Giant nonlinear conduction from inhomogeneous charge order in rapidly cooled θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$

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We find a giant nonlinear conduction in the charge-ordered organic salt θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$ in a rapidly cooled state from 34 to 99 K, which cannot be ascribed to a simple Joule heating. The observed voltage-current characteristics are essentially identical to those of the related organic salt θ-(BEDT-TTF)$_2$CsZn(SCN)$_4$, while the energy scale and the working temperatures are 1 order of magnitude higher. Considering that two kinds of charge-ordered domains coexist in this rapidly cooled state, we suggest that the inhomogeneity made of the two charge orders plays an essential role in the giant nonlinear conduction of the θ-type organic salts.

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I. INTRODUCTION

The θ-type BEDT-TTF salts are two-dimensional (2D) quarter-filled hole conductors and have been extensively studied as typical charge-ordered materials for the last decade.[1] A tight-binding band calculation shows that a single band crosses the Fermi energy to form an ellipsoidal Fermi surface in the a^*-c^* plane. This implies that the one-electron state is simple enough to be treated with an anisotropic 2D Drude model. The crystals of organic salts include little defects with no solid solutions, in which Shubnikov–de Haas oscillation is often observed in metallic phases.[2] The related charge-ordered salt α-(BEDT-TTF)$_2$I$_3$ shows a charge ordering below 135 K at ambient pressure[3] and becomes a zero-gap semiconductor with an ultrahigh mobility of 3×10^5 cm^2/Vs at 2 K in 2 GPa.[4] This example clearly shows that charge ordering takes place in an extremely pure crystal, offering an ideal playground for the studies of charge ordering.

Recently one of the salts θ-(BEDT-TTF)$_2$CsZn(SCN)$_4$ (the Cs salt) was found to show a giant nonlinear conduction below 10 K.[5–8] The resistivity of this salt rapidly increases below 20 K, which is driven by the growth of short-range charge-ordered domains indexed by $\mathbf{q}_2=(0,k,1/2)$.[9] In other words, the holes are gradually localized at every two molecules along the c axis below 20 K to be a charge-ordered insulator. Sawano *et al.*[6] observed that the diffuse scattering at $\mathbf{q}_2$ lost its intensity with current and associated the nonlinear conduction with current-induced melting of the $\mathbf{q}_2$-type charge order. They further associated the microscopic mechanism for the gigantic response with intrinsic inhomogeneity arising from competing orders. The $\mathbf{q}_2$-type charge order is believed to compete with another charge order indexed by $\mathbf{q}_1=(2/3,k,1/3)$ at low temperatures in the Cs salt, which was directly observed as two kinds of scattering peaks in x-ray diffraction images at low temperatures.[9] Watanabe *et al.*[10] showed that the suppression of the $\mathbf{q}_2$ peak is not simply due to Joule heating and found a possible nonequilibrium state in a high external current that cannot be achieved in thermal equilibrium. Sawano *et al.*[11] found that the activation energy and the $\mathbf{q}_2$ intensity exhibit the identical current dependence, which strongly suggests that the energy gap seen in the charge transport is the $\mathbf{q}_2$-type charge order. Ito *et al.*[12] measured and analyzed the temperature and current dependence of the $\mathbf{q}_1$ and $\mathbf{q}_2$ intensities and suggested that these two diffuse peaks should be associated with spatially different charge ordering domains. If so, the charge-ordered patterns will be spatially inhomogeneous, which can be susceptible to the external field as is suggested by Burgy *et al.*[13] However, the spatial distribution of the inhomogeneity is not yet observed, and some theories suggest that the two diffuse peaks can appear without the spatial inhomogeneity.[14,15]

To see whether or not an inhomogeneous state is related to the giant nonlinear conduction, we focus on θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$ (the Rb salt); another class of the θ-type organic salt. This particular salt shows the long-range $\mathbf{q}_2$-type charge order below a first-order transition temperature of $T_{CO}=190$ K.[1] Miyagawa *et al.*[16] clarified that the charge is disproportionated on two inequivalent molecules by a ratio of 0.8:0.2 below T_{CO}. Watanabe *et al.*[17] determined the crystal structures below and above T_{CO}, where the BEDT-TTF molecules slightly rotate below T_{CO} to minimize the intersite Coulomb repulsion. This clearly indicates the existence of strong electron-phonon interaction in this salt. In this respect, it may be controversial whether this charge disproportionated state belongs to the charge order or charge-density wave; the former is driven by the long-range Coulomb repulsion, whereas the latter is driven by a nesting vector with electron-phonon interaction. Within random-phase approximations of an extended Hubbard Hamiltonian, Kuroki[14] and Udagawa and Motome[15] independently found two peaks in the charge susceptibility, one of which comes

