

A NEW HEAVY FERMION SUPERCONDUCTOR: THE FILLED SKUTTERUDITE COMPOUND $\text{PrOs}_4\text{Sb}_{12}$ *

M.B. MAPLE, P.-C. HO, N.A. FREDERICK, V.S. ZAPF, W.M. YUHASZ,
AND E.D. BAUER

Department of Physics and Institute for Pure and Applied Physical Sciences
University of California, San Diego, La Jolla, CA 92093, USA

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The filled skutterudite compound $\text{PrOs}_4\text{Sb}_{12}$ exhibits superconductivity below a critical temperature $T_c = 1.85$ K that develops out of a non-magnetic heavy Fermi liquid with an effective mass $m^* \approx 50 m_e$, where m_e is the free electron mass. Analysis of magnetic susceptibility, specific heat, electrical resistivity and inelastic neutron scattering measurements within the context of a cubic crystalline electric field yields a Pr^{3+} energy level scheme that consists of a Γ_3 nonmagnetic doublet ground state that carries an electric quadrupole moment, a low lying Γ_5 triplet excited state at ~ 10 K, and Γ_4 triplet and Γ_1 singlet excited states at much higher temperatures. The superconducting state appears to be unconventional and to consist of two distinct superconducting phases. An ordered phase of magnetic or quadrupolar origin occurs at high fields and low temperatures, suggesting that the superconductivity may occur in the vicinity of a magnetic or electric quadrupolar quantum critical point.

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1. Introduction

The filled skutterudite compound $\text{PrOs}_4\text{Sb}_{12}$ has been reported to exhibit superconductivity below a critical temperature $T_c = 1.85$ K that develops out of a heavy Fermi liquid with an effective mass $m^* \approx 50 m_e$, where m_e is the free electron mass [1–4]. To our knowledge, $\text{PrOs}_4\text{Sb}_{12}$ is the first heavy fermion superconductor based on Pr; all of the other known heavy fermion superconductors (about 20) are compounds of Ce or U. The superconducting state appears to be unconventional in nature and may consist of two distinct superconducting phases [3, 5]. An ordered phase, presumably

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of magnetic or quadrupolar origin, occurs at high fields > 4.5 T and low temperatures < 1.5 K [3,4,6–9], suggesting that the superconductivity may occur in the vicinity of a magnetic or quadrupolar quantum critical point (QCP). Analysis of magnetic susceptibility $\chi(T)$, specific heat $C(T)$, electrical resistivity $\rho(T)$ and inelastic neutron scattering measurements within the context of a cubic crystalline electric field (CEF) yields a Pr^{3+} energy level scheme that consists of a Γ_3 nonmagnetic doublet ground state that carries an electric quadrupole moment, a low lying Γ_5 triplet excited state at ~ 10 K, and Γ_4 triplet and Γ_1 singlet excited states at much higher temperatures (~ 130 K and ~ 313 K, respectively) [1–4]. This scenario suggests that the underlying mechanism of the heavy fermion behavior in $\text{PrOs}_4\text{Sb}_{12}$ may involve the interaction of Pr^{3+} electric quadrupole moments with the charges of the conduction electrons, rather than the interaction of Pr^{3+} magnetic dipole moments with the spins of the conduction electrons, the interaction that is widely believed to be responsible for the heavy fermion state in most Ce- and U-based compounds. It also raises the possibility that electric quadrupole fluctuations play a role in the superconductivity of $\text{PrOs}_4\text{Sb}_{12}$. In this paper, we briefly review the current experimental situation regarding the heavy fermion state, the superconducting state, and a high field, low temperature phase that is apparently associated with magnetic or electric quadrupolar order in $\text{PrOs}_4\text{Sb}_{12}$.

