Galvanomagnetic properties and the Fermi surface of the organic superconductor β-$(ET)_2IBr_2$

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The field and angular dependences of the magnetoresistance of high-quality β-$(ET)_2IBr_2$ single crystals are studied in strong magnetic fields. The Shubnikov–de Haas oscillations are observed and investigated for various orientations of the field. The main motif of the Fermi surface of β-$(ET)_2IBr_2$ was found to be a weakly corrugated cylinder with its axis perpendicular to the conducting *ab* plane of a crystal. Other properties of the Fermi surface of this compound were also investigated.

INTRODUCTION

A major research effort on the synthesis of superconducting organic compounds, which began about 20 years ago, led to a discovery–in 1980–of superconductors belonging to the tetramethyltetraselenofulvalene (TMTSeF) family[1] and later–in 1984 those belonging to the bis(ethylenedithio)tetrathiofulvalene (BEDT–TTF or ET) family.[2] In spite of a large number of papers on the subject of organic metals, little has been done so far on their electron structure. Moreover, there are some unresolved problems relating to the mechanism of the superconductivity in these compounds, which are distinguished by a very strong anisotropy, a low density of charge carriers, and a strong interaction between electrons and molecular vibrations.

One of the specific problems is the fivefold difference between the temperatures of the superconducting transition T_c of the two modifications (β_L and β_H) of the organic metal β-$(ET)_2I_3$. It was found[3–6] that at temperatures below 120 K this compound may exist in different states: one of them is a metastable β_L phase with $T_c \approx 1.5$ K and the other is a β_H phase with $T_c \approx 8$ K. The β_H phase can be produced by cooling to temperatures $T < 100$ K under a pressure of $P > 200$–300 bar, and subsequent removing of the pressure. Measurements of the magnetic susceptibility[7–9] indicated that the density of states was little affected by the $\beta_L \to \beta_H$ transition. On the other hand, the only difference between the crystal structures[10] was the presence in β_L (but not in β_H) of an incommensurate superstructure associated with the distribution of the ethylene groups of the ET molecule relative to the plane of the molecule. It was shown in Refs. 4 and 11 that this superstructure strongly affects the residual electrical resistance. However, the role of the superstructure in the fivefold increase in T_c as a result of the $\beta_L \to \beta_H$ transition is not clear.[12]

The crystal lattice of the β_H phase, i.e., the phase without the superstructure, is exhibited also by two other organic metals of the ET family: β-$(ET)_2IBr_2$ (Ref. 13) and β-$(ET)_2AuI_2$ (Ref. 14). It can exist at any temperature under atmospheric conditions. Consequently, these two compounds are the closest analogs of the β_H phase of $(ET)_2I_3$ and a comparative study of their properties should shed light on the problem in hand.

Recent measurements of the magnetoresistance[15–19] provided some information on the electron structure of organic crystals based on ET. We describe the results of a detailed investigation of the angular and field dependences of the magnetoresistance of high-quality β-$(ET)_2IBr_2$ single crystals subjected to strong magnetic fields. We observed the Shubnikov–de Haas (SdH) oscillations over a wide range of angles between the direction of the field and the **c*** axis (the **c*** axis is perpendicular to the highly conducting *ab* crystal plane). The experimental results enable us to propose a possible shape for the Fermi surface of this metal.

EXPERIMENTS

Our measurements[2)] were made on single-crystals characterized by a ratio $R_{300\,K}/R_{4.2\,K} > 2000$ at temperatures from 1.5 to 4.2 K. The synthesis method and the main characteristics of the crystals were described earlier.[20] The values of the resistance R_a along the **a** axis and of R_c at right-angles to the highly conducting *ab* plane were determined using a current of $I = 1$ mA alternating at a frequency of 11.7 Hz. Samples with preevaporated gold stripe contacts were bonded to or pressed against platinum electrodes with a diameter of 30 μm. The resistance of the pressure contacts did not exceed 1–3 Ω, so that it was possible to determine the low-temperature resistances 10–30 mΩ with an error of at most 5×10^{-4}. A magnetic field up to 150 kOe was created in a superconducting solenoid. A sample could be rotated relative to the magnetic field in the *ab*, *ac**, and *b′c** planes (**c***⊥*ab*, **b′**⊥*ac**).

