Magnetic-field-induced superconductivity and phase diagrams of λ-$(BETS)_2FeCl_{4-x}Br_x$

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Resistance measurements have been performed in two- dimensional organic alloys λ-$(BETS)_2FeCl_{4-x}Br_x$ (x=0.4, 0.5, and 0.7), where BETS is bis(ethylenedithio)tetraselenafulvalene, to investigate the effect of the strong correlation between the Fe 3d and BETS π electrons on the electronic states. The ground state at low fields is an insulating phase, which becomes more stable as x increases. In high magnetic fields along the c axis, superconductivity is induced for all the samples. The critical temperature T_c has a maximum at 31–33 T, showing that the internal field created by the Fe 3d moments is almost independent of x. The analysis of the phase diagrams based on Fischer theory shows that both the orbital critical field and the spin-orbit scattering rate are enhanced by the Br substitution. All of these features are consistently understood in terms of the anisotropic negative chemical pressure and disorder induced by the Br substitution.

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I. INTRODUCTION

The phase diagrams of the organic alloys, λ-$(BETS)_2Fe_xGa_{1-x}Cl_4$, where BETS is bis(ethylenedithio) tetraselenafulvalene, are good examples of low-dimensional magnetic conductors where rich ground state properties appear.[1–4] In the absence of the magnetic field, λ-$(BETS)_2FeCl_4$ shows a transition from a paramagnetic metal (PM) to an antiferromagnetic insulator (AFI) around 8 K.[5–7] The AFI phase for λ-$(BETS)_2FeCl_4$ is destabilized by the magnetic field of ~10 T, where at higher fields the PM phase is recovered. When the magnetic field is applied parallel to the conducting layers, a superconducting (S) phase is induced above 17 T below 1 K.[1–3] The critical temperature T_c has a maximum ~5 K at 31 T. This superconductivity is easily destroyed when the magnetic field is tilted from the conduction layers (ac planes). As x decreases in λ-$(BETS)_2Fe_xGa_{1-x}Cl_4$, the magnetic field induced S phase simply shifts to a lower field whereas the AFI phase shrinks. The ground state in zero magnetic field becomes superconducting for $x \leqslant 0.35$.[8] The appearance of these different phases is ascribed to the x dependence of the effective exchange interaction between the Fe 3d moments and the π electron spins on the BETS molecules.[4,9–11]

The overall features of the field-induced S phases in λ-$(BETS)_2Fe_xGa_{1-x}Cl_4$ (Refs. 4 and 12) are well understood by Fischer theory,[13] based on the Jaccarino-Peter (JP) effect.[14] In the PM phase of λ-$(BETS)_2FeCl_4$, the localized Fe moments are aligned along the external field (H). Because of a strong negative d-π exchange interaction J, the π electron spins experience a strong internal field ($-H_{int}$) created by the Fe 3d moments, whose direction is antiparallel to H. Therefore when both external and internal fields are compensated ($H=H_{int}$), the Zeeman effect, one of the mechanisms that destroys superconductivity, is completely suppressed. Moreover, when H is parallel to the conducing layers, the orbital effect, which is the other destructive mechanism of superconductivity, is also suppressed. Therefore superconductivity can be induced by parallel fields H of order $\sim H_{int}$.

When the field is tilted from the layers, the orbital effect breaks the superconductivity. As the Fe concentration x decreases, the average value of H_{int} decreases. This effect causes the field-induced S phase to shift to a lower field. Recently, a similar field-induced S phase has been observed in another BETS system, κ-$(BETS)_2FeBr_4$.[15,16]

