Pressure-induced superconductivity in the spin-Peierls compound $(TMTTF)_2PF_6$

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Abstract: Electrical resistivity experiments on $(TMTTF)_2PF_6$ show that pressure induces the phase sequence spin-Peierls, antiferromagnetism (commensurate and incommensurate spin-density wave state), and superconductivity. This (T, p) phase diagram is generic for the organic salts of the $(TM)_2X$ family. The comparison of experimental and theoretical results supports a common origin for magnetism and superconductivity.

Keywords: 1-D conductor, superconductivity, antiferromagnetic fluctuations

1. INTRODUCTION

The $(TM)_2X$ charge transfer salts have been investigated intensively over the last two decades. This was partially triggered by the observation of the pressure-induced superconductivity in $(TMTSF)_2PF_6$, where TMTSF stands for tetramethyltetraelenafulvalene (Jérome, 1980). In this quasi-1-dimensional system superconductivity emerges after an antiferromagnetic ground state has been destabilized. In the course of the research other members of the $(TM)_2X$ salts (where TM is either TMTSF or its sulphur based analogue TMTTF = tetramethyltetrathio-fulvalene) revealed that the competition between antiferromagnetism and superconductivity is a common feature for the Bechgaard (TM = TMTSF) and Fabre (TM = TMTTF) salts.

The compounds of the $(TM)_2X$ series exhibit a remarkable variety of phases and electronic ground states that are tunable, either by changing the anion X = PF_6, Br, BF_4, ClO_4, AsF_6,...), magnetic field or pressure (for a review see: (Jérome, 1995)). The origin for the different electronic states is strongly related to the crystal structure. It consists of blocks which open the possibility for the charge to delocalize throughout the crystal. Stacking the planar molecules on top of each other gives rise to an overlap of the molecular orbitals being the strongest along the stacks (along the a-direction). This leads to the best electrical conductivity along the stacking axis. The anisotropy of the overlap along the three directions t_a : t_b : t_c is about 100 : 10 : 1, with t_a ranging from 0.15 eV to 0.30 eV.

The diversity of physical properties has been summarized in a (T, p) phase diagram where different members of the $(TM)_2X$ salts have been arranged depending on their ambient and high pressure ground states (Jérome, 1991). The Fabre salts are located on the low pressure side of the phase diagram. In these compounds the molecular stacks can be considered as weakly coupled chains resulting in a charge localization, i.e., the opening of a charge gap. Its value ranges from 100 K to 220 K for X = Br and PF_6, respectively (Creuzet, 1987; Coulon, 1982). Structural properties and the magnetic susceptibility for X = PF_6 are not affected by this spin-charge decoupling. The spin susceptibility is weakly temperature dependent but below 60 K strong spin-Peierls (SP) fluctuations appear which eventually lead to the opening of a gap in the magnetic structure at T_{SP} = 19 K. In $(TMTTF)_2Br$ the long range order establishes already at ambient pressure below the spin-density wave (SDW) temperature $T_{SDW} \approx 13$ K. A moderate pressure of 2.6 GPa stabilizes a superconducting ground state with T_c = 0.8 K (Balicas, 1994). A slightly higher pressure (3.7 GPa) had to be applied to $(TMTTF)_2BF_4$ to induce the superconducting state (T_c = 1.5 K), which has been discovered very recently (Auban-Senzier, 2001). The Bechgaard salts $(TMTSF)_2X$ show metallic behavior at high temperature and most of them exhibit a SDW ground state at ambient pressure. A small applied pressure of 0.8 GPa transforms $(TMTSF)_2PF_6$ into a superconductor with T_c = 0.9 K (Jérome, 1991). The only member of the $(TM)_2X$ family to show superconductivity at ambient pressure is $(TMTSF)_2ClO_4$ (T_c = 2 K) (Bechgaard, 1981). It is located at the right end of the (T, p) phase diagram.