EXPERIMENTAL RESULTS

1. Angular dependence of the magnetoresistance

Figures 1 and 2 show the angular dependence of the magnetoresistance R_a and R_c of a β-$(ET)_2IBr_2$ single crystal recorded in a field $H = 150$ kOe at $T = 1.45$ K. The field was in the *ac** (**H**⊥**b′**, Fig. 1) and *b′c* (**H**⊥**a**, Fig. 2) planes; here φ is the angle between the field direction and the **c** axis. We determined the values of $[R(150\,\text{kOe}) - R(0)]/R(0)$, which are plotted in Fig. 1. We found $R(0)$ by extrapolating the temperature dependence of the resistance in $H = 0$ from $T > T_c$ to $T = 1.45$ K.

The angular dependence in fields $H > 40$ kOe lying in the *ac** and *b′c** planes was characterized by strong oscillations that were periodic functions of the tangent of the angle between the direction of **H** and the **c*** axis (Fig. 3). Increasing temperature from 1.5 to 4.2 K reduced the amplitude of these oscillations by a factor of about 1.5 but did not affect their positions. A change in the field intensity did not alter

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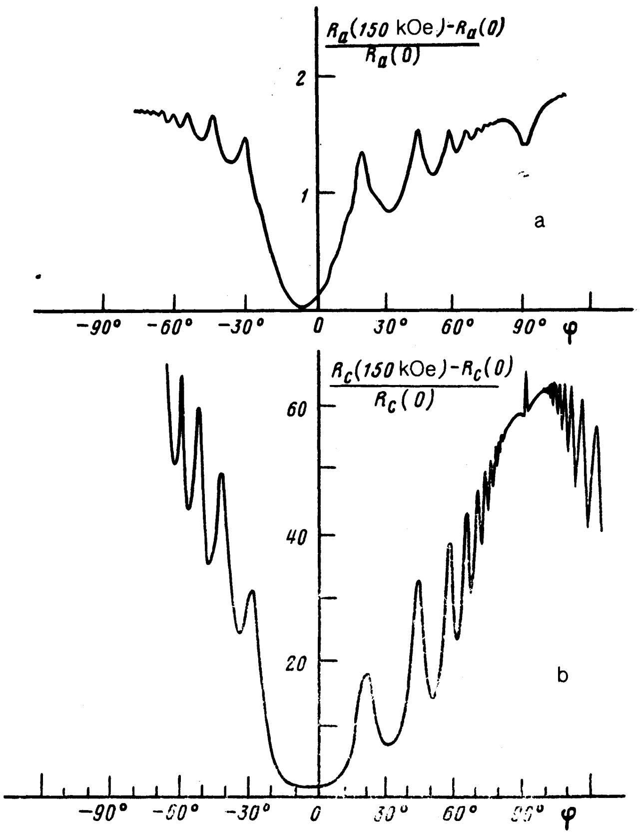

FIG. 1. Dependence of the magnetoresistance on the angle φ between the direction of the field in the ac^* plane ($\mathbf{H} \perp \mathbf{b}'$) and the $\mathbf{c}^*$ axis; $H = 150$ kOe, $T = 1.45$ K. a) Resistance R_a along the **a** axis; b) resistance R_c across the ab plane.

the positions of the local resistance maxima and minima and, consequently, this nonmonotonic behavior did not represent the SdH oscillations. The true "angular" SdH oscillations appeared in certain parts of the $R(\varphi)$ curves, but had a much smaller amplitude. The small sharp peaks obtained in the $\mathbf{H}\perp\mathbf{c}^*$ configuration corresponded to the Fermi surface which is open along the $\mathbf{c}^*$ axis.[17]

Figure 4 gives the dependence of the relative transverse magnetoresistance $[R_c(150\ \text{kOe}) - R_c(0)]/R_c(0)$ on the angle θ between the field direction and the **a** axis when $\mathbf{H} \perp$

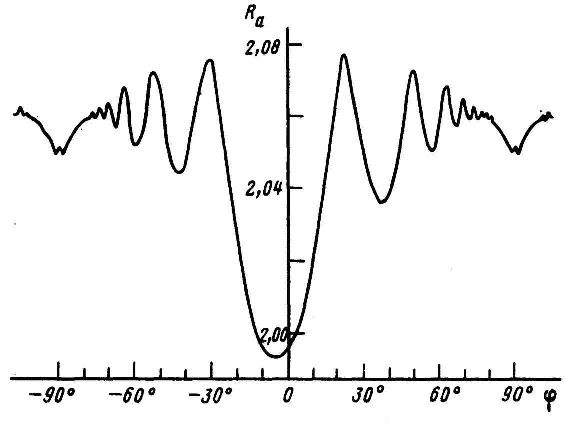

FIG. 2. Dependence of the resistance R_a ($10^{-2}\ \Omega$) on the angle φ between the direction of the field in the $b'c^*$ plane ($\mathbf{H} \perp \mathbf{a}$) and the $\mathbf{c}^*$ axis; $H = 150$ kOe, $T = 1.45$ K.

