Superconductivity in the charge-density-wave state of the organic metal α-(BEDT-TTF)$_2$KHg(SCN)$_4$

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The superconducting transition in the layered organic compound α-(BEDT-TTF)$_2$KHg(SCN)$_4$ has been studied in the two hydrostatic pressure regimes where a charge-density wave is either present or completely suppressed. Within the charge-density-wave state the experimental results reveal a network of weakly coupled superconducting regions. This is especially seen in a strong enhancement of the measured critical field and the corresponding positive curvature of its temperature dependence. Further, it is shown that on lowering the pressure into the density-wave state traces of a superconducting phase already start to appear at a much higher temperature.

I. INTRODUCTION

The organic metal α-(BEDT-TTF)$_2$KHg(SCN)$_4$ has already raised great attention due to a variety of physical phenomena found in its low-temperature charge-density-wave (CDW) state.[1–4] Of particular interest have been, for example, modulated CDW states existing in magnetic fields above the paramagnetic limit[5–8] and phase transitions induced by high magnetic fields due to a specific interplay between the Pauli paramagnetic and orbital effects.[7,9] Apart from the high-field phenomena there are other interesting properties, such as the coexistence/competition of CDW and superconductivity which have not been thoroughly addressed so far.

Owing to a strongly anisotropic electron system, the Fermi surface (FS) of this compound consists of coexisting open sheets and cylinders.[10,11] The slightly warped sheets correspond to a quasi-one-dimensional (Q1D) electron band. The latter emerges due to an enhanced electron transfer integral t_a in the crystallographic **a** direction between the organic BEDT-TTF molecules resulting in a chainlike coupling within the conducting **a**-**c** plane.[11] At about 8 K there is a phase transition to the CDW state,[5,6,12,13] in which these sheets of the FS become nested and the Q1D carriers are gapped. The system, however, keeps its metallic character due to the second, quasi-two-dimensional (Q2D) band.

Remarkably, the iso-structural salt α-(BEDT-TTF)$_2$NH$_4$Hg(SCN)$_4$ (hereafter we refer to both compounds as NH$_4$ and K salt) does not undergo the density wave transition but instead becomes superconducting (SC) at ≈ 1 K.[14,15] The absence of a density wave is interpreted to be due to a higher inter- to intrachain-coupling ratio t_c/t_a of the organic molecules within the layers, that strongly deteriorates the nesting conditions of the open sheets of the FS.[16,17] Moreover, it has been shown[17] that by tuning the ratio of the lattice constants c/a under uniaxial strain a density wave can be even (i) induced in the NH$_4$ salt and (ii) suppressed in the K salt, a SC state being stabilized at ≈ 1 K. Based on combined uniaxial strain measurements and band structure calculations Kondo *et al.*[16] have proposed that the major contri-

bution to superconductivity comes from the Q1D band.

Similarly, hydrostatic pressure turns out to worsen the nesting conditions in the K salt.[9] The increase of the interchain coupling leads to a decrease of the density wave transition temperature, and at the pressure $P_c \approx 2.5$ kbar the density wave is completely suppressed, a normal metallic (NM) state being stabilized.[9] Hydrostatic pressure studies[18] have also revealed superconductivity in the K salt but at temperatures much lower than it was observed in the uniaxial strain experiments. Remarkably, the superconductivity was shown to persist over the whole pressure range studied, from 0 up to 4 kbar, i.e., it exists both in the NM and in the CDW regimes. This offers a direct opportunity to study the influence of a CDW on a SC system.

Basically, the SC pairing competes with the density-wave instability for the FS.[19,20] Therefore one would expect the SC transition to be suppressed upon entering the CDW region of the phase diagram since the Q1D carriers, which are supposed to be responsible for superconductivity,[16] become completely gapped below P_c. On the other hand, it was predicted recently[21,22] that density-wave fluctuations can even stimulate the SC pairing in the vicinity of the CDW ground state.

In this paper we present experimental studies of the SC transition in the K salt at different pressures, temperatures, and magnetic fields. We argue that below the critical pressure P_c the SC phase exists in the form of an array of weakly coupled small SC regions or filaments embedded in the metallic CDW matrix. Moreover, we show that the SC onset temperature becomes drastically enhanced on lowering the pressure across the CDW/NM boundary which is likely a sign of a nontrivial effect of the CDW on the superconductivity in this compound.