The phase diagram constructed in this way represents a patchwork of ground states observed in various compounds even if they belong to the same family. Although the phase sequence SP, SDW (commensurate and incommensurate), and superconductivity was widely accepted its experimental verification resisted until recently (Jaccard, 2001; Adachi, 2000). To put the idea of a generic phase diagram on solid experimental grounds, $(TMTTF)_2PF_6$ was a good candidate because it is one of a few charge-transfer salts to show a SP ground state. According to the above sketched phase sequence, external hydrostatic pressure should transform the SP ground state into a superconducting phase. This idea received experimental support after the stabilization of the SDW phase has been reported (Moser, 1998). The experiments presented here were motivated by this success. They were performed in a wider pressure range and revealed that the

pressure-induced SDW state can also be suppressed above 4 GPa where a superconducting phase becomes the stable ground state. In this way, the universality of the (*T, p*) phase diagram was established for the Bechgaard and Fabre salts.

2. EXPERIMENTAL DETAILS

The single crystals of $(TMTTF)_2PF_6$ were grown by a galvanostatic electrocrystallization on Pt-electrodes at a constant current of 2 µA. A 15 ml U-shape electrochemical cell containing a 0.1 M solution of tetrabutylamonium hexafluorophosphate (NBu_4PF_6) in dry THF (15 ml) was used as electrolyte and pure TMTTF (Mora, 1992) (13 ml), preliminary recrystallized three times in ACN, was added in the anodic compartment of the cell. For both pressure experiments samples of the same batch were used.

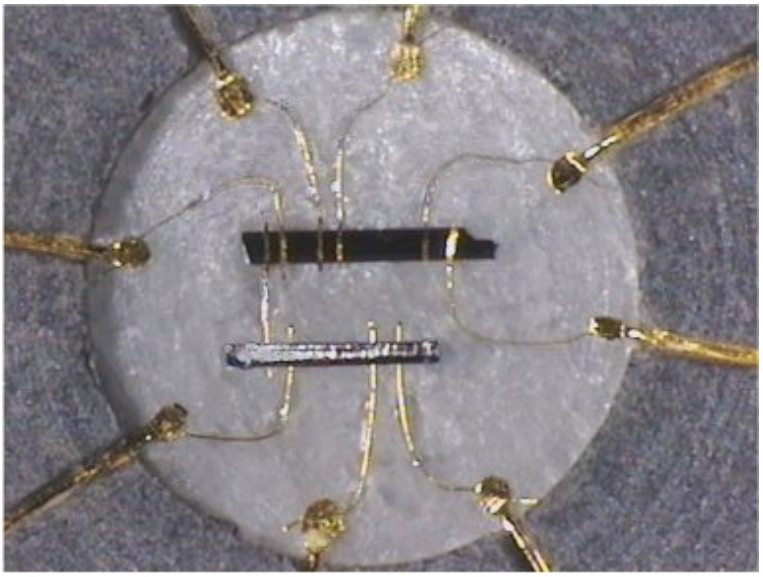

Figure 1: The sample chamber (ϕ = 2 mm) before pressurization. Thin Au wires (ϕ = 5 μm) are attached to the $(TMTTF)_2PF_6$ single crystal (black bar) and the pressure gauge (Pb foil). The cell is closed with a disk of a soft solid pressure transmitting medium (steatite) on top of this arrangement and then pressurized between the two tungsten-carbide anvils.

The pressure dependence of the electrical resistivity was studied with a piston-cylinder double-walled NiCrAl alloy clamped cell capable to reach pressures of $p_{max} \approx 4$ GPa at low temperature with a manganin-gauge pressure calibration performed at room temperature. The sample resistance was measured by the usual four-contact lock-in technique. This cell was located in a dilution refrigerator. For higher pressures a Bridgman anvil cell ($p_{max} \approx 10$ GPa) designed for temperatures as

low as T = 25 mK was used (see Fig. 1) (Jaccard, 1998). The electrical resistance was obtained by the four-point method. The voltage was measured for opposite current directions to cancel out thermoelectrical voltages. The pressure gauge is a thin Pb foil and its pressure dependent superconducting transition temperature yields the pressure. The upper limit for the inevitable, but small pressure gradient is estimated to be about 0.1 GPa. The anvils are enclosed by copper rings to ensure good thermalization of the sample. Thermometers attached on one of these rings were used to determine the sample's temperature. The very low magnetization of the whole device allows the transverse magnetoresistance ($\boldsymbol{B} \,||\, \boldsymbol{c}$) to be measured up to B = 8 T (Wilhelm, 1999).

