Thermopower and specific heat of the organic molecular salt $(TMTSF)_2ClO_4$: observation of the narrow band response

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Abstract
Measurements of thermopower $S_a(T)$ along the highly conducting a axis and specific heat of the Bechgaard salts (TMTSF)$_2$ClO$_4$ for various cooling rates through the anion ordering temperature $T_a = 24$ K were carried out. Sign reversal in $S_a(T)$ is found below T_a and it decreases with increasing cooling rate, which is attributed to the change of a narrow band filling level as the temperature and the cooling rates change. The crossover from 2D to 3D in $S_a(T)$ is observed around 15 K. The onset temperature of anion ordering in $S_a(T)$ decreases from 29.8 to 24.2 K as the cooling rate increases. Meanwhile, the electronic specific heat coefficient γ has a pronounced change within this temperature region, giving strong evidence for a narrow band contribution. The difference in the specific heat between the quenched and relaxed states follows a T-cubic law from 5 to 24 K, implying a lattice distortion by the ordered anion only. The entropy estimated from the specific heat peak between 28 and 15 K is $R \ln(4/3)$ lower than the value $R \ln 2$, consistent with the thermopower result that some anions have been ordered far above T_a for the relaxed state.

1. Introduction

The normal state properties of low-dimensional conductors have attracted much attention because electron–electron interactions in these materials lead to a complete breakdown of the usual Fermi liquid (FL) picture involving well-defined electron- or hole-like quasiparticle excitations [1, 2]. In contrast, concepts such as a Tomonaga–Luttinger liquid (TLL), in which interactions play a crucial role [3], or a marginal Fermi liquid are expected to be more appropriate for one- and two-dimensional (1D and 2D) materials, respectively. In a Luttinger liquid the electrons combine to form separate spin and charge excitations, with different velocities, while in a marginal Fermi liquid the quasiparticle relaxation rate is comparable to its energy. In such cases the physical properties are expected to obey certain characteristic non-Fermi liquid power laws as a function of temperature and

frequency. For example, the unusual normal state pseudogap may well be a manifestation of non-Fermi liquid behavior in quasi-2D superconducting cuprates [4]. At the same time, in 1D interacting electron systems (TMTSF)$_2$X, where TMTSF denotes tetramethyltetraselenafulvalene and X = PF$_6^{-1}$, AsF$_6^{-1}$ or ClO$_4^{-1}$, it has been pointed out that the resistivity and optical conductivity can be described by a TLL [5]. However, because of interchain hopping, or interchain electron–electron or electron–phonon interactions, such materials are never strictly 1D. In particular, although one expects the 1D theory to hold at high temperature, at lower temperatures interchain hopping is expected to drive the system toward a more isotropic (2D or 3D) behavior. It has been argued that the transport properties of the metallic state of the TMTSF analogs can be accounted for in terms of a weakly interacting FL [6]. Hence, these compounds are ideal candidates for a study of the dimensionality crossover between a non-Fermi-liquid regime (TLL in this case) and a more conventional Fermi-liquid state.

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Optical conductivity results [7] have revealed both a clear-cut gap feature (finite-energy (FE) mode) around 200 cm^{-1} ($\sim$25 meV) and a zero-energy (ZE) mode in all TMTSF salts at low temperatures. The ZE mode, although of the order of 1% of the total spectral weight, is responsible for the large conductivity, whereas the gap feature is close to a doped 1D Mott insulator in that the interchain hopping can be viewed roughly as an effective doping. But the origin of the 1% intensity of the ZE mode is still unclear because it does not result in a 100-times increase of the Hall coefficient as one would expect [8, 9]. Whether or not other physical properties are dominated by the ZE mode is an issue which has been widely debated.

In order to get more detailed information on the above issues, (TMTSF)$_2$ClO$_4$, a representative organic compound, is studied in this paper. (TMTSF)$_2$ClO$_4$ exhibits superconducting transition at ambient pressure with a slowly cooled state (about 0.1 K min^{-1}; the relaxed state) and an insulating antiferromagnetic state with rapidly cooled state ($>$30 K min^{-1}; the quenched state). The appearance of a superconducting ground state in the relaxed state is related to a structural phase transition at 24 K dimerizing the system along the *b* direction characterized by a wavevector $q = (0, 1/2, 0)$ due to ordering of the noncentrosymmetric perchlorate anions [10]. For the quenched state, the direction of the anions is frozen in two directions at random probability during rapid cooling through the temperature of the anion ordering transition [10–12]. Intermediate states with ordering regions and disordering regions, where a large number of disconnected superconducting regions eventually appear at quite low temperatures, can be created experimentally either by using intermediate cooling speeds or by quenching from different temperatures in the critical region around 24 K with a rapid cooling rate [13].