II. EXPERIMENT

The main results presented in the paper were obtained from interlayer resistance measurements using the standard four probe geometry and a.c. measuring technique. Two samples, hereafter referred to as samples 1 and 2, were mea-

sured simultaneously. The samples had the dimensions of $\sim 0.6 \times 0.5 \times 0.2$ mm^3 and $1.0 \times 0.3 \times 0.05$ mm^3, respectively, the smallest dimension being in the interlayer direction. Additionally, measurements with the current applied along the biggest dimension, i.e., nominally parallel to the layers, were done on sample 2. Of course, due to the extremely high anisotropy of our compound this measured "in-plane" resistance includes a mixture of the intra- and interlayer components of the resistivity tensor.[23] To minimize the influence of the interlayer component the thinnest sample was chosen. The in- and interplane resistances were measured in the same run by using the standard six-probe geometry (four contacts were made to one of the biggest surfaces of the platelike sample and two contacts to the opposite surface). Thus, after comparing the measured in- and interplane resistances we were able to make reasonable conclusions about the temperature dependence of the intralayer resistivity.

Hydrostatic pressure was applied using a conventional berillium-copper clamp cell. The latter was mounted on a dilution refrigerator allowing the sample to be cooled down to 20 mK. The pressure value at low temperatures was determined from the resistance of a calibrated manganin coil to an accuracy better than ±100 bar.

At the lowest temperatures, special care was taken to control and minimize overheating due to the transport current and field-sweep induced eddy currents. On measuring the interlayer resistance with the applied current of 50 nA the overheating of the sample was found to be <5 mK at 20 mK. The sweep rates of the magnetic field were chosen extremely low, ≈ 1 mT/min, so that eddy currents had no visible effect on the sample temperature.

Further, since the SC properties are extremely sensitive to magnetic fields, the superconducting magnet used in the experiment was always carefully demagnetized before the measurements, so that the remanent field was below 0.5 mT.

III. RESULTS AND DISCUSSION

A. Resistive SC transition at zero field

In Fig. 1 several temperature sweeps of the interlayer resistance for sample 1 measured at different pressures show the already reported behavior.[18] At $P = 3$ kbar the resistance exhibits a normal metallic behavior on cooling until at 110 mK a sharp SC transition ($\Delta T \approx 10$ mK) occurs. Above $P_c \approx 2.5$ kbar the SC transition remains sharp and the critical temperature T_c, defined as the midpoint of the transition, shows a negative pressure dependence of about -30 mK/kbar.[18] This value is 1–2 orders of magnitude lower than measured in other BEDT-TTF-based superconductors, where a strong linear suppression of superconductivity with hydrostatic pressure is commonly observed.[19,24]

Kondo *et al.*[16] performed uniaxial strain experiments on the NH$_4$ salt, with a combined x-ray determination of the lattice parameters. Their tight binding band structure calculation proposed the changes of the SC transition temperature to be reasonably described by the changing density of states (DOS) at the Fermi level within the BCS model. However, they mention that such a simple description fails as one ap-
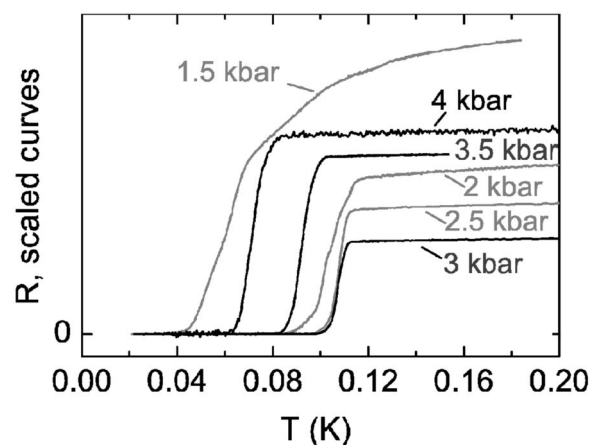

FIG. 1. Temperature sweeps of the interlayer resistance of sample 1 at different pressures. For clarity, each curve has a different resistance scale. At $P > 2.5$ kbar, there are sharp transitions from the NM state to superconductivity. Within the CDW state, $P \lesssim 2.5$ kbar, the superconducting transitions are broadened and the zero-resistance temperature decreases.

