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Shubnikov-de Haas oscillations in an organic conductor tetramethyltetraselenafulvalene-2,5-dimethyl-7,7', 8,8' tetracyanoquinodimethane (TMTSF-DMTCNQ)

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Résumé. — L'observation d'oscillations de Haas-Shubnikov dans la phase métallique (Metal-II) de TMTSF-DMTCNQ stabilisée sous haute pression ($P > 10$ kbar) démontre le caractère tridimensionnel de la surface de Fermi à basse température ($T < 2$ K). Les sections de la surface de Fermi sont respectivement de $1,16 \times 10^{17}$ et $2,6 \times 10^{17} \text{ m}^{-2}$ pour les plans ab et ac . La masse cyclotron $m_c/m_0 \simeq 0,56$ et la température de Dingle $T_D = 0,8$ K sont obtenues à partir de la dépendance des oscillations SdH en fonction du champ et de la température. Enfin, nous examinons ces résultats en fonction de la conductivité électronique qui s'élève à $3 \times 10^5 (\Omega\text{cm})^{-1}$ à $T = 1,2$ K.

Abstract. — The observation of Shubnikov-de Haas (SdH) oscillations in the metallic, high pressure phase (Metal-II) of TMTSF-DMTCNQ ($P > 10$ kbar) demonstrates the 3-dimensional, closed nature of the Fermi surface at low temperature ($T < 2$ K). We observe very small Fermi surface cross-sections of $1.16 \times 10^{17} \text{ m}^{-2}$ and $2.6 \times 10^{17} \text{ m}^{-2}$ in the ab and ac planes respectively. From the field and temperature dependence of the SdH oscillations we extract a cyclotron mass $m_c/m_0 \simeq 0.56$ and a Dingle temperature $T_D = 0.8$ K. Finally we discuss the discrepancy between these results and the very high dc conductivity ($\sigma_{1,2K} \simeq 3 \times 10^5 (\Omega\text{cm})^{-1}$).

The topology of the Fermi Surface (FS) of quasi one dimensional (Q-1-D) organic conductors is a topic of current interest [1]. In materials consisting of stacks of planar molecules one may *a priori* expect an open FS, but with some deviation from planarity, related to the interchain hopping integral $t_{\perp}$. This parameter is essential if we want to describe for example the observation of superconductivity in such materials, because it is used to account for a finite Josephson coupling ($t_{\perp}^2/t_{\parallel}$) between the chains and thus allows a long range ordered superconducting state at finite temperature.

In some cases however the FS of Q-1-D conductors do display closed orbits. Consequently

the models used to describe the transport properties change from the 1-D picture with chains coupled by weak tunneling to a more conventional 2D (or 3D) picture.

A closed FS in Q-1-D conductors has been observed in two phenomenologically different situations.

(i) In two-chain compounds such as HMTSF-TCNQ the closed orbits is a result of hybridization of electronic states [2-4] with wave vectors near $\pm k_F$ on the two different chains.

(ii) Closed orbits have been reported in single chain conductors (the TMTSF₂X-family), but in these materials they are observable only after a magnetic field induced phase transition has occurred [5].

HMTSF-TCNQ is so far the only two-chain material where Shubnikov-de Haas oscillations in the magnetoresistance have been observed [6]. In this material it was argued that short Se-N contacts are weakening the Peierls instability occurring at 24 K [7]. Thus a gap does not extend over the whole FS and this leads to semimetallic behaviour at helium temperature. At the same time lattice disorder and a hydrostatic pressure of 4 kbar can suppress the transition, but the maximum conductivity observable at low temperature is of the order of $10^4 (\Omega\text{cm})^{-1}$ [8].

The material TMTSF-DMTCNQ at ambient pressure undergoes a Peierls transition at 41 K [9]. The conducting phase, stable down to 41 K at ambient pressure, has been called the Metal-I phase.

A hydrostatic pressure of about 10 kbar abruptly suppresses the Peierls insulating state [10]. The predominant feature of the so-called Metal-II state of TMTSF-DMTCNQ (at $P > 10$ kbar) is a conductivity at least one order of magnitude larger than that of HMTSF-TCNQ [8].