2. Heavy fermion state in $\text{PrOs}_4\text{Sb}_{12}$

The first evidence for a heavy fermion state in the filled skutterudite compound $\text{PrOs}_4\text{Sb}_{12}$ emerged from specific heat $C(T)$ measurements on a $\text{PrOs}_4\text{Sb}_{12}$ pressed pellet (formed by pressing a collection of small single crystals in a cylindrical die) at low temperatures. Specific heat data in the form of a plot of C/T vs T between 0.5 K and 10 K for the $\text{PrOs}_4\text{Sb}_{12}$ pressed pellet from Refs. [1] and [2] are shown in Fig. 1. The $C(T)$ data have been corrected for excess Sb derived from the molten Sb flux in which the crystals were grown. The line in the figure represents the expression $C(T) = \gamma T + \beta T^3 + C_{\text{Sch}}(T)$, where γT and βT^3 are electronic and phonon contributions, respectively, and $C_{\text{Sch}}(T)$ is a Schottky anomaly for a two level system consisting of a doublet ground state and a triplet excited state at an energy Δ above the ground state. The best fit of this expression to the data yields the values $\gamma = 607$ mJ/mol K², $\beta = 3.95$ mJ/mol K⁴ (corresponding to a Debye temperature $\theta_D = 203$ K), and $\Delta = 7.15$ K. Superimposed on the Schottky anomaly is a feature in the specific heat due to the onset of superconductivity at $T_c = 1.85$ K which is also observed as an abrupt drop in $\rho(T)$ to zero and as a sharp onset of diamagnetism in $\chi(T)$. The feature in $C(T)/T$ due to the superconductivity is also shown

in the top inset of Fig. 1 along with an entropy conserving construction from which the ratio of the jump in specific heat ΔC at T_c , $\Delta C/T_c = 632$ mJ/mol K², has been estimated. Using the BCS relation $\Delta C/\gamma T_c = 1.43$, this yields a value for γ of 440 mJ/mol K². The value of $\Delta C/T_c$ is larger than that reported in Ref. [2] due to the correction of the $C(T)$ data for the excess Sb (about 30 percent of the total mass). This value is comparable to that inferred from the fit to the C/T vs T data in the normal state above T_c , and indicative of heavy fermion behavior. A similar analysis of the $C(T)$ data taken at the University of Karlsruhe on several single crystals of PrOs₄Sb₁₂ prepared in our laboratory yielded $\gamma = 313$ mJ/mol K², $\theta_D = 165$ K, $\Delta = 7$ K, and $\Delta C/\gamma T_c \approx 3$, much higher than the BCS value of 1.43 and indicative of strong coupling effects [6]. It is interesting that we also find a large value of $\Delta C/\gamma T_c \approx 3$ in recent $C(T)$ measurements on one single crystal of PrOs₄Sb₁₂ at UCSD. Although the values of γ determined from these experiments vary somewhat, they are all indicative of a heavy electron ground state and an effective mass $m^* \approx 50 m_e$.