In contrast to λ-$(BETS)_2Fe_xGa_{1-x}Cl_4$, the ground states of the alloys λ-$(BETS)_2FeCl_{4-x}Br_x$ remain insulating (Fig. 1).[17] For $x \leqslant 0.2$, the antiferromagnetic transition (T_N) of the Fe 3d moments and the metal-insulator (MI) transition (T_{MI}) take place almost simultaneously, showing that the Fe 3d moments and π electron spins are strongly coupled. However, in the range $0.3 < x < 0.5$, two successive magnetic transitions are observed: a small anomaly in the magnetization curves at T_{MI}, followed by the strong suppression of the magnetization at a lower temperature T_N which is assigned to the long range AF order of the Fe 3d moments. The differ-

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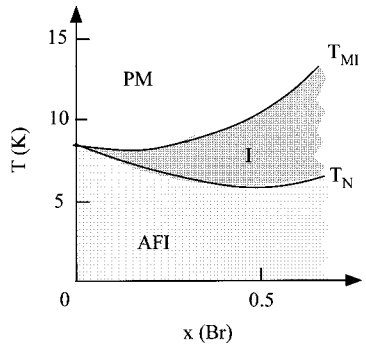

FIG. 1. Phase diagram of λ-$(BETS)_2FeCl_{4-x}Br_x$ determined by resistance and magnetization measurements (Ref. 17). PM: paramagnetic metal, I: insulator, and AFI: antiferromagnetic insulator.

ence between T_{MI} and T_N increases with increasing x.

For $x>0.6$, the anomaly in the magnetization at T_{MI} becomes less pronounced, whereas the AF order is clearly observed at T_N. Therefore the electronic state for $T_N<T<T_{MI}$ is expected to be an insulating (I) state with paramagnetic Fe moments. These features have been interpreted in terms of the reduction of the d-π coupling by the Br substitution.[17]

The AFI transition in λ-$(BETS)_2FeCl_4$ (i.e., for $x=0$) has been theoretically discussed in terms of a Mott transition induced by the antiferromagnetic order of the 3d moments, where the strong d-π coupling is crucial.[9,10] However, for $x\neq 0$, the presence of the I phase for $T_N<T<T_{MI}$ is not interpreted in this picture, but is a spin-Pierls-like transition.[17] Since the field-induced superconductivity strongly depends on the d-π coupling, it is of interest to investigate the high field electronic states in λ-$(BETS)_2FeCl_{4-x}Br_x$. In this paper, we report systematic investigation of λ-$(BETS)_2FeCl_{4-x}Br_x$ and discuss the effects of the Br substitution on the high field electronic states.

II. EXPERIMENT

Single crystals of λ-$(BETS)_2FeCl_{4-x}Br_x$ were synthesized electrochemically.[17] The resistance measurements were performed by a conventional *ac* technique with the electric current along the c axis in the hybrid magnet at NHMFL. The field-induced superconductivity is strongly suppressed by the field component perpendicular to the conducting layers. Therefore the samples were carefully aligned so that the magnetic field is parallel to the conducting *ac* plane within the accuracy of ~0.05°. In the *ac* plane, the magnetic field is parallel to within a few degrees of the c axis. The samples were cooled from the room temperature to 4 K for more than 10 h to avoid any cracks, which make the superconducting transitions broad.

III. RESULTS

Figure 2(a) presents the semilog plot of the resistance as a function of the magnetic field for $x=0.4$ in λ-$(BETS)_2FeCl_{4-x}Br_x$. At 0.45 K, as the field increases, the resistance shows a steep decrease at 15 T due to the AFI-PM transition, and then the S phase is induced. As the temperature increases, the field-induced S phase, which is associated with the resistance minimum at ~31 T, is suppressed. This indicates that the superconductivity is most stable at 31 T, i.e., the internal field $\mu_0 H_{int}=31$ T. (T_c has a maximum for $H=H_{int}$.) As temperature further increases, a broad peak around 31 T appears, the details of which will be discussed later. The AFI-PM transition field monotonically decreases with increasing temperature. Figure 2(b) presents the resistance for $x=0.5$. At low temperatures below 1.45 K, the field-induced S phase is stabilized around 20 T, following the AFI phase. At higher temperatures, the PM phase intervenes between the AFI and S phases. A resistance minimum is also visible at 31 T. Above 4 K, a broad peak similar to that for $x=0.4$ is seen at 31 T. For $x=0.7$, the AFI phase is destroyed at ~27 T for $T<2.1$ K [Fig. 2(c)], and then the S phase is immediately stabilized. A resistance minimum appears at 33 T, which is slightly higher in field than the others. For $T>2.1$ K, the AFI-PM transition becomes broad, and the resistance monotonically decreases with increasing field. The results above show that the internal field is almost independent of x, $\mu_0 H_{int}=31-33$ T, whereas T_{MI} strongly depends on x.