In the present work we have determined the FS topology of TMTSF-DMTCNQ in the Metal-II state, by observing SdH oscillations in magnetic fields transverse to the highly conducting (*a*) axis. A highlight of this work is the derivation, from the SdH measurements of a single particle conductivity which is several orders of magnitude smaller than the observed values of $3 \times 10^5 (\Omega\text{cm})^{-1}$ at helium temperature [10]. We propose an interpretation of this discrepancy in terms of a contribution of short range 1-D superconducting fluctuations.

1. Experimental.

The magnetoresistance of high purity TMTSF-DMTCNQ single crystals was measured by a classical four contacts and low frequency method. The resistance was recorded *versus* magnetic field, without field modulation. The samples were pressurized up to 12 kbar in a copper-beryllium cell and cooled slowly in a dilution refrigerator. The magnetic field (up to 7 Tesla) was aligned either along the *b* or *c* direction of the crystals. The accuracy of the alignment was not better than 10 degrees.

2. Results.

As previously reported, [10] a dramatic increase of the conductivity is observed on cooling. In this work our best result was $\sigma(1.2 \text{ K})/\sigma(300 \text{ K}) = 135$ at $P = 12$ kbar. Together with the pressure increase at room temperature by a factor 4.75 between ambient and 12 kbar pressure, the conductivity at low temperature culminates at $\approx 3 \times 10^5 (\Omega\text{cm})^{-1}$, [$\sigma(300 \text{ K}, 1 \text{ bar}) \approx 500 (\Omega\text{cm})^{-1}$].

In spite of the large and still somewhat temperature dependent resistivity at helium temperature the present study and some previous attempts have failed to detect superconductivity down to 80 mK.

Raw magnetoresistance data are displayed on figure 1, for temperatures from 155 mK to 3 K and with the magnetic field along the *c*-axis. The oscillations are most clearly observed below 1 K, but their amplitude never grows above a few percent of the non-oscillating background. Data qualitatively similar were obtained with the magnetic field along the *b*-direction.

Fig. 1. — Magnetoresistivity of TMTSF-DMTCNQ under 12 kbar at different temperatures. The applied magnetic field is parallel to the c -axis.

The results of figure 1 clearly indicate a quantum origin to the high-field magnetoresistance. Indeed they can be analysed following the expression of the SdH effect [11] :

$$\frac{\Delta\rho}{\rho} \approx \left(\frac{\hbar S_m}{2\pi eB} \right)^{-1/2} \cdot \sum_{r=1}^{\infty} \frac{(-1)^r}{r^{1/2}} \cdot \frac{kTm_c}{\hbar eB} \cdot \frac{\exp\left(-\frac{2\pi^2 rm_c kT_D}{\hbar eB}\right)}{\sinh\left(\frac{2\pi^2 rm_c kT}{\hbar eB}\right)} \cdot \cos\left(\frac{r\hbar S_m}{eB} - \frac{\pi}{4}\right) \quad (1)$$

where m_c is the cyclotron mass, S_m the extremal cross section of the FS in a plane perpendicular to the magnetic field and T_D the Dingle temperature ($kT_D \sim \hbar/\pi\tau$).

Magnetic fields corresponding to minima and maxima of the oscillations are plotted *versus* integers and half-integers in figure 2. The plot shows that equation 1 accounts well for the oscillatory nature of the magnetoresistance. Furthermore, the results of figure 2 allows a determination of the periods 0.086 T^{-1} and 0.24 T^{-1} for the oscillations with B along the c and b -axis respectively. The corresponding cross-sections $1.16 \times 10^{17} \text{ m}^{-2}$ and $2.6 \times 10^{17} \text{ m}^{-2}$ in the $(a-b)$ and $(a-c)$ planes respectively amount to about 0.1 % of a Brillouin zone cross section.

The measurement of the temperature dependence of the amplitude at fixed field, figure 3, gives an estimate of the cyclotron mass whereas the field dependence at constant temperature leads to T_D . We obtain $m_c = (0.56 \pm 0.05) m_0$ and $T_D \sim 0.8 \text{ K}$ for $B \parallel c$.

Fig. 2. — Successive integers and half-integers corresponding to maxima and minima of the oscillations versus H^{-1} . The upper curve is for $H//c$ and the lower for $H//b$.