proaches the density wave state. Under hydrostatic pressure, the pressure dependence of T_c in the K compound is found to be an order of magnitude lower than observed[25] in the NH$_4$ salt. This is quite unusual: Normally isostructural organic superconductors with different anion layers display approximately the same pressure dependence of T_c.[19] Thus, also in the hydrostatic pressure case the proximity to the density-wave instability in the K-salt seems to affect the SC transition in the metallic state. A direct comparison of the SC properties between the two compounds may, therefore, be inappropriate. Indeed, the value $dT_c/dP = -30$ mK/kbar is closer to that observed in the Q1D TMTSF (or TMTTF) based organic metals in which the SC state exists in the hydrostatic pressure range right next to the spin-density-wave state.[19] Obviously, in the vicinity of a density-wave transition a detailed consideration of different carrier interactions, due to which different instabilities of the metallic ground state compete with each other, becomes necessary.

As mentioned in the Introduction, on entering the CDW state, i.e., with lowering the pressure below P_c, the superconductivity does not vanish. At 2.5 kbar T_c remains at the value observed at 3 kbar, instead of further increasing, as would be expected from an extrapolation from higher P. With further decreasing the pressure, the transition broadens and gets a kind of a steplike structure as can be seen in Fig. 1. This leads to a strong suppression of the temperature T_0 at which zero resistance is reached; at ambient pressure the resistance does not vanish down to 20 mK. Thus, there is a clear effect of the CDW on the resistive SC transition. We note that the observed data are also very well in line with the former proposal[9] of 2.5 kbar being about the critical pressure P_c for the complete suppression of the CDW state.

The overall behavior described above was also observed on sample 2, measured simultaneously. The superconducting transition temperature, however, appears to be sample dependent. The difference between the resistively measured transition temperatures of samples 1 and 2 is approximately 10%
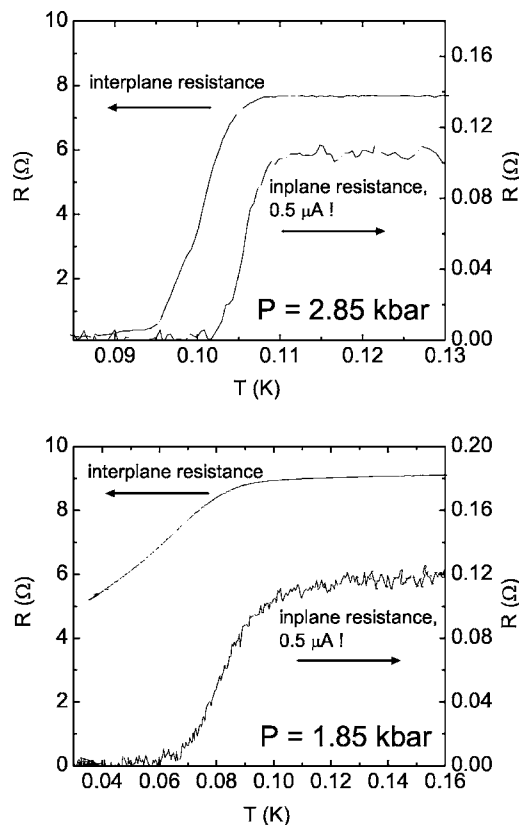
FIG. 2. Comparison of the in- and interplane resistances of sample 2 at pressures above (upper graph) and below (lower graph) the critical value P_c=2.5 kbar.

FIG. 3. Temperature dependence of the interlayer resistance at ambient pressure for three different samples.

at $P>2.5$ kbar, and becomes even stronger in the CDW state, at $P \lesssim 2.5$ kbar. This suggests the impact of the CDW on the superconductivity to be also dependent on impurities or defects.

