

Thermodynamic Evidence for the Fulde-Ferrell-Larkin-Ovchinnikov State in the KFe_2As_2 Superconductor

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We investigate the magnetic phase diagram near the upper critical field of KFe_2As_2 by magnetic torque and specific heat experiments using a high-resolution piezorotary positioner to precisely control the parallel alignment of the magnetic field with respect to the FeAs layers. We observe a clear double transition when the field is strictly aligned in the plane and a characteristic upturn of the upper critical field line, which goes far beyond the Pauli limit at 4.8 T. This provides firm evidence that a Fulde-Ferrell-Larkin-Ovchinnikov state exists in this iron-based KFe_2As_2 superconductor.

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While the upper critical field (H_{c2}) of type-II superconductors is usually determined by the orbital limit for superconductivity [1], there are rare cases in which the Pauli paramagnetic limit, at which the Zeeman-split Fermi surfaces suppress Cooper pairing, occurs instead [2,3]. When a superconductor approaches the Pauli limit, an exotic superconducting state was predicted by Fulde, Ferrell, Larkin, and Ovchinnikov (FFLO) [4,5]. Under certain conditions, a superconductor could overcome the Pauli limitation by forming the FFLO state in which the Cooper pairs have a finite center-of-mass momentum. Cooper pairing between the Zeeman-split Fermi surfaces is possible only with an oscillating component of the order parameter amplitude in the real space. Only a few observations of the FFLO state have been reported to date despite its high technological relevance for high magnetic field applications. The Q phase in the CeCoIn_5 heavy-fermion superconductor is regarded as a possible realization [6–8], although superconductivity coexists with an incommensurate spin-density wave [8]. In some layered organic superconductors, evidence has been provided for an FFLO state without magnetic order [9–19]. The rarity of the FFLO state is due to the strict requirements on its occurrence. It needs a very large Ginzburg-Landau parameter $\kappa \equiv \lambda/\xi \gg 1$ (λ , penetration depth; ξ , coherence length) and a Maki parameter α greater than 1.85 [20,21]. Only in this case is the Pauli limit below the orbital limit. Low dimensionality and anisotropic Fermi surfaces can further stabilize the FFLO state [22]. The H_{c2} line in the magnetic (H - T) phase diagram shows a characteristic saturation at the Pauli limit, where the second-order transition (SOT) changes into a discontinuous first-order transition (FOT). However, as soon as an FFLO state is formed, a characteristic increase of H_{c2} occurs to fields beyond the Pauli limit.

KFe_2As_2 is the overdoped end member of the $\text{Ba}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$ family of the multiband Fe-based superconductors with T_c of ~ 3.4 K. In recent magnetostriction

experiments, it was found that H_{c2} is Pauli limited for fields applied parallel to the FeAs layers [23,24]. However, no sign of an FFLO state has so far been reported. One possible reason may be that its observation requires high precision in the parallel field orientation [11]. Any finite perpendicular field component can produce the formation of a vortex state and suppress the FFLO state. KFe_2As_2 is a superconductor in the very clean limit with $l/\xi \sim 15$ and a large Maki parameter of 1.7–3.4 [23,24] and with the possibility of a nodal d -wave order parameter [25]. In this Letter, we provide thermodynamic evidence for an FFLO state in KFe_2As_2 by investigating its H - T phase diagram close to the Pauli limit by magnetic torque (τ) and specific heat (C_p) experiments. Only for a precise field alignment in the plane do we observe a characteristic upturn of the H_{c2} line beyond the Pauli limit, as well as the double transition from the homogeneous superconducting state via the FFLO state to the normal state.

The experiments were performed on a KFe_2As_2 single crystal. Details of sample growth and characterization can be found elsewhere [23,24]. The thin foil-like sample was flattened carefully between two glass plates. All experiments were carried out on an Attocube ANR51 piezorotary stepper positioner, which offers a millidegree fine positioning resolution. The rotator was mounted on a ^3He probe in a 15 T magnet cryostat. The torque was measured using a homemade capacitance torque magnetometer with a General Radio 1615A capacitance bridge in combination with a SR830 digital lock-in amplifier. The specific heat was measured with a modulated-temperature ac calorimeter [26] using an SR830 digital lock-in amplifier. Since the transition into the FFLO state often occurs as a nearly horizontal line in the H - T phase diagram, we have carried out all experiments at a fixed base temperature as a function of the field, while the temperature was periodically modulated with a millidegrees-Kelvin amplitude. The calorimeter chip is suspended on a pair of thin nylon wires that

