Transport properties in $CeOs_4Sb_{12}$: Possibility of the ground state being semiconducting

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We have measured both magnetoresistance and Hall effect in $CeOs_4Sb_{12}$ to clarify the large resistivity state ascribed to the Kondo insulating one and the origin of the phase transition near 0.9 K reported in the specific-heat measurement. We found unusual temperature (T) dependence both in the electrical resistivity $\rho \sim T^{-1/2}$ and the Hall coefficient $R_H \sim T^{-1}$ over the wide temperature range of about two orders of magnitude below ~30 K, which can be explained as a combined effect of the temperature dependences of carrier density and carrier scattering by spin fluctuation. An anomaly related with the phase transition has been clearly observed in the transport properties, from which the H-T phase diagram is determined up to 14 T. Taking into account the small entropy change, the phase transition is most probably the spin density wave one. Both the electrical resistivity and Hall resistivity at 0.3 K is largely suppressed about an order of magnitude by magnetic fields above ~3 T, suggesting a drastic change of electronic structure and a suppression of spin fluctuations under magnetic fields.

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I. INTRODUCTION

Filled-skutterudite compounds RT_4Pn_{12} (R: rare earth, T: Fe, Ru, Os, and Pn: pnictogen),[1] exhibit a wide variety of exotic phenomena associated with the unique body-centered cubic structure.[2–6] Strong hybridization between $4f$ electrons and conduction electrons enhanced by the large coordination number; 12 Pn and 8 T ions surrounding R, is thought to realize such exotic phenomena.[4,7] In fact, the energy gap ΔE_g estimated from the temperature dependence of electrical resistivity $\rho(T)$ for the Ce-based filled skutterudites can be roughly scaled with the lattice constant as shown in Fig. 1; the smaller lattice constant ones such as $CeRu_4P_{12}$ and $CeFe_4P_{12}$ have the larger energy gap.[8–12,14] Note the metal-insulator transition in $PrRu_4P_{12}$ and the apparent Kondo-like behaviors in $PrFe_4P_{12}$ also reflect strong c-f hybridization,[2,3,5] which are unusual as Pr compounds. In contrast, $CeRu_4Sb_{12}$ with a larger lattice constant is a metal exhibiting non-Fermi-liquid (NFL) behaviors at low temperatures,[13,14] and $PrRu_4Sb_{12}$ is an ordinary superconductor exhibiting no exotic behaviors.

Thus, only from the lattice constant, $PrOs_4Sb_{12}$ was expected to be an ordinary metal, however, it was found to be the first Pr-based heavy-fermion superconductor.[6] $CeOs_4Sb_{12}$, predicted to be a metal by the band structure calculation,[15] was first reported to be a semiconductor with a small gap ($\Delta E_g/k_B \sim 10$ K) estimated from $\rho(T)$,[12] while the finite value of the Sommerfeld coefficient $\gamma \simeq 0.2 \mathrm{J/K^2\,mol}$ suggests metallic ground state.[12,16] Recently, in a far-infrared measurement,[17] an apparent decrease of the optical conductivity at low temperatures, indicating the development of gap structure near the Fermi level E_F with decreasing temperature. The another controversial feature is the sharp peak observed in the specific heat $C(T)$ at ~1.1 K in zero field, that was first ascribed to a phase transition of some impurity phase by Bauer *et al.*[12] The field dependence of $C(T)$ reported recently by Namiki *et al.* rules out such a possibility of impurity effect and suggests the existence of intrinsic phase transition in this compound.[16] However, neither the origin of the peak nor the ground state of this material has been clarified at this stage. In this paper, we report the extended study of electrical resistivity (ρ) and Hall coefficient (R_H) on high-quality single crystals, to deepen the understanding of the ground-state properties in $CeOs_4Sb_{12}$.

