Transport properties of the heavy-fermion superconductor $PrOs_4Sb_{12}$

H. Sugawara,* M. Kobayashi, S. Osaki, S. R. Saha, T. Namiki, Y. Aoki, and H. Sato

Graduate School of Science, Tokyo Metropolitan University, Minami-Ohsawa, Hachioji, Tokyo 192-0397, Japan

We have measured the electrical resistivity, thermoelectric power, Hall coefficient, and magnetoresistance (MR) on single crystals of $PrOs_4Sb_{12}$, $LaOs_4Sb_{12}$, and $NdOs_4Sb_{12}$. All the transport properties in $PrOs_4Sb_{12}$ are similar to those in $LaOs_4Sb_{12}$ and $NdOs_4Sb_{12}$ at high temperatures, indicating the localized character of $4f$ electrons. The transverse MR both in $LaOs_4Sb_{12}$ and $PrOs_4Sb_{12}$ tends to saturate for wide field directions, indicating these compounds to be uncompensated metals with no open orbit. We have determined the phase diagram of the field induced ordered phase by the MR measurement for all the principle field directions, which indicates an unambiguous evidence for the Γ_1 singlet crystalline electric field ground state.

I. INTRODUCTION

$PrOs_4Sb_{12}$ with the filled skutterudite structure ($Im\bar{3}$) was reported to be the first Pr-based heavy-fermion (HF) superconductor.[1,2] The heavy mass has been suggested by the large specific-heat jump $\Delta C/T_C \sim 500$ mJ/K^2 mol at the superconducting (SC) critical temperature $T_C = 1.85$ K, and directly confirmed by the de Haas–van Alphen (dHvA) experiments.[3] From the various microscopic measurements; i.e., Sb-nuclear quadrupole resonance (NQR) and muon spin relaxation (μSR), etc.,[4–6] this material has been recognized as an unconventional superconductor which differs from Ce- and U-based HF superconductors reported to date. Specific heat and magnetic susceptibility measurements in $PrOs_4Sb_{12}$ indicate the nonmagnetic crystal electric field (CEF) ground state of Pr^{3+} ions,[1,2,7–10] which is a sharp contrast with the existing HF superconductors; the existing HF superconductor has magnetic ground state and the magnetic fluctuation is believed to mediate the superconducting pairing. The unconventional nature of this material is also inferred from the unusual SC multiple phase diagram evidenced by the double SC transitions in the specific heat and also by the thermal conductivity measurements which indicates anisotropic SC gap.[2,8,11–13] Recent zero-field μSR measurements have revealed the appearance of spontaneous internal fields below T_C, providing an evidence for the breaking of time reversal symmetry in the SC state.[6] As another interesting feature of this material, an anomalous field-induced ordered phase (FIOP) was first observed in the magnetoresistance (MR) measurement[14] and confirmed by the specific-heat measurement under magnetic fields.[7] Recent neutron-diffraction experiments has suggested this ordered phase to be an anitiferroquadrupolar (AFQ) ordering.[15] For better understanding of this exotic SC state, it is quite important to clarify the nature of the field-induced ordered state.

The exotic superconducting properties of $PrOs_4Sb_{12}$ have promoted intense research activities based on various experimental techniques mentioned above. In contrast, the physical properties of the reference compounds $LaOs_4Sb_{12}$ and $NdOs_4Sb_{12}$ have been only poorly investigated despite its importance for understanding the unusual properties of $PrOs_4Sb_{12}$. For the transport properties of $PrOs_4Sb_{12}$, the re-

ported data until now have been limited mostly to the electrical resistivity $\rho(T)$ and MR.[2,14,16–18] Ho *et al.* have reported the MR and an anomaly related to FIOP in $PrOs_4Sb_{12}$,[14,17,18] although their measurement is limited only on MR without any description on the angular dependence. Frederick and Maple analyzed their $\rho(T)$ and MR data based on the CEF theory and suggested that the CEF ground state to be Γ_3 (Γ_{23} in T_h notation[19]) doublet.[20] Their interpretation of CEF as an origin of both the decrease (or roll-off) in $\rho(T)$ below 5 K and the FIOP may be correct, however, recent experiments show inconsistency with the Γ_3 ground state model; the results of recent specific-heat, magnetization, and neutron experiments,[7,9,10,15,21,22] are well describable by the Γ_1 singlet ground state model. Therefore, it is important to investigate anisotropy of FIOP to settle the CEF ground state.