3. RESULTS

The longitudinal electrical resistivity $\rho_a(T)$ of $(TMTTF)_2PF_6$ studied with the piston-cylinder clamped cell is shown in Fig. 2. Pressure strongly decreases the resistivity at room temperature from 400 mΩcm at ambient pressure (not shown) to ρ_a = 11 mΩcm at p = 4.05 GPa. A similar reduction is observed as a function of temperature: ρ_a decreases monotonously upon cooling to temperatures of the order of 10 K. In this temperature regime, ρ_a passes through a minimum at T_{min} and the upturn in $\rho_a(T)$ is related to a transition into an insulating state, attributed to the onset of itinerant antiferromagnetism at T_{SDW} (Moser, 1998). The temperature T_{SDW} is deduced from the $\rho_a(T)$-curves using either the maximum of $\delta \ln\rho(T)/\delta T$ (for the low pressure curves) or the criterion $\rho_a(T) = 2 \times \rho_a(T_{min})$. The onset temperature T_{SDW} is rapidly suppressed by pressure (inset Fig. 2). The extrapolation of $T_{SDW}(p)$ for $T \rightarrow 0$ would predict the formation of a metallic state at a pressure of about 4.5 GPa.

Access to this pressure region is provided by the Bridgman-type of high pressure cell. The resistivity $\rho(T)$ of the single crystal at p = 3.7 GPa measured with this pressure apparatus shows a similar low temperature behavior as the sample used in the low pressure device. Thus, we are confident that the results of the two experiments match satisfactorily. The strong increase of $\rho(T)$ below 20 K is disrupted by the onset of a sharp drop in resistivity at T_c = 1.8 K and p = 4.35 GPa (see Fig. 3). The residual resistivity ρ_0 = 20 mΩcm, measured at 40 mK, is still high, and is as high as the resistivity at room temperature. The drop in resistivity is even more pronounced at slightly higher pressure (at p = 4.73 GPa) where $\rho(T)$ starts to decline already at T_c = 2.2 K. Upon further pressure increase the temperature T_c as well as the magnitude of the drop in resistivity decrease. The residual resistivity, measured at the lowest temperature reached in each run, amounts to 1-2 mΩcm. Beyond 7 GPa no evidence of a drop in resistivity is found.