II. EXPERIMENTAL

High-quality single crystals of $CeOs_4Sb_{12}$ and $LaOs_4Sb_{12}$ were grown by Sb-flux method starting from a composition of R:Os:Sb=1:4:20 using raw materials 3N5 (99.95%)- La, Ce, 3N—Os, and 6N—Sb.[13,14] The lattice constants determined by x-ray powder diffraction agree with the reported values,[18] and absence of impurity phases was confirmed within the experimental accuracy. ρ and R_H were measured by the ordinary dc four-probe method. The voltage measurements were made by Keithley 182 nanovoltmeters. In order to reduce the heating effect, samples were directly immersed in liquid ^{3}He in the magnetoresistance (MR) and Hall resis-

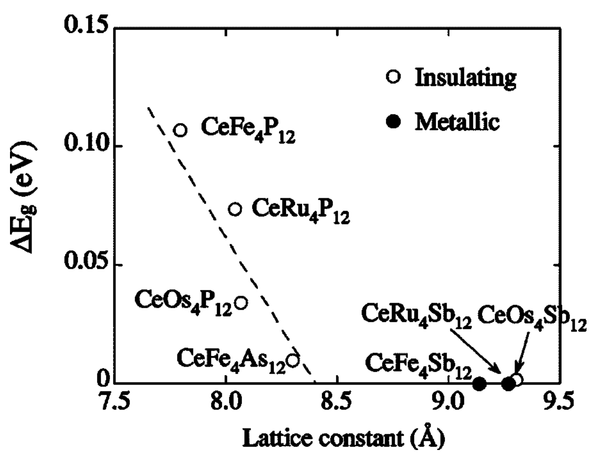

FIG. 1. Energy gap ΔE_g vs lattice constant in $CeTr_4Pn_{12}$.

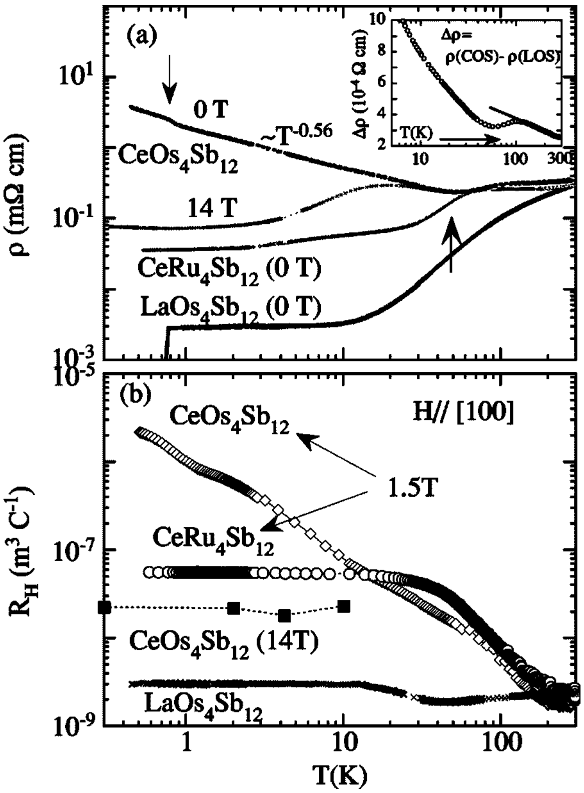

FIG. 2. Temperature dependence of (a) the electrical resistivity $\rho(T)$ and (b) Hall coefficient $R_H(T)$ in $LaOs_4Sb_{12}$ along with $CeOs_4Sb_{12}$ and $CeRu_4Sb_{12}$. The inset of Fig. 2(a) shows the $4f$ component $\Delta\rho = \rho(CeOs_4Sb_{12}) - \rho(LaOs_4Sb_{12})$.

tivity (ρ_H) measurements using an Oxford Instrument top loading ^{3}He cryostat, down to 0.3 K and up to 14 T. The magnetic measurements were made by a Quantum Design SQUID magnetometer up to 7 T. Resistivity for $CeOs_4Sb_{12}$ at room temperature (RT) is $\sim$500 $\mu\Omega$ cm which is more than twice as large as $\sim$200 $\mu\Omega$ cm for $LaOs_4Sb_{12}$. The large residual resistivity ratio (RRR) of $\sim$100 and successful observation of de Haas–van Alphen (dHvA) oscillation for $LaOs_4Sb_{12}$,[19] could be an indirect evidence of the high quality of $CeOs_4Sb_{12}$ single crystals grown in the same manner.