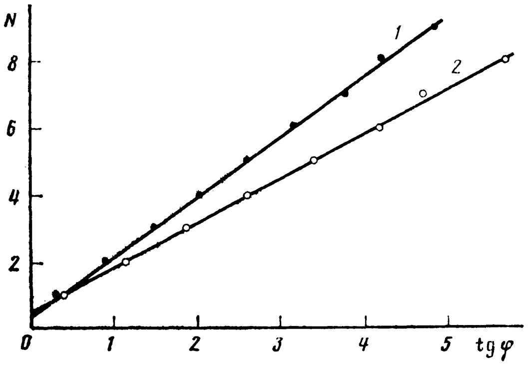

FIG. 3. Dependence of the positions of the local maxima: 1) $\mathbf{H} \perp \mathbf{b}'$; 2) $\mathbf{H} \perp \mathbf{a}$ (compare with Figs. 1 and 2); N is the number of the maximum.

$\mathbf{c}^*$. In this case the field was $H = 150$ kOe and the temperature was $T = 1.45$ K. We included (in the inset) the same dependence replotted using polar coordinates and indicating the crystal axes. It is clear from this figure that the magnetoresistance anisotropy in the ab plane was unexpectedly strong and the ratio R_{max}/R_{min} reached 10–15, depending on the quality of the crystal. A minimum of the dependence $R_c(\theta)$ corresponded to $\mathbf{H}\perp\mathbf{a}$, while a maximum corresponded to a field perpendicular to the direction of the stacks of the ET molecules (i.e., it was perpendicular to the $\mathbf{a} + \mathbf{b}$ direction in the crystal).

2. Shubnikov–de Haas oscillations

Over a wide range of angles φ the field dependences of the resistance exhibited the SdH oscillations in the range $H > 80$ kOe (Figs. 5–7). Figure 5 shows clearly two types of oscillations.

The first type should be called "slow" oscillations of frequency $F_{slow} \approx 0.5$ MHz, which were significant in the $\mathbf{H}\perp\mathbf{b}'$ case within the range of angles approximately from $-25°$ to $+5°$ between the field and the $\mathbf{c}^*$ axis. The amplitude of these oscillations in the $\mathbf{H}\|\mathbf{c}^*$ configuration was

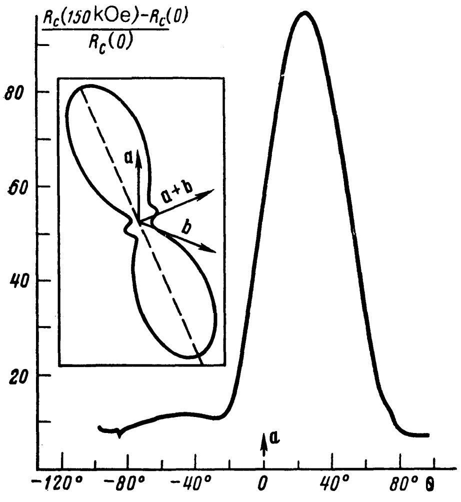

FIG. 4. Dependence of the transverse magnetoresistance R_c on the angle θ between the direction of the field in the ab plane ($\mathbf{H} \perp \mathbf{c}^*$) and the **a** axis; $H = 150$ kOe, $T = 1.45$ K. The inset shows the same dependence, but plotted using polar coordinates and indicating the directions of the crystallographic axes.