from the nesting of the Fermi surface (charge-density wavelike), while the other comes from the intersite Coulomb repulsion (charge orderlike). The real situation is perhaps affected by both, so that one cannot strictly distinguish charge order from charge-density wave in this salt. Thus we will use the term of "charge order" to mean that the charge disproportionated state below T_{CO} is not a simple charge-density wave because (i) the lattice modulation is doubly periodic for quarter filling (quadruple periodicity is expected for charge-density wave), (ii) no sliding motion or narrow-band noise are reported, and (iii) the susceptibility above T_{CO} is of Heisenberg type (Pauli paramagnetism is expected for charge-density wave).[1]

An important feature of this transition is that the normal state above T_{CO} is not normal; diffuse scattering peaks indexed at $\mathbf{q}_1'=(1/3,k,1/4)$ are visible in the x-ray diffraction images at 225 K.[18] This observation indicates that the $\mathbf{q}_1'$-type charge order grows above T_{CO} and is suddenly replaced by the $\mathbf{q}_2$-type charge order below T_{CO}. Miyagawa *et al.*[16] observed the fluctuation of the charge order below 264 K in the NMR spectra and Chiba *et al.*[19] found that a typical time scale for the fluctuation is on the order of 100 MHz. Inagaki *et al.*[20] and Nad *et al.*[21] found a large dielectric constant above T_{CO} which is expected as an ac response of the charge order/charge-density wave. Since the transition at T_{CO} is of first order, one can overcool the Rb salt by quenching it through T_{CO} to make an inhomogeneous state composed of the $\mathbf{q}_1'$- and $\mathbf{q}_2$-type charge orders. Actually this rapidly cooled state is similar to the low-temperature state of the Cs salt; Watanabe *et al.*[18] observed the $\mathbf{q}_1'$ and $\mathbf{q}_2$ peaks in an x-ray diffraction image at 90 K. Nakamura *et al.*[22] did not find any significant anomaly associated with magnetic orders in $1/T_1$ below 100 K and Nad *et al.*[23] found no ferroelectric anomaly at T_{CO}. According to these results, this rapidly cooled state is formed with the first-order transition suppressed and can be a good reference to the "intrinsic" inhomogeneous state in the Cs salt. Here we show a giant nonlinear conduction in the rapidly cooled state in the Rb salt from 34 to 99 K, which is essentially identical to that in the Cs salt except for the working temperature and the energy scale. This work is an experimental demonstration that the inhomogeneous state made of the two charge orders plays an essential role in the giant nonlinear conduction.

II. EXPERIMENTAL

Single crystals were prepared by a galvanostatic anodic oxidation method and the detailed growth conditions and their characterization were described elsewhere.[1] The samples were thermally anchored to a copper block with two Au wires of 20-μm diameter and 3-mm length and were exposed to a He gas flow in a liquid He cryostat.

The resistivity was measured with sample C1 ($0.24\times0.1\times0.98$ mm^3) along the c axis using a two-probe method from 4.2 to 300 K. The nonlinear resistance was measured with sample C2 ($0.25\times0.1\times0.3$ mm^3) along the c axis from 34 to 99 K by the two-probe method using a pulse current source (Keithley 6221). The reason to use the two-probe method was that the input power was easily monitored from the sample voltage. Essentially the same results were obtained in a four-probe configuration. The voltage-current characteristics along the b axis were measured at 78 K with sample B1 just for comparison. The sample voltage V_{sample} was recorded with a nanovoltmeter (Keithley 2182) electronically synchronized with the current source. All the measurements were done with a single current pulse I_{ext} of 10 ms, and the signal at 8 ms was taken as V_{sample}. The wave forms of V_{sample} were recorded with a digital oscilloscope for a few representative pulses, where those of I_{ext} were simultaneously recorded as a voltage across an Ohmic resistor of 500 Ω connected to the sample in series. The samples were cooled at a rate of 8 K/min from 300 down to 210 K and then at a rate of 18 K/min around T_{CO}.