III. RESULTS AND DISCUSSION

The temperature dependence of electrical resistivity $\rho(T)$ for $CeOs_4Sb_{12}$ is compared with those for $LaOs_4Sb_{12}$ and $CeRu_4Sb_{12}$ in Fig. 2(a).[20] $\rho(T)$ for $CeOs_4Sb_{12}$ increases approximately as $T^{-1/2}$ over the wide temperature range below $\sim$30 K at 0 T, which qualitatively agrees with the previous report.[12] A small but apparent bend in $\rho(T)$ curve is foundat around 0.8 K where some phase transition has been reported in the specific-heat measurements.[16]

Hedo *et al.* reported the resistivity under high pressures,[21] where they fitted as $\rho(T) \sim \exp[(T^*/T)^{1/2}]$ and found the reciprocal characteristic temperature $1/T^*$ to be proportional to the applied pressure up to 8 GPa. The dependence of ρ on both temperature and pressure has been analyzed on the base of variable range hopping model,[22] though the intrinsic mechanism has not been well described.

Recently, Yogi *et al.* performed the Sb nuclear quadrupole resonance (NQR) on this compound and found that the temperature dependence of nuclear-spin-lattice-relaxation-rate $1/T_1$ obeys a relation $1/T_1 \sim T^{1/2}$ approximately in the same temperature range (below 25 K).[23] In the self-consistent renormalization (SCR) theory for the spin fluctuations in itinerant antifferomagnetic (AFM) metals,[24] $1/T_1$ is expected to be proportional to $T^{1/2}$ well above Néel temperature. However, the contribution from AFM fluctuation to ρ is expected to decrease with decreasing temperature as T or $T^{3/2}$ depending on the temperature range,[25] which is inconsistent with $\rho(T)$ in the present measurements. It should be noted that the theories up to now assume a metal without temperature dependence of carrier densities. The more systematic studies on different properties are necessary to settle the origin of the temperature dependence on ρ.

The inset of Fig. 2(a) shows the $4f$ component $\Delta\rho = \rho(CeOs_4Sb_{12}) - \rho(LaOs_4Sb_{12})$, assuming their Fermi surface to be basically the same. Such an assumption might be reasonable at high temperatures, taking into account the closeness of R_H near RT. $\Delta\rho$ increases logarithmically with decreasing temperature down to $\sim$100 K, where it shows a faint maximum. Above 60 K, the transport properties are similar to those of ordinary heavy-fermion compounds. After showing a shallow minimum at 60 K, $\Delta\rho$ increases and varies approximately as $T^{-1/2}$ below $\sim$30 K, that is consistent with the fact that the optical conductivity in the low-energy range starts to decrease below about 60 K.[17]

As origins of this resistivity maximum, there are two possibilities depending on the magnitude of Kondo temperature T_K compared with the crystalline electric field (CEF) splitting. In the Ce compounds such as $CePd_3$ with relatively high T_K comparable with the CEF excitation Δ, the temperature of the resistivity maximum ($\sim$100 K) roughly corresponds to T_K.[26] In such systems, the magnetic susceptibility χ exhibits a peak near T_K, however, χ in $CeOs_4Sb_{12}$ monotonously increases with decreasing temperature without any sign of peak structure.[12] On the other hand, in low T_K Ce compounds such as $CeAl_2$, double peaks in $\rho(T)$ have been observed as a function of temperature,[27] and are related with the two Kondo temperatures; T_K^h at high temperatures associated with all the CEF split levels and T_K^l for low temperatures resulting from only the CEF ground state for Ce-heavy fermion compounds. According to Hanzawa *et al.*,[28] T_K^h and T_K^l are related as

$$T_K^h = (T_K^l \Delta_1 \Delta_2)^{1/3}, \tag{3.1}$$

where Δ_1 and Δ_2 are the CEF-splitting between the ground state and the first and the second excited states, respectively. Putting $\Delta = \Delta_1 = \Delta_2 = 327$ K (between Γ_7 ground state and Γ_8 excited state estimated from the temperature dependence of magnetic susceptibility[12]) and $T_K^h = 100$ K at the resistivity maximum, T_K^l is estimated as $\sim$10 K, leading to the specific-heat coefficient $\gamma \sim 1000$ mJ/K^2 mol.[29] The experimental value of specific-heat coefficient $\gamma \sim 180$ mJ/K^2 mol,[16] is rather close to that in the high T_K scenario of $\gamma \sim 100$ mJ/K^2 mol, which contradicts with the experimental result. However, it should be noted that the peak temperature

