAs method. What our study shows here is the presence of the second peak even in the underdoped FeAs-122 grown with the Sn flux. As both FeAs-122 crystals show the second peak regardless of the growth flux used, it can be inferred that the normal core, if any, cannot come from the remaining impurities from the flux materials in the sample. Therefore, the second peak is likely rather caused by vortex pinning by intrinsic defects in the FeAs-122 samples. This observation in turn suggests that the second peak effect in the magnetization loop is rather common in the FeAs superconductors and further studies are required to understand the related vortex pinning mechanism.

To investigate the anisotropic H_{c2} behavior and related vortex physics, we have measured the dc transport properties in magnetic fields applied along and perpendicular to the c axis, as shown in Fig. 3. We estimated $H_{c2}(T)$ using the same criterion used to determine the zero-field T_c through resistivity: linear extrapolated lines in the normal state and just below T_c were used to find the intersection temperature ($\equiv T_c$). Figure 4 shows the temperature dependence of $H_{c2}(T)$ as determined from the $\rho(T)$ curves in Fig. 3. When the slope of $H_{c2}(T)$ near T_c is calculated, $-dH_{c2}(T)/dT|_{T_c} = 3.14$ and 6.58 T/K for $H \parallel c$ and $H \perp c$, respectively. The anisotropy

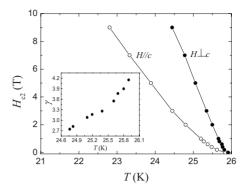


FIG. 4. The temperature dependence of the upper critical field H_{c2} for the (Ba,K)Fe₂As₂ single crystal. The open and solid symbols represent H_{c2} parallel and perpendicular to the c axis, respectively. Inset: temperature dependence of anisotropy ratio γ determined from the upper critical fields.

ratio γ near T_c of 4.2 shown in Fig. 4 is comparable³¹ to recent results for (Ba, K)Fe₂As₂ of $\gamma \sim 3.5$ for a T_c =30 K sample¹⁹ and $\gamma \sim 2$ for a T_c =28 K sample.²⁵

The temperature dependence of γ calculated with our data up to 9 T decreases from 4 to 2.7 as T decreases from T_c = 26.8 to 24.8 K, as summarized in the inset of Fig. 4. This characteristic decrease in γ from \sim 4 to lower values with the temperature decrease from T_c to lower temperature is consistent with the γ behaviors found by several groups in the FeAs-122 systems with $T_c \approx$ 30 K.^{17-19,25}

To gain further insight into the vortex-glass physics in our underdoped FeAs-122 single crystal, we measured the E-J characteristics up to 9 T (Fig. 5). The E-J curves show very similar features to those of YBa₂Cu₃O₇ single crystals and thin films²² and MgB₂ thin film²⁴ around T_g . According to the VG theory,²¹ the I-V curves in a 3D system show positive curvature for T> T_g , negative curvature for T< T_g , and a power-law behavior at T_g . As shown in Fig. 5, we found that all of these features around T_g exist in our I-V results at H=9 T with T_g =20.7 K with the proviso that only the low-dissipation regime (E<4 \times 10⁻⁴ V/cm) data are considered. As seen in high- T_c work (see, e.g., Ref. 32), systems quite often show a linear log E vs log E1 (power-law) behavior only below a certain threshold, and this is the case for our data in

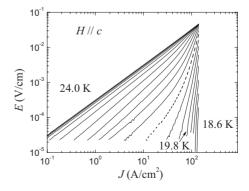


FIG. 5. *E-J* characteristics in the range T=18.6-24.0 K with a 0.3 K step under H=9 T. The dashed line indicates $T_g=20.7$ K. The negative curvature $\log E$ vs $\log J$ data expected below T_g can be seen in our data starting at about 19.8 K (arrow).

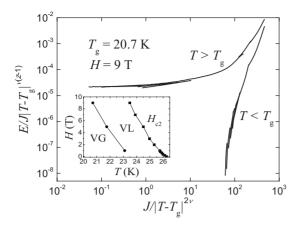


FIG. 6. The vortex-glass scaling behavior for the low-dissipation regime ($E < 4 \times 10^{-4} \text{ V/cm}$). The *I-V* curves collapse very nicely onto two scaling curves, one for above T_g and the other for below T_g , near the vortex-glass phase-transition temperature of 20.7 K. The inset shows the phase diagram revealing the VG to VL transition.