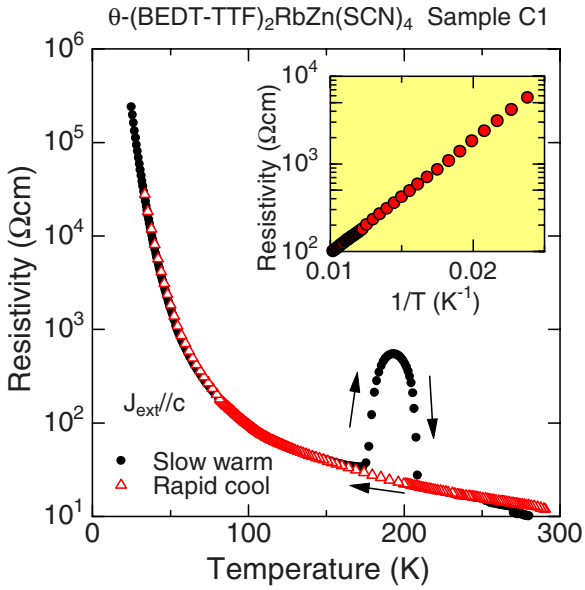

FIG. 1. (Color online) The c-axis resistivity of sample C1 of θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$ for a rapidly cooled and slow-warmed states. The inset shows the Arrhenius plot of the resistivity in the slowly warmed state from 34 to 100 K.

III. RESULTS AND DISCUSSION

Figure 1 shows the c-axis resistivity of sample C1 with an external current of 1.0 μA. The resistivity shows clear hysteresis from 170 to 210 K between a rapidly cooled and slowly warmed states, which was observed in the dc resistivity measured by Kanoda[24] and in the ac conductance measured by Nad *et al.*[23] The hump in the slowly warmed state corresponds to a crossover from the quenched state (the nonequilibrium state) to the thermally equilibrium state. The resistivity curves resemble the magnetization curves in colossal magnetoresistive manganites[25] and the polarization curves in relaxor ferroelectrics.[26] Since the two systems are composed of inhomogeneous mixture of two phases, we expect that the rapidly cooled Rb salt has a similar inhomogeneous state made from two charge-ordered domains. Kanoda[24] first recognized the importance of the similarity to spin-glass systems, after which they named "charge glass." Note that V_{sample} changes with time near T_{CO}, as is similar to

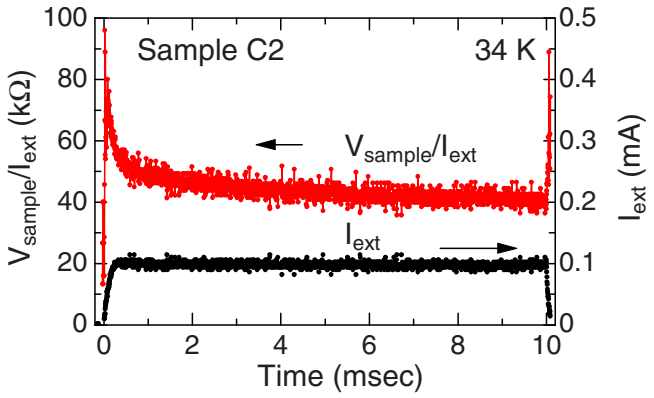

FIG. 2. (Color online) The two-probe resistance V_{sample}/I_{ext} and the external current I_{ext} of sample C2 of θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$ for a rapidly cooled state plotted as a function of time.

the magnetization relaxation in spin glasses. The relaxation is slower at lower temperatures, and the resistivity is essentially independent of time below 100 K in comparison with an experimental duration time of 5–6 h.

The inset of Fig. 1 shows the Arrhenius plot of the same data from 34 to 100 K, clearly showing that the resistivity ρ obeys an activation-type transport given by $\rho = \rho_0 \exp(\Delta/k_B T)$, where ρ_0 is the resistivity in the high-temperature limit, and Δ is an activation energy. The activation energy $\Delta/k_B T$ is evaluated from the slope to be 300 K, being consistent with the value reported in the literature.[7,23]

