LETTER TO THE EDITOR

Evolution of superconducting order in 
Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$

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Abstract
We report measurements of the magnetic penetration depth $\lambda$ in single crystals of 
Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ down to 0.1 K. Both $\lambda$ and superfluid density $\rho_s$ exhibit an 
exponential behaviour for the $x \geq 0.4$ samples, going from weak ($x = 0.4, 0.6$) 
to moderate coupling ($x = 0.8$). For the $x \leq 0.2$ samples, both $\lambda$ and $\rho_s$ 
not vary as $T^2$ at low temperatures, but $\rho_s$ is s-wave-like at intermediate to high 
temperatures. Our data are consistent with the presence of an additional nodal 
low-temperature phase at $T_c < 0.6$ K, for small values of $x$.

(Some figures in this article are in colour only in the electronic version)

The recent discovery [1, 2] of the heavy-fermion (HF) skutterudite superconductor (SC) 
PrOs$_4$Sb$_{12}$ has attracted much interest due to its differences from the other HFSCs. Early 
work suggested that the ninefold degenerate $J = 4$ Hund’s rule multiplet of Pr is split by the 
cubic crystal electric field, such that its ground state is a nonmagnetic $\Gamma_1$ doublet, separated 
from the first excited state $\Gamma_5$ by $\sim$10 K. Hence its HF behaviour, and consequently the origin 
of its superconductivity, might be attributed to the interaction between the electric quadrupolar 
moments of Pr$^{3+}$ and the conduction electrons [1]. More recent results appear to rule this 
mechanism out, giving strong evidence for a singlet $\Gamma_1$ ground state with a $\Gamma_5$ triplet state at 
slightly higher energy [3, 4]. In this scheme, aspherical Coulomb scattering [4] and spin-
fluctuation scattering [5] have been proposed as mechanisms leading to superconductivity.

Surprisingly, replacement of Os by Ru, i.e. in PrRu$_4$Sb$_{12}$, yields a superconductor with 
$T_c \approx 1.25$ K [6] and significantly different properties. The effective mass of the heavy 
electrons calculated from de Haas–van Alphen (dHvA) and specific-heat measurements [1, 7] 
show that, while PrOs$_4$Sb$_{12}$ is clearly an HF material, PrRu$_4$Sb$_{12}$ is at most, a marginal HF.

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Various experimental results suggest that these two materials have different order-parameter symmetry. Firstly, there is no Hebel–Slichter peak in the nuclear quadrupole resonance (NQR) data [8] for PrOs$_4$Sb$_{12}$, while a distinct coherence peak was seen [9] in the Sb-NQR $1/T_1$ data for PrRu$_4$Sb$_{12}$. Secondly, the low-temperature power-law behaviour seen in specific heat [1] and penetration depth [10], and the angular variation of thermal conductivity [11], suggest the presence of nodes in the order parameter of PrOs$_4$Sb$_{12}$. For PrRu$_4$Sb$_{12}$, however, exponential low-temperature behaviour was seen in $1/T_1$ [9] and penetration depth [12] data. The latter data were fitted with an isotropic zero-temperature gap of magnitude $\Delta(0) = 1.9k_BT_c$. Thirdly, muon spin rotation ($\mu$SR) experiments on PrOs$_4$Sb$_{12}$ reveal the spontaneous appearance of static internal magnetic fields below $T_c$, providing evidence that the superconducting state is a time-reversal-symmetry-breaking (TRSB) state [13], consistent with the presence [10, 11] of point nodes on the Fermi surface (FS). Adding to the puzzle, a recent paper [14] reported an unexpected enhancement of the lower critical field $H_{c1}(T)$ and the critical current $I_c(T)$ deep in the superconducting state below $T \approx 0.6$ K ($T/T_c \approx 0.3$) in PrOs$_4$Sb$_{12}$. The authors suggest a transition into another superconducting phase that occurs below $T_{c3} \approx 0.6$ K that may explain such anomalies in other measurements as the levelling off of Sb-NQR $1/T_1$ below 0.6 K following its exponential decrease [9], the small downturn of penetration depth below 0.62 K and its deviation from point-node-$T^2$-behaviour above $\sim 0.6$ K [10]. The discrepancy between different experiments at $H = 0$, concerning the nature of the superconducting gap, can also be reconciled if the temperature interval covered in the analysis is taken into account [14]—the NQR analysis [9], consistent with an isotropic gap, was performed for $T \geq 0.6$ K, while the penetration depth analysis [10], consistent with nodes in the gap, was done for $T < 0.55$ K.