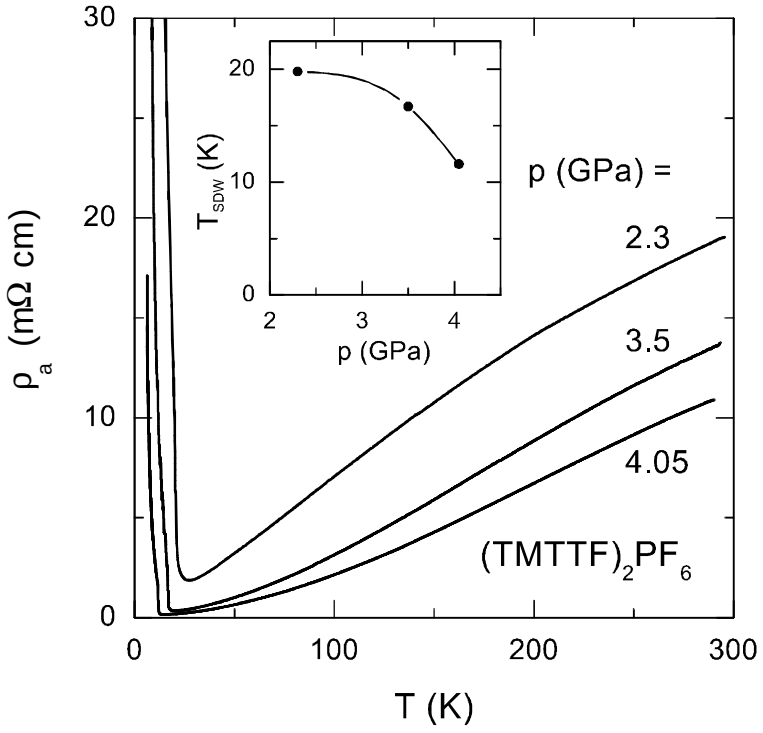

Figure 2. Temperature dependence of the longitudinal resistivity ρ_a obtained in the clamped cell. Inset: pressure dependence of T_{SDW}. Its extrapolation for $T \rightarrow 0$ would predict the formation of a metallic state at a pressure of about 4.5 GPa.

The influence of an external magnetic field along the *c*-axis is shown in the inset of Fig. 3. The drop in resistivity, which occurred at T_c = 2.2 K, is completely suppressed in a field of $\mu_0 H$ = 0.8 T. A strong argument to identify T_c as a superconducting transition temperature is provided by the field dependence of $\rho(T)$. The value of the critical field $H_{c_2}^c$ determined by the recovery of the normal state resistivity, increases together with T_c as pressure decreases. It increases even further after T_c passes its maximum (Jaccard, 2001). The upper critical fields of 1-D organics have been interpreted within the framework of type II superconductors in the clean limit (Gorkov, 1985). The latter assumption is justified in most $(TMTSF)_2X$ salts since the electron mean free path is of the order of $10^4 \times a$, with a the lattice parameter. In this limit, the upper critical field is given by

$$H_{c_2}^c = \frac{A}{t_a t_b} T_c (T_c - T) \qquad (1)$$

where A is a constant independent of the field orientation and pressure. The variation of $dH_{c_2}^c / T_c dT$ between 4.45 and 6.14 GPa leads to a pressure dependence of

20%/GPa for $(t_a t_b)^{1/2}$ which is in fair agreement with the optical measurements of the bare band parameters of organic conductors under pressure (Jérome and Schulz, 1982).

