Critical field and Shubnikov-de Haas oscillations of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ under pressure

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A tuned tank circuit in combination with a nonmetallic diamond anvil cell has been successfully used to measure the change in critical field with angle in κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ at pressures up to 1.75 kbar and at temperatures down to 70 mK. The critical field has been found to decrease by more than 90% within less than 2 kbar and at a much higher rate for the field applied parallel to the conducting planes. For this orientation, at 1.75 kbar, we have seen a clear change from the ambient pressure behavior of the critical field with temperature at low temperatures. Up to $P = 1.75$ kbar, the $H_{c2}(\theta)$ phase diagram is in good agreement with the theoretical prediction for weakly coupled layered superconductors. We have also succeeded in measuring oscillations in the resistivity of the normal state at higher magnetic field. The $\alpha$-orbit Shubnikov-de Haas frequency was found to increase at a rate of 44 T/kbar. Our experiment opens the possibility for further investigations of the effective mass with pressure, especially because the setup is suitable for pulse fields as well.

I. INTRODUCTION

Due to its strong two-dimensional character, the charge-transfer organic salt κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ is a suitable material for studying the various theories put forth for anisotropic superconductivity in magnetic fields. The electronic structure of organic superconductors is very similar to that of the cuprate high $T_c$ superconductors, consisting of stacks of alternating conducting and insulating sheets. In contrast to the cuprates, however, the critical fields of organic superconductors are much lower making them easier to study. Among the organics, κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ with a $T_c = 10.4K$, has been shown to be one of the compounds with the highest critical fields. Even with its conducting planes parallel to the applied field, $H_{c2}$ is less than 40 tesla (T)\(\uparrow\). Furthermore, high-purity single crystals of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ are available which make for reliable studies of the Fermi surface. For example, the samples used in this study have mean free paths from 600-900 Å, and a superconducting coherence length in the layers of $\sim 100$ Å. These parameters put κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ clearly in the clean limit. YBCO in comparison has a mean free path less than 100 Å and a superconducting coherence length in the layers of $\sim 50$ Å\(\uparrow\).

The strong effect of the pressure on the band structure via modification of the carrier effective mass and Fermi surface (and hence on the superconducting properties) already reported in the literature\(\uparrow\) has motivated our work. Our innovation was to combine a nonmetallic diamond anvil cell (DAC)\(\uparrow\) with an rf penetration depth technique\(\uparrow\). The plastic pressure cell design overcomes the difficulties of using metals in magnetic field, can be made of a relatively small size to fit on a rotating platform and, by placing a ruby chip inside the cell, the pressure can be measured in-situ. The penetration depth was measured using the TDO technique, which offers the

![FIG. 1: The rf penetration (proportional to the change in frequency) as a function of magnetic field for the orientation perpendicular to the conducting planes (a) and the parallel orientation (b). $H_{c2}$ is determined by the intersecting point between the linear extrapolation of the normal state and the superconducting transition.](image-url)
advantage of not requiring contacts on the sample, and therefore, eliminates problems like contact resistance and additional stress on the sample. It is particularly well suited for use in the diamond anvil cell because the coil and the sample can be of arbitrarily small size. In a recent advance, we have succeeded in using this combination of techniques in the pulsed field environment to 50 T at He-3 temperatures.

In the present paper, we focus on the change in critical field under pressure for different orientations of the applied dc magnetic field with respect to the conducting planes (we will refer to $\theta$ as the angle between the magnetic field and the normal to the conducting planes). The study of reduced dimensional systems is important, because of the different mechanisms which destroy the superconductivity when the magnetic field is applied perpendicular or parallel to the conducting layers. In the perpendicular orientation, or whenever the magnetic field has a nonzero component in this direction, the upper critical field is determined by orbital magnetic effects (through the in-plane kinetic energy of electrons). However, when the applied magnetic field is parallel or very close to the parallel direction, the orbital effects can be suppressed, because the vortices can fit outside the conducting planes. In this case, the spin-magnetic field interaction may become important. In the absence of any other mechanisms (spin-orbit scattering, many body effects), the maximum critical field in the parallel orientation, called the BCS Pauli paramagnetic limit, $H_{BCS}^P$, is driven by the spin polarization effect, where the condensation energy is overcome by the Zeeman splitting energy.\textsuperscript{5,9} For a comprehensive summary of organic superconductors in the Pauli paramagnetic limit region we refer the reader to Ref.\textsuperscript{12}.