To explore why the substitution of Ru for Os (same column in the periodic table) causes PrRu$_4$Sb$_{12}$ to differ in so many respects from PrOs$_4$Sb$_{12}$, particularly in the symmetry of the superconducting gap, Frederick et al performed x-ray diffraction, magnetic susceptibility and electrical resistivity measurements [15] on single crystals of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$. They found a smooth evolution of the lattice constant and $T_c$ with $x$, albeit with a deep minimum (0.75 K) in $T_c$ at $x = 0.6$, and an increased splitting between the ground and excited states of the Pr ion. These data do not clarify measurements [11, 10, 13, 16] that indicate point-node gap structure, TRSB and a double superconducting transition $T_{c2} \lesssim T_c$ [15] in PrOs$_4$Sb$_{12}$, none of which are seen for $x > 0$.

In this letter, we present high-precision measurements of the penetration depth $\lambda(T)$ of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ ($x = 0.1, 0.2, 0.4, 0.6, 0.8$) at temperatures down to $\sim 0.1$ K, using the same experimental conditions as for PrOs$_4$Sb$_{12}$ and PrRu$_4$Sb$_{12}$ [10, 12]. For the $x \geq 0.4$ samples, both $\lambda(T)$ and superfluid density $\rho_s(T)$ exhibit exponential behaviour at low temperatures, supporting the presence of an isotropic superconducting gap on the FS. The $\rho_s(T)$ data agree with the theoretical curve over the entire temperature range. The values of $\Delta(0)$ used in the fits suggest an increase in coupling strength from weak coupling ($x = 0.4, 0.6$) to moderate coupling ($x = 0.8$). On the other hand, the $x \leq 0.2$ samples exhibit a low-$T$ power law, implying the existence of low-lying excitations. However, the $\rho_s$ data fit a fully gapped theoretical curve from intermediate temperatures up to $T_c$, but not curves based on a superconducting gap with line or point nodes. This is consistent with the scenario depicted by Cichorek et al [14], where for the $x \leq 0.2$ samples the fully gapped high-$T$ phase undergoes a transition into a nodal low-$T$ phase below $T_{c3}(x)$. As $x$ increases, the low-$T$ phase is suppressed ($T_{c3}$ decreases) such that for the $x \geq 0.4$ samples $T_{c3}$ falls below the base temperature of our experiment, and we are left with a fully gapped phase over our entire experimental temperature range. Taken together with other data, we suggest that there is an additional superconducting phase at $T_{c3}$ that exhibits point nodes, thus providing an independent confirmation of the conclusion of [14].
The single-crystal samples were grown by the Sb self-flux method [6]. The observation of dHvA effect [7] both in PrOs$_4$Sb$_{12}$ and PrRu$_4$Sb$_{12}$ are indicative of the high quality of these samples grown in the same manner. Measurements were performed utilizing a 21 MHz tunnel diode oscillator [17] with a noise level of two parts in $10^9$ and low drift. The magnitude of the ac field is estimated to be less than 40 mOe. The sample was mounted, using a small amount of GE varnish, on a single-crystal sapphire rod. The other end of the rod is thermally connected to the mixing chamber of an Oxford Kelvinox 25 dilution refrigerator. The sample temperature is monitored using a calibrated RuO$_2$ resistor at low temperatures ($T_{\text{base}}$–1.3 K) and a calibrated Cernox thermometer at higher temperatures (1.2–1.8 K).