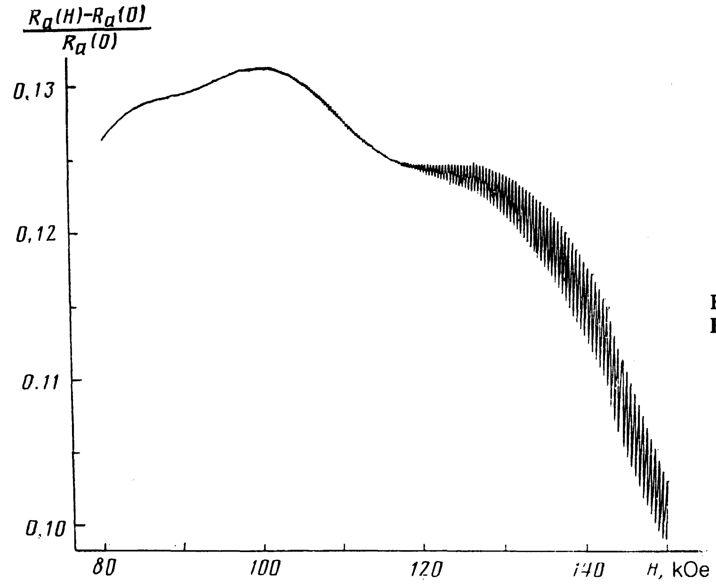

FIG. 5. Field dependence of the magnetoresistance along the **a** axis in the $\mathbf{H} \perp \mathbf{b}'$ configuration; $\varphi \approx -17°$, $T = 1.45$ K.

about 0.5% of the total resistance of the sample. The parameters of the oscillations obtained along this direction of the field agreed with those published earlier.[15]

The second type of oscillations, which should be called "fast," were characterized by a frequency $F_{\text{fast}} \approx 40$ MHz and were observed over a wide range of angles φ. For some angles φ these oscillations exhibited characteristic beats. The beats were particularly clear at low angles φ i.e., in the region of the principal maximum of the angular dependence of the magnetoresistance (Fig. 6); in this case the frequency of the beats was $F_{\text{beat}} \approx 0.3$ MHz. The beats at angles lying in the interval between the first and second local maxima of the dependence $R(\varphi)$ were much weaker. The occurrence of the beats most probably indicated the existence of two extremal sections of the Fermi surface with similar areas. For the field directions corresponding to the local maxima of the $R(\varphi)$ curves we found that the field dependence $R_a(H)$ and $R_c(H)$ exhibited oscillations of just one frequency and there were no beats (see, for example, Fig. 7).

Figure 8 shows the angular dependence of the amplitude of the fast oscillations observed in a field $H = 150$ kOe in the $b'c^*$ plane. Two large maxima coincided with the first local maxima of the $R_a(\varphi)$ curve. We identified the beat range in the figure. In this range the oscillation amplitude depended in a complicated manner on the angle because of the presence of beat nodes or antinodes.

The angular dependence of the oscillation frequencies obtained in the $\mathbf{H} \perp \mathbf{b}'$ and $\mathbf{H} \perp \mathbf{a}$ configurations is plotted in Fig. 9. When polar coordinates are used, the dependence is approximated satisfactorily by a straight line perpendicular to the $\mathbf{c}^*$ axis, indicating that the oscillations in question were associated with a Fermi surface sheet in the shape of a slightly corrugated cylinder with its axis along $\mathbf{c}^*$. The oscillation frequency 39 MHz for the $\mathbf{H} \perp ab$ direction corresponded to the Fermi surface section of 3.7×10^{15} cm^{-2} area, i.e., $\approx 55\%$ of the Brillouin zone area in the ab plane. The Fermi surface section responsible for the slow oscillations was known to be 5×10^{13} cm^{-2} (Ref. 15) or 0.6% of the Brillouin zone area.

At moderately low values of T the temperature dependence of the oscillation amplitude made it possible to estimate readily the cyclotron mass for the extremal sections of the

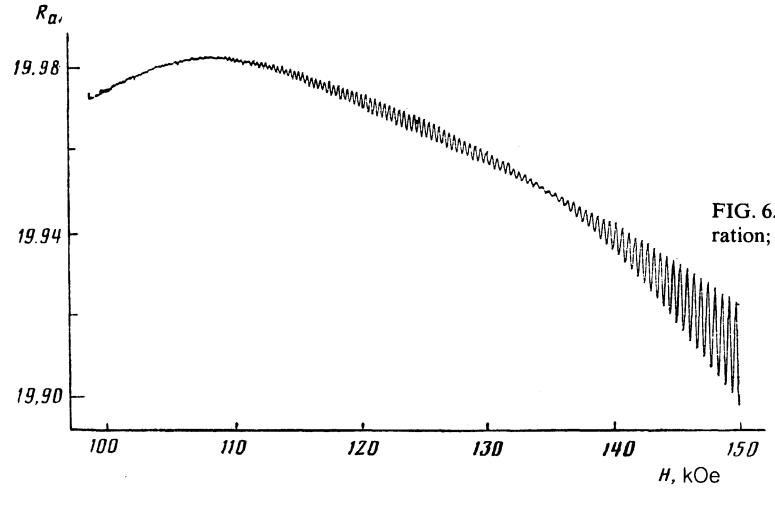

FIG. 6. Field dependence of the resistance R_a (mΩ) in the $\mathbf{H} \perp \mathbf{a}$ configuration; $\varphi \approx -11°$, $T = 1.45$ K.