Fig. 3. — Amplitude of three extrema (4.42, 3.7 and 3.14 Tesla) of the oscillations as a function of the temperature. The solid lines show the theoretical results with a cyclotron mass of $0.56 m_0$.

As demonstrated in figure 4 the field and temperature dependence of the oscillations is in agreement with the derived values of m_c and T_D .

In table I, we compare the parameters of TMTSF-DMTCNQ with the parameters obtained for HMTSF-TCNQ [6] and $(\text{TMTSF})_2\text{PF}_6$ [5]. We emphasize that SdH oscillations of the 1-chain materials are observed only above a threshold field, the amplitude of which depends on the orientation with respect to crystal axis and temperature. In $(\text{TMTSF})_2\text{PF}_6$ for instance, the

Fig. 4. — Oscillatory magnetoresistivity of TMTSF-DMTCNQ under 12 kbar ($H//c$) versus $1/H$ for different temperatures. The solid lines show the best fit (see the text).

threshold field is 65 kOe ($H//c$ (¹)) at 1 K under 7 kbar [5]. A similar threshold field effect has been reported for (TMTSF)₂ClO₄ under a very small pressure [12] or after slow cooling at ambient pressure. No threshold field is observed for TMTSF-DMTCNQ and HMTSF-TCNQ.

Table I.

	S_m (10^{16} m^{-2})	m_e/m_0	T_D (K)
TMTSF-DMTCNQ	11.6 ($//c$) 26.7 ($//b$)	0.56	0.8
HMTSF-TCNQ	7 ($//c$)	0.05	(1) (¹)
(TMTSF) ₂ PF ₆	73 ($//c$)	1.1	3

(¹) The Dingle temperature was not reported in reference [6] but the domain of temperatures in which oscillations are observed is consistent with $T_D \approx 1$ K.

3. Interpretation.

The data of the present work prove conclusively the existence of closed orbits on the FS of the Metal-II phase of TMTSF-DMTCNQ at low temperature. These closed orbits are provided by nearly ellipsoidal pockets of very small volume. If we assume parabolic bands and use the SdH parameters (Table I) and figure 2 we obtain the Fermi energy of a pocket $\varepsilon_F \approx 50$ K. Moreover, the volume enclosed within such small pockets is about 3×10^{-4} times smaller than the volume enclosed by the original high temperature 1-D Fermi surface. Thus, the density of carriers seen by SdH effects at low temperature in the Metal-II phase is about 10^{-4} per formula-unit, i.e. $3 \times 10^{18} \text{ cm}^{-3}$ (assuming two pockets of such carriers per Brillouin zone). The Fermi energy which can be derived from the density of carriers and the data in table I, $\varepsilon_F = \frac{\hbar^2}{2 m^*} (3 \pi^2 n)^{2/3}$, within a nearly free electron approximation ($m^* \sim 0.5 m_0$), amounts to ≈ 180 K.

We infer that the present SdH results can be interpreted within the framework of the model [4] proposed for the interpretation of the galvanomagnetic data of HMTSF-TCNQ at low temperature [13]. In this model a 1-D to 3-D band structure transformation occurring gradually upon cooling to low temperature was suggested.

Starting our description with a (hypothetical) situation where we assume a zero transverse coupling between adjacent 1-D chains, the bands derived from the donor and acceptor chains respectively, will cross at the Fermi level and the FS is planar. This planar geometry is however slightly modified by the conjugated effects of weak couplings both between like and unlike chains (Q-1-D situation) which transforms the original planar FS derived from non-interacting chains into a semimetallic three-dimensional FS. The semimetallic FS appears as small pockets of electrons and holes comprising a volume which is much smaller than the original Brillouin zone. The energy spread of the small pockets is typically of the order of $t_\perp$. Therefore as long as the smearing $\hbar/\tau_\parallel$ of the FS by the finite electron life time $\tau_\parallel$ is larger than $t_\perp$, we do not expect to observe 3-D Fermi surface characteristics. Consequently, a semimetallic FS may be observed only when the condition

$$\hbar\tau_\parallel^{-1} < t_\perp, \quad (2)$$

is fulfilled.