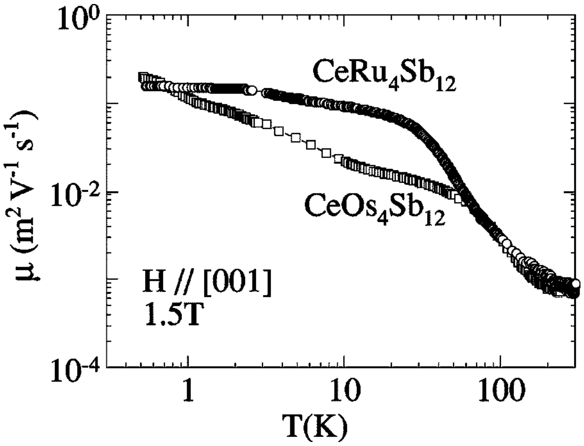

FIG. 3. Temperature dependences of Hall mobility μ in $CeOs_4Sb_{12}$ and $CeRu_4Sb_{12}$.

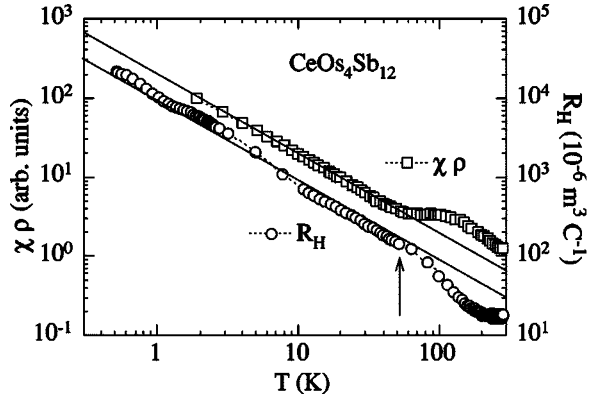

FIG. 4. Temperature dependences of $\chi\rho$ in $CeOs_4Sb_{12}$: analysis of the anomalous Hall effect (skew scattering).

~18 K under 14 T is close to the estimated T_K^l and the magnetic contribution to the resistivity at 0 T shown in the inset of Fig. 2(a) follows a $-\ln T$ dependence only within the narrow temperature range 10–30 K.

Both the dc and the optical conductivities indicate the decreasing carrier number below ~60 K, which suggests the model to explain the low-temperature properties of this material must have the temperature-dependent carrier number and electronic density of states. $\rho(T)$ above ~60 K can be understood as of the ordinary Ce Kondo compound with a peak at ~100 K corresponding to T_K or T_K^h. At lower temperatures, the increases in ρ and R_H indicate the reduction of density of states at E_F. However, the approximate $T^{-1/2}$ dependence of ρ in such a wide temperature range (of about two orders of magnitude) down to 0.6 K rules out the simple semiconducting state as an origin.

Such a temperature dependence of ρ could be ascribed to the temperature dependence of carrier density n and scattering lifetime of electrons τ, even if we assume the simplest single FS model. By combining R_H, where only the temperature dependence of n plays a role, we might be able to separate the two contributions. Figure 2(b) shows the temperature dependence of R_H for $CeOs_4Sb_{12}$ along with those for the reference compounds $LaOs_4Sb_{12}$ and $CeRu_4Sb_{12}$. At high temperatures, R_H is positive for all the three compounds and the magnitude is not much different. The estimated carrier density is between 1.0–1.4 holes/f.u. at RT. The T dependence of R_H for $LaOs_4Sb_{12}$ is not so large, but have a broad minimum near ~40 K. The decrease down to 40 K is ascribed to the change in the main scattering centers from the isotropic phonon-scattering with large wave vectors (**q**) to the anisotropic phonon scattering with smaller **q**, and the increase below ~40 K reflects the recovery to the isotropic scattering by impurities.[30,31]