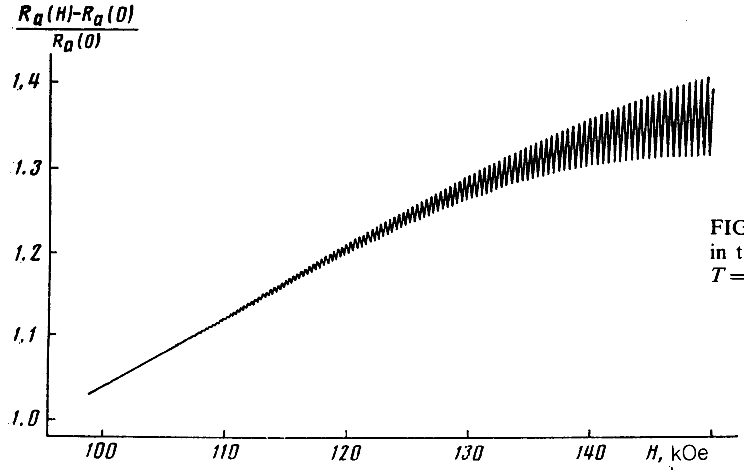

FIG. 7. Field dependence of the resistance R_a in the $\mathbf{H} \perp \mathbf{b}'$ configuration in the region of the first local maximum shown in Fig. 1a ($\varphi \approx 17°$); $T = 1.45$ K.

Fermi surface:

$$\ln(A/T) = \text{const} - 2\pi^2 c k_B m^* (T + T_D)/e\hbar H,$$

where A is the oscillation amplitude and T_D is the Dingle temperature. Figure 10 shows the corresponding dependence for the fast oscillations in a field $H = 150$ kOe directed in the ac^* plane for $\varphi \approx 17°$ (i.e., at the point of the first local maximum). If we ignore the temperature dependence of T_D, then estimates of the effective mass give $m^* \approx 4.5 m_e$ and $\approx 0.5 m_e$ for the fast and slow oscillations, respectively. An estimate of the Dingle temperature deduced from the field dependence of the amplitude of the fast oscillations at $T = 1.45$ K gives $T_D \approx 0.4$ K.

3. Semiclassical part of the magnetoresistance

The semiclassical part of the field dependence of the magnetoresistance $R_A(H)$ showed a tendency to saturate irrespective of the field direction. In fields exceeding ≈ 100 kOe directed close to $\mathbf{H} \| \mathbf{c}^*$ there was even a small (about 5%) fall whose origin was unclear. This fall could be due to a parasitic Hall emf.

The behavior of the magnetoresistance $R_c(H)$ measured across the ab plane was more complicated. If the field was not in the ab plane, the dependence $R_c(H)$ could be as follows. In the region of local minima of the angular dependence $R_c(\varphi)$ (Fig. 1b) the $R_c(H)$ curves showed a tendency to saturation. Near local maxima the $R_c(H)$ curves had a positive curvature. By way of example, we plot in Fig. 11 the dependence $R_c(H)$ for $\mathbf{H} \perp \mathbf{b}'$ and $\varphi \approx 17°$ (first local maximum) at $T = 1.45$ K (curve 1). The points corresponding to the same values of $R_c(H)$ but after subtraction of the absolute value of the magnetoresistance peak are also plotted in this figure (curve 2).

In the $\mathbf{H} \perp \mathbf{c}^*$ case the field dependences of R_c show no tendency to saturation (Fig. 12). For $\mathbf{H} \| \mathbf{a}$, i.e., in the region

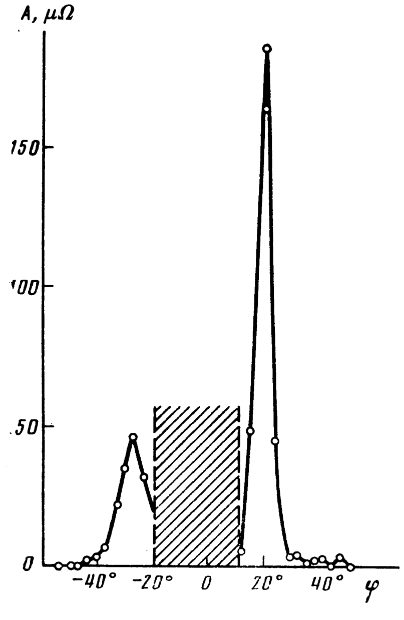

FIG. 8. Amplitude of the Shubnikov de Haas oscillations obtained in a field lying in the $b'c^*$ plane; $H = 150$ kOe, $T = 1.45$ K. The shaded region is where significant beats occur.