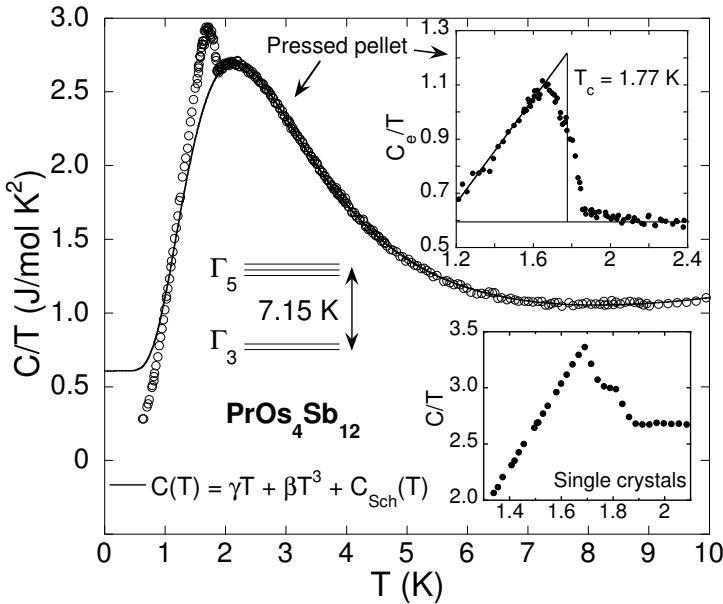


Fig. 1. Specific heat C divided by temperature T , C/T , vs T for a PrOs₄Sb₁₂ pressed pellet. The line represents a fit of the sum of electronic, lattice, and Schottky contributions to the data. Upper inset: C_e/T vs T near T_c for a PrOs₄Sb₁₂ pressed pellet (C_e is the electronic contribution to C). Lower inset: C/T vs T near T_c for PrOs₄Sb₁₂ single crystals, showing the structure in ΔC near T_c . Data from Ref. [1, 2].

Further evidence of heavy fermion superconductivity is provided by the upper critical field H_{c2} vs T curve which is shown in Fig. 2 [2, 3]. The orbital critical field $H_{c2}^*(0)$ can be derived from the slope of the H_{c2} curve near T_c and used to estimate the superconducting coherence length $\xi_0 \approx 116$ Å via the relation $H_{c2}^*(0) = \Phi_0/2\pi\xi_0^2$, where Φ_0 is the flux quantum. The Fermi velocity v_F can be obtained from the BCS relation $\xi_0 = 0.18\hbar v_F/k_B T_c$ and used to determine the effective mass m^* by means of the expression $m^* = \hbar k_F/v_F$. Using a simple free electron model to estimate the Fermi wave vector k_F , an effective mass $m^* \approx 50 m_e$ is obtained [2, 3]. Calculating γ from m^* yields $\gamma \sim 350$ mJ/mol K², providing further evidence for a heavy fermion state in PrOs₄Sb₁₂.

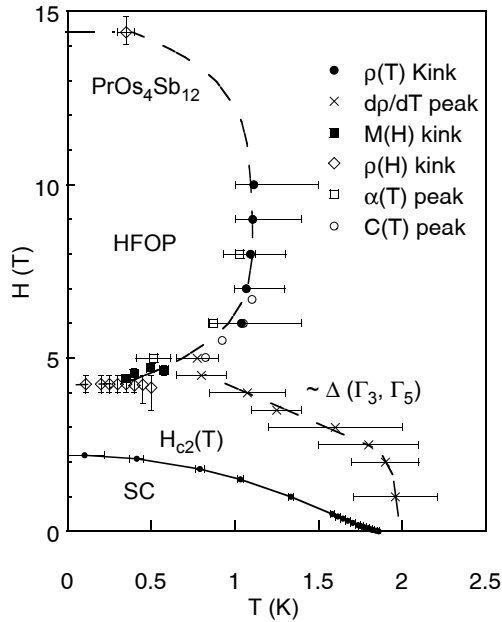


Fig. 2. Magnetic field–temperature (H – T) phase diagram of PrOs₄Sb₁₂ showing the regions exhibiting superconductivity (SC) and the high field ordered phase (HFOP). The dashed line is a measure of the splitting between the Pr³⁺ Γ_3 ground state and Γ_5 excited state (see text for further details). Data from Refs. [3, 4, 6, 7, 23].

Recently, Sugawara *et al.* [10] performed de Haas–van Alphen effect measurements on PrOs₄Sb₁₂. They found that the topology of the Fermi surface is close to that of the reference compound LaOs₄Sb₁₂ and is explained well by band structure calculations. In contrast to the similarity in the Fermi surface topologies of PrOs₄Sb₁₂ and LaOs₄Sb₁₂, the cyclotron effective masses m_c^* of PrOs₄Sb₁₂ are up to ~ 6 times enhanced compared to those of LaOs₄Sb₁₂. The Sommerfeld coefficient γ estimated from the Fermi surface

volume and the value of m_c^* , assuming a spherical Fermi surface, is ~ 150 mJ/mol K², which is two to three times smaller than the value of γ inferred from the normal and superconducting properties of PrOs₄Sb₁₂. Our studies of LaOs₄Sb₁₂ single crystals reveal superconductivity with a T_c of 1 K.