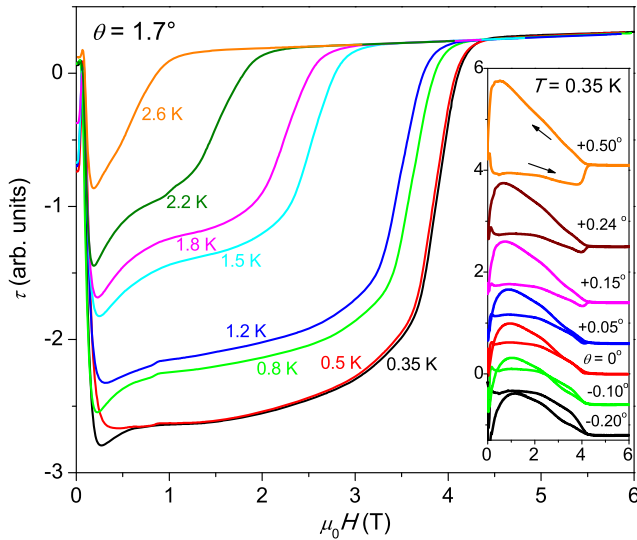


FIG. 1. The magnetic torque as a function of the applied magnetic field of KFe_2As_2 , measured at fixed temperatures. The field was aligned with a small angle of $\theta = 1.7^\circ$ to the FeAs layers. The data were measured with an increasing field. Inset: Magnetic torque at various angles close to $\theta = 0^\circ$. The linear normal state background was removed here for clarity.

provide firm support at a low temperature to avoid torque artifacts. Further details on the experimental methods and a basic characterization of the sample quality in zero field can be found in Supplemental Material [27].

In Fig. 1, we show τ data measured at various fixed temperatures as a function of the field with a small $\theta = 1.7^\circ$ misalignment of the field with respect to the FeAs layers. The torque shows the characteristic change from a kinklike SOT at H_{c2} (2.6 K) to a jumplike FOT at lower temperatures. The data at 0.5 and 0.35 K are nearly identical, indicating that H_{c2} saturates at low temperatures at a constant field value. This is in contradiction to the expected behavior of an orbital limited H_{c2} , for which $H_{c2}(T)$ would continue to increase monotonically with a decreasing temperature, and a clear sign that H_{c2} becomes Pauli limited. The behavior is similar to magnetostriction data reported previously [24]. Note that the large jump in the torque at the FOT results mostly from an abrupt decay of irreversible screening currents when approaching the Pauli limit but may occur at somewhat lower fields than the true H_{c2} in a purely thermodynamic quantity. H_{c2} should be determined by the first deviation from the normal state background, which may be represented by a smaller reversible component. No evidence for an FFLO state is observed here.

In the next step, we aligned the FeAs layers precisely parallel to the field. For this case, the torque would be expected to disappear completely and then change sign as the sample is further rotated. However, during field sweeps, there is always some hysteresis, as shown in the inset in Fig. 1 for 0.35 K data at different orientations. For this

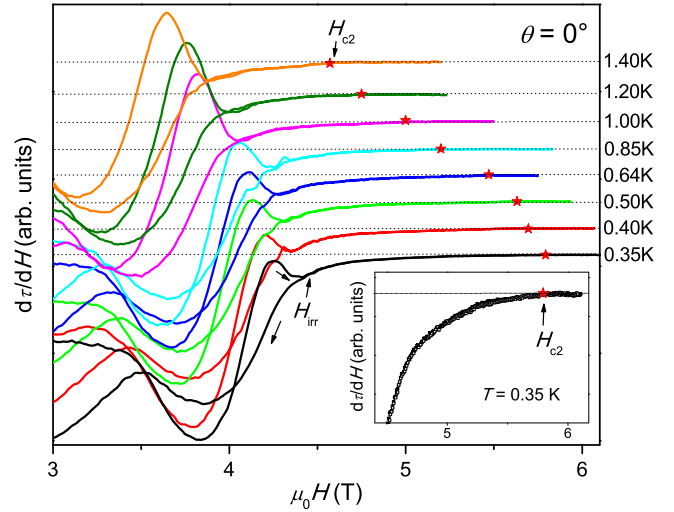


FIG. 2. The first-order derivative of the magnetic torque as a function of the applied field at different temperatures for the field strictly parallel to the FeAs layers ($\theta = 0^\circ$). The stars mark the upper onset of the superconducting signal and the dotted lines the constant normal state value. Vertical offsets have been added to the data. The inset shows an enlarged region of the $T = 0.35$ K data at H_{c2} .