In this paper, we report the systematic study of transport measurements; i.e., electrical resistivity ρ, thermoelectric power (TEP) S, and Hall coefficient R_H in comparison with those on the reference compounds $LaOs_4Sb_{12}$ and $NdOs_4Sb_{12}$. We have also measured the MR for the magnetic fields along all the principle crystalline directions to investigate the anomalous FIOP.

II. EXPERIMENTAL

High-quality single crystals of ROs_4Sb_{12} [R (rare earth): La, Pr, and Nd] were grown by Sb-self-flux method starting from a composition $R:Os:Sb = 1:4:20$,[23,24] using high-purity raw materials 4N (99.99% pure)-La, 4N-Pr, 4N-Nd, 3N-Os and 6N-Sb. The typical forms of the single crystals are cubic or rectangular shape with a largest dimension of about 3 mm. By powder x-ray-diffraction experiments, we confirmed that the lattice constants agree with the reported values,[25] and the absence of impurity phases within the experimental accuracy. The residual resistivity ratios (RRR) of the present samples are ~ 100 for $LaOs_4Sb_{12}$, ~ 50 and ~ 36 for $PrOs_4Sb_{12}$ (No. 1) and $PrOs_4Sb_{12}$ (No. 2), respectively, indicating the high quality of the present samples, as was confirmed by the observation of the dHvA oscillations.[3] The quality of $NdOs_4Sb_{12}$ is slightly lower (RRR ~ 18).
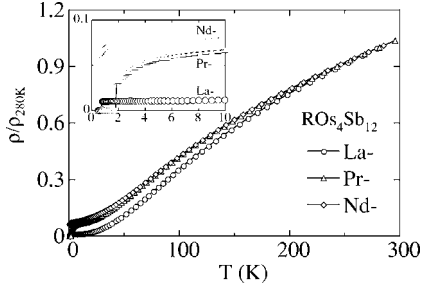
FIG. 1. Temperature dependence of the electrical resistivity normalized at 280 K in ROs$_4$Sb$_{12}$ (R: La, Pr, and Nd). The inset shows the expanded view below 10 K.

ρ and R_H were measured by the ordinary four-probe dc method. S was measured by the differential method using Au-0.07% Fe versus Chromel thermocouples. The high field MR was measured in a top loading ^{3}He-refrigerator cooled down to 0.3 K with a 16 T superconducting magnet.

III. RESULTS AND DISCUSSION

A. Electrical resistivity

Figure 1 shows the temperature dependence of electrical resistivity $\rho(T)$ normalized at 280 K for ROs$_4$Sb$_{12}$ (R: La, Pr, and Nd).[26] $\rho(T)$ for PrOs$_4$Sb$_{12}$ is qualitatively the same as the previous reports.[1,2] The similar metallic behavior of $\rho(T)$ for these compounds above $\sim$10 K suggests a localized nature of 4f electrons in ROs$_4$Sb$_{12}$ (R; Pr and Nd). As shown in the inset of Fig. 1, we have found the superconductivity in LaOs$_4$Sb$_{12}$ below 0.74 K, which was also confirmed by the NQR and specific-heat measurements.[4,27] It should be noted that the T_C of PrOs$_4$Sb$_{12}$ is higher than that of LaOs$_4$Sb$_{12}$. That is unusual, since PrOs$_4$Sb$_{12}$ contains Pr ions with 4f electrons. Actually, for the ordinary Pr-based filled skutterudite superconductor PrRu$_4$Sb$_{12}$($T_C \sim$ 1 K), T_C is lower than that for LaRu$_4$Sb$_{12}$ ($T_C \sim$ 3.5 K).[28,29]

Figure 2 shows the SC H-T phase diagram for LaOs$_4$Sb$_{12}$ determined by the field dependence of electrical resistivity at selected temperatures and the temperature dependence of ac susceptibility χ_{ac} under selected magnetic fields. Maple *et al.* also reported the superconductivity of LaOs$_4$Sb$_{12}$,[17] although
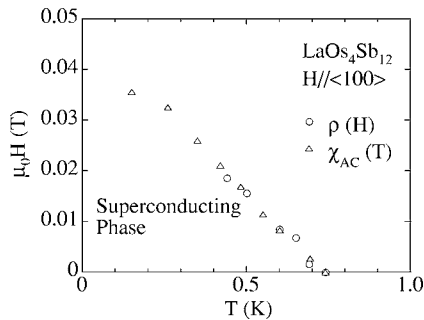

FIG. 2. Superconducting phase diagram in LaOs$_4$Sb$_{12}$, determined by the field dependence of electrical resistivity and ac susceptibility. The dotted line is guide for eyes.