3. The Pr³⁺ energy level scheme in the crystalline electric field

Magnetic susceptibility $\chi(T)$ data between ~ 1 K and room temperature for PrOs₄Sb₁₂ from Ref. [2] are shown in Fig. 3. These $\chi(T)$ data have been corrected for excess Sb by assuming the effective moment μ_{eff} was equal to the full Hund's rule value of $3.6 \mu_B$. This led to an estimation of the mass of the excess Sb to be $\sim 25\%$ of the total mass. The $\chi(T)$ data exhibit a peak at ~ 3 K and saturate to a value of ~ 0.11 cm³/mol as $T \rightarrow 0$, indicative of a nonmagnetic ground state. At temperatures above ~ 5 K, $\chi(T)$ is strongly T -dependent, as expected for well defined Pr³⁺ magnetic moments. In the analysis of the $\chi(T)$ data, interactions between Pr³⁺ ions and hybridization of the Pr $4f$ and conduction electron states were neglected, while the degeneracy of the Hund's rule multiplet of the Pr³⁺ ions was assumed to be lifted by a cubic crystalline electric field (CEF) and to have a nonmagnetic ground state. According to Lea, Leask, and Wolf [11], in a cubic CEF, the Pr³⁺ $J = 4$ Hund's rule multiplet splits into a Γ_1 singlet, a Γ_3 nonmagnetic doublet that carries an electric quadrupole moment, and Γ_4 and Γ_5 triplets. It was assumed that the nonmagnetic ground state of the Pr³⁺ ions corresponds to either a Γ_1 singlet or a Γ_3 nonmagnetic doublet [2]. Although reasonable fits to the $\chi(T)$ data could be obtained for both Γ_1 and Γ_3 ground states, as shown in Fig. 3, the most satisfactory fit was obtained for a Γ_3 nonmagnetic doublet ground state with a Γ_5 first excited triplet state at 11 K and Γ_4 and Γ_1 excited states at 130 K and 313 K, respectively (see the inset to Fig. 3). Inelastic neutron scattering measurements on PrOs₄Sb₁₂ [3] reveal peaks in the INS spectrum at 0.71 meV (8.2 K) and 11.5 meV (133 K) that appear to be associated with transitions between the Γ_3 ground state and the Γ_5 first and Γ_4 second excited states, respectively, that are in good agreement with the Pr³⁺ CEF energy level scheme determined from the analysis of the $\chi(T)$ data. As noted above, the Schottky anomaly in the $C(T)$ data on PrOs₄Sb₁₂ taken at UCSD and at the University of Karlsruhe [6] can be described well by a two level system consisting of a doublet ground state and a low lying triplet excited state with a splitting of ~ 7 K, a value that is comparable to the values deduced from the $\chi(T)$ and INS data. However, a Γ_1 ground state cannot, at this point, be completely excluded.