To obtain the critical field parameters that determine the phase diagrams, we define H_{c2} as the field where $R/R_n=0.5$. Here R_n is the normal state resistance obtained by

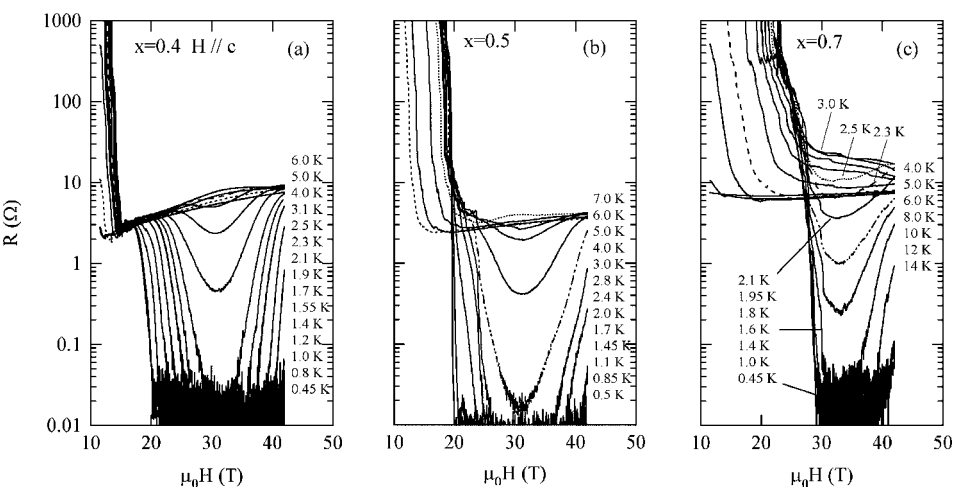

FIG. 2. Magnetic field dependence of the resistance for (a) $x=0.4$, (b) $x=0.5$, and (c) $x=0.7$ in λ-$(BETS)_2FeCl_{4-x}Br_x$. The field is applied along the c axis.

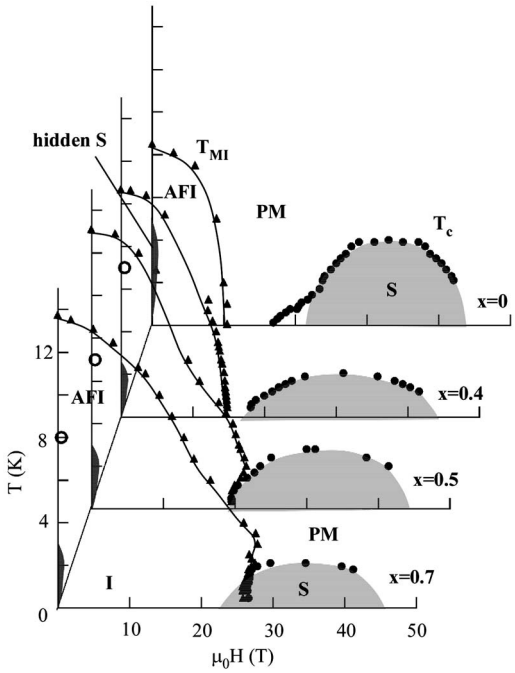

FIG. 3. Phase diagrams of λ-$(BETS)_2FeCl_{4-x}Br_x$ for $H \| c$. The open circles show T_N from the magnetization measurements. The shaded areas are the S phases calculated by Fischer theory. In the low field region, the hidden S phases are present.