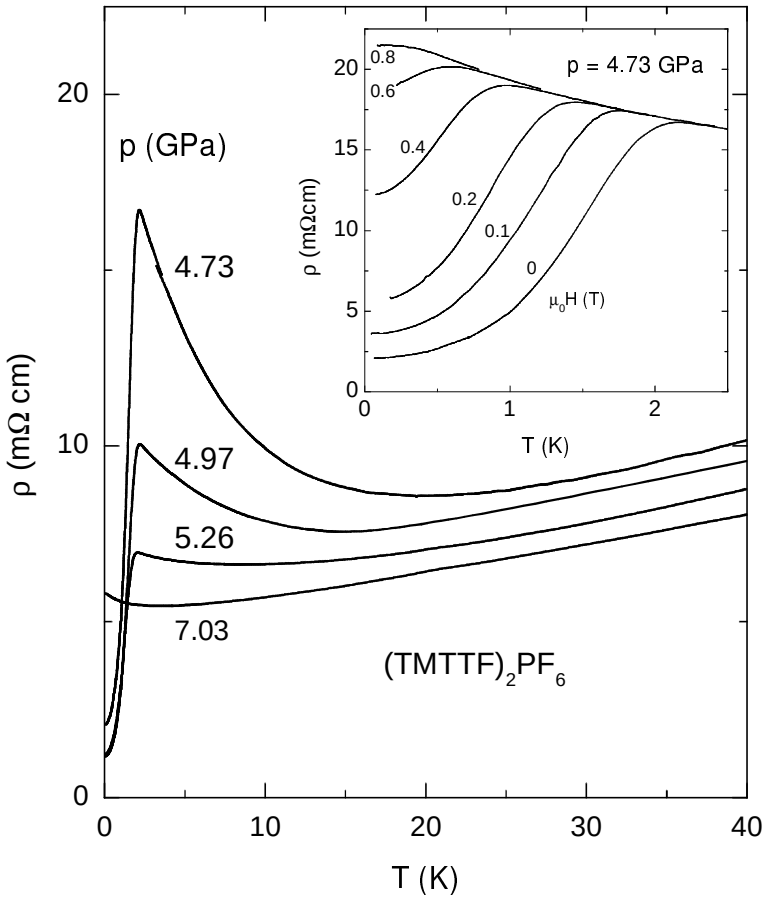

Figure 3. Electrical resistivity ρ(T) of $(TMTTF)_2PF_6$ obtained in a Bridgman-type of high pressure cell. The drop in ρ(T) at low temperature marks the entrance into a superconducting ground state. The influence of a magnetic field on ρ(T) is depicted in the inset. The superconducting phase at 4.73 GPa is suppressed in a field of $\mu_0 H = 0.8$ T.

4. DISCUSSION

The presented data provide the information for the (*T, p*) phase diagram of $(TMTTF)_2PF_6$ to be constructed (see Fig. 4). At low pressure metallic properties are observed at high temperatures. Below the charge transfer gap, where $\rho(T)$ exhibits a

minimum, the charge degrees of freedom are lost. Then for $T < T_{SP}$ also the spin degrees of freedom are lost and the compound enters the SP phase (Moser, 1998). Towards higher pressure the electron-phonon interaction becomes less and less dominant. After the suppression of the SP phase an antiferromagnetic state (commensurate SDW) is present and then the incommensurate SDW phase is stable up to about 4 GPa. Here, the electron-electron interaction remains important. At even higher pressure the system remains metallic below room temperature and enters a superconducting state at low temperature ranging from slightly above 4 GPa to almost 7 GPa. The superconducting transition temperature reaches a maximum value of T_c = 2.2 K at p = 4.73 GPa. It is worth noting that at this pressure the temperature T_{SDW} of the magnetic ordering has decreased sharply.

The shaded region in the M-state (Fig. 4) indicates the presence of antiferromagnetic spin fluctuations. This region has an upper bound in temperature defined by T_{min} and a lower limit given by either T_{SDW} or T_c. In this temperature interval $\rho(T)$ shows an upturn. It can be seen that the width in temperature of this interval increases with decreasing pressure and is largest where $T_c(p)$ reaches its optimum value. Thus, T_{min} appears to be closely linked to the critical temperature T_c. Critical antiferromagnetic fluctuations seem to be enhanced when the SDW ground state is approached from high pressure, i. e., where the system is close to the border between the SDW and superconducting phases. The correlation between the T_c-value and the spin fluctuation regime is taken as a strong experimental argument in favor of a pairing mechanism involving antiferromagnetic fluctuations. At slightly lower pressure the decrease of T_c is clearly related to the occurrence of the SDW phase at a higher temperature. Similar behavior is encountered in the competition between charge density wave and superconducting instabilities (Friedel, 1975). The correlation between the fall of T_{SDW} and the rise in T_c reflects the suppression of the SDW with pressure. This restores areas of the Fermi surface lost by the creation of magnetic gaps, thereby increasing the density of states at the Fermi level and hence T_c.

The universality of this phase diagram for the Bechgaard and Fabre salts becomes obvious if the location of several $(TM)_2X$ members in Fig. 4 is regarded. Therefore, some representative compounds are arranged in the phase diagram (see top axis in Fig. 4) according to their ambient pressure properties. Two assumptions have been made to locate them within a particular phase: (i) In all compounds pressure leads to similar elastic and electronic changes; (ii) The critical pressure for the appearance of superconductivity in $(TMTTF)_2PF_6$ is assumed to be about 4.0 GPa. With these crude assumptions the known pressure to induce superconductivity in these $(TM)_2X$ compounds determines roughly the distance from the superconducting phase $(TMTTF)_2PF_6$. The excellent agreement with the experimental phase diagram is a convincing argument that Fig. 4 represents the generic phase diagram for the $(TM)_2X$ charge-transfer salts.