In particular, $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ has been found to have an unusual evolution of the parallel critical field with temperature. In spite of their differences in the measured values of the critical field and in the curvature of the phase diagram at higher temperatures, all the experimental results at ambient pressure agree that the absolute value of $H_{c2}^\parallel$ not only exceeds $H_{BCS}^P$, but it also shows no tendency of saturation at low temperatures\textsuperscript{10,14,15} and displays positive curvature toward 0 K. However, the reason for this behavior is not well understood. We cite two recent hypotheses, one that explains the lack of saturation as a first order phase transition into the FFLO state\textsuperscript{22} and another one that claims the high critical fields are indeed beyond the BCS Pauli paramagnetic limit, but comparable to the paramagnetic limit calculated from thermodynamic quantities\textsuperscript{18}. Both references seem to ignore the spin-orbital scattering effect, which also can be responsible for the enhancement of the upper critical field. Moreover, in the absence of spin-orbital scattering, the transition from normal to superconducting state at low temperatures should turn into a first-order phase transition.\textsuperscript{14} Although we do not fully agree with the analysis of Ref.\textsuperscript{12}, we independently agree that there is a first order transition below 4 K in $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$. Even if this first order transition were not there, we suggest that spin-orbit scattering cannot be responsible for the total enhancement of the critical field. If this was the case, following Ref.\textsuperscript{12} we calculated that the spin-orbit scattering time would be between 0.46 and 0.62 ps, which is much less than the total measured scattering time of about 3 ps determined from magnetoresistance oscillations.\textsuperscript{13,14} It is thus valuable to study the effect of the pressure on $H_{c2}^\parallel(T)$ phase diagram, especially because existing data suggests a tendency of saturation at 1.5 kbar.\textsuperscript{13,14}

At ambient pressure, it is often claimed that $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ is very anisotropic. Although the anisotropy ratio $\gamma = H_{c2}^\parallel/H_{c2}^\perp$ is only about 6\textsuperscript{10} the anisotropy of the London penetration depths is $\approx 160-330$.\textsuperscript{16,17} The reason why the anisotropy determined by the critical fields is misleading is that the mechanism that limits the superconductivity when the applied field is parallel to the layers is not related to the coherence length, because this critical field is Pauli limited. Hence, the parallel and perpendicular critical fields cannot be used to find the ratio of the parallel and perpendicular coherence lengths, as is common with other anisotropic superconductors. Nevertheless, the $H_{c2}^{\theta}$ diagram for $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ fits the Lawrence-Doniach 2D model of weakly coupled layered superconductors despite the Pauli limiting.\textsuperscript{10,15} And, as we will show, even under moderate pressure we never see results completely consistent with anisotropic 3D Ginzburg-Landau theory.\textsuperscript{18}

Thus, we hope to discover if the effect of pressure will eventually induce a transition from one type of superconductor (2D) to another (3D).