extrapolating the resistance curves from the low or high field range for $x=0.4$ and 0.5. For $x=0.7$, we cannot make a reliable extrapolation, so a simple functional form giving the $R(H)$ curve at 14 K is used to obtain R_n. Although some ambiguity remains in determining the critical field H_{c2} from the resistive transitions,[18] the H_{c2} values obtained are sufficiently reliable for semiquantitative analysis.[19] From the data in Figs. 2(a)–2(c), we obtain the phase diagrams for λ-$(BETS)_2FeCl_{4-x}Br_x$ (Fig. 3). For comparison, we also present the results for $x=0$.[2,11] For the Br substituted salts ($x>0.3$), T_{MI} does not coincide with T_N.[17] The value of T_N obtained from the magnetization measurements is shown by the open circle for each salt. As x increases, we note that the I phase expands monotonically. For $x=0$ and 0.4, the AFI and field-induced S phases are well separated, but the S phase is next to the AFI phase for $x=0.5$. For $x=0.7$, there seems a strong competition between the AFI and S phases at ~27 T. The maximum of T_c is reduced by the Br substitution.

IV. DISCUSSION

A. Phase diagram

The global H-T phase diagrams of λ-$(BETS)_2Fe_xGa_{1-x}Cl_4$ in parallel fields ($H \| c$) have been qualitatively understood by Fischer theory based on the Jaccarino-Peter (JP) effect.[13] Therefore we also try to explain the phase diagrams of λ-$(BETS)_2FeCl_{4-x}Br_x$ according to Fischer theory. In the formula there are four adjustable parameters, the critical temperature T_c, the orbital critical field H^*_{c2}, the exchange field H_J whose field dependence is given by Brillouin function, and the dimensionless spin-orbit scattering parameter λ_{so}. The scattering parameter due to magnetic impurities λ_m is taken to be zero for simplicity. The shaded areas in Fig. 3 show the calculated results with the parameters listed in Table I. All the field-induced S phases except for $x=0$ are well reproduced by the theory.

TABLE I. Parameters of λ-$(BETS)_2FeCl_{4-x}Br_x$. T_c: critical temperature; λ_{so}: dimensionless spin-orbit scattering parameter; H^*_J: saturated exchange field due to the Fe moments; H^*_{c2}: orbital critical field for $T=0$ in the absence of magnetic impurities; and RRR: residual resistance ratio, $R(300\text{ K})/R(15\text{ K})$.

x (Br)	T_c (K)	$\mu_0 H^*_{c2}$ (T)	$\mu_0 H^*_J$ (T)	λ_{so}	RRR
0	5.5	55	36	4.3	29
0.4	2.3	140	31	18	6.7
0.5	3	110	33	14	7.3
0.7	2.6	100	34	12	6.6

The parameters T_c and H^*_J give the highest T_c of the phase diagram and the field where T_c roughly has the maximum, respectively. Therefore we can determine these values with less ambiguity. However, the other parameters, H^*_{c2} and λ_{so}, which mainly give the width of the superconducting phase, include large errors because the width of the phase is not very sensitive to these parameters. Therefore the differences of H^*_{c2} and λ_{so} among the Br substituted salts are not significant.

In the low field regions, superconducting phases can be stabilized because H_{int} is not so strong to cause the pair breaking. However, the I and AFI phases are stabilized at higher temperatures in the low field regions, so that the low field S phases (hidden S phases) are not observable experimentally. Here we should note that the internal field H_{int} is lower than the exchange field H_J,[20] $H_{int}=H_J-1/\eta$ in the high field limit, where $\eta \approx \alpha/\lambda_{so}T_c$. The value α is a Maki parameter given by $\alpha=\sqrt{2}H^*_{c2}/H_p$, where H_p is the Pauli limit.[21,22] Only in a pure two-dimensional (2D) case, we obtain $H_{int}=H_J$, because H^*_{c2} is infinite ($1/\eta=0$).

