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LETTER TO THE EDITOR

Evolution of superconducting order in $Pr(Os_{1-x}Ru_x)_4Sb_{12}$

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Abstract
We report measurements of the magnetic penetration depth λ in single crystals of $Pr(Os_{1-x}Ru_x)_4Sb_{12}$ down to 0.1 K. Both λ and superfluid density ρ_s exhibit an exponential behaviour for the $x \geqslant 0.4$ samples, going from weak ($x = 0.4, 0.6$) to moderate coupling ($x = 0.8$). For the $x \leqslant 0.2$ samples, both λ and ρ_s vary as T^2 at low temperatures, but ρ_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 $T_{c3} < 0.6$ 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) $PrOs_4Sb_{12}$ 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* Γ_3 doublet, separated from the first excited state Γ_5 by ~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 Γ_1 ground state with a Γ_5 triplet state at a 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 $PrRu_4Sb_{12}$, 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 $PrOs_4Sb_{12}$ is clearly an HF material, $PrRu_4Sb_{12}$ 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 $PrOs_4Sb_{12}$, while a distinct coherence peak was seen [9] in the Sb-NQR $1/T_1$ data for $PrRu_4Sb_{12}$. 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 $PrOs_4Sb_{12}$. For $PrRu_4Sb_{12}$, 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 (μSR) experiments on $PrOs_4Sb_{12}$ 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 $PrOs_4Sb_{12}$. 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 ~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 $T \geqslant 0.6$ 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 $PrRu_4Sb_{12}$ to differ in so many respects from $PrOs_4Sb_{12}$, 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 $Pr(Os_{1-x}Ru_x)_4Sb_{12}$. 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 $PrOs_4Sb_{12}$, none of which are seen for $x > 0$.

In this letter, we present high-precision measurements of the penetration depth $\lambda(T)$ of $Pr(Os_{1-x}Ru_x)_4Sb_{12}$ ($x = 0.1, 0.2, 0.4, 0.6, 0.8$) at temperatures down to ~0.1 K, using the same experimental conditions as for $PrOs_4Sb_{12}$ and $PrRu_4Sb_{12}$ [10, 12]. For the $x \geqslant 0.4$ 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 $x \leqslant 0.2$ samples exhibit a low-T power law, implying the existence of low-lying excitations. However, the ρ_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 $x \leqslant 0.2$ 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 $x \geqslant 0.4$ 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].