TABLE I. Comparison of the superconducting critical temperature T_C, upper critical field $H_{C2}(0)$, coherence length ξ_0, penetration depth λ, and Ginzburg-Landau parameter κ between LaOs$_4$Sb$_{12}$ and PrOs$_4$Sb$_{12}$.

	LaOs$_4$Sb$_{12}$	PrOs$_4$Sb$_{12}$
T_C(K)	0.74[a]	1.85[b]
$H_{C2}(0)$(T)	0.035[a]	2.45[b]
ξ_0(Å)	970[a]	116[b]
λ(Å)	4700[d]	3440[c]
$\kappa=\lambda/\xi_0$	4.8	30

[a]Present work.
[b]Reference 1.
[c]Reference 5.
[d]Reference 27.

$T_C \sim$ 1 K is slightly higher and $H_{C2} \sim$ 0.6 T is more than an order of magnitude larger than that of our results in their experiment. At the present stage, we do not know the origin of such a large discrepancy, though we can say that the sample dependence is not so large within the samples we have grown; we have confirmed the almost the same value of T_C and H_{C2} for different several samples and also by different measurements (i.e., ρ, χ_{ac}, NQR, and specific-heat measurement). From the slope of the upper critical field H_{C2} near $T_C(-dH_{C2}/dT)_{T_C}$=0.068 T/K, the critical field at 0 K $H_{C2}(0)$ and coherence length ξ_0 are obtained following Ref. 1, which are summarized in Table I compared with PrOs$_4$Sb$_{12}$. The rapid decrease in $\rho(T)$ for NdOs$_4$Sb$_{12}$ below $\sim$0.8 K as shown in the inset of Fig. 1 might indicate a ferromagnetic transition, since it shifts to higher temperatures with increasing magnetic field.

B. Thermoelectric power

Figure 3 shows the temperature dependence of TEP $S(T)$ for ROs$_4$Sb$_{12}$ (R: La, Pr, and Nd). The TEPs of all the compounds are not describable only by a simple combination of the diffusion and the phonon drag TEP. For LaOs$_4$Sb$_{12}$, the TEP shows a broad negative peak near 80 K, for which there are two possible explanations. The peak temperature is not so far from $\Theta_D/5(\sim$60 K) expected for the phonon drag peak (one fifths of the Debye temperature Θ_D), taking into account the reported value of Θ_D=304 K.[24] However, PrOs$_4$Sb$_{12}$
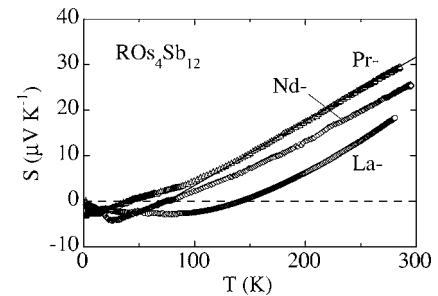

FIG. 3. Temperature dependence of the thermoelectric power in ROs$_4$Sb$_{12}$ (R: La, Pr, and Nd).
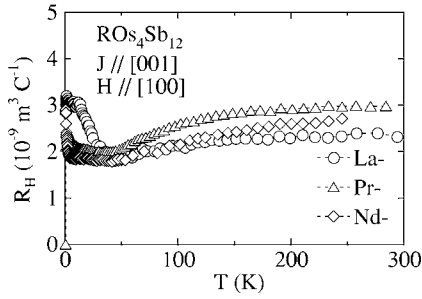
FIG. 4. Temperature dependence of the Hall coefficient in ROs_4Sb_{12} (R: La, Pr, and Nd).