In this paper, thermopower and specific heat experiments were performed with different cooling rates through the anion ordering transition temperature. Narrow band behavior is revealed from both the thermopower and specific heat experiments at low temperatures in this paper. This sheds light on the transport mechanism which can be described just from energy-band theory for (TMTSF)$_2$ClO$_4$ at low temperatures (below 40 K). At the same time, the dimensional crossover from 2D to 3D is indicated for the first time by our thermopower data. This demonstrates that the system is 3D before superconductivity occurs for the relaxed state, which resolves the fundamental issue about whether superconductivity occurs in 2D or 3D systems. On the basis of the high precision of our thermopower data, it is observed for the first time that the melting temperature of the anion changes as the cooling rate changes. This leads to the suggestion that the entropy change related to the anion ordering transition is lower than the theoretical value $R \ln 2$, which has been a controversial issue up to now.

2. Experimental details

Needle-like single crystals of the Bechgaard salts (TMTSF)$_2$ClO$_4$ (which we abbreviate in the following text as ClO$_4^{-1}$)
were prepared by a conventional electrochemical method [14] with a typical size of $(a \times b \times c) = 3 \times 0.1 \times 0.05$ mm^3. The steady-state technique of alternating the temperature gradient was employed in measuring S_a [15]. Furthermore, in order to effectively remove the potential tension during the cooling and warming processes and avoid irreversible jumps which are typical in resistivity measurements on all organic conductors, one end of the crystal was glued to a fixed Cu plate using VGE-7031 varnish (Lakeshore) while the other end of the crystal was attached to another very small Cu plate which could move freely along the sample direction. The temperature, monitored by Cernox thermometer at the two ends of the crystal, was controlled automatically within a precision of 0.004 K and the temperature gradient between them was kept at 2 K at room temperature, and decreased as the temperature was lowered. Each data point is the average of 100 points and the error of the thermopower measurement system was less than 0.1 μV K^{-1}. The high precision of our TEP measurement system gives us the opportunity to observe the more detailed behavior of the thermopower. First, the sample was cooled down by performing the measurement from 290 to 5 K at an average rate of 0.0005 K s^{-1}, which leads to a relaxed state. Next, to produce intermediate states and the quenched state, the sample was warmed up to 40 K to ensure that the anion disorder at high temperatures was completely developed and then cooled down to the lowest temperature with different cooling rates of 0.022 and 1 K s^{-1}, entering the intermediate states and the quenched state, respectively. The thermopower data for the intermediate and the quenched states were always measured by slowly warming up from the lowest temperature. Finally, the thermopower of the Cu wire was subtracted. In order to make sure the data used in the paper are believable, we measured S_a on different samples and the data presented in this paper were obtained from a representative sample. All the data were obtained from the same sample which is also one of the samples used in the specific heat measurement. The reproducibility of the thermopower during the cooling and heating cycle was also confirmed. The specific heat measurement for the slow cooling state and the quenched states was performed between 1.8 and 35 K on a ^{4}He cryostat with a transient-heat-pulse technique, which is described in detail in [14].