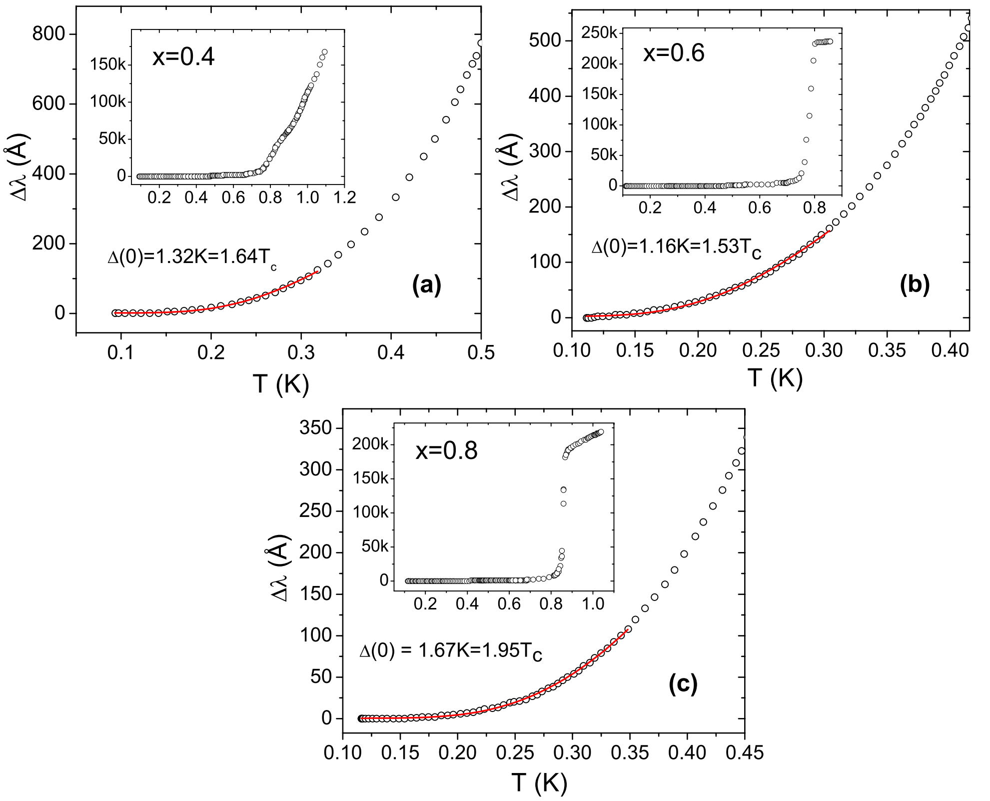

Figure 1. (○) Low-temperature dependence of $\Delta\lambda(T)$ for (a) $x = 0.4$, (b) $x = 0.6$ and (c) $x = 0.8$. Lines: fits to BCS low-T expression from T_{base} to $0.4T_c$. The parameters of the fits are described in the text. Insets show $\Delta\lambda(T)$ over the full temperature range.

The single-crystal samples were grown by the Sb self-flux method [6]. The observation of dHvA effect [7] both in $PrOs_4Sb_{12}$ and $PrRu_4Sb_{12}$ 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_{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 $x \geqslant 0.4$ samples. Figure 1 (○) 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

Table 1. Parameters used to calculate curves in figures 2 and 3. Values for $x = 0$ and 1 are included for comparison.

Sample x	0	0.1	0.2	0.4	0.6	0.8	1.0
$\Delta(0)/k_B T_c$	2.6	1.76	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

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_c^* 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 $0.3T_c$, implying activated behaviour in this temperature range. We fit these data to the BCS low-temperature expression in the clean and local limit, from $T_{\rm base}$ (~0.1 K) to $0.4T_c$, 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 $PrRu_4Sb_{12}$ ($x = 1$).

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 $PrOs_4Sb_{12}$) [20] and 290 nm (for $PrRu_4Sb_{12}$) [12]. We compute ρ_s for an isotropic s-wave superconductor in the clean and local limits using $\rho_s = 1 + 2\int_0^\infty \frac{\partial f}{\partial E}\,d\varepsilon$, where $f = [\exp(E/k_B T) + 1]^{-1}$ 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] $\Delta(T) = \delta_{sc} k_B T_c \tanh\{(\pi/\delta_{sc})\sqrt{(2/3)[(\Delta C)/C][(T_c/T) - 1]}\}$, where $\delta_{sc} \equiv \Delta(0)/k_B T_c$ is the only variable parameter. The specific heat jump $\Delta C/C$ can be obtained from $\Delta(0)/k_B T_c$ 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 (○) and calculated (solid line) values of ρ_s as a function of temperature for the $x \geqslant 0.4$ 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 $x \leqslant 0.2$ 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_c^* 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 $\approx 0.3T_c^*$) 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 (○) 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