FIG. 5. Comparison of the field dependence of magnetoresistance both for the transverse ($\rho_\perp$) and longitudinal ($\rho_\parallel$) geometry in $PrOs_4Sb_{12}$.

does not show such a structure though it has almost the same Debye temperature, that in turn suggests the phone drag is not the main contribution for such a low-temperature structure in $LaOs_4Sb_{12}$. Another possible origin is some fine structure in the density of states $N(\varepsilon)$ near the Fermi level ε_F, since the diffusion themoelectric power can be represented as $-(\pi^2 k_B^2 T/3|e|)[dN(\varepsilon)/d\varepsilon]_{\varepsilon_F}$ in the simplest free electron model.[30] According to the band structure calculations,[31] ε_F is located in between a large peak and a small peak in the density of states; the energy difference between these two peaks is $\Delta E/k_B \sim 100$ K. At low temperature, the positive slope of $N(\varepsilon)$ at ε_F leads to the negative $S(T)$. With increasing temperature, the small peak structure becomes thermally smeared, and the averaged slope of $N(\varepsilon)$ near ε_F becomes negative, reading to the positive $S(T)$ at high temperature. Such temperature dependence of averaged slope of $N(\varepsilon)$ may be an origin of the low-temperature structure of TEP in ROs_4Sb_{12}. The slope of $S(T)$ at higher temperatures is not much different among the three compounds, indicating basically the same electronic structure. The slope $dS/dT \sim 0.14$ μV/K^2 is an order of magnitude large compared to ordinary sp metals and is similar to $3d$-transition metals such as Ni, Pd, and Pt,[32] that is consistent with the large electronic density of states at Fermi level predicted by the band structure calculation.[3,31]

C. Hall coefficient

Figure 4 shows the temperature dependence of Hall coefficient $R_H(T)$ for ROs_4Sb_{12} (R: La, Pr, and Nd). The Hall coefficient is almost temperature independent at high temperatures irrespective of R. The absolute value is also not much different among the three compounds, taking into account relatively large error in the geometrical determination. The carrier number at higher temperatures for $PrOs_4Sb_{12}$ is $n = 2.1 \times 10^{27}$/m^3 (0.85 holes/molecular unit) assuming a single carrier model. That is consistent with the result of dHvA experiments,[3] which is well explained by the band structure calculation predicting two pieces of holelike Fermi surfaces (FS) and a multiply connected one; the number of holes within the two hole-like surfaces gives $n = 1.1 \times 10^{27}$/m^3. The weak temperature dependence below ~ 150 K could be ascribed to the temperature dependent anisotropy of relaxation time,[34] which is not unusual as was observed even in simple metals such as Al and Pb.[35] For
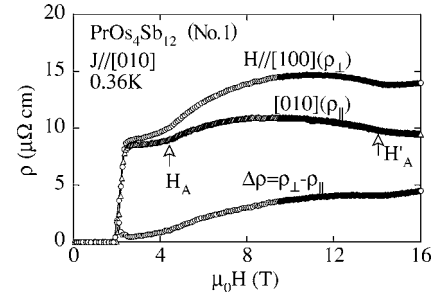
$LaOs_4Sb_{12}$, the decrease down to 40 K is ascribed to the change in the main scattering centers from the isotropic phonon scattering with large wave vectors $\mathbf{q}$ to the anisotropic phonon scattering with smaller $\mathbf{q}$, and the increase below ~ 40 K reflects the recovery to the isotropic scattering by impurities. The rapid changes both in $PrOs_4Sb_{12}$ and $NdOs_4Sb_{12}$ at low temperature are ascribed to the superconducting and ferromagnetic transition, respectively.

On $\rho(T)$, $S(T)$, and $R_H(T)$ in $PrOs_4Sb_{12}$, qualitative comparison to those in $PrFe_4P_{12}$ might be of interest, since both compounds exhibit HF behaviors which are very rare as Pr-based compounds. Among Ce-based dense Kondo compounds, $-\ln T$ dependence in $\rho(T)$, a large positive peak in $S(T)$, anomalous Hall effect in $R_H(T)$ are well known as common features associated with the Kondo scattering (cooperated with the crystal field). For $PrFe_4P_{12}$, an apparent $-\ln T$ dependence in $\rho(T)$, huge negative TEP peak (~ 70 μV/K), and the skew scattering in $R_H(T)$ have been found above the AFQ transition temperature,[33] which are thought to be supportive evidences of an intense Kondo effect. In contrast, no such drastic features appear in $PrOs_4Sb_{12}$ indicative well localized nature of $4f$ electrons. For $PrFe_4P_{12}$ with the smaller lattice constant ($\simeq 7.8$ Å), the stronger c-f hybridization is expected. On the other hand, for $PrOs_4Sb_{12}$ with larger lattice constant ($\simeq 9.3$ Å), the c-f hybridization is expected to be weaker. The difference of c-f hybridization strength might be the origin of large differences of transport properties between these two compounds.