The deviation $\Delta \lambda(T) = \lambda(T) - \lambda(0.1\,\text{K})$ is proportional to the change in resonant frequency $\Delta f(T)$ of the oscillator, with the proportionality factor $G$ dependent on sample and coil geometries. We determine $G$ for a pure Al single crystal by fitting the Al data to extreme nonlocal expressions and then adjust for relative sample dimensions [18]. Testing this approach on a single crystal of Pb, we found good agreement with conventional BCS expressions. The value of $G$ obtained in this way has an uncertainty of $\pm 10\%$ because our samples have a rectangular, rather than square, basal area [19].

We first discuss the $x \geq 0.4$ samples. Figure 1 (O) shows $\Delta \lambda(T)$ for the three samples ($x = 0.4, 0.6, 0.8$) as a function of temperature in the low-temperature region. The insets show $\Delta \lambda(T)$ for the entire temperature range. The onsets of the superconducting transitions $T_C^*$ are
0.81 K (x = 0.6) and 0.88 K (x = 0.8). These values are consistent with those of [15]. We could not obtain $T^*\lambda$ for the x = 0.4 sample as the ac losses were so large that oscillation was lost before $T_c$ was reached; its large transition width is also consistent with the ac susceptibility data of Frederick et al [15], though the origin is unknown. The values of $T_c$, determined from the point where the experimental superfluid density almost vanishes and fits the theoretical curves (described later), are 0.8 K (x = 0.4), 0.76 K (x = 0.6) and 0.86 K (x = 0.8).

For all three samples the data points flatten out below 0.3$T_c$, implying activated behaviour in this temperature range. We fit these data to the BCS low-temperature expression in the clean and local limit, from $T_{\text{base}}$ (~0.1 K) to 0.4$T_c$, using the expression $\Delta\lambda(T) \propto \sqrt{\pi \Delta(0)/2k_B T} \exp(-\Delta(0)/k_B T)$, with the proportionality constant and $\Delta(0)$ as parameters. The best fits (solid lines) are obtained when $\Delta(0)/k_B T_c = 1.64$ (x = 0.4), 1.53 (x = 0.6) and 1.95 (x = 0.8). This implies that the x = 0.4 and 0.6 samples are weak coupling, while the x = 0.8 sample is a moderate-coupling superconductor. The x = 0.8 result is consistent with that for PrRu$_4$Sb$_{12}$ (x = 1).

The experimental superfluid density is defined as $\rho_s(T) = \lambda^2(0)/\lambda^2(T)$. To extract $\rho_s(T)$ from our data, we need to know $\lambda(0)$. Absent published data on $\lambda(0)$, we assume that it lies in the vicinity of 344 nm (for PrOs$_4$Sb$_{12}$) [20] and 290 nm (for PrRu$_4$Sb$_{12}$) [12]. We compute $\rho_s$ for an isotropic s-wave superconductor in the clean and local limits using $\rho_s = 1 + 2 \int_x^\infty \frac{\delta}{\pi} \frac{1}{\exp(E/k_BT) + 1} \, d\varepsilon$, where $f = [\exp(E/k_BT) + 1]^{-1}$ is the Fermi function, and $E = [\varepsilon^2 + (\Delta(T))^2]^{1/2}$ is the quasiparticle energy. The temperature dependence of $\Delta(T)$ can be obtained by using [21] $\Delta(T) = \delta_{sc}k_BT_c\tanh[(\pi/\delta_{sc})\sqrt{(2/3)[(\Delta C)/C][(T_c/T) - 1]]}$, where $\delta_{sc} \equiv \Delta(0)/k_BT_c$ is the only variable parameter. The specific heat jump $\Delta C/C$ can be obtained from $\Delta(0)/k_BT_c$ using strong-coupling equations [22, 23]. Note, however, that large values of $\Delta C/C$ interpreted as ‘strong coupling’ may also be produced by aspherical Coulomb scattering from crystal field excitations, resulting in the enhancement of conduction electron mass [24, 4].

Figure 2 shows the experimental (O) and calculated (solid line) values of $\rho_s$ as a function of temperature for the x ≥ 0.4 samples. The theoretical curves fit the data very well using the parameters shown in table 1. Fitted values for $\lambda(0)$ are reasonable, considering the uncertainty in obtaining the calibration factor $G$.