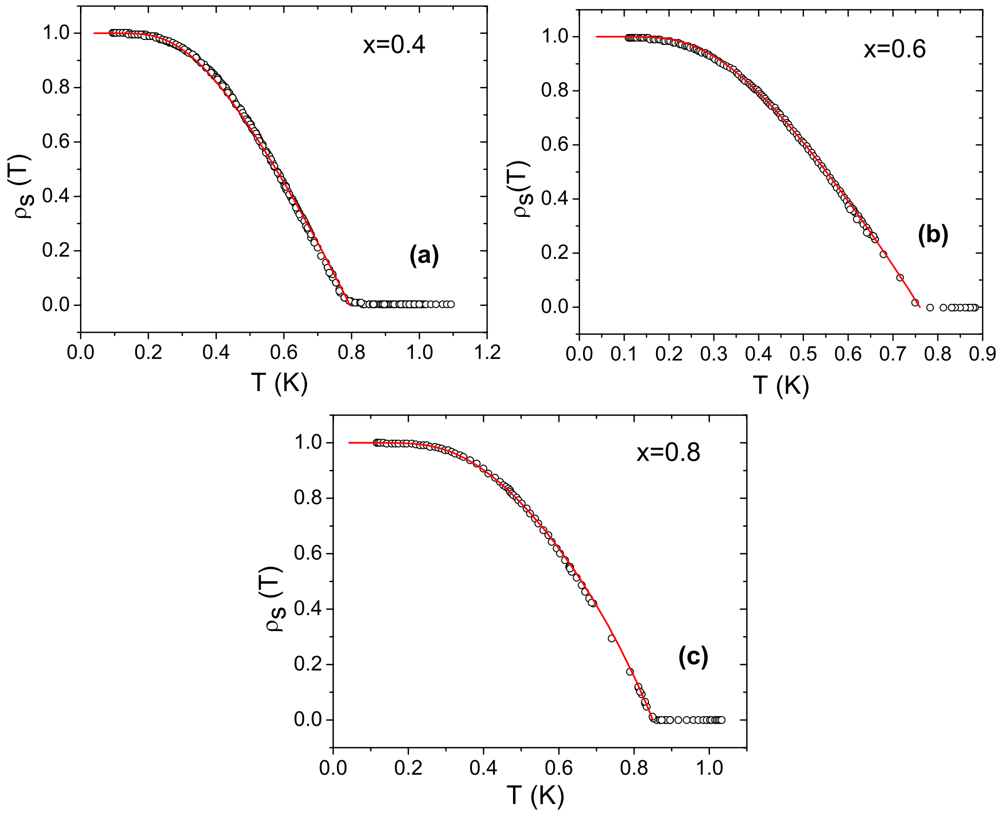

Figure 2. (○) Superfluid density $\rho_s(T) = [\lambda^2(0)/\lambda^2(T)]$ calculated from $\Delta\lambda(T)$ data in figure 1, for (a) $x = 0.4$, (b) $x = 0.6$, and (c) $x = 0.8$. Lines: theoretical $\rho_s(T)$ with parameters $\Delta(0)/k_B T_c$ and $\Delta C/\gamma T_c$ mentioned in the text.

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 ~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 $T_{c3}(x = 0.2) = 0.17 \pm 0.01$ 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 $x \geqslant 0.4$ 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 Γ_1–Γ_5 spacing decreases (observed as x is decreased from 1 to 0 in [15]), superconductivity

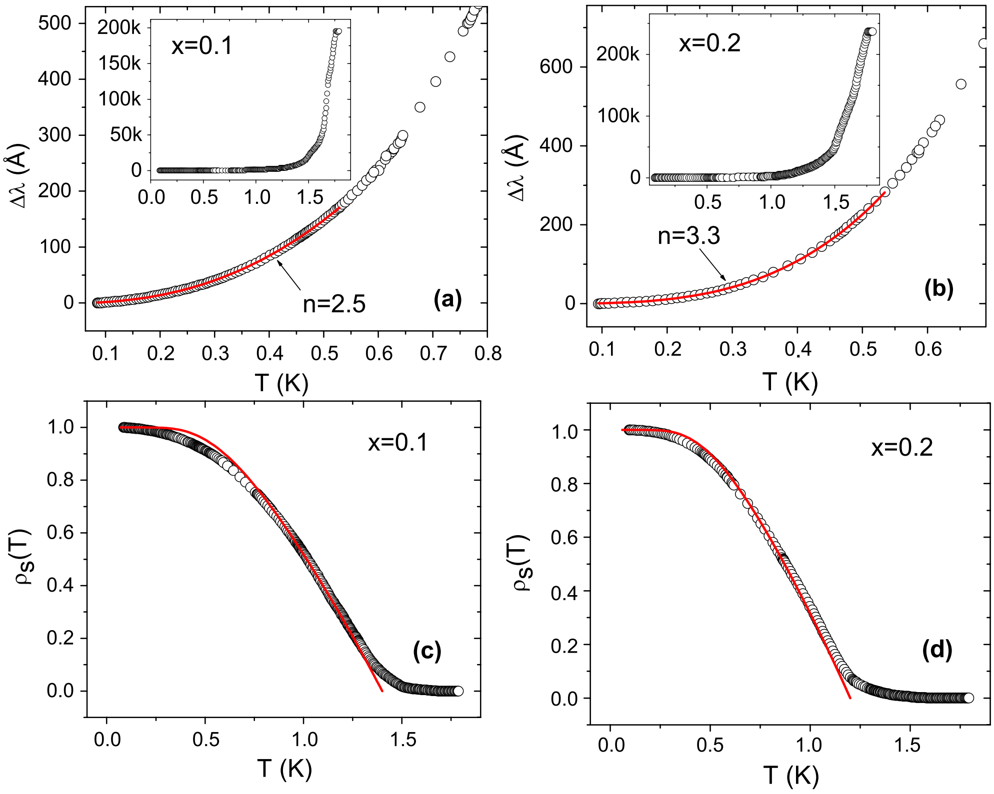

Figure 3. (○) Low-temperature $\Delta\lambda(T)$ for (a) $x = 0.1$ and (b) $x = 0.2$. Lines: fits to $\Delta\lambda(T) = A + BT^n$ from 0.1 to 0.53 K. Insets show $\Delta\lambda(T)$ over the full temperature range. (○) Superfluid density $\rho_s(T)$ calculated from $\Delta\lambda(T)$ data for (c) $x = 0.1$ 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 $x = 0.2$.