**II. EXPERIMENTAL**

The samples were single crystals of $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ approximately $210 \times 175 \times 40 \ \mu m^3$. They were placed in a four turn coil (56 AWG wire) with an inner diameter of 300 $\mu$m with their conducting planes perpendicular the axis of the coil. To minimize the background signal, a nonmetallic diamond anvil cell was used with a diamond filled epoxy gasket reinforced by a Zylon overband.\textsuperscript{20} The plastic DAC freely rotated in a top-loading dilution refrigerator with an ID of 21.5 mm. The coil rested in the 350 $\mu$m diameter hole of the gasket that was filled with the quasi-hydrostatic pressure medium glycerin. Ruby was used to calibrate the pressure at the operating temperature.\textsuperscript{21} The TDO setup has been explained in detail elsewhere.\textsuperscript{8} The oscillating frequency of the circuit at 70 mK was 290 MHz, and the change in frequency during the sweep of the magnetic field was about 2 MHz, less than 1% percent. Fig.\textsuperscript{4} shows typical field dependences of the frequency and amplitude when the field is applied parallel and perpendicular to the conducting layers. The overlap of the inverse ampli-
tude, which is a direct measure of the dissipation in the circuit, and the frequency, attests to the integrity of the data. We define the critical field as the intersection point between the linear extrapolation of the normal state and the superconducting transition. The data reported in the present work were taken in a top loading dilution refrigerator and an 18 T superconducting magnet system at NHMFL in Tallahassee. The present configuration of the TDO electronics limits the lowest achievable temperature to 70 mK.

III. RESULTS

Fig. 2 shows the critical field, both parallel and perpendicular, for ambient pressure and three other values: 1.5, 1.67, and 1.75 kbar at T=90 mK. The critical fields in the different orientations decrease linearly as the pressure increases. However, the rates of change are very different for the two orientations. We found \( \frac{dH_{c2}^{\perp}}{dP} \approx -2.8 \) T/kbar\(^{-1} \) whereas \( \frac{dH_{c2}^{\parallel}}{dP} \approx -14.75 \) T/kbar\(^{-1} \). Extrapolating the fitting lines, we found a critical pressure \( P_c \) of about 1.8 kbar for \( H_{c2}^{\perp} \) and 2.1 kbar for \( H_{c2}^{\parallel} \). The perpendicular value is less than half of the value reported in Ref. 11 where \( P_c \approx 5 \) kbar. It is possible that beyond 1.75 kbar the variation of \( H_{c2} \) with pressure is strongly nonlinear which would make our estimations invalid, although we doubt this is the case. The suppression of superconductivity by more than 90% within less 1.5 kbar underlines the importance of a careful study of the effective mass under pressure. However, the very high linear rate of change of the parallel critical field with pressure is also striking, because while the change in the effective mass directly influences orbital effects, \( H_{c2}^{\parallel} \) is not orbital limited. At this point, we only question the conclusion of Ref. 4 that the enhancement of the effective

mass is directly associated with superconductivity in \( \kappa \)- (BEDT-TTF)\(_2\)Cu(NCS)\(_2\) and suggest that other parameters, such as the \( V_{BCS} \) interaction term (the electron-phonon coupling matrix element)\(^{22} \), the density of states and/or the phonon characteristic energy may be very sensitive to the applied pressure.

We measured the change in critical field with temperature at 1.75 kbar, both in the perpendicular and parallel orientation (Fig. 3). Although both diagrams show a saturation of the critical field at very low temperature, it may not happen for the same reason as we will show later in the paper. In the perpendicular orientation, \( \kappa \)- (BEDT-TTF)\(_2\)Cu(NCS)\(_2\) is orbitally limited at ambient pressure, the orbital critical field(\( \approx 5 T \)) being well below the Pauli limit (\( \approx 18 T \)), and we found the same situation at 1.75 kbar. As can be seen in Fig. 3(a), our experimental data falls nicely on the theoretical Ginzburg-Landau result, \( H_{c2} \approx (1 - (T/T_c)^2) \) albeit for only the lower half of the temperature range. A fit and extrapolation yields \( T_c \approx 1.75 K \pm 0.5 K \).

As mentioned in the introduction, ambient pressure studies show that \( H_{c2}^{\parallel} \) exceeds the Pauli paramagnetic limit \( H_{BCS}^{P} \), and shows no tendency of saturation as \( T \to 0 K \). In contrast, at \( P = 1.75 \) kbar we found no change

FIG. 2: Pressure dependence of parallel (circles) and perpendicular (triangles) critical field. The error is approximately the size of the symbols. The two ambient pressure points come from Ref. 11.