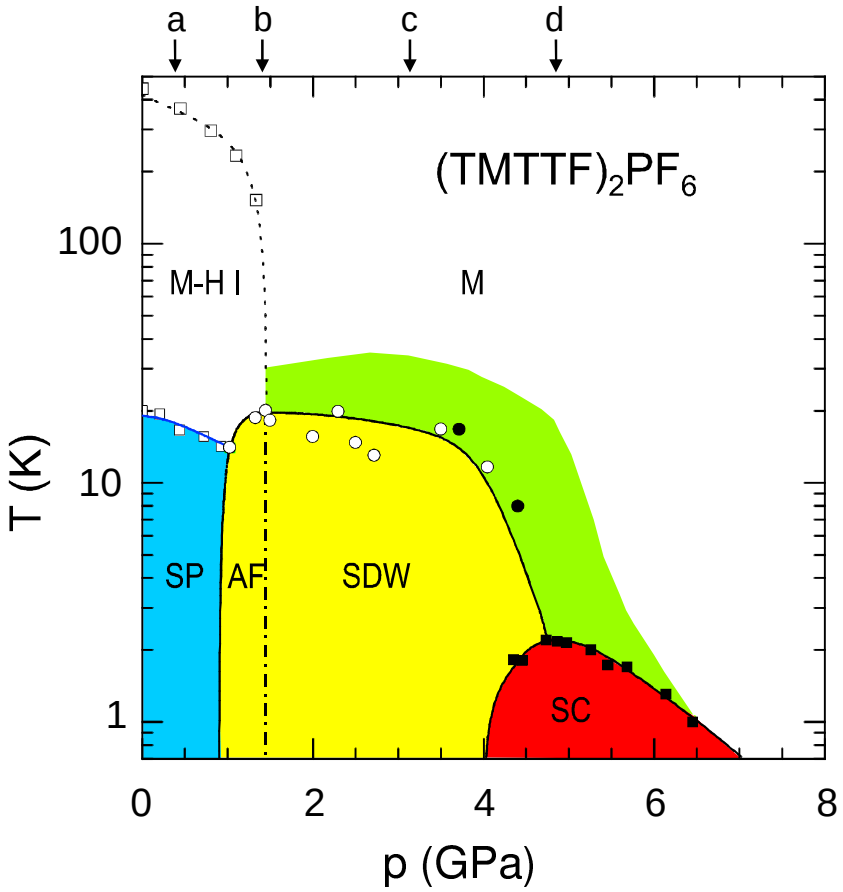

Figure 4. Temperature vs pressure phase diagram of $(TMTTF)_2PF_6$. M-H-I: Mott-Hubbard insulating state, M: metallic, SC: superconducting state, SP: spin-Peierls, AF: antiferromagnetic, and SDW: spin-density wave phase. Open symbols represent data from (Moser, 1998). The characters at the top axis indicate the location of other $(TM)_2X$ compounds according to their ambient pressure ground states: a = $(TMTTF)_2BF_4$, b = $(TMTTF)_2Br$, c = $(TMTSF)_2PF_6$, and d = $(TMTSF)_2ClO_4$ (see text).

The stabilization of superconductivity in $(TMTTF)_2PF_6$ at high pressure provides an interesting opportunity to estimate the strength of the electron-phonon coupling in the sector of the phase diagram where Cooper pairing becomes favorable (Duprat, 2001; Wilhelm, 2001). The SP phase transition in $(TMTTF)_2PF_6$ is found to occur at $T_{SP} \approx 19$ K at ambient pressure (Pouget, 1982; Creuzet, 1987), and it is preceded by 1-D precursors (lattice softening) up to about 60 K, as borne out by X-ray diffuse scattering data (Pouget, 1982). The SP character ascribed to both the lattice softening and the ordered state comes from the fact that at higher temperature, namely below $T_\rho \approx 220$ K (Coulon, 1982) (Fig. 4), $(TMTTF)_2PF_6$ presents a Mott