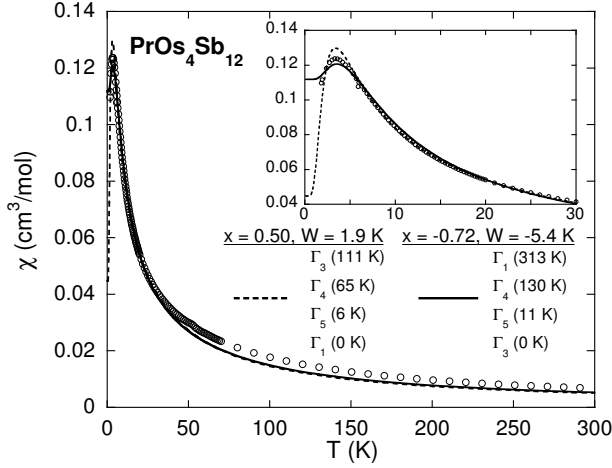


Fig. 3. Magnetic susceptibility χ vs T for $\text{PrOs}_4\text{Sb}_{12}$ single crystals. Fits of a CEF model to the $\chi(T)$ data in which the ground state is either a Γ_3 nonmagnetic doublet (solid line) or a Γ_1 singlet (dashed line) are indicated in the figure. Inset: $\chi(T)$ below 30 K. After Ref. [2].

While a magnetic Γ_4 or Γ_5 Pr^{3+} ground state could also produce a nonmagnetic heavy fermion ground state via an antiferromagnetic exchange interaction (Kondo effect), the behavior of $\rho(T)$ of $\text{PrOs}_4\text{Sb}_{12}$ in the normal state does not resemble the behavior of $\rho(T)$ expected for this scenario. For a typical magnetically-induced heavy fermion compound, $\rho(T)$ often increases with decreasing temperature due to Kondo scattering, reaches a maximum, and then decreases rapidly with decreasing temperature as the highly correlated heavy fermion state forms below the coherence temperature. At low temperatures, $\rho(T)$ typically varies as AT^2 with a prefactor $A \approx 10^{-5} [\mu\Omega \text{ cm K}^2(\text{mJ/mol})^{-2}] \gamma^2$ that is consistent with the Kadowaki-Woods relation [12]. In contrast, $\rho(T)$ of $\text{PrOs}_4\text{Sb}_{12}$, shown in Fig. 4 [1,2], exhibits typical metallic behavior with negative curvature at higher temperatures and a pronounced ‘roll off’ below ~ 8 K before it vanishes abruptly when the compound becomes superconducting (upper inset of Fig. 4). The temperature of the ‘roll off’ in $\rho(T)$ is close to that of the decrease in charge or spin dependent scattering due to the decrease in population of the proposed first excited state (Γ_5) as the temperature is lowered. The $\rho(T)$ data are shown in the lower inset of Fig. 4 and can be described by a temperature dependence of the form AT^2 between ~ 8 K and 45 K, but with a prefactor $A \approx 0.009 \mu\Omega \text{ cm/K}^2$ that is nearly two orders of magnitude smaller than that expected from the Kadowaki-Woods relation ($A \approx 1.2 \mu\Omega \text{ cm/K}^2$ for $\gamma \approx 350 \text{ mJ/mol K}^2$) [12]. Interestingly, $\rho(T)$ is consistent with T^2 behavior

with a value $A \approx 1 \mu\Omega \text{ cm}/\text{K}^2$ in fields of $\sim 5 \text{ T}$ [4] in the high field ordered phase discussed in Section 5. The zero-field temperature dependence of $\rho(T)$ is similar to that observed for the compound PrInAg_2 , which also has a low value of the coefficient A , an enormous γ of $\sim 6.5 \text{ J/mol K}^2$, and a T_3 non-magnetic doublet ground state [13]. The compounds $\text{PrOs}_4\text{Sb}_{12}$, PrInAg_2 , and another Pr-based skutterudite, $\text{PrFe}_4\text{P}_{12}$ [14], may belong to a new class of heavy fermion compounds in which the heavy fermion state is produced by electric quadrupole fluctuations. In contrast, magnetic dipole fluctuations are widely believed to be responsible for the heavy fermion state in most Ce and U heavy fermion compounds (with the possible exception of certain U compounds such as UBe_{13}). Another possible source of the enhanced effective mass in $\text{PrOs}_4\text{Sb}_{12}$ may involve excitations from the ground state to the low lying first excited state in the Pr^{3+} CEF energy level scheme [15].