FIG. 3: Critical fields [(a)-\( H_{c2}^{\perp} \) and (b)-\( H_{c2}^{\parallel} \)] as a function of temperature at \( P = 1.75 \) kbar. The continuous line in (a) is the Ginzburg-Landau equation of the critical field at low temperature: \( H_{c2} = \text{Const.} \times (1 - (T/T_c)^2) \).
in the parallel critical field as the temperature increases from 70 mK to 240 mK. Above 240 mK it drops with a negative curvature (Fig. 3). Studying how the ratio between the measured $H_{c2}^\parallel$ and the BCS Pauli limit change under pressure, we had to sort out the very different values obtained for the highest $H_{c2}^\perp$ at ambient pressure. Using the upper critical field between 30 T and 35 T, the ratio $H_{\text{meas}}/H_{\text{BCS}}^\perp$ is between 1.67 and 1.91. At 1.75 kbar, if we conservatively estimate $T_c$ to be 2.00 K (from the perpendicular diagram), then $H_{\text{BCS}}^\perp$ would be equal to 3.7 T and the ratio $H_{\text{meas}}/H_{\text{BCS}}^\perp$ would be about 1.47. These numbers suggest that the effect of the pressure is to bring the parallel critical field toward the Pauli limit. However, TDO measurements performed on two different sets of samples, the one used in the present experiment and another identical to those used in Ref. 16, led to a maximum critical field of 24.4 T at 450 mK. To be consistent, we compare data obtained by the same method on the same set of samples. This data shows that the ratio $H_{\text{meas}}/H_{\text{BCS}}^\perp$ is 1.35 at ambient pressure, which is surprisingly less than the value of 1.47, found at 1.75 kbar.

Based on $H_{c2}$ studied in previous experiments, one could expect either an increase or decrease in the parallel critical field as $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ is subjected to pressure. If the conducting layers are decoupled and the layers are squeezed, the parallel critical field should increase as was found in single layers of aluminum. If the insulating layers are squeezed, the parallel critical field should decrease as the the orbital limiting is enhanced due to the increased coupling of the layers, and the increased perpendicular coherence length. It is unclear how these two phenomena will combine in a given situation, although both phenomena are tied to the energy gap and critical temperature through the density of superconducting electrons. All other parameters being equal $H_{c2}^\parallel$ and $H_{c2}^\perp$ will shift together. The fact that the ratio $H_{\text{meas}}/H_{\text{BCS}}^\parallel$ is higher at higher pressures suggests that the conducting layers are getting thinner faster than the insulating layers, and the material is getting closer to its real Pauli limit. It should also be mentioned that, up to 1.75 kbar and at temperatures down to 70 mK, we have seen no evidence for the FFLO phase, claimed to be present at ambient pressure, and the transition is always of second order (Fig. 1(b)).

Concluding the above discussion, to understand the mechanism of superconductivity in $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ it becomes very important to complete the ambient pressure phase diagram below 500 mK, but it is experimentally difficult to obtain magnetic fields higher than 30-35 T and temperatures below 500 mK at the same time. We have also shown that a very low pressure, probably less than 1 kbar, would make this experimental investigation much more facile.

However, one must always make sure that, under pressure, $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ does not suffer a transition from a layered quasi 2D superconductor toward an anisotropic 3D superconductor. If that were the case, then the orbital effects would no longer be negligible in parallel orientation, and could even become the dominant factor. In the case that the major effect of the pressure is to increase the coupling between layers, this should cause a transition from a Lawrence-Doniach type of superconductor to an anisotropic 3-D Ginzburg-Landau one. We can experimentally verify this change by measuring the change in the critical field with angle. The Ginzburg-Landau theory for an anisotropic 3-D superconductor predicts a variation of the critical with angle after the following equation:

$$\frac{(H_{c2}(\theta)\cos(\theta))^2}{H_{c2}^\parallel} + \frac{(H_{c2}(\theta)\sin(\theta))^2}{H_{c2}^\perp} = 1,$$

where $\theta$ is the angle between the field and the normal to the layers. For weakly coupled layered superconductors, Tinkham and Schneider and Schmidt found that the angular dependence is given by:

$$\frac{(H_{c2}(\theta)\cos(\theta))^2}{H_{c2}^\parallel} + \frac{(H_{c2}(\theta)\sin(\theta))^2}{H_{c2}^\perp} = 1,$$

which leads to a cusp-like behavior.