Figure 2 shows the two-probe resistance V_{sample}/I_{ext} and the external current I_{ext} of rapidly cooled sample C2 plotted as a function of time. The external current rapidly increases from 0 to 0.2 ms owing to a finite response time of the pulse current source and is constant after 0.5 ms. Similarly the external current decreases to zero in 0.05 ms after 10 ms. The resistance V_{sample}/I_{ext} slowly changes by 10% after 1 ms, so that the resistance data are essentially independent of time after 1 ms within an accuracy of 10%. Here we evaluate the temperature increase due to the self-heating of the sample. We first calculate the heat capacity of sample C2 to be 5.4 μJ/K by using the specific-heat value of θ-(BEDT-TTF)$_2$CsZn(SCN)$_4$ (450 J/mol K at 35 K) (Ref. 27) and the number of moles (1.2×10^{-8} mol) calculated from the sample dimension. We then evaluate a total amount of heat below 1 ms in Fig. 2 to be 0.1 mA$\times$5 V$\times$1 ms $=5\times10^{-7}$ J. This value shows that the temperature increase would not exceed 0.1 K, even if the input heat was fully confined in the sample. The nonlinear resistance is clearly observed at this current (see Fig. 3) and is too large to be explained from the change in the sample temperature. Consequently, we conclude that the nonlinear conduction observed here is not due to the self-heating of the sample. The maximum input power was approximately 5 mW in the present experiment, which corresponds to a maximum temperature increase of 1 K at 1 ms.

Next we evaluate the amount of heat flow in this experimental setup because the pulse width of 10 ms is long enough to let the heat flow steadily. The samples were thermally anchored with two 4-mm-long Au wires (the diameter

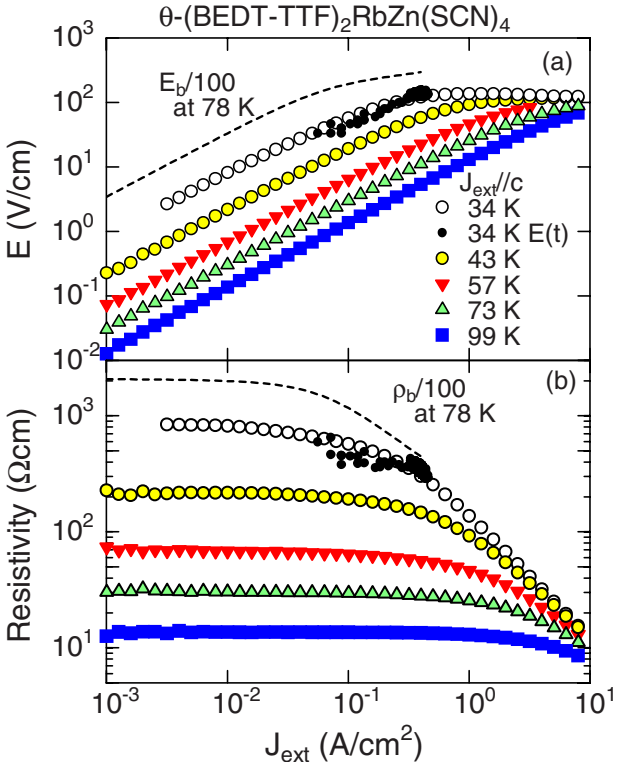

FIG. 3. (Color online) (a) The electric field E and (b) the nonlinear resistivity of rapidly cooled sample C2. The closed circles are the same data in Fig. 2 using the measured time as a parameter. The dotted curves represent the data along the cross-layer (the b axis) direction at 78 K.

of 50 μm) in a He gas flow of $10^{-2}\sim10^{-1}$ cm/s. According to the standard heat-transfer theory,[28] the coefficient of heat conduction α is given by $N_u\kappa/d$ for a cylinder with a diameter of d in a cross flow, where N_u is the Nusselt number, and κ is the thermal conductivity of the coolant. In the present case, N_u can be assumed to be 0.5 which is a minimum value experimentally observed for a small flow velocity of the coolant. Then α is calculated to be 500 W/Km^2. The effective thermal conductance for the heat transfer is given as αS, where S is the surface area of sample and Au wires, and is eventually calculated to be 7×10^{-4} W/K. We should consider the thermal conduction through the Au wires as well, which gives a thermal conductance of 3×10^{-4} W/K for a thermal conductivity of 300 W/mK. Thus the total thermal conductance will be 10^{-3} W/K, which means that a power of 1 mW increases the sample temperature by 1 K. This evaluation indicates that the maximum power of 5 mW used in the present experiment may increase the temperature by 5 K. The temperature intervals for the nonlinear conduction measurements were chosen to be 9–16 K (see Fig. 3) that is larger than the maximum temperature increase estimated above. We further note that the data used for analysis (see Fig. 4) were chosen in order that the applied power should be less than 2 mW.