D. Magnetoresistance

Figure 5 shows the comparison of the field dependence of MR between the transverse ($\rho_\perp$) and longitudinal ($\rho_\parallel$) geometry in $PrOs_4Sb_{12}$. The difference between the two geometries $\Delta\rho = \rho_\perp - \rho_\parallel$ is positive and could be mainly ascribed to the ordinary Lorentz MR contribution. Figures 6 and 7 show the angular and field dependences of transverse MR for the field along principal crystallographic directions in $PrOs_4Sb_{12}$ and $LaOs_4Sb_{12}$, respectively, where θ both in the insets of Figs. 6 and 7 represents to the field angle from the [001] direction. As shown in Fig. 5, $\Delta\rho$ shows a saturating tendency with increasing field. Combined with the small angular dependence in the inset of Fig. 6, $PrOs_4Sb_{12}$ is judged to
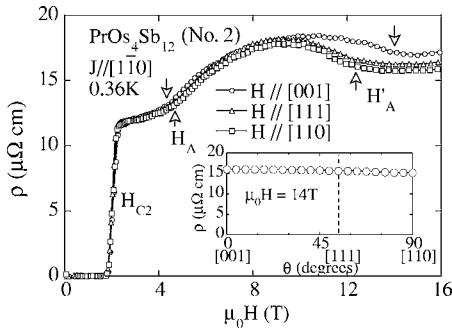
FIG. 6. Field dependences of the transverse magnetoresistance for field along the all principal directions in $PrOs_4Sb_{12}$. The inset shows the angular dependence of the transverse magnetoresistance.

FIG. 8. Comparison of the field dependence of Hall resistivity between $PrOs_4Sb_{12}$ and $LaOs_4Sb_{12}$. The inset shows the comparison of magnetization curves between these two compounds.

be an uncompensated metal with no open orbit. That is also confirmed in $LaOs_4Sb_{12}$ as shown in Fig. 7, whose FS topology is almost the same as that in $PrOs_4Sb_{12}$.[3] The difference in angular dependence between those insets of Figs. 6 and 7 may be ascribed to $4f$ contribution in $PrOs_4Sb_{12}$ where the larger residual resistivity suppresses the ordinary MR contribution compared to $LaOs_4Sb_{12}$.

As shown in Fig. 5, for both the transverse and longitudinal geometries in $PrOs_4Sb_{12}$, there exists positive contribution due to the scattering with magnetic and/or orbital degree of freedom, which is dominating in the field induced ordered phase inferred from the two anomalies H_A and H'_A, indicated by the arrows. Important finding in this experiments is the existence of the anomaly in MR indicating the phase boundary of the FIOP for all the principal field directions as shown in Fig. 6. The result for $H \| [100]$ agrees with that reported by Ho *et al.* except the absolute value of the resistivity.[18] The domelike structure between H_A and H'_A has been explained by Frederick and Maple as due to the conduction electron scattering associated with the CEF excitation.[20] Taking into account the fact that the magnetic moment of Pr is enhanced in the FIOP,[15] the resistivity increase in the FIOP could be most naturally ascribed to the enhancement of electron scattering associated with the CEF excitation. The first CEF excited state is reported to locate above $\Delta_1/k_B \sim 8$ K both in Γ_3 and Γ_1 ground-state models,[1,7,10,15] which naturally explains the gradual decrease of $\rho(T)$ below ~ 5 K under 0 T as
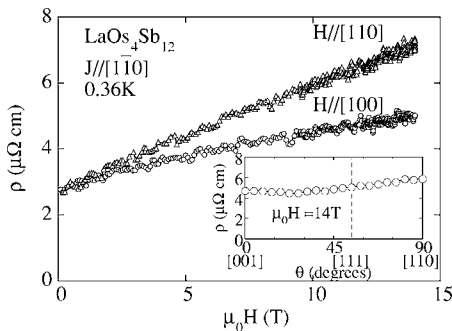