reason, we field cooled the sample to a point slightly below the upper critical field at 0.35 K and minimized the torque by gradually rotating the sample in small increments to find the $\theta = 0^\circ$ orientation.

The normal state background in high fields is completely linear. At $\theta = 0^\circ$ there remains a slight deviation from the linear background up to 5.8 T, as will be demonstrated by the first field derivative of the torque at $\theta = 0^\circ$ in Fig. 2. Here, we define H_{c2} from a deviation point from the constant normal state value (dotted lines) as shown in the inset. The main change in τ initiates at a characteristic irreversibility field (H_{irr}), below which the ascending and descending branches deviate. However, a reversible superconducting contribution in the form of a gradual downturn is observed above H_{irr} up to 5.8 T at 0.35 K. Note that the torque is directly related to the anisotropic component of the magnetization, which in the absence of irreversibility is a bulk thermodynamic probe. The onset corresponds to the start of Cooper pairing and thus best represents H_{c2} . Additional data showing τ measured at three different angles can be found in Supplemental Material [27]. H_{c2} is increased from 5 to 5.8 T when the angle varies from 1.7° to 0° (Supplemental Fig. S1 [27]). This may indicate that an FFLO state associated with this reversible tail in torque goes far beyond the Pauli limit [24] but develops only for accurate parallel field orientation, while for $\theta > 1^\circ$ it disappears and the superconductor remains Pauli limited.

In order to further test the possible existence of an FFLO state, we shall now investigate the specific heat in the vicinity of H_{c2} . Figure 3(a) shows the field dependence of

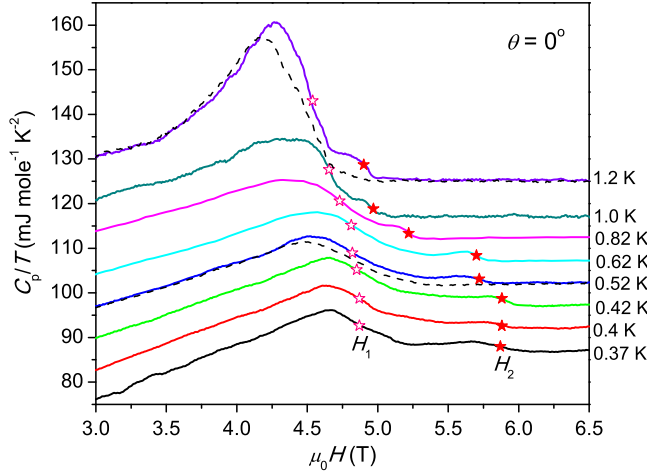


FIG. 3. Specific heat C_p/T measured as a function of the magnetic field aligned with $\theta = 0^\circ$ with respect to the FeAs layers at different fixed temperatures. Solid stars mark the additional phase transitions at H_2 above the main transitions at H_1 (empty stars). Vertical offsets were added for clarity. The additional dashed lines were measured under identical conditions at 0.52 and 1.2 K but with a misalignment angle of $\theta = 3^\circ$ instead.