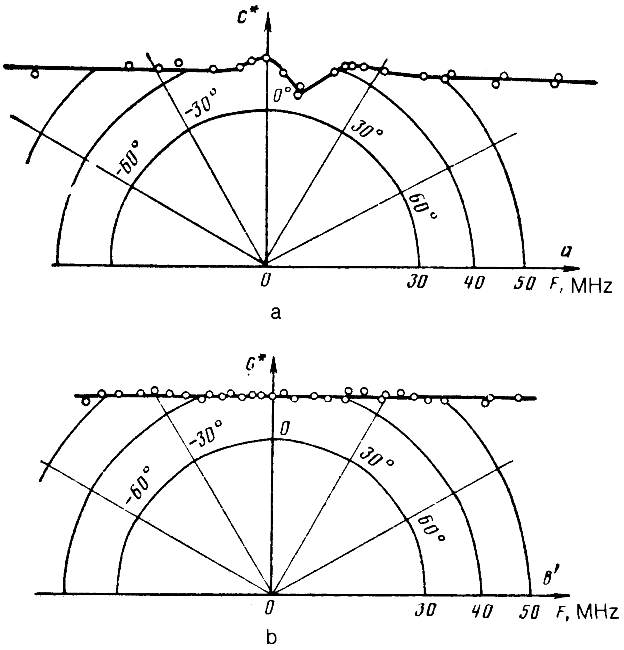

FIG. 9. Dependence of the frequency of fast Shubnikov–de Haas oscillations on the angle between the field and the $\mathbf{c}^*$ axis, plotted in polar coordinates; (a) $\mathbf{H} \perp \mathbf{b}'$; b) $\mathbf{H} \perp \mathbf{a}$.

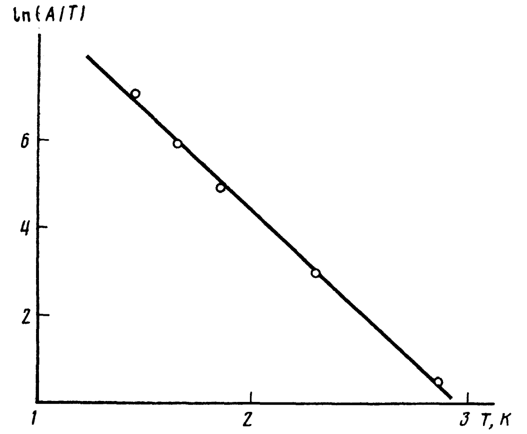

FIG. 10. Reduced temperature dependence of the amplitude of the Shubnikov–de Haas oscillations in the $\mathbf{H} \perp \mathbf{b}'$ configuration, $\varphi \approx 17°$. The slope of the line determines the cyclotron mass on an extremal orbit (described by an expression given in the text).

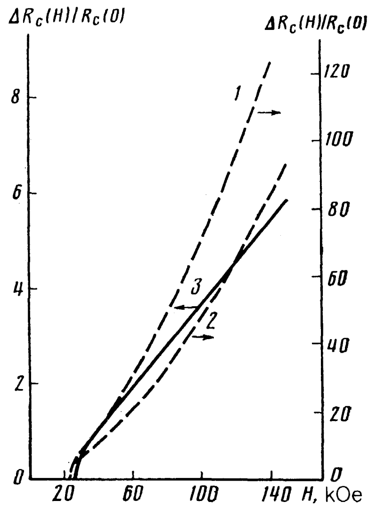

FIG. 12. Dependence of the magnetoresistance $\Delta R_c(H)/R_c(0)$ on the field lying in the *ab* plane: 1) $\mathbf{H} \perp (\mathbf{a}+\mathbf{b})$; 2) $\mathbf{H} \parallel \mathbf{a}$; 3) $\mathbf{H} \perp \mathbf{a}$.

of a "spike" in Fig. 1b, and also for $\mathbf{H} \perp (\mathbf{a}+\mathbf{b})$ the $R_c(H)$ curves have a clear positive curvature. At the points corresponding to both minima and a "small" maximum of the dependence $R_c(\theta)$ the dependence $R_c(H)$ was linear.