3. Results and discussion

Figure 1 shows the temperature dependence of the thermopower $S_a(T)$ along the highly conducting *a* axis in (TMTSF)$_2$ClO$_4$ for the relaxed state in the temperature range of 5–35 K. At first glance, a small abnormal signal is observed at a temperature of about 29.8 K which is defined as T_a, implying that some anions have been ordered. We consider T_a to be the onset temperature corresponding to the occurrence of anion ordering, which is not only consistent with the temperature at which the specific heat curve departs from the background in the following text but is also in good agreement with the temperature where the weak superlattice peaks appear due to anion ordering in the XRD experiment [10]. As the temperature decreases, it must be noticed that the thermopower S_a
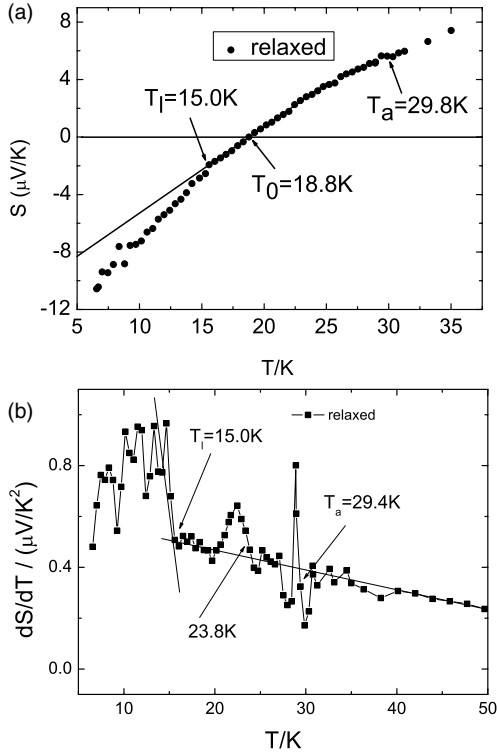
Figure 1. (a) Temperature dependence of the thermopower for ClO$_4$ single crystal in the *a* direction for the relaxed state. (b) Temperature derivative of the thermopower for (a) (for details see text).

Figure 2. (a) Temperature dependence of the thermopower S_a for cooling rates of 0.22 K s^{-1} through the anion ordering transition. (b) Temperature derivative of the thermopower for (a).

changes its sign around 18 K (defined as T_0), suggestive of a change in the sign of the charge carriers, which is consistent with the results observed in the Hall coefficients [16], and the explanation for the sign reversal in the thermopower as will be discussed later. With the temperature reaching about 15.0 K (defined as T_l) the thermopower $S_a(T)$ drops more sharply with decreasing temperature, which is attributed to 2D to 3D crossover. Since the thermopower is a measure of the entropy transported per carrier, the entropy, corresponding to the number of microscopic states in the system, is increasing distinctly as the system of electron begins to change from 2D to 3D, and thus the increase of the absolute value of $S_a(T)$ is ascribed to the increase in the entropy transported per carrier below the temperature T_l. At the same time, the increase in the absolute value of $S_a(T)$ could be interpreted in another way. As mentioned in the introduction, the gap feature in optical conductivity results is close to a doped 1D Mott insulator in that the interchain hopping can be viewed roughly as an effective doping [7]. As the system changes from 2D to 3D, the interchain hopping t_c will play an important role in thermopower, that is to say both t_b and t_c can be considered as an effective doping. So the change in the interchain hopping in the *c* direction will also affect the thermopower along the *a* direction. Furthermore, according to [17, 5], the transfer integrals in the *b* and *c* crystallographic directions, t_b and t_c, are quite different from each other, with the ratio of t_b to t_c being about 25:1 meV. Thus the small value of t_c implies that at temperatures below about 13 K (t_c/k_B, where k_B is the Boltzmann constant) the system presents a weak 3D character. Below T_l the thermal
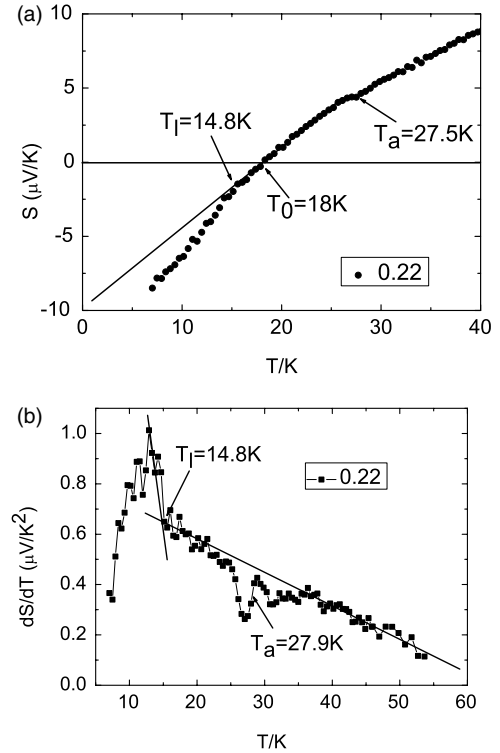
fluctuations are too small to destroy the coherent transport that could cause the system to have weak three-dimensionality.