Near RT, R_H for both $CeOs_4Sb_{12}$ and $CeRu_4Sb_{12}$ increases drastically with decreasing temperatures. Such an increase in R_H have been observed in many Ce-based Kondo compounds related with the Kondo-like increase in resistivity,[32] however, the magnitude is unusually larger than that was reported previously; usually R_H shows a peak of the magnitude less than 10^{-8} m^3/C near T_K. For $CeRu_4Sb_{12}$, R_H tends to saturate to 5.7×10^{-8} m^3/C below ~50 K, which is consistent with the temperature dependence of the electronic density of states at E_F in the optical measurement.[33] Interestingly, R_H in $CeOs_4Sb_{12}$ shows approximately T^{-1} dependence in the same temperature range where ρ varies approximately as $T^{-1/2}$. This fact automatically rules out the simple assignment of the origin of the $T^{-1/2}$ dependence in resistivity to the T dependence of carrier density. In the case of Kondo insulator CeNiSn, the inconsistent temperature dependence between ρ and R_H (the decrease in ρ contradicts with the decreasing carrier number estimated from R_H with decreasing temperature) has been ascribed to the increase in the carrier mobility with decreasing temperature.[34] The temperature dependences of Hall mobility $\mu = R_H/\rho$ in $CeOs_4Sb_{12}$ and $CeRu_4Sb_{12}$ are compared in Fig 3. Both the magnitude and the temperature dependence of μ for the two compounds are very close above ~60 K, where no drastic change in the electronic density of states at E_F has been reported.[17,33] Thus, the initial rise in μ with decreasing temperature in Fig. 3 could not be ascribed to the increase in the carrier mobility within the normal Hall effect contribution, but is more naturally ascribed to the anomalous Hall effect (the skew scattering). In addition, for $CeOs_4Sb_{12}$, taking into account $T^{1/2}$ dependence of $1/T_1$ associated with the spin fluctuation near AFM critical point, the anomalous Hall component is expected to dominate also at lower temperatures. In many Kondo compounds, the T dependence of Hall coefficient roughly follows Eq. (3.2):[35]

$$R_H(T) = R_{H0} + R_S(T)\chi(T), \tag{3.2}$$

where $R_S(T)$ is a function of the magnetic part of electrical resistivity. $R_S(T)$ in Kondo materials has been well described by the skew component under selected conditions. For rough estimation, we have calculated the skew component based on the simplest assumption of $R_S(T) = \alpha\rho(T)$; the coefficient α takes different values above and below T_K depending on the phase shift associated with the scattering channels,[35] $\rho(T)$ is the electrical resistivity under 1.5 T simultaneously measured with Hall coefficient. The result is compared with the experimental one in Fig. 4, where the main characteristic of $R_H(T)$; the almost T^{-1} dependence on temperature, is roughly reproduced, taking into account the oversimplification of the

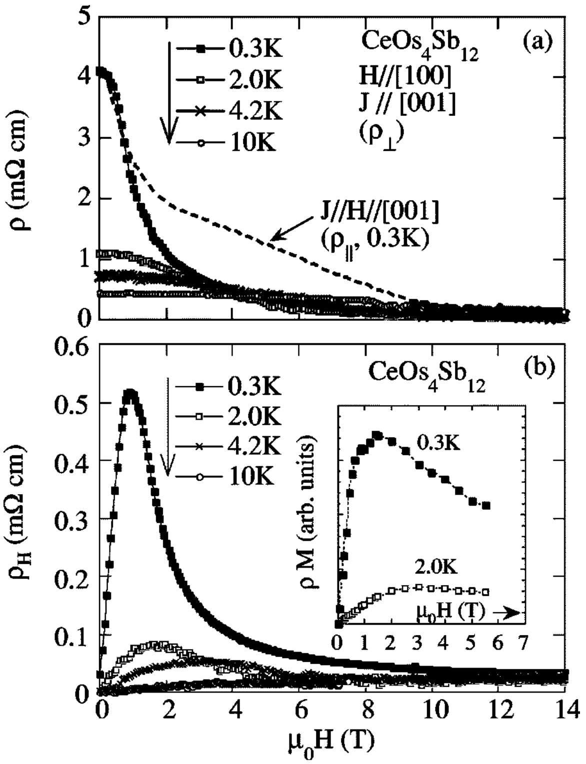

FIG. 5. Field dependences of (a) the electrical resistivity $\rho(H)$ and (b) Hall resistivity $\rho_H(H)$ in $CeOs_4Sb_{12}$.

model. The difference in curvature above $\sim$50 K may be ascribed to the change in sign of α above and below T_K.