The above discussion is based on the assumption that the external current flows homogeneously through the sample. Since the inhomogeneous state studied here is regarded as a mixture of nanometer-size domains of $\mathbf{q}_1'$- and $\mathbf{q}_2$-type or-

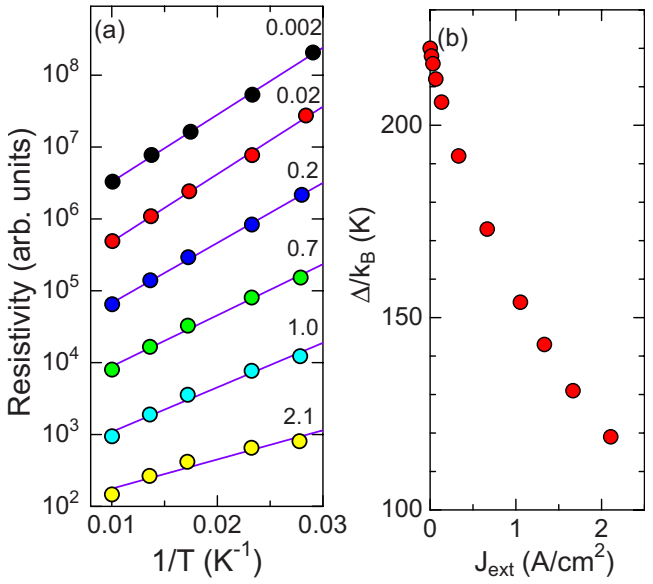

FIG. 4. (Color online) (a) The Arrhenius plots of the resistivity for various current densities J_{ext} indicated by the numbers at the right. Note that each data set is properly shifted to the vertical direction in order to see the change in the slope with J_{ext}. (b) The energy gap obtained from (a) plotted as a function of external current density.

ders, it will act as a homogeneous medium with respect to macroscopic currents. Nevertheless, when the thermal conductivity κ is fairly low [2 W/mK (Ref. 29)], the temperature and the current path may be inhomogeneous, as was observed in amorphous semiconductors.[30] Mercone *et al.*[31] found an analytical expression for the inhomogeneous distribution of current and temperature for such cases. According to their theory,[32] the current can be inhomogeneous above a critical current of $I_c = 2w\sqrt{2\kappa T_0/\beta\rho_0}$, where w is the width of the rectangular-shaped sample, T_0 is the lowest measured temperature, and ρ_0 is the resistivity at T_0. The constant β is roughly evaluated as $\ln[\rho(T_0)/\rho(2T_0)]$. Substituting $w = 0.25$ mm, $\rho_0 = 10\ \Omega$ m, $T_0 = 30$ K, and $\beta = 2.5$, we get $I_c = 10^{-3}$ A. As shown in Fig. 3(b), the resistance is expected to be Ohmic below I_c, but our sample shows nonlinear conduction above 0.1 A/cm^2 (0.025 mA) at 34 K, which is approximately 100 times smaller than I_c. Thus, we can safely conclude that the inhomogeneous current flow is not the origin for the nonlinear conduction.

Figure 3(a) shows the electric field E of rapidly cooled sample C2 as a function of external current density J_{ext}. E begins to saturate above 0.1 A/cm^2 at 34 K and shows the negative derivative resistance above 1 A/cm^2. The nonlinear conduction is more clearly visible in the form of nonlinear resistivity as shown in Fig. 3(b). The resistivity dramatically decreases by a factor of 60 at 34 K, from which we may call this phenomenon "giant nonlinear conduction." Takahide *et al.*[7] first reported nonlinear conduction in a rapidly cooled sample of the RbZn salt, but the measured temperature was limited below 30 K. We also plot $E(t)$ as a function of $J_{ext}(t)$ using the data near the end of the pulse in Fig. 2, expecting that the transient data after 10 ms can give momentary $E-J$ characteristics. The open and closed circles coincide reasonably, indicating that the same J_{ext} gives the same E. From this result, we conclude that the nonlinear resistance is instantaneous and essentially nonthermal.

The giant nonlinear conduction observed here shares common features with that in the Cs salt.[5,6] First, the nonlinear conduction starts from a small current density and a small electric field, in which threshold currents/voltages cannot be defined. This makes a sharp contrast with the nonlinear conduction of the density waves, in which the conduction is Ohmic below a threshold electric field.[33] Second, the nonlinear conduction appears without any thermodynamic phase transition, which indicates that no collective mode can be the origin for this phenomenon. Note that the rapidly cooled state studied here is realized without any anomalies in the thermodynamic quantities[22,23] because the first-order transition temperature is quickly passed through. One may notice that hot electrons[34] can show giant nonlinear conduction as an individual particle-hole excitation. We can rule out this possibility because the mobility of the Cs and Rb salts is far below 1 cm^2/Vs at room temperature,[5] being too poor for hot electrons. Third, the nonlinear conduction occurs with similar x-ray diffraction images where two diffuse peaks are observed at the same time. Preliminarily, we measured the $E-J$ characteristics of a slowly cooled Rb salt and found that the conduction remained Ohmic until the sample was broken. This means that electric field cannot break long-range-ordered $\mathbf{q}_2$-type charge order. Fourth, the conduction along the cross-layer (b^* axis) direction is nonlinear, as shown by the dotted curves in Fig. 3, which is again difficult to understand from the sliding motion of density waves.