For λ-$(BETS)_2FeCl_4$, the experimental result of the field-induced S phase is significantly wider than the theoretical fit below 2 K. For this salt, an inhomogeneous superconducting state with a spatially modulated order parameter, the so-called FFLO state,[23] is expected to be stabilized.[2,24,25] The necessary conditions for the FFLO state are quenched orbital effect, large paramagnetic susceptibility, and clean limit superconductors. All the conditions are well fulfilled for λ-$(BETS)_2FeCl_4$. At present, no strong evidence of the FFLO state has been obtained experimentally, but the wide S phase below 2 K is well reproduced by the theoretical models taking account of the FFLO state.[2,24,25] For the Br substituted salts, however, the last condition is probably not fulfilled because of some disorder due to the Br ions. This is a possible reason why the S phases for the Br substituted salts are well reproduced by the Fischer theory. The possibility of the FFLO state is also argued for λ-$(BETS)_2GaCl_4$.[26]

Although the H_{c2} values are rather ambiguous, and T_c slightly depends on the sample quality, we can qualitatively conclude as follows.

(1) The critical temperature T_c is reduced by the Br substitution. Disorder in the structure caused by the Br substitution may decrease T_c since the ionic radius of Br^-(1.96 Å) is slightly larger than that of Cl^-(1.81 Å). This is consistent with the increase of the residual resistance ratio (RRR) (Table I).

(2) The spin-orbit scattering λ_{so} is significantly enhanced by the Br substitution, again probably because of Br disorder.

(3) The internal field H_{int} is almost independent of the Br substitution. This shows that the d-π interaction does not change with x.

(4) The orbital critical field H_{c2}^{*} is enhanced by the Br substitution. This suggests that the interlayer coupling is effectively reduced by the enhancement of the spin-orbit scattering.

The scattering time τ_{so} estimated from λ_{so}, $\tau_{so}=2\hbar/3\pi T_c\lambda_{so}$, is an order of 10^{-13} s for the Br substituted salts. It seems very short. However, we should note that the scattering time obtained here is of the electron motion in the plane perpendicular to the applied magnetic field (not only in the layers). It is likely that the scattering is predominant in the interlayer motion and that the interlayer motion is diffusive rather than coherent. This is consistent with the fact that these systems have highly 2D electronic states. We expect that the scattering in the in-plane motion is also enhanced by the Br substitution, which probably makes the superconductivity dirty.

According to the magnetization measurements,[17] the Weiss temperature decreases with increasing x (Fig. 1). The AF order of the Fe $3d$ moments is determined by both the direct exchange coupling J_{dd} and the indirect one via the d-π coupling $J_{\pi d}$. According to the molecular orbital calculation, J_{dd} is comparable to $J_{\pi d}$.[27] Since $J_{\pi d}$, which directly gives H_{int}, is x independent, J_{dd} should decrease with increasing x. This is consistent with the x-ray measurements, which show that the lattice constant of the a axis along the Fe-Fe bonds increases with increasing x at least five times more than those of the b and c axes.[17] Since the a axis is also the stacking direction of the BETS molecules, the intralayer transfer integral $t_{\parallel}$ should be significantly reduced with increasing x, whereas the $J_{\pi d}$ coupling, which is sensitive to the interlayer spacing b, does not change very much. The reduction of $t_{\parallel}$ increases T_{MI} with x because it effectively results in the enhancement of the correlation effect between the π electrons. Therefore, the I phase, which is regarded as a Mott insulator, becomes more stable with increasing x.[9]

B. Resistance maximum

Next, we briefly discuss the broad maximum of the resistance at ~31 T. Figure 4(a) presents a linear plot of the resistance for $x=0.4$. We note that the minimum at 31 T below 2.3 K changes to a maximum at higher temperatures. Such broad maxima have not been observed in λ-$(BETS)_2FeCl_4$ at ambient pressure, but are evident under a pressure of 1.4 kbar.[28] The temperature dependence of the resistance is shown in Fig. 4(b). The peaks near T_c seem sharpest at 31 T, where T_c has a maximum.