insulating behavior and that the pseudo-gap (resp. true gap) in the *spin* sector occurs only below $T_{SP}^0 \approx 40$ K << T_ρ (resp. T_{SP}) – as shown by spin susceptibility and NMR data (Wzietek, 1993; Creuzet, 1987; Dumm, 2000). T_{SP}^0 then marks the onset of strong coupling between 'bond' charge-density wave correlations and acoustic phonons of wavevector $2k_F^0$. In the framework of scaling theory (Bourbonnais, 1995; Caron, 1987; Bourbonnais, 1996), both temperatures T_{SP}^0 and T_ρ are connected through the static electron-electron interaction induced by the exchange of $2k_F^0$ acoustic phonons, which is denoted g_{ph} in the expression

$$T_{SP}^0 = c\left|g_{ph}\right|T_\rho \ , \qquad (2)$$

where c is a constant of the order of unity. Given the empirical values for T_{SP}^0 and T_ρ, one finds $g_{ph} \approx -0.15$ (in πv_F units, where v_F is the Fermi velocity) as the bare interaction induced by phonons in $(TMTTF)_2PF_6$ at ambient pressure. Thus, a small ratio for T_{SP}^0/T_ρ is congruent with an electron-phonon interaction that is weak in comparison to the Coulomb repulsion as it can be extracted from NMR data or quantum chemistry calculations (Wzietek, 1993; Castet, 1996).

The relation (2) is also useful to understand another key experimental finding: the suppression of the spin pseudo-gap after a sizable decrease of T_ρ (Fig. 4) – as shown by NMR in $(TMTTF)_2PF_6$ at 1.5 GPa ($T_\rho \approx 75$ K) and $(TMTTF)_2Br$ at ambient pressure ($T_\rho \approx 100$ K) (Wzietek, 1993; Creuzet, 1987). Assuming an increase of the Debye frequency ω_D under pressure that overcomes the one of v_F (or t_a) in $g_{ph} \propto t_a/\omega_D^2$ by taking the reasonable variations $(1/t_a)dt_a/dp \sim (1/\omega_D)d\omega_D/dp \approx$ 15 %/GPa, (Jérome and Schulz, 1982) we arrive at $|g_{ph}| \sim 0.05$ for $(TMTTF)_2PF_6$ at 4.5 GPa, a coupling that is weakly enhanced by charge-density wave correlations according to X-ray experiments in selenide compounds (Pouget, 1982). If this coupling is singled out in perturbation theory an instability of the normal state towards a *s*-wave superconducting ordering will occur at $T_c \sim \omega_D \exp(-1/|g_{ph}|) \sim 100 \exp(-20)$ K, which is vanishingly small. The prognostication is even worse for inter-chain pairing induced by phonons since the relevant coupling $g_{ph}^\perp \approx (t_\perp/t_a)^2 g_{ph}$ is reduced by a factor 100.

The coupling of electrons to $2k_F^0$ acoustic phonons, though singularly enhanced on the SP side by strong electronic correlations in the vertex part (Caron, 1987; Bourbonnais, 1996), becomes quickly non singular as the sequence of states unfolds under pressure – as confirmed by X-ray diffuse scattering experiments showing very weak, nearly inexistent, lattice precursors in the normal phase of $(TMTSF)_2X$ (Pouget, 1982; Pouget, 1996). It is worthwhile to mention, however, that a recent re-examination of the X-ray data revealed that charge-density wave and SDW ordering do coexist in few systems, $(TMTSF)_2PF_6$ being one example at

ambient pressure (Pouget, 1996; Pouget, 2000; Kagoshima, 1999). Numerical simulations on 1-D models indicate that coupling of electrons to phonons would be involved in this coexistence (Mazumdar, 1999; Riera, 2000), though purely electronic models have been proposed (Ogata, 1999). On experimental grounds this charge-density wave instability is apparently not the result of a soft mode mechanism that can lead to a strong renormalization of the electron-phonon matrix element in the normal phase (Pouget, 1996).