Sawano *et al.*[6,11] analyzed the giant nonlinear conduction in terms of the current-dependent activation energy. We apply their analysis to the nonlinear resistance in the Rb salt. Figure 4(a) shows the Arrhenius plots of the resistivity of sample C2 for various current densities. We are interested only in the slopes, so that the magnitude of the resistivity is arbitrarily taken to shift each curve properly. All the data are found to be linear in $1/T$, which indicates that the activation energy Δ is a function of J_{ext}. Figure 4(b) shows the value of Δ/k_BT thus evaluated is plotted as a function of J_{ext}. Δ/k_B for small J_{ext} is obtained to 220 K, being slightly smaller than that for sample C1. This may be because the inhomogeneous state is nonequilibrium, strongly depending on cooling conditions and sample quality. The activation energy decreases roughly linearly with J_{ext}. This current dependence is identical to that of the Cs salt, which means that the $E-J$ characteristics will be identical if they are normalized by proper values.

Finally, we comment on an origin of the nonlinear conduction. Sawano *et al.*[11] reported that the $\mathbf{q}_2$-type charge-order gap linearly decreases with current density, in which the gap reduces by 50% at 8 A/cm^2. This suggests that a current-density characteristic to the nonlinear conduction is of the same order ($\sim$1 A/cm^2) for the two salts. Kondo *et al.*[8] measured the nonlinear conduction in a sample of the Cs salt under various uniaxial pressures and found that the nonlinear conduction occurs near the same current of 2 mA below 40 K, while the sample voltage changes by ten times with pressure. Sawano *et al.*[11] discussed the current-induced reduction in the charge-order gap in analogy with nonequi-

librium superconducting states. It is known that the superconducting energy gap decreases with excess quasiparticle density.[35–37] Theoretical models indicate that the normalized energy gap $\Delta(n)/\Delta(0)$ decreases with the normalized excess quasiparticle density n as $\Delta(n)/\Delta(0)=1-2n$ for $n \ll 1$.[38,39] Since n is roughly proportional to the external current, one can rewrite this equation as $\Delta(J^*)/\Delta(0)=1-\alpha J^*$, where J^* is a properly normalized current density and α is a constant of the order of unity. Our finding in Fig. 4(b) is qualitatively similar to this relationship, which suggests that a flow of carriers may reduce the energy gap of the charge order in a nonequilibrium state. Such a state is theoretically predicted within a framework of steady-state thermostatistical physics.[40]

Let us briefly discuss how the inhomogeneous state is related to the nonequilibrium state formed by excess carrier injection. We suggest that necessary conditions for such states are (1) the sliding motions of the charge order (the collective modes of the phase) are prohibited and (2) injected carriers should flow in the $\mathbf{q}_2$-type charge-ordered domains. One can easily understand that the condition (1) is satisfied in the inhomogeneous state because the charge density has no long-range order. Since the sizes of the $\mathbf{q}_1'$ and $\mathbf{q}_2$ domains are tens of nanometers,[18] such nanoscale composites will work as an effectively homogeneous media for external currents because the length scale of the inhomogeneity is of the same order of the mean-free path.[5] As a result, we expect that a finite current will flow in the $\mathbf{q}_2$ domains, which will satisfy the condition (2).

IV. SUMMARY

We have measured the voltage-current characteristics in the rapidly cooled θ-(BEDT-TTF)$_2$RbZn(SCN)$_4$ from 34 to 99 K. The conduction is largely nonlinear and is essentially identical to that of θ-(BEDT-TTF)$_2$CsZn(SCN)$_4$ except for the working temperatures and the activation energies that are ten times higher. This result indicates that the nonlinear conduction of the θ-type charge-ordered salts is closely related to the inhomogeneous state composed of different charge-ordered domains. We suggest that the nonlinear conduction observed here is a prototypical example of a giant response from the inhomogeneous charge order.

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