To further understand the unusual temperature dependence of $\rho(T)$ and $R_H(T)$ in Figs. 2(a) and 2(b) at low temperatures, the resistivity and Hall resistivity at selected temperatures have been measured as a function of magnetic field in Figs. 5(a) and 5(b), respectively. $\rho(H)$ is dramatically suppressed; especially at 0.3 K, it is reduced to $\sim 1/70$ under 14 T. ρ_H exhibits a peak near 0.9 T above which it also shows a drastic reduction. In the form $R_H^* = \rho_H/\mu_0 H$, the reduction factor $\sim 1/350$ is about five times larger than that in ρ, which is unexplainable only by a carrier density change in the simplest single carrier model. This drastic field effect is in sharp contrast with the pressure effect on ρ reported to be minor in $CeOs_4Sb_{12}$.[21]

These field dependences might be ascribed to the combined effect of changes in carrier number and in carrier scattering intensity. The former alone in the simplest single carrier model is unable to explain both $\rho(T)$ and $R_H(T)$ as was already mentioned related with the mobility in Fig. 3. In magnetic systems, the anomalous Hall component sometime predominates over the normal one; i.e., the Hall resistivity composed of the normal component proportional to magnetic field H and anomalous one proportional to magnetization M as

$$\rho_H(H) = R_{H0}H + R_S M(H). \qquad (3.3)$$

Using $\rho(H)$ in Fig. 5(a) and $M(H)$ measured by a superconducting quantum interference device (SQUID) magnetometer, the second term in Eq. (3.3) is plotted in the inset of Fig. 5(b).[36] The peak structure can be reproduced, however, the agreement of both the position and the width are not satisfactory. It must be noted that the theory on the anomalous Hall effect assumes ordinary metallic Kondo systems with a basically constant carrier concentration. To make quantitative comparison, a model taking into account the temperature dependence of carrier number is necessary.

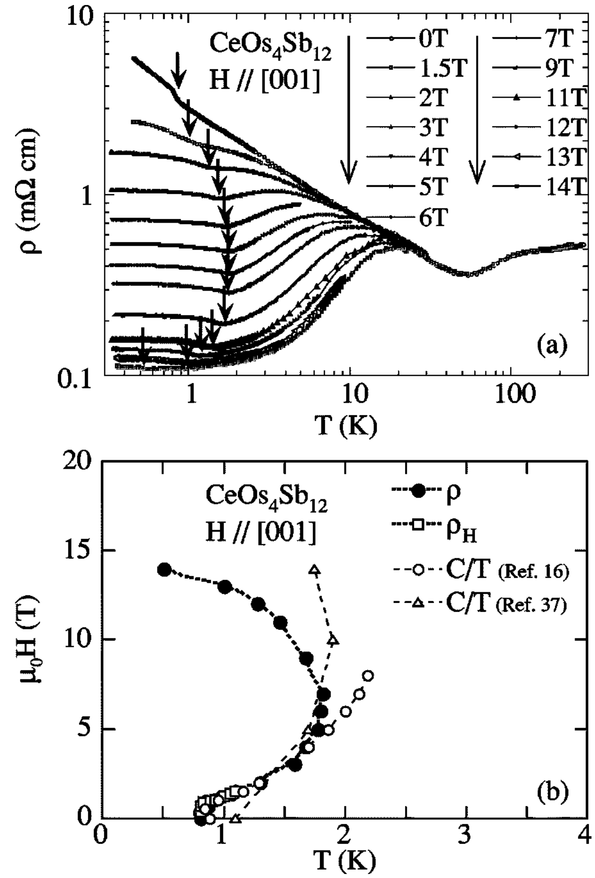

FIG. 6. (a) Temperature dependence of the electrical resistivity under selected magnetic fields and (b) *H-T* phase diagram in $CeOs_4Sb_{12}$. The higher field triangle point in the phase diagram was estimated from a faint peak in the specific heat in Ref. 37.

To understand another characteristic feature in Fig. 2, a small but clear anomaly at 0.8 K where some phase transition was found in the specific-heat measurements,[6,16] we have measured the temperature dependence of ρ as shown in Fig. 6(a) along with ρ_H (not shown) under selected magnetic fields. The position of the anomaly shifts with magnetic field, which is plotted in Fig. 6(b) as a *H-T* phase diagram where the anomalies found in the specific-heat measurements are also plotted.[16,37] Above about 4 T, the phase boundary determined by the transport measurements deviates from that by the specific-heat measurement, which might be due to the small misalignment of crystalline direction to the magnetic field.

Recently, Rotundu and Andraka also determined the *H-T* phase diagram for $H \parallel [100]$ up to 10 T based on the specific-heat measurements.[37] Their data points are in between those in the present experiment and those in the specific-heat measurement by Namiki *et al.* below 10 T above which the anomaly is almost invisible in $C(T)$. On the other hand, $\rho(T)$ in the present measurements exhibits an evident anomaly up to 14 T, though the magnitude becomes quite small.