In order to exhibit the above phenomena distinctly, the derivative plot for the thermopower $S_a(T)$ as a function of temperature is shown in figure 1(b), and a clear peak resulting from anion ordering and a change of the slope due to the 2D to 3D crossover are observed at $T_a \sim 29.4$ K and $T_l \sim 15.0$ K, respectively. It must be pointed out that there is still a peak at 23.8 K between T_a and T_l in the derivative plot, corresponding to the position where most of the anions become ordered, which is in agreement with the results of the specific heat in the following text as well as the previous reports [17–19].

In order to probe the influence on the thermopower of increasing the rate of cooling through the anion ordering transition temperature, the temperature dependence of the thermopower for different cooling rates (0.22 and 1 K s^{-1}) are plotted in figures 2(a) and 3(a) as well as their corresponding derivative plot in figures 2(b) and 3(b), respectively. It is noticed that T_a shifts toward a lower temperature as the cooling rate increases, and this phenomenon was first observed for thermopower. Nevertheless, this is incompatible with the results for resistivity [13], where the temperature dependence of resistivity for ClO$_4^{-1}$ overlap with each other just above 24 K. The differences between thermopower and resistivity may be because thermopower is more sensitive to the transition than resistivity, or because the thermopower is not related to the boundaries between the ordered regions and the disordered regions but to variations in the electrical nature of the charge carriers [20, 21]. The higher the cooing rate becomes, the
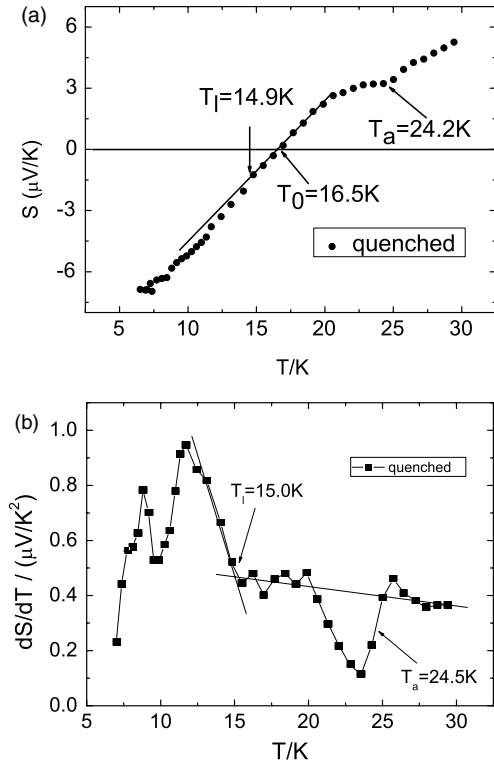
Figure 3. (a) Temperature dependence of the thermopower S_a for cooling rates of 1 K s^{-1} through the anion ordering transition. (b) Temperature derivative of the thermopower for (a).

Figure 4. Sketch showing the change of the band structure with respect to the transfer integrals t_b. The arrow represents increase of t_b.

more anions will be frozen in random directions, leading to the system becoming more disordered. It is known that the most ordered system will have the least energy. Therefore, little energy needs to be supplied to the system to melt the frozen anions when the cooling rate becomes high. This may lead to T_a for cooling rate of 1 K s^{-1} lower than that for a cooling rate of 0.22 K s^{-1}. However, we also note that T_0 shift to the lower temperature and T_l is unchanged as the cooling rate increases. We will discuss the former in detail later; for the latter, just as discussed in the previous paragraph, T_l, determined by the transfer integral t_c, is the critical temperature corresponding to the crossover from 2D to 3D. T_l is nearly unchanged as the cooling rate changes, suggesting that the anion ordering has a negligible effect on t_c, and this result is supported by XRD measurements [10]. At the same time, as shown in figure 4(b), it should be pointed out that the thermopower shows an abrupt increase below 6 K for the quenched state corresponding to the beginning of the metal–spin density wave transition temperature, in agreement with the results of the resistivity measurements [13] and the thermopower in (TMTSF)$_2$PF$_6$ [22].