The condition $kT < t_\perp$ must also be fulfilled, but we expect this latter condition to be less restrictive since at low temperature the growth of $\tau_\parallel$ is limited by residual impurities and kT becomes smaller than $\hbar/\tau_\parallel$. The finding of $t_\perp \approx \varepsilon_F \approx 50$ K from the SdH data shows *a posteriori* that the starting assumption of a Q-1-D Metal-II phase is verified, because unless $t_\parallel$ is « accidentally » diminished significantly under high-pressure, we obtain $t_\perp \ll t_\parallel$.

Other experimental data corroborate the existence of small pockets in the FS of TMTSF-DMTCNQ under pressure and at low temperature.

First, the thermopower at 12 kbar [10] exhibits semimetallic behaviour below 10 K showing in addition that the dominant carriers are holes.

ε_F is related to the Seebeck coefficient S by $S = \frac{\pi^2}{2} \frac{k}{e} \frac{kT}{\varepsilon_F}$ and $S = \frac{\pi^2}{6} \frac{k}{e} \frac{kT}{\varepsilon_F}$ in 3-D and 1-D band models respectively [14], with corresponding $\varepsilon_F(3D) = 300$ K and $\varepsilon_F(1D) = 100$ K. Both values are larger than the estimate 50 K based on the SdH oscillations. This apparent discrepancy is discussed below. Secondly the magnetic susceptibility of TMTSF-DMTCNQ shows strong diamagnetism under pressure at low temperature (below 30 K) with χ_{dia} reaching $-7 \times 10^{-4} \text{ emu/mole}$ at 4.2 K [15]. This behaviour was interpreted as the signature of a small FS giving rise to a strong Landau-Peierls diamagnetism.

The question which arises now is : how can the very few carriers of TMTSF-DMTCNQ explain

the highest conductivity ever found at helium temperature in an organic conductor (besides organic superconductors) ?

The Dingle temperature of 0.8 K gives a fairly long mean scattering time $\tau \sim 3 \times 10^{-12}$ s, about 100 times longer than the optical scattering times obtained from reflectance studies with light polarized along the chain axis. These measurements have not been done for TMTSF-DMTCNQ under pressure, but results are available for compounds exhibiting a similar high dc conductivity at low temperature. For instance $\tau = 10^{-14}$ s and 1.5×10^{-14} s in $(\text{TMTSF})_2\text{PF}_6$ at 25 K [16] and $(\text{TMTSF})_2\text{ClO}_4$ at 30 K [17] respectively.

Since the optical reflectance and the SdH effect refer to different shapes of the FS, the factor 100 is not surprising. The semimetallic carriers have long scattering time (but a much reduced Fermi velocity) and the mean free-path $\lambda = v_F \tau$ does not vary much through the 1-D to 3-D FS cross-over. Following the definition $\sigma = ne^2 \tau / m^*$ the semimetallic conductivity amounts to about $1500 (\Omega\text{cm})^{-1}$, $m^* = 0.5 m_0$, $n = 3 \times 10^{18} \text{ cm}^{-3}$, $\tau = 3 \times 10^{-12}$ s. This value may be underestimated by one order of magnitude or so because τ derived from SdH oscillations is frequently shorter than τ derived from dc conductivity. We are however still far from the experimental conductivity of $3 \times 10^5 (\Omega\text{cm})^{-1}$.

We may consider other carriers, not revealed by oscillatory magnetoresistance, as the source of the high conductivity. However we find this possibility rather unlikely since with the model of band hybridization [4] the density of carriers (electrons) must be similar to the density of holes, i.e. $\approx 3 \times 10^{18} \text{ cm}^{-3}$. Furthermore the former carriers which do not show up in SdH oscillations have presumably a mass larger than $0.5 m_0$. Therefore, unless their relaxation time is accidentally very long $\tau \sim 10^{-9}$ s, they cannot explain the conductivity of $3 \times 10^5 (\Omega\text{cm})^{-1}$.

Consequently, the semimetallic picture which can explain the low temperature state of HMTSF-TCNQ ($\sigma \sim 10^3\text{-}10^4 (\Omega\text{cm})^{-1}$) fails to give a reasonable estimate of the extraordinary large value of the conductivity of TMTSF-DMTCNQ.