We have determined the $H_{c2}(\theta)$ diagram for P=1.67 and 1.75 kbar at T=70 mK. The experimental result along with the fits by Eq.(1) and Eq.(2), are plotted in Fig. 3. The cusp-like feature observed experimentally at $\theta=90^\circ$ is the indication that Eq.(2) is a better fit up to 1.75 kbar. Therefore, we confirm experimentally that up to P=1.75 kbar there is no evidence for moving toward a more 3-D (or less 2-D) superconductor. We also found an enhancement of the anisotropy in critical field $\gamma=H_{c2}^\parallel/2H_{c2}^\perp$ from $\approx 6$ at ambient pressure to $\gamma \approx 23$ at P=1.75 kbar, but we attribute this increase in the apparent anisotropy to the different mechanisms that affect the critical fields in each orientation rather than to an enhancement of the 2-D character of the $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$, as discussed in the introduction. In reference to our comment earlier that $H_{c2}^\parallel$ at 1.75 kbar is Pauli limited, the cusp like character of the angular dependence is further evidence that the orbital effects are suppressed when the sample is at the parallel orientation. If there was significant transport through the layers, the angular dependence would have a rounded top near 90°.

Beyond the superconducting transition, the change in frequency (and amplitude) of the TDO is due to the resistivity of the normal state, and at higher fields we have measured the Shubnikov-de Haas oscillations in magnetoresistance as shown in Fig. 4. Our limit of 18 tesla did not allow for a careful analysis of the oscillation frequency with pressure and temperature, but we found an increase in the frequency of $\alpha$-orbit ($F_o$) from 694.1 T at 1.5 kbar to 703.0 T at 1.67 kbar ($T=90$ mK), while the ambient value ($F_o$) is about 595 T. (At 1.75 kbar an experimental
FIG. 4: Angular dependence of $H_{c2}$ at 1.67 kbar (a), and 1.75 kbar (b). For both graphs, the continuous line represents a fit with Lawrence-Doniach equation and the dotted curve is a fit to the anisotropic 3D Ginzburg-Landau equation.

error prevented us from seeing the SdH oscillations.) The ratio $\frac{F_\alpha}{F_0}$ is therefore, 1.17 at 1.5 kbar and 1.18 at 1.67 kbar. The linear increase of the frequency of oscillations with pressure is due to the change in size of the unit cell and the inverse effect on the Brillouin zone.

IV. CONCLUSIONS

In summary, we have proven that the combination of the TDO technique and the nonmetallic pressure cell can provide a very useful tool in the study of superconductivity.

Measurements on $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ revealed that the pressure strongly affects the critical field, by more than 90% within 1.5 kbar, both in the perpendicular and parallel orientations. The superconductivity is suppressed at a much higher rate in the parallel direction, although in this case the orbital critical field, directly proportional to the effective mass, is not the limiting factor.

At 1.75 kbar, we found a clear change in the behavior of the parallel critical field with temperature, from the ambient pressure phase diagram. The value of $H_{c2}$ still exceeds the BCS Pauli limit, but the lack of consistent data at ambient pressure confuses the issue of how the ratio $H_{meas}/H_{BCS}^{2D}$ evolves with pressure. According to our experimental evidence, up to 1.75 kbar, $\kappa$-(BEDT-TTF)$_2$Cu(NCS)$_2$ is still well described by the 2-D Lawrence-Doniach model for layered superconductors.

The frequency of magnetoresistance oscillations increases with pressure at a higher rate than previously reported in literature Ref.\textsuperscript{4}. In an effort to better understand the role played by different physical quantities (e.g. the effective mass, $V_{BCS}$, transfer integral, spin-orbit scattering rate) we are pursuing larger fields, lower temperatures, and higher pressures. Exploring the very low pressure gap in our data would be very useful as well.

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