Fig. 5. Lower-dissipation regime data would extend the linear behavior seen at T_g in Fig. 5. Furthermore, the E-J curves in the low-dissipation regime are well described (Fig. 6) by a universal scaling function with two common variables, $E_{\rm sc} = E/J|T - T_g|^{\nu(z-1)}$ and $J_{\rm sc} = J/|T - T_g|^{2\nu}$, with $T_g = 20.7$ K and the critical exponents of $\nu = 1$ and z = 4.1. The critical exponents obtained from the E-J data are indeed in good agreement with the predicted values ($\nu \sim 1-2$ and z > 4) in the VG theory. When the other E-J curves at different magnetic fields (not shown) are analyzed, we found that they also follow the same scaling behaviors predicted by the VG theory, with the similar critical exponents $\nu = 1$ and $z = 4.1 \pm 0.2$.

It is important to note that in the VG theory, the resistance (see above) should vanish as $(T-T_g)^{\nu(z-1)}$. Thus, Fig. 7 shows such a plot where indeed the ohmic resistivity vanishes with the appropriate slope at T_g . The predicted slope of the vortex-glass theory $[1/\nu(z-1)]$ results in 0.32, which is close to the slope of 0.42 ± 0.20 in Fig. 7 within error bars. A further check of the validity of our scaling analysis is whether the glass correlation length (see Ref. 33) calculated from our data is with the 3D regime. We arrive at a value of

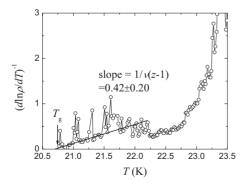


FIG. 7. A plot of $[d(\ln \rho)/dT]^{-1}$ vs temperature T shows an intercept near T_g determined from the scaling (Fig. 6), with a slope consistent with the scaling-determined exponents as discussed in the text.

965 Å, certainly large enough to indicate three-dimensional behavior. Therefore, these results clearly show that a vortex liquid-glass phase transition exists in our underdoped FeAs-122 crystal, clear evidence of such a vortex liquid-glass transition in the FeAs-122 system.

The inset of Fig. 6 summarizes the vortex phase diagram showing T_g variation with H as obtained from the scaling analyses (not shown) in each field. As discussed above when considering the magnetization data, the 20 K magnetic hysteresis curve can be extrapolated to meet at about 10 T; this irreversibility point is consistent with the vortex liquid-glass transition line shown in the inset of Fig. 6. This inset also includes the low-field H_{c2} curve along $H \| c$. The observation of the vortex-glass phase implies that the FeAs-122 single crystals have strong flux-pinning centers. A priori, either intrinsic or extrinsic defects could be the source for the flux pinning in the type-II superconductors. As for intrinsic defects, the FeAs-122 single crystal could have a structural deficiency, as in the case of the cuprate superconductors forming the collective pinning centers due to oxygen vacancy.^{21,22} As for extrinsic defects, the FeAs-122 sample might have small normal cores caused by inclusions of Sn flux (see, e.g., Ref. 18) or external ion impurities that can naturally come into the sample during the flux growth. 18 However, the observation discussed above of the second peak effect in M vs H [Fig. 2(a)], irrespective of the fluxgrowth method, suggests that the vortex pinning mechanism in the FeAs-122 single crystals might come from intrinsic defects rather than an extrinsic one such as the Sn flux.

In a recent comparative study of polycrystalline specimens of NdFeAsO_{0.85} and NdFeAsO_{0.85}F_{0.15}, ³⁴ the former has shown a clear vortex-glass scaling similar to the observation made here while the latter did not show any scaling behavior. This observation also supports the idea that intrinsic defects such as oxygen vacancies can play a role in creating the vortex liquid-glass transition in the FeAs-based superconductors.

In this regard, more systematic research is necessary to understand the origin of the vortex liquid-glass transition reported here. For example, synthesis of FeAs-122 superconducting single crystals with systematic control of intrinsic defect levels and studies of the vortex liquid-glass transition would be quite useful.

IV. CONCLUSIONS

We have measured magnetization, magnetotransport, and I-V characteristics in the underdoped FeAs-122 single crystal with T_c =26.2 K to study its superconducting properties upper critical fields, and to investigate the possibility of a vortex-glass phase. Analyzing the data using the 3D vortex-glass phase-transition theory, we found scaling of the I-V curves in each magnetic field up to 9 T, with critical exponents of ν =1 and z=4.1, constituting evidence of a vortex-glass to vortex-liquid transition in the (Ba,K)Fe₂As₂ single crystal. The decrease in the upper critical-field anisotropy was found to exist with decrease in temperature from T_c to 24.8 K, consistent with the observation made in other FeAs-122 crystals with different T_c values.

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