The feature of phase diagram is reminiscent of the antiferroquadrupole (AFQ) transition observed in CeB_6.[39] However, as already pointed out from the $C(T)$ measurements,[6,16] the electronic part of the entropy release ($0.05R \ln 2$ at 0 T and $0.06R \ln 2$ at 4 T) below the transition temperature is too small to be attributed to localized f-electron contributions. Itinerant nature of $4f$ electrons is also suggested by Bauer *et al.* in relation to the Wilson ratio $R_W=(\pi^2 k_B^2/3\mu_{\rm eff}^2)(\chi_0/\gamma)$ which is of the order of unity for $CeOs_4Sb_{12}$.[12] Here $\mu_{\rm eff}$ is the effective magnetic moment, χ_0 is the Pauli susceptibility, and γ is the Sommerfeld coefficient. Moreover, the possibility of an AFQ transition in cubic system requires that the CEF ground state is Γ_8 quartet, however, from the magnetic susceptibility measurements,[12] the Γ_7 ground state is suggested for $CeOs_4Sb_{12}$ inconsistent with the AFQ scenario. Taking into account the minor change in ρ and R_H across the transition along with the anomalous Hall contribution reflecting the conduction electron scattering by magnetic instability, the transition at 0.8 K may be ascribed to the spin-density-wave (SDW) order. Yogi *et al.* have found a clear anomaly at $\sim$0.9 K in a recent Sb-NQR experiment,[23] which was ascribed to the onset of SDW order.

Rotundu and Andraka pointed out that their finding of a sizable decrease of Sommerfeld coefficient above 5 T is in sharp contrast with the Kondo insulators,[37] such as CeNiSn; where magnetic fields increase the Sommerfeld coefficient by destroying the c-f hybridization and closing the energy gap.[38] The quenching of spin fluctuations by magnetic fields is a possible explanation for the apparent decrease of Sommerfeld coefficient,[40] which also indirectly suggests the origin of phase transition to be the SDW order. However, the relation between the SDW order and the AFQ-like phase diagram is still unclear. $Ce(Ru_{1-x}Rh_x)_2Si_2$ ($x=0.05-0.25$) is also reported to exhibit a SDW order below 6 K.[41] However, there exists a clear difference between the two compounds; in $Ce(Ru_{1-x}Rh_x)_2Si_2$, the change in carrier number may be minor, even if it exits, since it has a metallic ground state and the H-T phase diagram exhibits an ordinary AFM-like one. Therefore, one can infer that the change in carrier density might play some role in the unusual H-T phase diagram of $CeOs_4Sb_{12}$. To elucidate the origin of the phase transition and to establish the phase diagram including anisotropy, more intense studies on other physical properties are necessary.

Finally, we discuss the comparison of the transverse and longitudinal magnetoresistance as shown in Fig. 5(a). ρ is larger in the longitudinal ($\rho_\parallel$) than in the transverse geometry ($\rho_\perp$) above $\sim$1 T. If only the Lorenz magnetoresistance plays a role, $\rho_\perp$ is never smaller than $\rho_\parallel$. If one assumes the conventional form of exchange interaction $V=-J\mathbf{s}\cdot\mathbf{S}$ between a conduction electron with spin **s** and a magnetic impurity with spin **S**, the magnetoresistance is isotropic; independent of the relative orientation of the current and magnetic field directions. Fert reported experimental results of anisotropic magnetoresistance due to heavy rare-earth ions in Au,[42] which is explained by taking into account the quadrupolar interaction. He has also calculated the anisotropic magnetoresistance for Ce impurities in La,[43] which qualitatively explains the experimental result. At this stage, we cannot say any conclusive remark on the anisotropic magnetoresistance in Fig. 5(a), however, it should be noted that some contribution from the orbital angular moment is necessary to explain such a anisotropic magnetoresistance. That might be also related with the unusual H-T phase diagram, in which the strong correlation between $4f$ and conduction electrons is expected.

In summary, we have found an unusual temperature dependence of both electrical resistivity and Hall coefficient below $\sim$30 K in $CeOs_4Sb_{12}$, which may be ascribed to the combined effect of carrier density decreasing and spin fluctuations. The anomalous low-temperature state with large electrical resistivity is considerably suppressed by the magnetic fields, suggesting a drastic change of electronic structure and suppression of spin fluctuations by the magnetic field.

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