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 ρ_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 $Pr_{1-y}La_yOs_4Sb_{12}$ [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_c^* 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

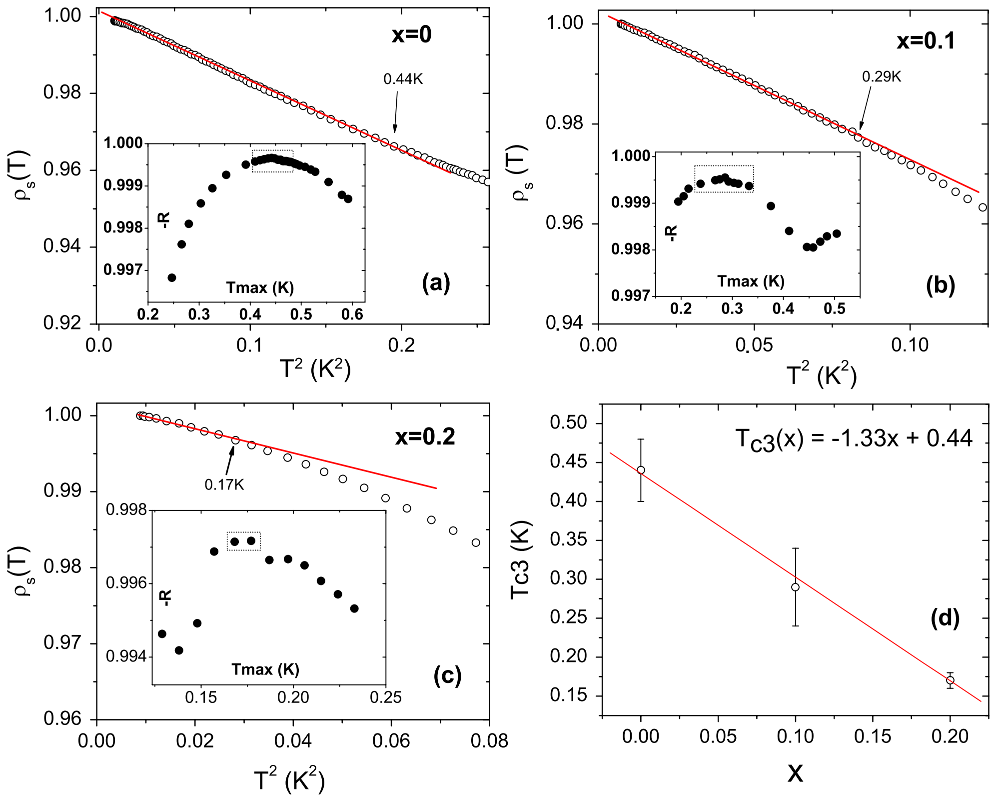

Figure 4. (○) Low-temperature $\rho_s(T)$ versus T^2 for (a) $x = 0$ (data taken from [10]), (b) $x = 0.1$ and (c) $x = 0.2$. The solid lines are visual aids to determining the range of linear fit. Insets: value of $-R$ versus T, where R is the correlation coefficient of the straight-line fit. $R = +1$ (-1) represents a perfect positive (negative) linear relationship between ρ_s and T^2. T_{c3} is defined to be the point of maximum (absolute) R, close to the temperature where ρ_s starts to depart from T^2-behaviour. (d) (○) $T_{c3}(x)$ for $x = 0, 0.1, 0.2$. Line: best linear fit to the three data points. Note that the line extrapolates to zero near $x = 0.33$.

Γ_5 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 Γ_1–Γ_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 λ in single crystals of $Pr(Os_{1-x}Ru_x)_4Sb_{12}$ down to ~0.1 K. Both λ and superfluid density ρ_s exhibit an exponential behaviour for the $x \geqslant 0.4$ samples, going from weak coupling ($x = 0.4, 0.6$) to moderate coupling ($x = 0.8$). For the $x \leqslant 0.2$ samples, both λ and ρ_s vary as T^2 at low temperatures, but ρ_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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