We now turn to the x ≤ 0.2 samples. Figures 3(a) and (b) show $\Delta\lambda(T)$ in the low-temperature region. The insets show $\Delta\lambda(T)$ for the entire temperature range. $T^*\lambda$ is measured to be 1.76 K (x = 0.1) and 1.77 K (x = 0.2), while $T_c$ is 1.4 K (x = 0.1) and 1.2 K (x = 0.2). It is possible to fit the low-temperature data (up to 0.53 K ≈ 0.3$T_c$) to a variable power law $\Delta\lambda(T) = A + BT^n$ yields $n = 2.5$ (x = 0.1) and 3.3 (x = 0.2), indicating the existence of low-lying states. There is no theoretical basis for fractional power laws—these are simply effective values indicating a crossover between an integral power of temperature and an exponential increase, which we will describe later.

Figures 3(c) and (d) show the experimental (O) values of $\rho_s(T)$. The solid lines represent the theoretical curve based on an isotropic weak-coupling gap as in table 1. Note that the data do not agree with the theoretical curve at low temperatures, but agree from intermediate temperatures up to near $T_c$. The deviation of data from the theoretical curve at low temperatures

| Sample x | 0     | 0.1   | 0.2   | 0.4   | 0.6   | 0.8   | 1.0   |
|----------|-------|-------|-------|-------|-------|-------|-------|
| $\Delta(0)/k_BT_c$ | 2.6   | 1.76  | 1.76  | 1.76  | 1.95  | 1.90  |
| $\Delta C/C$       | 3.0   | 1.43  | 1.43  | 1.43  | 1.43  | 2.04  | 1.87  |
| $\lambda(0)$ (nm)  | 344   | 320   | 380   | 340   | 380   | 400   | 290   |
Superfluid density $\rho_s(T)$ calculated from $\Delta \lambda(T)$ data in figure 1, for (a) $x = 0.4$, (b) $x = 0.6$, and (c) $x = 0.8$. Lines: theoretical $\rho_s(T)$ with parameters $\Delta(0)/k_B T_c$ and $\Delta C/\gamma T_c$ mentioned in the text.

is more pronounced going from $x = 0.1$ to $0.2$, showing non-exponential behaviour. We assert this to be a continuation of the transition to a nodal low-$T$ phase reported to occur at $\sim 0.6$ K for $x = 0$ by Cichorek et al [14]. We label this transition $T_{c3}(x)$ and explore its concentration dependence. Because it has been established that the low-$T$ phase at $x = 0$ is characterized by point nodes [10, 11], we track the range over which the expected $T^2$ temperature dependence holds. Therefore, we plot $\rho_s(T)$ versus $T^2$, shown in figures 4(b) and (c), where we then fit a straight line to the data from $T_{base}$ to various temperatures $T_{max}$. $T_{c3}(x)$ is determined from the temperature where the fit yields the largest absolute value of the correlation coefficient $R$, as shown in the insets, from which we obtain $T_{c3}(x = 0.1) \approx 0.29 \pm 0.05$ K and $T_{c3}(x = 0.2) \approx 0.17 \pm 0.01$ K. Applying the same criterion to our $x = 0$ data [10], we find $T_{c3}(x = 0) \approx 0.44 \pm 0.04$ K (figure 4(a)). This is compatible with the features deduced in [14], but suggest that our estimation of $T_{c3}(x)$ may only place a lower limit on its position, since the $T^2$ dependence of $\rho_s(T)$ is expected to hold only for temperatures $T \ll \Delta$. We plot $T_{c3}$ versus $x$ in figure 4(d). Extrapolating the best-fit line yields $T_{c3} \approx 0$ when $x \approx 0.33$. This implies that the low-$T$ nodal phase disappears, perhaps at a quantum critical point, when $x \gtrsim 0.3$, i.e. one only sees a fully gapped behaviour over the whole temperature range, agreeing with our $x \gtrsim 0.4$ data sets. A preliminary analysis of a $x = 0.05$ sample from another source gives $T_{c3} \approx 0.37$ K, close to the line in figure 4(d). A theory by Hotta [5] predicts that as the $\Gamma_1-\Gamma_5$ spacing decreases (observed as $x$ is decreased from 1 to 0 in [15]), superconductivity
changes from conventional to unconventional, supporting our scenario. Finally, we wish to point out that though the number of low-temperature points used to determine $T_{c3}$ in our $x = 0.2$ data is small the fact that $T_{c3}(x = 0.2)$ lies on the same straight line as that of $x = 0, 0.05$ and 0.1 allows us to place some level of confidence in the accuracy of its value.