In Fig. 2 a comparison between the in- and interplane resistances is shown for sample 2 at pressures above and below P_c. Note that in order to measure the inplane resistance to a reasonable accuracy the applied current had to be at least 0.5 μA. However, despite this high current, that caused a small, $\sim$1–2 mK, overheating at the transition temperature, it is seen that the SC transition in the plane occurs at a higher temperature in comparison to the interlayer one. This difference in the transition temperatures originates most likely from the layered character of superconductivity: The SC ordering is first established within the layers whereas the interlayer coherence develops at lower temperatures. Such a scenario has also been proposed for the NH$_4$-compound,[15] where the interlayer coherence length $\xi_\perp$ is found to be smaller than the interlayer spacing of 20 Å.[19] This can also be assumed for the K salt, since, although T_c is here an order of magnitude lower, the in- to interplane anisotropy of the Fermi velocity is considerably higher than the one in the NH$_4$ compound.[26]

At 1.85 kbar the inplane resistance is zero below 50–60 mK whereas the interlayer transition does not vanish down to the lowest temperature. A clear broadening of the inplane transition within the CDW state is, however, also observed. We therefore presume that the evolution, with
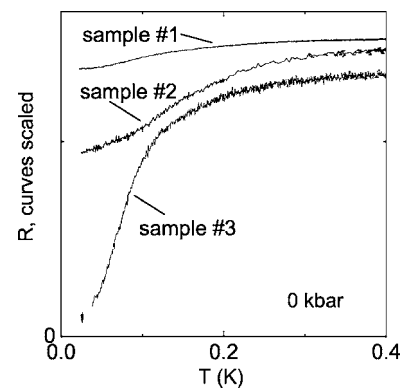
pressure, of the SC transition in the intralayer resistance is similar to that described above for the interlayer resistance. This is supported by the previous report by Ito *et al.*[27] on the incomplete transition in the inplane resistance at ambient pressure.

We now discuss a possible reason for broadening the SC transition. First, we note that for all measured samples the transition width is maximum at zero pressure and decreases as the pressure is increased until the critical value P_c is reached; at $P>P_c$ the transition width is relatively small and approximately constant, $\Delta T_c \approx 10$ mK. Thus, the broadening cannot be ascribed to pressure inhomogeneity. Generally one can think of phase fluctuations, typical of highly anisotropic electron systems with small superfluid density, that leads to a suppression of the bulk superconductivity[28] as has been observed in high T_c superconductors.[29] However, in our system the SC transition temperature is of the order of 100 mK. In this case, the zero-temperature phase stiffness of superconductivity is high enough, so that effects of phase fluctuations on T_c are negligible.[28]

A clue to finding the real nature of the strongly broadened resistive transition lies in a comparison of transport and magnetization measurements. In Fig. 3 we show the temperature dependence of the interlayer resistance for three different samples at ambient pressure. As can be seen, sample 3 almost reaches zero resistance on cooling down to 20 mK, reflecting the already mentioned sample dependence of the SC transition.[18] This, however, does not mean that the whole sample at lower temperatures is in the SC state. D.C. magnetization measurements on the same sample made on a superconducting quantum interference device (SQUID) magnetometer could not resolve any Meissner effect, even down to 6 mK. Therefore, the zero resistance most likely originates from a network of weakly coupled SC regions or filaments. Thus, we suggest that the SC and CDW phases are separated in space. This is also supported by theoretical predictions that a CDW leads to a suppression of superconductivity.[20] We consider an inhomogeneous system of SC islands embedded in a metallic (actually CDW) matrix to be more likely. The SC coherence, thus, develops within the islands until at lower temperatures they couple to each other via the proximity effect, providing a percolation network. At ambient pressure the islands are strongly separated, so that a com-
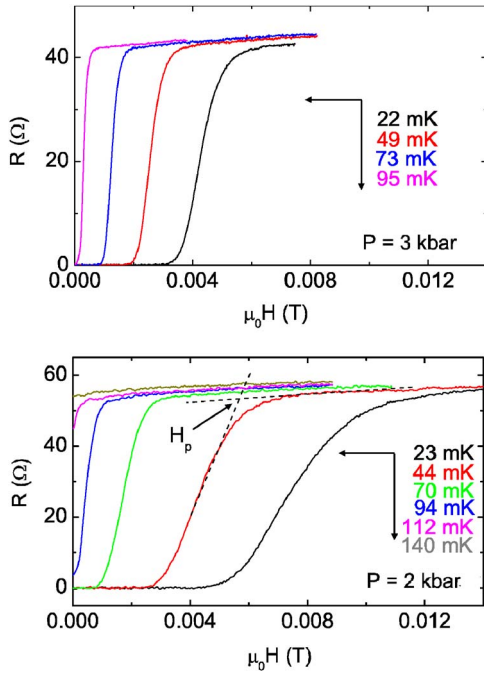
FIG. 6. Critical fields at $P=2$ kbar (solid symbols) and $P=3.5$ kbar (open symbols) determined from the resistive transition in the field sweeps using different criteria. The inset shows how the criteria are chosen: the onset (squares), the inflection point (circles), and the end of the SC transition where the resistance is $\approx 10\%$ of the normal metallic value (triangles).