DISCUSSION

The nature of the angular dependences of the frequency of the fast SdH oscillations (Fig. 9) indicated that the main motif of the Fermi surface of this compound is a cylinder with its axis along **c***. The cylinder can be slightly corrugated. A small amplitude of the corrugations is supported also by the low frequency of the beats of the Shubnikov oscillations and by the strong anisotropy of the resistance (in $H = 0$) in the *ac** plane typical of β-$(ET)_2X$ compounds.[21] The open nature of the Fermi surface along the **c*** axis was confirmed, as pointed out already, by sharp peaks exhibited by the angular dependence of the magnetoresistance for $\mathbf{H} \perp \mathbf{c}^*$ (Figs. 1 and 2) and by an absence of saturation of the field dependence of R_c (Fig. 12).

The strongly nonmonotonic nature of the dependence $R(\varphi)$ (Figs. 1 and 2) and the qualitative difference in the nature of the $R(H)$ dependence at the local minima and maxima of the magnetoresistance (Fig. 11) may be due to a change in the topology of the carrier orbits as a result of a change in the magnetic field direction. For example, at the point of a local maximum in the angular dependence $R(\varphi)$ one of the extremal orbits may be self-intersecting. The period of revolution on this orbit will then tend to infinity. The semiclassical part of the magnetoresistance then becomes proportional to H^2 and the contribution of this orbit to the SdH oscillations vanishes. The observed nature of the magnetoresistance and the absence of beats of the SdH oscillations at the local maxima are in agreement with this hypothesis. Obviously, this dependence should be periodic in the reciprocal lattice, as manifested by the periodicity of the nonmonotonic features of the angular dependence of the magnetoresistance (Fig. 3).

The angular dependence of the oscillation amplitude and the absence of beats in certain field directions can in principle be explained by the nature of corrugations of the Fermi surface cylinder. In fact, if we assume that the isotropic cylinder is corrugated in accordance with the cosine law [i.e., $\varepsilon_F = \hbar^2(k_x^2 + k_y^2)/2m + 2t\cos(ck_z)$, where t is the transverse hopping integral], it follows from Ref. 22 that at angles φ between the magnetic field direction and the **c*** axis such that $ck_F \tan(\varphi) = \pi(n - 1/4)$ holds all the orbits have the same area. The beats should then disappear and the oscillation amplitude should be maximal. However, the reason for the abrupt change in the field dependence of the semiclassical part of the magnetoresistance from saturation to a quadratic rise (Fig. 11) is then not quite clear.

The corrugated nature of the Fermi surface cylinder may also give rise to self-intersecting orbits, but it is not very likely that in our case the corrugations can be sufficient for the manifestation of the effect in the case of relatively small angles between the field and the **c*** axis. Another possibility of the formation of self-intersecting orbits is associated with the necks which join the Fermi surface cylinders in neighboring unit cells of the reciprocal lattice. A strong anisotropy of the magnetoresistance in the *ab* plane supports the conclusion that the base of the Fermi surface cylinder is far from a circle. Therefore, if we allow for the large Fermi surface cross section, exceeding 50% of the Brillouin zone area,

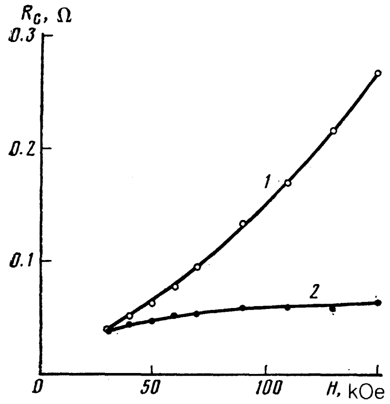

FIG. 11. Field dependence of the resistance R_c obtained in the $\mathbf{H} \perp \mathbf{b}'$ configuration at the point of the first local maximum ($\varphi \approx 17°$). Curve 1 represents the total resistance and curve 2 is obtained by subtracting the absolute value of the peak of the dependence $R(\varphi)$.

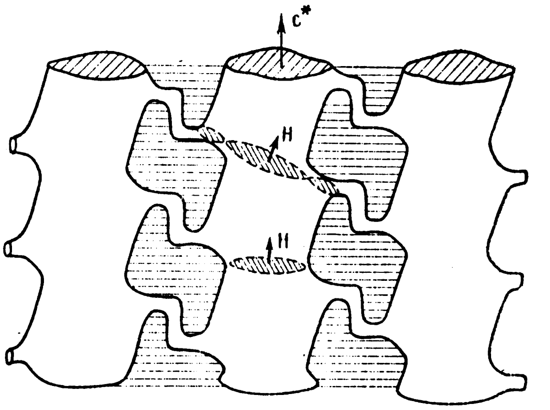

FIG. 13. Possible variant of the Fermi surface of $\beta\sim(ET)_2IBr_2$ shown in the repeated zone scheme. One of the extremal sections for **H** ∥ **c*** and the maximum section in a field directed along one of the local maxima in Fig. 1 are shown.

we find that intersections of the Fermi surface with the Brillouin zone boundary in this plane are highly likely.