A conventional electron-phonon mechanism seems not appropriate to explain the pressure-induced superconductivity in $(TMTTF)_2PF_6$ at temperatures of the order of 2 K and therefore other approaches have to be considered. The correlation between the T_c-value and the width in temperature of the (insulating) spin-fluctuation regime is taken as a strong experimental argument in favor of a pairing mechanism involving antiferromagnetic fluctuations (Duprat, 2001). Furthermore, the maximum value of T_c is about twice as large in $(TMTTF)_2PF_6$ as in the parent selenium compound (Jérome and Schulz, 1982) which is in agreement with the larger antiferromagnetic fluctuation regime observed in our investigation. This supports the idea that antiferromagnetic fluctuations are involved in the microscopic pairing mechanism similar to the scenario proposed for Ce- and U-based strongly correlated electron multiband systems (Julian, 1996; Mathur, 1998; Jourdan, 1999).

SDW fluctuations have been considered by Emery (Emery, 1986) as another possibility to form bound states of charge carriers. This idea was worked out in the context of nearly antiferromagnetic itinerant fermion systems (Béal-Monod, 1986). As far as 1-D organic conductors are concerned, it was shown that the exchange of SDW fluctuations between carriers belonging to the same stack does not lead to attractive pairing and thus, the development of a 1-D attractive pairing appears to be hopeless. Additionally, in 1-D systems the electron-phonon coupling is opposed to the Coulomb repulsion of carriers moving in a restricted phase space.

In a conventional approach, using the spin fluctuation exchange model in quasi-1-D organic superconductors, *d*-wave pairing in the vicinity of the SDW phase has been predicted (Kino, 1999). This theory, however, does not take fully into account the entire temperature regime and in particular the non-Fermi-liquid features, observed in DC transport (Moser, 1998; Danner, 1994) and optical conductivity (Vescoli, 1998) at high temperature, which persist down to low temperature. An attractive inter-stack pairing can be the outcome of the exchange of antiferromagnetic spin fluctuations between electrons located on neighboring stacks (Bourbonnais, 1988; Duprat, 2001). This would lead to an anisotropic superconducting gap. An inescapable prediction of scaling theory is that within the framework of the low-energy quasi-1-D model, singlet superconductivity is by far the most stable pairing state SDW fluctuations can stabilize giving in turn support for such an order parameter. Triplet pairing – which has been recently considered on both experimental and theoretical basis as a possible candidate for the superconducting state in the Bechgaard salts (Lee, 2000; Gorkov, 1985; Belin, 1997;

Lee, 1997; Lebed, 1986; Dupuis, 1993; Lebed, 1999) – does exist in the present model between electrons on *next* to nearest-neighbor chains, but its enhancement is vanishingly small compared to singlet pairing (Duprat, 2001). In a model for superconductivity, based on an inter-chain attractive pairing, the existence of lines of nodes in the superconducting gap should be a natural outcome. Consequently, low lying quasi-particle states would govern the thermodynamical properties of the superconducting phase. Experiments such as thermal conductivity, sound attenuation or nuclear spin lattice relaxation should be able to probe this assumption. These measurements should however, be performed on a compound such as $(TMTSF)_2PF_6$ in which no additional anion ordering is likely to spoil the quasi-1-D Fermi surface at low temperature (Bourbonnais, 1999; Belin, 1997).

5. CONCLUSION

External pressure stabilizes a superconducting phase in $(TMTTF)_2PF_6$ in the pressure range 4.3 GPa < p < 7 GPa *in spite* of its spin-Peierls ground state at ambient pressure. This important result establishes the universality of the $(TM)_2X$ phase diagram with a single compound spanning all possible ground states of the $(TM)_2X$ series. Furthermore, the suppression of the SP ground state makes a phonon-mediated Cooper-pair formation unlikely to explain the existence of superconductivity at temperatures as high as T_c = 2.2 K. The manifestation of critical SDW fluctuations above the onset of superconductivity and the close connection between their amplitude and the value of T_c speaks strongly in favor of an inter-stack pairing mechanism mediated by the exchange of these fluctuations between neighboring stacks. Thus, our findings supply an important input for theoretical models of magnetic coupling in quasi-1-D conductors and may even shed light on superconductivity in strongly correlated electron systems including high-temperature superconductors.

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