FIG. 4. (Color online) Field dependence of the interlayer resistance of sample 1 at various constant temperatures for pressures above and below P_c.

pletely coupled system does not exist at $T>20$ mK. A strong broadening of the "bulk" SC transition is indeed known to exist in a two-dimensional array of SC islands which are embedded in a metallic matrix.[30,31] After the islands become SC the decrease of the resistance is determined by the growth of the normal metallic coherence length on lowering the temperature, i.e., the proximity effect. Since we have no possibility at the moment to study the magnetization under pressure, we cannot directly verify the absence of the Meissner effect. However, as we shall see next, the inhomogeneous nature of superconductivity under hydrostatic pressure is supported by measurements of the SC transition in magnetic fields.

B. Magnetic field effect

In Fig. 4 we show the magnetic field sweeps made on
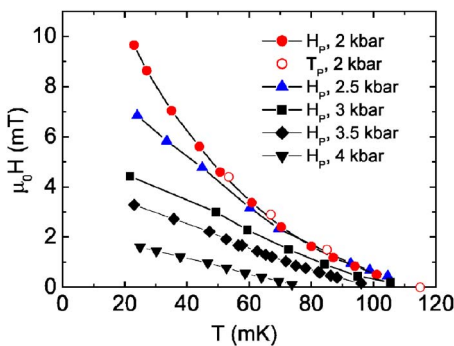

FIG. 5. (Color online) Critical fields and temperatures determined at pressures around the critical value $P_c=2.5$ kbar. Filled symbols are obtained from field sweeps, see Fig. 4, and open circles from temperature sweeps, see Fig. 7.
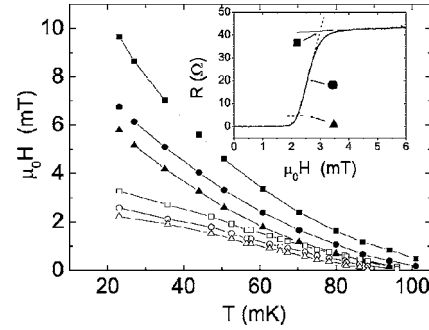
sample 1, with the field directed perpendicular to the planes, at different temperatures and two pressures, above and below the critical value P_c. At zero field the transition temperature at these two pressures is approximately the same (see Fig. 1). While at 3 kbar the transitions remain relatively sharp over the whole temperature range, at 2 kbar they become somewhat broadened at lower T. The critical fields H_p determined as shown in the lower panel of Fig. 4 are plotted in Fig. 5 for five different pressures. At $P \geqslant 3$ kbar the critical field displays a nearly linear dependence on temperature that can be expected for coupled SC planes in the 3D limit.[29] On entering the CDW state, H_p at low temperatures becomes dramatically enhanced, leading to a pronounced positive curvature of its temperature dependence as seen in the 2 kbar curve. It is important to note that this behavior does not depend on the way we determine H_p. To illustrate this, Fig. 6 shows the critical fields obtained by three different methods for two different pressures, above and below P_c. Obviously, all criteria lead to the same qualitative behavior.

In principle, the positive curvature of H_p might be related to the melting of the superconducting vortex lattice. However, this can be ruled out by looking at the temperature dependence of the interlayer resistance at different constant magnetic fields that is shown in Fig. 7. The fact that at 2 kbar the resistive transition in the temperature sweep does not
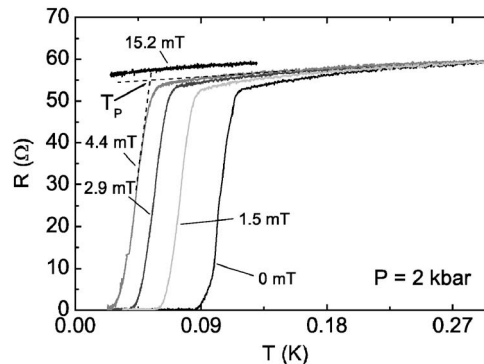

FIG. 7. Temperature dependent interlayer resistance at different constant magnetic fields, at $P=2$ kbar.

broaden with applying a magnetic field rules out any considerable flux flow effect. It would, anyway, be surprising if the vortex motion were important in a material with such a low T_c.