C_p/T at $\theta = 0^\circ$ at various fixed temperatures. The 1.2 K data show a main transition at characteristic fields H_1 in the form of a broad triangular peak at 4.3 T. At lower temperatures, it is transformed into a broad steplike anomaly with a midpoint which increases to 4.8 T at 0.37 K. In all data, another small steplike phase transition anomaly occurs above the main transition and reaches a field $H_2 (= H_{c2}) = 5.8$ T at 0.37 K. The characteristic fields H_2 agree well with the onset of the superconducting τ contribution. We added two sets of data at 0.52 and 1.2 K in which we had a misalignment angle $\theta = 3^\circ$ for comparison. The 3° data show only one transition, and the additional step at H_2 is completely absent. A pronounced double transition develops only for a precisely parallel field alignment. The transition at H_1 corresponds to H_{irr} in τ and initiates the rapid increase in the torque magnitude at lower fields. Note that it is difficult to judge the phase transition order without careful consideration. C_p data as a function of the field are rarely shown and should not be confused with the magnetocaloric effect [26,28]. The latter represents the calorimetric response with respect to a field change, while C_p is measured in response to a temperature modulation during a field sweep. Both transitions at H_1 and at H_2 appear, at first sight, as a SOT in the form of a characteristic jump. However, the transition at H_1 sharpens at 1.2 K and evolves into a more symmetrical shape, which is probably the signature of a FOT. The absence of latent heat could be a result of the low temperature and the vanishing slope of the phase boundaries, which make the entropy differences between the phases disappear, or due to a limitation of the ac technique, which is known to underestimate latent heats [29].

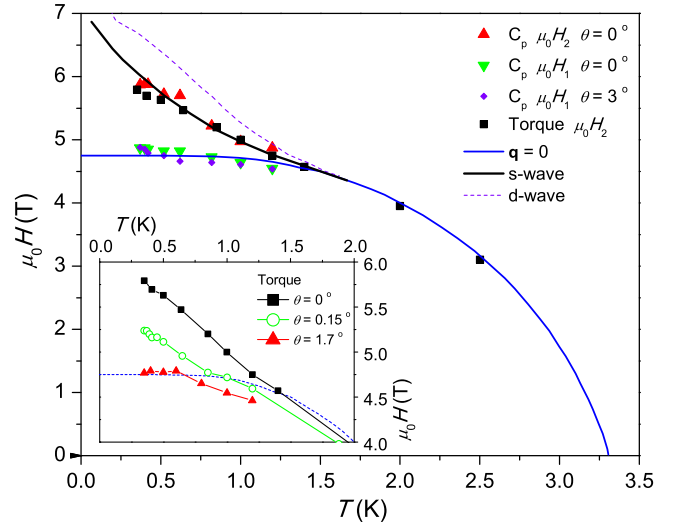


FIG. 4. H - T phase diagram of KFe_2As_2 from magnetic torque and specific heat. $\mu_0 H_1$ represents the transition between the homogeneous superconducting state ($\mathbf{q} = 0$) and the FFLO state. The upper transition at $\mu_0 H_2$ separates the FFLO state from the normal state. The additional lines represent the $H_{c2}(T)$ lines predicted for a purely Pauli-limited superconductor ($\mathbf{q} = 0$) [30] and for an in-plane isotropic s -wave and a d -wave superconductor with the FFLO state [31]. The inset shows an enlargement with the upper onset of the superconducting contribution to torque for different field orientations (0° , 0.15° , and 1.7°).

In Fig. 4, we compile an H - T phase diagram from our τ and C_p data showing the presence of an additional superconducting phase below ~ 1.5 K in high magnetic fields above the Pauli limit, with the characteristics of an FFLO state. In the following, we compare this phase diagram with theoretical predictions for the FFLO state. There are numerous theoretical descriptions of the H_{c2} transition with orbital and Zeeman pair breaking effects in clean superconductors [30–33]. Gurevich provided a recent model of the H_{c2} transition in anisotropic multiband superconductors [30], and we include his H_{c2} prediction for the purely Pauli-limited case without the FFLO state in Fig. 4. It agrees well with our C_p data in which we had a 3° field misalignment, which appears to be purely Pauli limited and thus approaches a constant field value of $H_1 = 4.8$ T below ~ 1.5 K, without forming the FFLO state in higher fields. Meanwhile, for the perfect parallel orientation, both the τ and C_p data first follow the line of the Pauli-limited case above ~ 1.5 K, but then the H_2 line gradually turns up to higher fields. In addition, the characteristic double transition in C_p is observed as a signature of the transition sequence from the ordinary superconducting phase to an FFLO state at H_1 and from an FFLO state to the normal conducting state at H_2 . The main C_p transition at H_1 remains at the Pauli limiting field, which therefore initiates the field-driven transition into the FFLO state. For a perfect alignment of the field parallel to the

FeAs planes, the phase diagram thus shows all the features expected for the formation of the FFLO state, with its boundary being represented by the lines marked as H_1 and H_2 as the lower and upper field boundary, respectively, while the FFLO phase completely vanishes for small field misalignments.