The reason for the appearance of the slow SdH oscillations is not yet clear. They may be associated with extremal sections of the necks joining the main Fermi surface cylinders or with some other sheet of the Fermi surface.

We can summarize the results obtained by plotting some variants of the Fermi surface of β-$(ET)_2IBr_2$ crystals. One of the likely variants is plotted in Fig. 13 (not to scale) in the repeated zone scheme. The main motif of the Fermi surface is a corrugated cylinder with the axis along **c***. The cylinders located in neighboring chains are joined by narrow necks. The directions of the necks are selected so as to account for slow SdH oscillations.

It would be of interest to compare the behavior of β-$(ET)_2IBr_2$ and β-$(ET)_2I_3$ crystals in a magnetic field. It was reported in Ref. 18 that β_L-$(ET)_2I_3$ crystals exhibit slow SdH oscillations of frequency ≈ 1 MHz; possible reasons for the absence of the fast SdH oscillations and of angular oscillations of the magnetoresistance were discussed. Recent data on the fast SdH oscillations in the β_{H^*}-$(ET)_2I_3$ phase[19] confirm the hypothesis that the Fermi surface of this phase is similar to that of β-$(ET)_2IBr_2$. Observations of oscillations in β_L-$(ET)_2I_3$ and β-$(ET)_2IBr_2$ with similar parameters are reported in Refs. 23 and 24. However, these results differ very greatly both from our results and from those reported in Ref. 19. The reason for such discrepancies is not yet clear; we cannot exclude the possibility that the energy band structure of β-$(ET)_2X$ compounds is affected significantly by certain impurities.

Let us note in conclusion that the actual discovery of the SdH oscillations in these samples was somewhat unexpected. In fact, if we estimate the order of magnitude of the relaxation time from the conductivity using the Drude–Lorentz relationship $\tau = \sigma m^*/ne^2$, then using the stoichiometric value of the carrier density $n = 1/V_{\text{u.c.}} \approx 1.2\times10^{21}\ \text{cm}^{-3}$ (with one electron per unit cell–u.c.), we find that for a sample with typical room-temperature conductivity $\sigma(300\ \text{K}) \approx 30\ \Omega^{-1}\cdot\text{cm}^{-1}$ and with the ratio $R_{300\,\text{K}}/R_{1.5\,\text{K}} \approx 2500$, we obtain $\tau(1.5\ \text{K}) \approx 1.1\cdot10^{-12}$ s. If we assume that the mass of the carriers dominating the conductivity is $m^* \approx 4.5 m_e$, deduced from the fast oscillations, then in a field of $H = 100$ kOe we have $\omega\tau \approx 0.4$. This value obviously does not correspond to the condition for the observation of quantum oscillations.

On the other hand, an estimate of the relaxation time (from the Dingle temperature) of the fast oscillations gives $\tau_D \approx \hbar/k_B T_D \approx 6.10^{-11}$ and $\omega\tau_D \approx 7$. The product $\omega\tau_D$ for the slow oscillations (if we assume that $T_D \approx 2.2$ K and $m^* \approx 0.5\, m_e$) amounts to about 11. The nature of the behavior of the magnetoresistance, namely saturation when **H**∥**c*** and the high value of R_c when **H**⊥**c***, also supports the conclusion that for $H \approx 100$ kOe the product in question is $\omega\tau \approx 10$. Therefore, an estimate of the relaxation time and of the mean free path from the Drude–Lorentz expression, i.e., in the approximation of the quadratic dispersion law is in this case invalid. The data on the SdH oscillations allow us to estimate the mean free path in the *ab* plane as follows. If we take the approximate value of the Fermi momentum in this plane $p_F \approx \hbar(S_F)^{1/2}/\pi \approx 2.10^{-20}$ g.cm.s^{-1} (S_F the Fermi surface cross section) and if we postulate that the effective mass is $m^* \approx 4.5 m_e$ and the relaxation time is $\tau \approx 6\times10^{-11}$ s, we find that the mean free path is $l \approx p_F\tau/m^* \approx 2.5\ \mu$m.

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2) The measurements were carried out at the International Laboratory of Strong Magnetic Fields and Low Temperatures, Wrocław, Poland.

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