The upper critical field H_{c2} is generally known to be enhanced in a superconductor if at least one of the dimensions perpendicular to the field direction becomes less than the coherence length.[29] A dimensional crossover with lowering T then also leads to a strong positive curvature of the upper critical field. A similar scenario might also occur in our compound. This means that there is a possibility that the size of the superconducting regions within the plane becomes less than the coherence length. However, the field H_p determined from the resistive transition does not necessarily match the upper critical field H_{c2} at $P < P_c$. As was argued above, the superconductivity is most likely inhomogeneous in this pressure range. Therefore, the resistive transition may be largely determined by the coupling between the randomly separated SC islands rather than by H_{c2} inside the islands. This means that not only the value of H_p defined above can differ from the real H_{c2} but also its temperature dependence. Although an exact theoretical description of the resistive transition of a proximity coupled random array of SC islands in a magnetic field still has to be worked out, a comparison to existing inhomogeneous superconductors shows that a strong positive curvature of H_p can be expected.

As an example one can mention polymeric sulfur nitride $(SN)_x$, a compound that consists of bundles of SC filaments. For a magnetic field applied perpendicular to the fiber axis the temperature dependence of the resistive transition was shown to exhibit a positive curvature.[32] Another, and probably more relevant example is the well-known CDW compound $NbSe_3$. It has been reported[33] that within the CDW state of $NbSe_3$ a small fraction of the sample becomes SC and it has been proposed to emerge within the boundaries of CDW domain walls, where the CDW order parameter is supposed to become zero. This would then indeed be a system of SC regions separated by the metallic CDW phase similar to our present case. At higher pressures the CDW gap becomes smaller and the domain wall fraction, where ungapped Q1D electrons exist, is expected to become bigger. Moreover, a strong sample dependence of the SC properties would not be surprising in such a model, since crystal defects or impurities very likely affect the domain structure. Whether such a domain structure really exists in the title compound we cannot judge from our data, but the similarities between both compounds with respect to their SC properties suggest the nature of the critical field behavior to be the same. The possibility of domains within a Q1D CDW system has indeed been predicted.[34] Furthermore, Gor'kov and Lebed[34] and Gor'kov and Grigoriev[35] mention that the superconductivity would be expected to survive in the domain walls perpendicular to the conducting chain direction.

Noteworthy, there might exist a narrow pressure region in the vicinity of P_c, in which the system becomes inhomogeneous, irrespective of the CDW domain structure.[36] Such an inhomogeneous system, associated with a first-order phase transition, was also shown to have an enhanced SC upper critical field[37] in the spin density wave compound $(TMTSF)_2PF_6$.
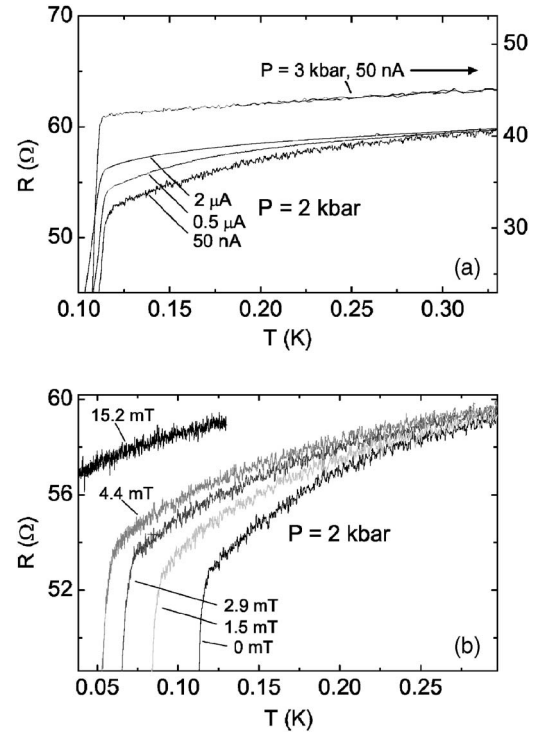

FIG. 8. Within the CDW state ($P=2$ kbar) the decrease of the interlayer resistance accelerates at much higher temperatures than in the NM state ($P=3$ kbar). This decrease strongly depends on the level of the applied current (a) and magnetic field perpendicular to the layers (b).