To explain the sign reversal in the thermopower and the dependence of T_0 on the cooling rate, we will discuss their possible origins in detail. In generally, the sign reversal in $S_a(T)$ is considered a result of either the existence of two conduction bands with opposite carrier signs (the so-called two-carrier conduction model), or the change of the band filling as a function of temperature. Experimentally, the single band model is appropriate for this system at high
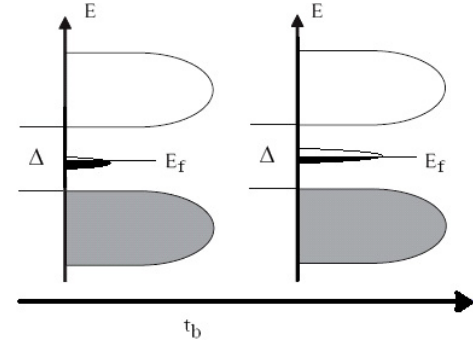
temperatures, as verified by optical experiment [23] and Hall measurement [8, 9]. At the same time, for a conventional conduction band, the filling level scarcely changes with the cooling rate, inconsistent with the filling level changing with cooling rate in ClO$_4^{-1}$ at low temperatures. So the sign reversal in $S_a(T)$ at low temperatures cannot be interpreted by the conventional single band model as at high temperatures, and the sign reversal in $S_a(T)$ is most probably a result of the narrow band, and the filling of the narrow band changes as temperature decrease for ClO$_4^{-1}$. According to the narrow band model, the sign reversal of $S_a(T)$ could be described by the Heike formula when the width of the narrow band is less than $k_B T$:

$$S = \frac{k_B}{e}\left(\ln\frac{c}{1-c}\right),$$

where c is the filling level in the narrow band. As shown in figure 1(a) for the relaxed state, that S_a is positive at temperatures above T_0 indicates that c is larger than 1/2. Since the transfer integral t_b has an effect similar to doping [7], a finite density of states is introduced at the Fermi level in the narrow band. The change in the band structure with respect to t_b is shown in figure 4. It is known that t_b arrives at the maximum when almost all the anions are completely ordered, implying that t_b increases as temperature decreases. In addition, the number of carriers in the narrow band is nearly unchanged [7, 24]. Hence, c decreases as temperature decreases. That is to say, c decreases to 1/2 at temperature T_0 and to less than 1/2 at temperatures below T_0, which results in sign reversal in $S_a(T)$ according to the Heike formula. Furthermore, it can be found that T_0 shifts to low temperature as the cooling rate increases from figures 1(a), 2(a) and 3(a). Since the fraction of the ordered regions decreases as the cooling rate becomes high, and reaches about 30% even in the state with a cooling rate of 100 K min^{-1} as pointed out in [25], the transfer integral t_b for the quenched state still shows a slight increase compared to that at high temperatures, but it increases more slowly than for the relaxed state. Therefore, the transfer integral t_b for the state with a high cooling rate increases more slowly than that for the relaxed state. c decreases more slowly in the state with a high cooling rate than for the relaxed state. The temperature corresponding to the band filling c decreasing to 1/2 is delayed to a temperature lower than that
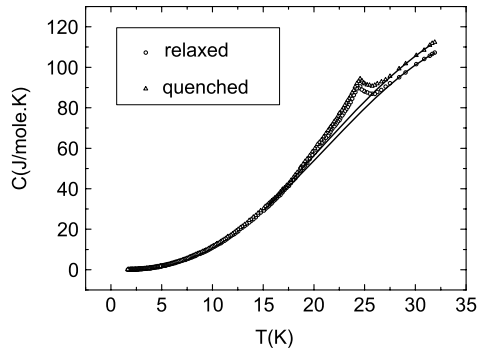
Figure 5. The temperature dependence of the specific heat for the slow cooling state and the fast cooling state in the temperature range 1.8–35 K. The solid lines are the background.

for the relaxed state, which gives a good explanation for the shifting of T_0 to lower temperature as the rate of cooling increases. Furthermore, as stated in previous reports [17, 5], a narrow mode at zero frequency with a small spectral weight in the optical conductivity is observed, resulting in high dc conductivities. Moreover, the narrow mode at zero frequency change with reduction in temperature. All of the characteristics are in qualitative agreement with our narrow band picture.