The H_{c2} transition with the FFLO state for mixtures of s - and d -wave superconductors has been predicted in Ref. [31]. An s -wave superconductor, which is isotropic in the plane, should exhibit a pronounced convex H_{c2} upturn at low temperatures, and this model fits best our data. The H - T phase diagram thus appears identical to that expected for a simple s -wave superconductor and does not reflect the unconventional multiband structure of Fe-based superconductors. This may be due to the fact that, in KFe_2As_2 , the electronic band structure is simpler than in most other iron-based superconductors and contains only four α , β , ζ , and ε hole bands [34]. At the lowest temperatures, however, our data slightly deviate from the fit with a saturating concave trend. The d -wave model describes such a change in the curvature well, but the corresponding fit in Fig. 4 results in an FFLO phase greater than that which we observe. A mixture of s - and d -wave order parameter symmetry would cause an even larger FFLO phase [31]. On the other hand, a slightly saturating behavior of the H_2 line was also predicted for anisotropic single-band and for multiband s -wave order parameters [30]. In this case, however, the predicted FFLO phases are much smaller than what we observe here. However, the enlarging effect of a nodal d -wave order parameter symmetry in the FFLO phase may eventually be canceled out by a downsizing effect of the multiband nature of superconductivity and thus, by chance, mimic the H_2 transition line of a simple s -wave superconductor with the FFLO state.

The effect of multiband superconductivity with finite intraband pairing interaction on an FFLO state in KFe_2As_2 was predicted to cause a separation of the FFLO phase into a Q_1 and a Q_2 phase characterized by different order parameter modulation vectors [32]. While we do not see evidence of such an additional phase transition within the FFLO phase, it is possible that it may occur at an even lower temperature which was not accessible in our cryostat.

The H - T phase diagram of KFe_2As_2 (Fig. 4) is very similar to κ -(BEDT-TTF) $_2\text{Cu}(\text{NCS})_2$ [11] and β' -(BEDT-TTF) $_2\text{SF}_5\text{CH}_2\text{CF}_2\text{SO}_3$ [12,13], although the experimental features are quite different. In these layered organic superconductors, there is only a small τ [11] and C_p [12,28] anomaly at H_1 , while the greatest change occurs at H_2 where the transition into the normal state takes place. This is different in KFe_2As_2 , for which the main transition anomaly in C_p occurs at H_1 , with only a small superconducting contribution of $\sim 15\%$ of the total C_p jump persisting up to H_2 . This small contribution could indicate that the FFLO state forms only in one of the four bands [35]. The gap in the ε band is much greater than the others [36],

and, while the smaller gaps are largely suppressed in high fields, only the ε band would remain gapped up to H_{c2} and thus dictate the characteristics of the FFLO state.

The transition into the FFLO state occurs at 4.8 T, while the theoretical weak-coupling paramagnetic limit $H_p(0) = 1.84T_c \approx 6$ T is slightly higher. A more precise estimation is obtained from $H_p(0) = \Delta_0/(\sqrt{2}\mu_B) \sim 7.4$ T, with $\Delta_0/k_B T_c \approx 1.9$ [36]. These estimations likely overestimate the Pauli limit because of the large normal-state Pauli susceptibility of KFe_2As_2 [37]. Using $H_p(0) = H_c(0)/(\sqrt{\chi_n - \chi_s}) \sim 7.4$ [38] (χ_n and χ_s are the normal-state and superconducting spin susceptibilities) yields a value of $H_p(0) = 3.6$ T [23], which may be enhanced by the multiband superconductivity to the observed value of 4.8 T.

Finally, it should be noted that, even with $\text{Ba}_{0.07}\text{K}_{0.93}\text{Fe}_2\text{As}_2$, a FOT with a certain upturn of the H_{c2} line was observed, although the latter was interpreted differently [39]. Furthermore, an unusual field-induced superconducting phase has been reported in FeSe [40]. It would therefore be interesting to carry out similar experiments on other Fe-based superconductors.

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