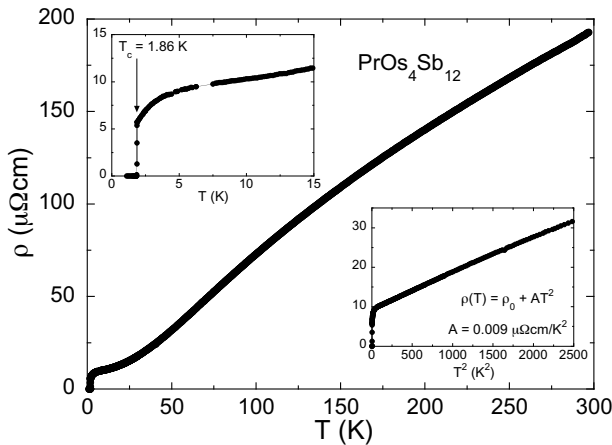


Fig. 4. Electrical resistivity ρ vs T for $\text{PrOs}_4\text{Sb}_{12}$ between 1.8 K and 300 K. Upper inset: $\rho(T)$ below 20 K. Lower inset: $\rho(T)$ below 50 K. After Ref. [1].

Two studies of the nonlinear magnetic susceptibility have been performed in an attempt to determine the CEF ground state of the Pr^{3+} ion in $\text{PrOs}_4\text{Sb}_{12}$ [8,16]. The nonlinear susceptibility χ_3 is the coefficient of an H^3 term in the expansion of the magnetization M in a series of odd powers of H ; *i.e.*, $M \approx \chi_1 H + (\chi_3/6)H^3$, where χ_1 is the ordinary linear susceptibility. For an ionic situation, χ_3 is isotropic and varies as T^{-3} for a magnetic ground state, whereas for a non-Kramers T_3 doublet it is anisotropic and diverges at low temperatures for $H \parallel [100]$ and approaches a constant for $H \parallel [111]$ [17]. This type of study was previously employed in an attempt to determine the ground state of U in the compound UBe_{13} [18]. In the study by Bauer *et al.* [16], χ_3 was found to be anisotropic and exhibit a minimum followed by a maximum and a negative divergence as the temperature was decreased

for $H \parallel [100]$, while χ_3 exhibited a minimum and then increased down to the lowest temperature of the measurement ($T = 1.8$ K), for $H \parallel [111]$. Comparison with calculations based on the quadrupolar Anderson–Hamiltonian provided a qualitative description of the $\chi_3(T)$ data for $H \parallel [100]$, but did not describe the $\chi_3(T)$ data well for $H \parallel [111]$. It was concluded that the data were qualitatively consistent with a Γ_3 ground state, given the limits of the experiment and the complexity of the theory. On the other hand, a study by Tenya *et al.* [8] found $\chi_3(T)$ to be nearly isotropic. It was concluded that the a Γ_1 ground state could not be ruled out on the basis of this experiment. However, it should be noted that these $\chi_3(T)$ studies are difficult to interpret because of the curvature of $M(H)$ and the complications that arise at lower temperatures $T \leq T_c$ and lower fields $H \leq H_{c2}$ due to the superconductivity and at temperatures $T \leq 2$ K and higher fields $H \geq 4.5$ K by the onset of the high field ordered phase, discussed in Section 5.