In order to demonstrate that the narrow band model is appropriate in this system, we also carried out a specific heat experiment on the ClO_4^{-1} for relaxed (0.3–0.5 K h^{-1}) and quenched states (3.3 K s^{-1}), respectively. The molar specific heat C of $(TMTSF)_2ClO_4$ at constant pressure in the form of C against T from 1.8 to 35 K for the two states is shown in figure 5. The Debye temperatures are 205 ± 2 K for the quenched state and 216 ± 2 K for the relaxed state [14]. Generally speaking, the Debye function starts to deviate from T^3 law above $\theta_D/20 \sim \theta_D/15$. Therefore, the specific heat curves are expected to follow the formula $C = \gamma T + \beta T^3$ below 10 K (where γ, β are the electronic heat coefficient and phonon specific heat coefficient, respectively). At the same time, the influence of anion order on the specific heat could be ruled out in this temperature range, supported by [13] in which it was pointed out that almost 100% of the anions have been ordered below 15 K. However, as shown in figures 6(a) and (b), there is still a little departure from the fitting curve for both states, which suggests that the value of $\gamma \sim N(E_F)$ changing intensively with temperature. In order to estimate how γ changes as the temperature decreases, $\gamma(T)$ is calculated by subtracting βT^3 from the total specific heat with $\beta \approx 11.0$ and 12.5 mJ mol^{-1} K^{-4}, obtained by fitting the specific heat curves for the relaxed and quenched states in the temperature range below 2 K, respectively. It is worth emphasizing that $\beta \approx 11.0$ and 12.5 mJ mol^{-1} K^{-4} is in quite good agreement with that reported in [14]. Since β is determined by the Debye temperature θ_D, which is affected by the crystal structure and slightly affected by the temperature at low temperatures. As almost 100% of the anions have been ordered below 15 K [13], the crystal structure is nearly unchanged below 15 K. Hence, as the first approximation, β can be considered as a constant below 15 K. The temperature dependence of γ is shown in the insets of figures 6(a), (b). It is noticed that $\gamma(T)$, proportional
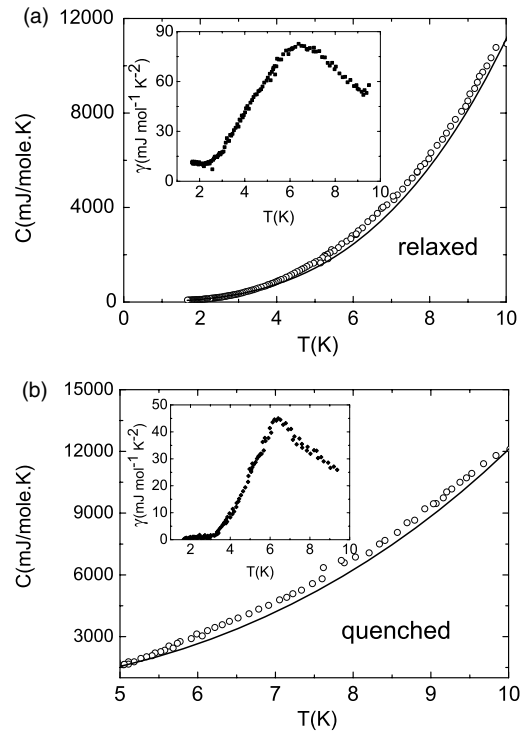

Figure 6. Temperature dependence of the specific heat in the temperature range 1.8–10 K for the relaxed state and 5–10 K for the quenched state. The solid line is the fitting curve by the formula $C = \gamma T + \beta T^3$. Inset: the temperature dependence of γ. (a) Relaxed state. (b) Quenched state.

to $N(E_F)$, increases distinctly as temperature increases. This is convincing evidence that the energy band is a narrow band and the band filling is changing with the temperature decrease in the middle temperature region just as discussed for the thermopower, though our thermopower data could not show where γ returns to the normal value at high temperatures. The temperature dependence of the specific heat for the different states shows a distinct difference at the temperatures above 20 K, and a sharp peak is observed in each curve at the anion ordering temperature of about 24 K shown in figure 5. There are some differences in the background for the two different states even at 30 K, consistent with the result for the thermopower, that some anions begin to become ordered at this temperature for the relaxed state, and the resistivity results [13]. The temperature dependence of ΔC, defined as the difference in the specific heat between the quenched state and the relaxed state, is shown in the form of C/T versus T^2 in figure 7, and the solid line is the fitting curve for ΔC by $\Delta C/T = AT^2$ which fits our experimental data quite well, where A is a fitting parameter with $A = 0.23 \pm 0.02$ mJ mol^{-1} K^{-4}. This suggests clearly that ΔC just derives from the contributions of the lattice specific heat, which may be attributed to the distortion of the organic molecule caused by anion order due to the interaction between the anion and the organic molecule.