FIG. 7. Field dependences of the transverse magnetoresistance for field along the [100] and [110] directions in $LaOs_4Sb_{12}$. The inset shows the angular dependence of the transverse magnetoresistance.
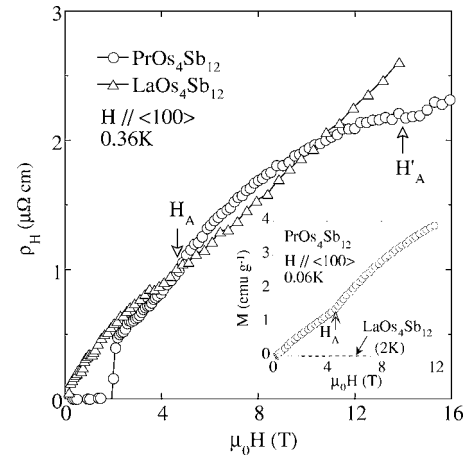
shown in the inset of Fig. 1. At low temperatures far below Δ_1/k_B, the electron scattering associated with CEF excitation is suppressed in zero field. For the Γ_1 ground state model,[7,10,15] with increasing field, one out of Γ_5 ($\Gamma_4^{(2)}$ in T_h notation[19]) triplet excited state comes down close to the ground state, resulting in enhancement of electrical resistivity associated with CEF excitation. After showing a maximum at a crossing field ~ 9 T of the two levels, the electrical resistivity decreases with increasing gap between the two levels. The closeness of the two levels is thought to be a key factor to stabilize the FIOP.[15] It should be noted that the level crossing is expected for field along the all principal crystallographic directions in the Γ_1 ground state model,[10,36,37] whereas no level crossing is expected for fields parallel to the $\langle 110 \rangle$ and $\langle 111 \rangle$ directions in the Γ_3 ground state model.[10] Thus the present result indicates an unambiguous evidence for the Γ_1 CEF ground state in $PrOs_4Sb_{12}$.

As Frederick and Maple discussed,[20] the domelike structure in $\rho(H)$ near 9 T at low temperatures may be ascribed to the CEF excitation. In addition, we discuss here the origin of the sharp anomaly across the phase boundary. One possiblity is the slight change of FS associated with the superzone gap formation of new periodicity; namely, the AFQ ordering as was clarified by the neutron-scattering experiment.[15] Actually, as shown in Fig. 8, the field dependence of Hall resistivity $\rho_H(H)$ exhibits the slight slope changes across H_A and H'_A, in contrast with the almost linear increase of $\rho_H(H)$ in $LaOs_4Sb_{12}$. Also in the dHvA experiments,[3] the frequency of the β branch originating from the 48th hole FS has been confirmed to exhibit slight changes across H_A and H'_A. However, it could not be a decisive evidence for the FS change because the magnetization also changes nonlinearly at H_A as shown in the inset of Fig. 8,[9,10] leading to a superficial change in FS. Such a slight change of the dHvA frequency can be also ascribed to the combined effect of the spin splitting and nonlinear magnetization.[38] Thus, the change in FS across the FIOP boundary has not been settled yet, however, taking into account the normal Hall contribution is dominating in the Hall resistivity as shown in Fig. 8; namely the Hall
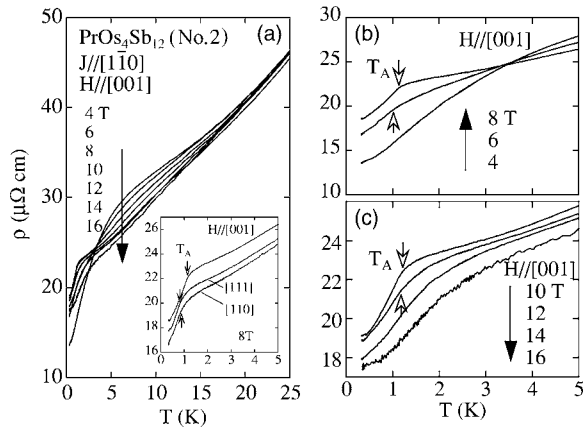
FIG. 9. Temperature dependence of the resistivity $\rho(T)$ at selected magnetic fields in $PrOs_4Sb_{12}$. The inset in (a) shows anisotropy of $\rho(T)$ at 8 T. (b) and (c) show $\rho(T)$ below 8 T and above 10 T, respectively. T_A represents to the transition temperature of FIOP.