Similar broad peaks near T_c have been observed in various superconductors, mesoscopic Al thin films,[29–31] high T_c superconductors,[32] and organic superconductors.[33–35] In mesoscopic Al thin films, peaks have been discussed by models based on the charge imbalance, which is created near phase slip centers[29,30] or superconductor/normal boundaries.[31] They are evident only for mesoscopic structures, where the sample size is comparable to the penetration depth or the coherence length. Therefore such possibilities are ruled out. In $Bi_2Sr_2CaCu_2O_8$ and κ-$(BEDT\text{-}TTF)_2Cu(NCS)_2$, semiconductive behavior of the normal resistance is assumed. In electron-doped superconductors, the peaks are ascribed to the superconducting granular islands, where the charging energy of the islands is comparable to Josephson coupling energy between the islands. At present, the origin of the broad peaks observed in λ-$(BETS)_2FeCl_{4-x}Br_x$ is unclear. The fact that the peaks are observed only for the Br substituted salts, or for the pressurized λ-$(BETS)_2FeCl_4$ suggest that some disorder, which may cause a distribution of T_c, is essential for the peaks. The magnetic field has two effects on the magnetoresistance as well as on the superconductivity: orbital and Zeeman. The magnetoresistance normalized by the linear background in Fig. 4(a) has an almost symmetric field dependence around 31 T. The result may suggest that the Zeeman effect, which is suppressed at 31 T and increases with decreasing or increasing field, is related to the maximum behavior.

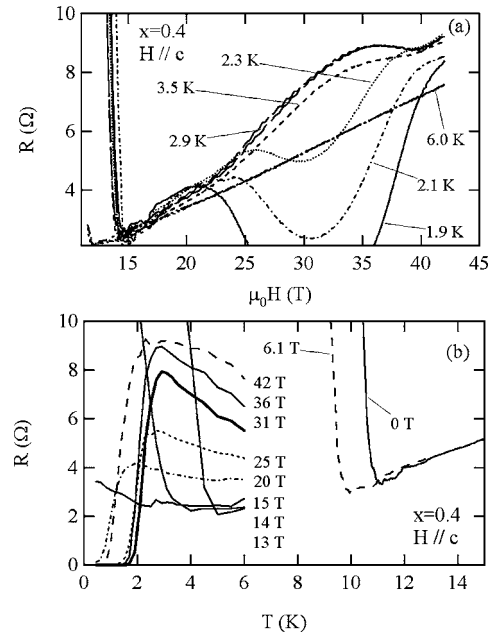

FIG. 4. (a) Field and (b) temperature dependence of the resistance for $x=0.4$.

V. CONCLUSIONS

The phase diagrams of the organic alloys λ-$(BETS)_2FeCl_{4-x}Br_x$ are determined for a magnetic field directed along the c axis. Superconductivity is stabilized in high magnetic fields for all samples, whereas the ground

states in low fields are insulating. The internal field H_{int} is almost independent of the Br substitution x, showing that the d-π interaction is x independent. The orbital critical field H_{c2}^{*} is enhanced by the Br substitution. These features are consistent with the fact that the crystal a axis along the stacking direction of the BETS molecules significantly increases with increasing x, causing the reduction of the intralayer transfer integral $t_{\parallel}$. The reduction of $t_{\parallel}$ also explains that the I phase is more stable with increasing x. Resistance maxima near T_c for fields in the range of H_{int} are observed, whose origin is not clear.

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