The temperature dependence of the specific heat δC owing to the anion ordering transition, obtained by subtracting a background (the solid lines in figure 6(a)) defined by
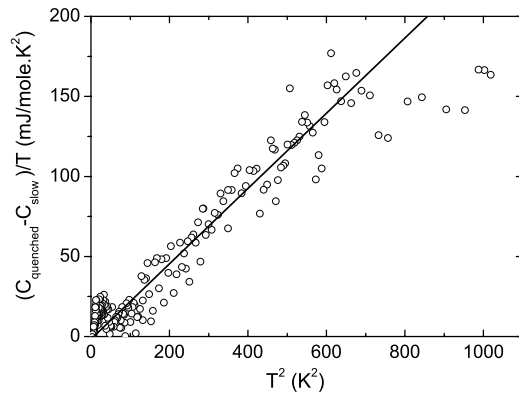
Figure 7. The difference in the specific heat between the quenched state and the slow cooling state. The dashed line is the fitting curve for $\Delta C/T = AT^2$.

Figure 8. The abnormal peak of the anion ordering transition for the slow cooling state is obtained by subtracting a background (the dashed line in figure 5(a)) from the total C.

the fit of the data outside the anion ordering transition temperature range from the total C [14], is plotted in figure 8. The asymmetric shape of the curve is a typical second-order transition [26]. It should be pointed out that the value of the entropy estimated from the specific heat peak is $R\ln(4/3)$, reported in detail in [14], which is smaller than $R\ln 2$ expected for an order–disorder transition with two possible configurations for the ClO_4^{-1} tetrahedron in the high-temperature disordered state. This could also be understood on the basis of some anions becoming ordered at a temperature around 30 K, just as discussed above for thermopower. At the same time, according to a report of the resistivity [13], intermediate states with various degrees of frozen anion disorder can be obtained by quenching from different temperatures, confirming that there are already some anions becoming ordered at temperatures around 30 K, which is also in agreement with magnetoresistance measurements as well as the x-ray diffraction studies. Therefore, a smaller entropy change is easy to understand in this way.

4. Conclusion

We have measured the thermopower $S_a(T)$ along the highly conducting a axis and the specific heat of the Bechgaard salts $(TMTSF)_2ClO_4$ with various cooling rates through the anion ordering temperature. We observed that there is a sign reversal in $S_a(T)$ at the temperature T_0 and the sign reversal temperature decreases with increasing the cooling rate, explained by the change of the narrow band filling as the function of the temperature and the cooling rate. The change of the slope in thermopower can be attributed to the crossover from 2D to 3D. As far as the specific heat is concerned, that the γ changes intensively with temperature is good evidence for the existence of the narrow band in this system. In addition, a well-defined asymmetric peak characteristic of a second-order transition was found with a maximum at 24 K. However, after subtracting a background from the total specific heat, the value of the entropy from the specific heat between 28 and 15 K is $R\ln(4/3)$, lower than the value $R\ln 2$ expected for an anion ordering transition with two possible configurations for the
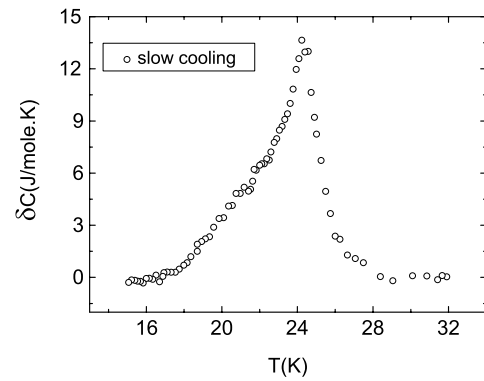
$(TMTSF)_2ClO_4$, which could be the result of some anions becoming ordered around 30 K as discussed for thermopower. Moreover, from the difference in the specific heats between the above two different states, we can conclude that the lattice is slightly distorted owing to anion ordering.

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