In order to explain the intriguing question of extra conduction in TMTSF-DMTCNQ a contribution provided from short range 1-D superconducting pairing has been suggested [10]. This fluctuating channel σ' acts in parallel with the single particle channel σ_0 and the total conductivity becomes

$$\sigma = \sigma' + \sigma_0. \quad (3)$$

The strong sensitivity of the σ' channel to a low concentration of irradiation induced defects has allowed a qualitative evaluation of the contributions [19]. At helium temperature $\sigma'/\sigma_0 \sim 10\text{-}20$ and σ' is dominant in the definition (3) below 30 K.

Therefore, as far as SdH experiments are concerned the paraconductivity σ' could account for the high dc conductivity and its large field dependence, whereas the small hole pockets would provide the small amplitude magnetoresistance oscillations. When the concept of superconducting fluctuations is introduced into the definition of the thermopower a two-fluids model can be used. The thermopower thus reads :

$$S = \frac{\sigma' S' + \sigma_0 S_0}{\sigma' + \sigma_0}. \quad (4)$$

On the one hand the fluctuating part of the thermopower S' is expected to be small if not zero, since no heat is carried by a current of superconducting origin. On the other hand the normal contribution, which follows a linear T -dependence can reach fairly large values in a semimetal with a very small Fermi energy. Consequently equation (4) reduces to :

$$S = \frac{\sigma_0}{\sigma'} S_0, \quad (5)$$

and the Fermi energy derived from the thermopower data drops to $\varepsilon_F \approx 30$ K (with the 3-D approximation) in much better agreement with the SdH results.

The final question, to which this article is addressed, is why no superconductivity is observed in TMTSF-DMTCNQ ?

There are no clear answers to this question, but we may suggest some possibilities :

(i) The phase of TMTSF-DMTCNQ which shows at low temperature the high conductivity behaviour is the Metal-II phase, of which very little is known. This phase may be incommensurate since pressure is known to enhance the band filling in two-chain conductor [20, 21]. It is characteristic that the $(\text{TMTSF})_2\text{X}$ series in which superconductivity is observed, is commensurate. Commensurability seems to be relevant for the stabilization of superconductivity through electron Umklapp scattering [22, 23], thus possible effects of non-commensurability should be considered for the phase Metal-II of TMTSF-DMTCNQ.

(ii) Weak and non-magnetic defects suppress very easily the 3-D ordered superconducting state of Q-1-D superconductors with much less perturbation of the 1-D divergence [24, 25]. Therefore, the possible existence of such defects in the phase II should also be considered.

(iii) In the semimetallic state the density of carriers is very small and the Coulomb interaction is too weakly screened to allow the establishment of superconductivity.

4. Conclusion.

In summary, the present report of well-defined SdH oscillations in an organic conductor have bearing on several key questions regarding its electronic properties. The coupling between molecular orbitals of like and unlike chains transforms the high temperature planar FS into a low temperature semimetallic 3-D surface. Direct observation of the FS surface at low temperature is possible because a highly conducting state is stabilized under pressure. The reason for the suppression of the Peierls state of TMTSF-DMTCNQ under pressure is still an unresolved problem. Horowitz *et al.* [26] have suggested that an enhancement of the transverse coupling under pressure can lead to a suppression of the Peierls transition in a Q-1-D conductor when $t_\perp$ becomes comparable with $t_\parallel$ say $t_\perp \approx (0.2-0.5) t_\parallel$.

We consider this model inapplicable to account for the suppression of the Peierls state of TMTSF-DMTCNQ since the present SdH studies show that the band structure of the Metal-II phase appears to be nearly one-dimensional ($t_\perp/t_\parallel \ll 1$).

With these results in mind we reemphasize that the semimetallic two-dimensional Fermi surface of the $(\text{TMTSF})_2\text{X}$ series is related to the occurrence of magnetic ordering driven by a magnetic field above 50 kOe ($H \nparallel c$) [27].

We also emphasize that in the one chain materials such as $(\text{TMTSF})_2\text{X}$ a closed FS would require a much stronger $t_\perp$. In the present two chains material, the closed FS is a result of inter-band hybridization ; even though $t_\perp$ is very small.

Finally the low density of carriers in TMTSF-DMTCNQ fails to explain the experimental conductivity of $3 \times 10^5 (\Omega\text{cm})^{-1}$ at low temperature ($P = 12$ kbar). The coexistence of a fluctuating superconducting contribution, important below 30 K [28], with a single-particle contribution is not contradictory with the present results.

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