FIG. 10. H-T phase diagram of $PrOs_4Sb_{12}$.

resistivities of two compounds with the same sign and similar magnitude naturally suggest the anomalous Hall effect[39] is not dominant in $PrOs_4Sb_{12}$, the FS does not drastically change in FIOP, that is in contrast with $PrFe_4P_{12}$ in which the apparent FS reconstruction has been observed across the AFQ ordering.[38]

In order to determine the H-T phase diagram for field along the three principal field directions, we have measured $\rho(T)$ for selected fields as shown in Fig. 9. We have also measured $\rho(T)$ for $H\|[110]$ and $H\|[111]$, which are basically the same as that for $H\|[100]$ except the difference in transition temperature T_A as shown in the inset of Fig. 9(a). A shoulder (or roll off) of $\rho(T)$ around $\sim$5 K associated with the CEF excitation is strongly suppressed by magnetic fields as shown in Fig. 9(a), suggesting the CEF excited state comes down approaching the ground state. As shown in Figs. 9(b) and 9(c), above 6 T, we can see a bend below T_A $=1.25$ K, which is apparently related with FIOP. With increasing magnetic fields, T_A increases and the bend becomes sharper (i.e., at 8 and 10 T). After showing a maximum around 10 T, T_A decreases rapidly and the anomaly disappears above 14 T. Figure 10 shows the H-T phase diagram determined by the present MR measurements along with specific-heat and dHvA experiments. The similar phase diagram has already reported by other groups,[17,18,40] however, there are no description about the field anisotropy in their reports. In the anisotropy determination of FIOP based on the magnetization measurements,[10] the field range was limited to only below 13 T. In the present MR measurements up to 16 T, the upper boundary H'_A of FIOP including the anisotropy has been determined for all the principal field directions: $H'_A \sim 14$ T for $H\|\langle 100\rangle$ and $H'_A \sim 12$ T for $H\|\langle 110\rangle$ and $H\|\langle 111\rangle$. We again emphasize that the FIOP exists for the field along all the three principal crystallographic directions.

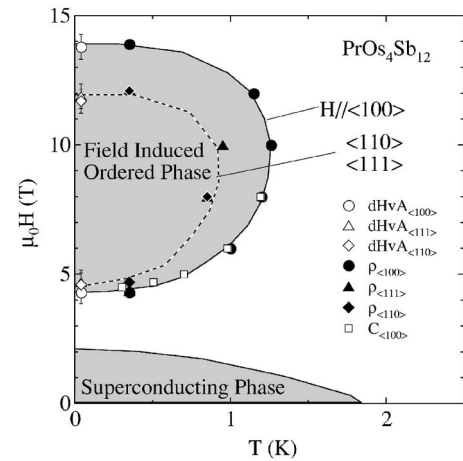The presence of AFQ phase close proximity to the superconducting phase remind us the Ce- and U-based HF superconductors and high-T_C oxides; in those systems the Cooper pairing is believed to be mediated by magnetic fluctuations.[41] By analogy, in $PrOs_4Sb_{12}$, the quadrupole fluctuations of Pr ions might play an important role in the HF-SC properties.

IV. CONCLUSIONS

All the transport properties of $PrOs_4Sb_{12}$, compared to those of the references $LaOs_4Sb_{12}$ and $NdOs_4Sb_{12}$, suggest the $4f$ electrons to be well localized at high temperatures, while the exotic behaviors associated with $4f$ electrons degree of freedom are observed below the temperature of order of CEF splitting between the ground state and the first excited state.

From the field dependence of MR both for the transverse and longitudinal geometry in $PrOs_4Sb_{12}$, we found that the ordinary MR contribution saturates for all the field directions. This fact suggests that $PrOs_4Sb_{12}$ is an uncompensated metal with no open orbit, which is consistent with the band structure calculation.

The dependences of MR on both field and temperature have revealed that the FIOP exists for the field along all the three principal crystallographic directions between 4.4 and 14 T below 1.25 K, which strongly supports the Γ_1 CEF ground-state model proposed based on the specific heat, magnetization, and neutron-diffraction measurements.

*Present address: Department of Mathematical and Natural Sciences, Faculty of Integrated Arts and Sciences. The University of Tokushima, Tokushima, 770-8502, Japan. E-mail: sugawara@ias.tokushima-u.ac.jp

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