4. Superconducting state

Several features in the superconducting properties of $\text{PrOs}_4\text{Sb}_{12}$ indicate that the superconductivity of this compound is unconventional in nature. First, $C_s(T)$ follows a power law T -dependence, $C_s(T) \sim T^{2.5}$, after the Schottky anomaly and βT^3 lattice contributions have been subtracted from the $C(T)$ data. As reported in Ref. [3], $C_s(T)$ follows a power law with $C_s(T) \sim T^{3.9}$ when the Schottky anomaly is not subtracted. Second, there is a ‘double-step’ structure in the jump in $C(T)$ near T_c in single crystals (lower inset of Fig. 1) that suggests two distinct superconducting phases with different T_c ’s: $T_{c1} \approx 1.70$ K and $T_{c2} \approx 1.85$ K [3, 6]. This structure is not evident in the $C(T)$ data taken on the pressed pellet of $\text{PrOs}_4\text{Sb}_{12}$ shown in the upper inset of Fig. 1, possibly due to strains in the single crystals out of which the pressed pellet is comprised that broaden the transitions at T_{c1} and T_{c2} so that they overlap and become indistinguishable. However, at this point, it is not clear whether these two apparent jumps in $C(T)$ are associated with two distinct superconducting phases or are due to sample inhomogeneity. It is noteworthy that all of the single crystal specimens prepared in our laboratory and investigated by our group and our collaborators exhibit this ‘double-step’ structure. Multiple superconducting transitions, apparently associated with distinct superconducting phases, have previously been observed in two other heavy fermion superconductors, UPt_3 [19] and $\text{U}_{1-x}\text{Th}_x\text{Be}_{13}$ ($0.1 \leq x \leq 0.35$) [20]. Measurements of the specific heat in magnetic fields reveal that the two superconducting features shift downward in temperature at nearly the same rate with increasing field, consistent with the smooth temperature dependence of the $H_{c2}(T)$ curve [6]. These two transitions have also been observed in thermal expansion measurements [7],

which, from the Ehrenfest relation, reveal that T_{c1} and T_{c2} have different pressure dependencies, suggesting that they are associated with two distinct superconducting phases.

Recent transverse field μ SR [21] and Sb-NQR measurements [22] on $\text{PrOs}_4\text{Sb}_{12}$ are consistent with an isotropic energy gap. Along with the specific heat, these measurements indicate strong coupling superconductivity. These findings suggest an s -wave, or, perhaps, a Balian–Werthamer p -wave order parameter. On the other hand, the superconducting gap structure of $\text{PrOs}_4\text{Sb}_{12}$ was investigated by means of thermal conductivity measurements in magnetic fields rotated relative to the crystallographic axes by Izawa *et al.* [5]. These measurements reveal two regions in the H – T plane, a low field region in which $\Delta(\mathbf{k})$ has two point nodes, and a high field region where $\Delta(\mathbf{k})$ has six point nodes. The line lying between the low and high field superconducting phases may be associated with the transition at T_{c2} , whereas the line between the high field phase and the normal phase, $H_{c2}(T)$, converges with T_{c1} as $H \rightarrow 0$. Clearly, more research will be required to further elucidate the nature of the superconducting state in $\text{PrOs}_4\text{Sb}_{12}$.

5. High field ordered phase

Evidence for a high field ordered phase was first derived from magnetoresistance measurements in the temperature range $80 \text{ mK} \leq T \leq 2 \text{ K}$ and magnetic fields up to 9 tesla [3,23]. The H – T phase diagram, depicting the superconducting region and the high field ordered phase is shown in Fig. 2. The line that intersects the high field ordered state represents the inflection point of the ‘roll-off’ in $\rho(T)$ at low temperatures and is a measure of the splitting between the Pr^{3+} ground state and the first excited state, which decreases with field (see Fig. 4). The high field ordered phase has also been observed by means of large peaks in the specific heat [6,9] and thermal expansion [7] and kinks in magnetization *vs* magnetic field curves [8,23] in magnetic fields $> 4.5 \text{ T}$ and temperatures $< 1.5 \text{ K}$.

Shown in Fig. 5 are $\rho(T)$ data for various magnetic fields up to 9 T for $\text{PrOs}_4\text{Sb}_{12}$, which reveal drops in $\rho(T)$ due to the superconductivity for $H \leq 2.3 \text{ T}$ and features in $\rho(T)$ associated with the onset of the high field ordered phase for $H \geq 4.5 \text{ T}$. Isotherms of electrical resistance R *vs* H and magnetization M *vs* H are shown in Figs. 6(a) and 6(b), respectively. The fields denoting the boundaries of the high field ordered phase, H_1^* and H_2^* , are indicated in the figure.