The continuity across the series of the first superconducting transition, that we label $T_{c1}$, and the BCS-like behaviour of $\rho_s$ over much of the $T$–$x$ plane suggest that conventional phonon-mediated superconductivity prevails, in agreement with the experimental result of [15] and the theoretical result of [5]. Nonetheless, there is ample evidence for a second superconducting transition at $T_{c2}$ at $x = 0$ below which unconventional superconductivity appears. Specific heat measurements on Pr$_{1-y}$La$_y$Os$_4$Sb$_{12}$ [25] showed that the second superconducting transition at $T_{c2}$ disappears between $y = 0.05$ and 0.1, leaving conventional superconductivity for larger values of $y$. Figures 1(a) and 3(a) and (b) show some changes in curvature in $\Delta \lambda$ close to $T^*$ for the $x = 0.1, 0.2$ and 0.4 samples that could be indicative of $T_{c2}$, but the positions and strengths of the curvature change vary from sample to sample, consistent with differences seen among bulk data, such as specific heat in [16] and [13]. As noted in the introductory paragraph, two mechanisms—spin-fluctuation and aspherical Coulomb scattering—have been proposed to explain the heavy-fermion behaviour and superconducting properties of the $x = 0$ skutterudite. One possibility is that the spin-fluctuation mechanism is active at high temperatures where the

\begin{figure}
\centering
\includegraphics[width=\textwidth]{fig3}
\caption{(O) Low-temperature $\Delta \lambda(T)$ for (a) $x = 0.1$ and (b) $x = 0.2$. Lines: fits to $\Delta \lambda(T) = A + BT^n$ from 0.1 to 0.53 K. Insets show $\Delta \lambda(T)$ over the full temperature range. (O) Superfluid density $\rho_s(T)$ calculated from $\Delta \lambda(T)$ data for (c) $x = 0.1$ and (d) $x = 0.2$. Lines: theoretical $\rho_s(T)$ with weak-coupling parameters. Note that the deviation of data from the theoretical curve at low temperatures is more pronounced for $x = 0.1$ than for $x = 0.2$.}
\end{figure}
\(\Gamma_3\) state is thermally populated on the Os-rich end of the phase diagram, but is suppressed by decreasing temperature or as Ru doping increases the \(\Gamma_1-\Gamma_5\) splitting. Aspherical Coulomb scattering may remain important at lower temperatures and at larger values of \(x\). Our data, when considered together with other data and theory, suggest three different superconducting phases: phonon driven (conventional) across the series at the upper transition \(T_c1\), but with spin-fluctuation and aspherical Coulomb scattering at the Os end giving rise to transitions to unconventional phases at \(T_c2\) and \(T_c3\). The agreement between our data and Cichorek’s bulk data, on the presence of an additional phase at \(T_c3\), shows that the features we see are intrinsic, not merely a surface effect.

In conclusion, we report measurements of the magnetic penetration depth \(\lambda\) in single crystals of \(\text{Pr(Os}_{1-x}\text{Ru}_x)_{4}\text{Sb}_{12}\) down to \(\sim 0.1\) K. Both \(\lambda\) and superfluid density \(\rho_s\) exhibit an exponential behaviour for the \(x \geq 0.4\) samples, going from weak coupling (\(x = 0.4\), 0.6) to moderate coupling (\(x = 0.8\)). For the \(x \leq 0.2\) samples, both \(\lambda\) and \(\rho_s\) vary as \(T^2\) at low temperatures, but \(\rho_s\) is s-wave-like at intermediate to high temperatures. Our data are consistent with the presence of an additional nodal low-\(T\) phase at \(T_c3\) for small values of \(x\). The \(x\)-dependence of \(T_c3\) suggests that the low-\(T\) phase disappears near \(x = 0.3\).
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