C. Enhanced SC onset temperature

Besides the broadening of the main SC transition, all temperature sweeps at pressures ≤ 2.5 kbar show an unusually strong decrease (negative curvature) of the resistance in a remarkably wide temperature range well above the T_c value that would be expected from its linear extrapolation from $P > P_c$. Figure 8 shows, in an enlarged scale, the resistance of sample 1 at 2 kbar, at temperatures right above the main transition, which is still rather sharp at this pressure. For comparison, the 3 kbar resistance is also shown in the upper panel. As can be seen from the figure, the decrease of the resistance strongly depends on the level of the applied current and field. With increasing the current or field the resistance decrease becomes suppressed. Note that the main transition shifts only slightly at higher currents in Fig. 8. Therefore, effects of overheating can be neglected.

The present data manifest that traces of superconductivity, occupying a small fraction of the crystal volume, exist already at much higher temperatures. The described behavior was found throughout the entire CDW pressure range. The onset temperature of superconductivity is ≈ 0.22 K at 2.5 kbar and 0.30 K at 2 and 0 kbar. These findings were reproduced on several samples. They are also consistent with the ambient pressure results of Ito *et al.*[27]

By contrast to the CDW pressure region, in the NM state such an accelerated decrease of the resistance above the bulk SC transition has not been detected (see the 3 kbar curve in Fig. 8). Hence, we conclude that the dramatic increase of the
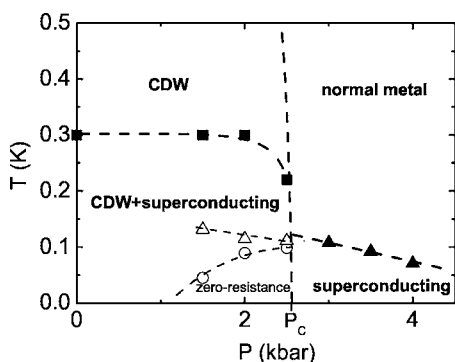
FIG. 9. Proposed *P-T* phase diagram. Filled symbols show the phase transitions between different states. Open symbols mark the onset and zero-resistance temperatures of the broadened main SC transition in the CDW state. The lines are guides for the eye.

SC onset temperature is a consequence of entering the CDW region of the phase diagram. The whole *P-T* phase diagram including all phases must, therefore, look as depicted in Fig. 9. Since the SC transition is sharp above P_c but becomes broadened in the CDW region, we take here the midpoint for the NM/SC transition (filled triangles in Fig. 9) and the onset and zero-resistance temperatures for the main SC transition in the CDW state (open triangles and circles, respectively). The onset temperature of small SC regions in the CDW state (filled squares) is determined by the inflection point in the temperature dependent resistance. Obviously, there is an extended range in the *P-T* phase diagram that includes both ground states, superconductivity, and density wave.

If the superconductivity is indeed spatially restricted to the CDW domain boundaries, as suggested above, one can understand why the CDW does not completely suppress the SC state, in contradiction to what has been theoretically proposed.[20] This will, however, not explain the enhanced SC

onset temperature. In principle, in the model above one would still expect the opposite effect, namely, that the SC island has a reduced onset temperature due to the proximity effect. On the other hand, we do not know in what way the superconductivity, located in the domain boundaries where the order parameter of the density wave reaches zero, is influenced by the CDW neighborhood. An interesting scenario to consider would be an additional stimulation of superconductivity in the CDW domain walls, such as, for example, a charge-fluctuation mediated pairing.[21,22,38] More investigations on this topic are highly desirable.

IV. CONCLUSION

In conclusion, pronounced differences in the superconducting properties are observed between the CDW and the NM pressure regions. The determined phase diagram further confirms that $P_c \approx 2.5$ kbar is the critical pressure at which the CDW state becomes completely suppressed. Below P_c, the broadening of the resistive SC transitions, the absence of the Meissner effect, as well as the pronounced enhancement and positive curvature of the critical magnetic field point to the formation of a network of coupled SC regions embedded in the CDW matrix. We propose that the superconductivity is located within CDW domain walls. Furthermore, it is found that traces of a SC phase exist in the CDW region already at temperatures much higher than expected from the NM state. The origin of this remarkable and unexpected expansion of the SC temperature range remains at present one of the most intriguing questions.

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