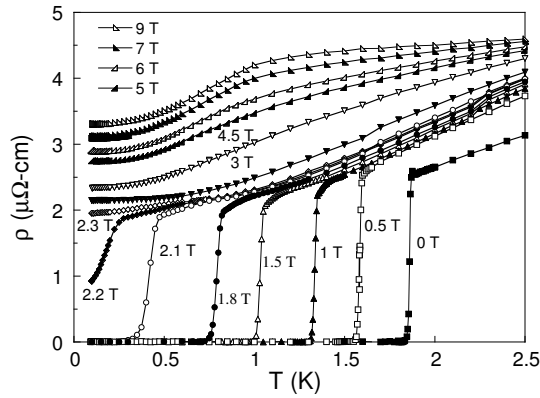


Fig. 5. Electrical resistivity ρ vs T for various magnetic fields up to 9 T for a $\text{PrOs}_4\text{Sb}_{12}$ single crystal. The rapid drop in $\rho(T)$ to zero for $H < 2.3$ T is due to the superconducting transition, while the decrease in $\rho(T)$ for $H \geq 4.5$ T below ~ 1 K appears to be due to a field induced phase, presumably due to magnetic or quadrupolar order. After Ref. [4].

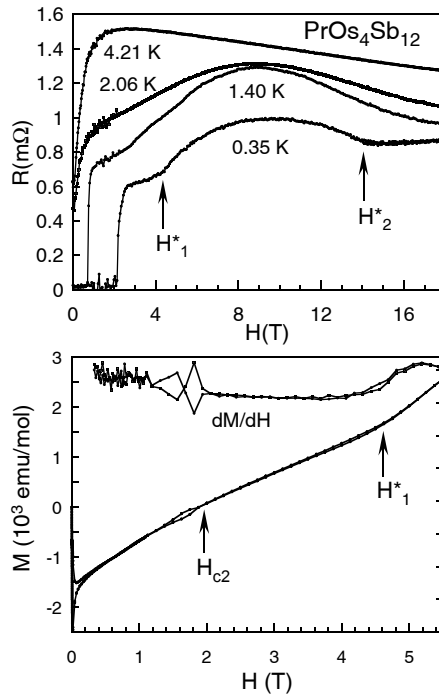


Fig. 6. (a) Electrical resistance R vs magnetic field H for $\text{PrOs}_4\text{Sb}_{12}$ at several different temperatures below 4.21 K for $0 \leq H \leq 18$ T. (b) Magnetization M vs H for $\text{PrOs}_4\text{Sb}_{12}$ at 0.34 K for $0 \leq H \leq 5.5$ T.

6. Summary

Experiments on the filled skutterudite compound $\text{PrOs}_4\text{Sb}_{12}$ have revealed a number of extraordinary phenomena: a heavy fermion state characterized by an effective mass $m^* \approx 50 m_e$, unconventional superconductivity below $T_c = 1.85$ K with two distinct superconducting phases, and a high field ordered phase, presumably associated with magnetic or electric quadrupolar order. Analysis of $\chi(T)$, $C(T)$, $\rho(T)$, and INS data indicate that Pr^{3+} has a nonmagnetic Γ_3 doublet ground state that carries an electric quadrupole moment, a low lying Γ_5 triplet excited state at ~ 10 K, and Γ_4 triplet and Γ_1 singlet excited states at much higher energies. This suggests that the interaction between the quadrupole moments of the Pr^{3+} ions and the charges of the conduction electrons, as well as the excitations between the Γ_3 ground state and Γ_5 low lying excited state may play an important role in generating the heavy fermion state and superconductivity in this compound. The heavy fermion state and unconventional superconductivity will constitute a significant challenge for theoretical description [24].

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