Double superconducting transition and low-temperature specific heat study of 
Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$

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The double superconducting transition in PrOs$_4$Sb$_{12}$, first observed in specific heat $C(T)$ measurements of single crystal samples, is the subject of an in-depth study. The double superconducting transitions of a batch of single crystals were measured before and after they were annealed for 5 days at 500°C, with no observed change. $C(T)$ measurements near $T_c$ for PrOs$_4$Sb$_{12}$ in several magnetic fields are also presented, detailing the evolution of the double transition up to 1 T. Samples of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ with $0.01 \leq x \leq 0.04$ also appear to display a double superconducting transition in specific heat. In addition, samples with the smallest Ru concentration measured ($x = 0.01$) may even display a different type of superconductivity than pure PrOs$_4$Sb$_{12}$ ($x = 0$).

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I. INTRODUCTION

The compound PrOs$_4$Sb$_{12}$ has attracted a great deal of attention in recent years as the first Pr-based heavy fermion superconductor, with a superconducting transition temperature $T_c \approx 1.85$ K and an electron effective mass $m^* \approx 50m_e$. Detailed measurements show the strongest support for a nonmagnetic $\Gamma_1$ singlet ground state in a tetrahedral crystalline electric field (CEF), separated from a $\Gamma_5$ (in the cubic notation) triplet first excited state by approximately 10 K. Quadrupolar ordering arises below 1.5 K and above 4.5 T, related to the crossing of the Zeeman-split CEF energy levels. The superconducting state appears to be highly unconventional, exhibiting time-reversal symmetry breaking and multiple superconducting phases and double superconducting transitions.

Upon substitution of Ru to form Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$, the transition temperature decreases to a minimum of $T_c \approx 0.75$ K at $x = 0.6$, whereupon it increases to $\sim 1.0$ K for PrRu$_4$Sb$_{12}$. This behavior was observed in measurements of electrical resistivity, magnetic susceptibility, and specific heat. All three measurement techniques indicated that the CEF energy level splitting between the ground state and first excited state increased monotonically from $\sim 10$ K for PrOs$_4$Sb$_{12}$ to $\sim 70$ K for PrRu$_4$Sb$_{12}$. The $C(T)$ experiments also revealed that the electronic specific heat coefficient $\gamma$ decreased from $\sim 500$ mJ/mol K$^2$ for $x = 0$ to a plateau of $\sim 100$ mJ/mol K$^2$ for $x \geq 0.6$. Upon subtracting the normal state $\gamma$ and Schottky anomaly due to CEF effects, we were able to investigate the structure of $C(T)$ below $T_c$. We performed phenomenological fits which suggested that the PrOs$_4$Sb$_{12}$ data below $T_c$ were more in agreement with power law behavior, indicating nodes in the energy gap, while the samples with $0.05 \leq x \leq 0.2$ suggested exponential behavior, consistent with an isotropic energy gap. In addition, no obvious double superconducting transition was observed for $x = 0.05$, the lowest substituted concentration measured in the previous study.

In this paper, we present the results of further probes into the nature of the double superconducting transition in PrOs$_4$Sb$_{12}$. We observe the effect of magnetic fields, annealing, and Ru substitution on the double transition through measurements of $C(T)$. These results are compared to the double superconducting transitions observed in the heavy fermion systems U(Pt$_{1-x}$Pd$_x$)$_3$ and U$_{1-x}$Th$_x$Be$_{13}$. The low-temperature specific heat of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ is investigated in order to determine a possible concentration at which the superconducting state changes from one possessing nodes in the energy gap to one that is more isotropic.

II. EXPERIMENTAL DETAILS

The single crystals of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ studied in this work were grown using an Sb flux method, and the crystal structure was confirmed via x-ray diffraction measurements, as described previously. The specific heat $C(T)$ measurements were performed in the same cryostat as for previous experiments using a semi-adiabatic method at temperatures between 0.6 K and 20 K. Magnetic fields were applied using a 5 T superconducting magnet attached to the outside of the vacuum can. The experiments in a magnetic field were performed on the same individual single crystal measured by Cichorek et al. with a mass of 8.17 mg. The field direction was along a principal crystal axis. The other measurements were made on collections of single crystals, with total masses between 20 and 30 mg.

The annealing was carried out by wrapping the crystals in tantalum foil, which was sealed inside a quartz tube with a piece of zirconium foil, under 150 Torr UHP Ar. The quartz tube was placed in a 500°C furnace for 5 days, and then quenched to room temperature. The mass of the sample was the same after annealing within the systematic error of the weighing scales.
III. RESULTS AND DISCUSSION

The only other stoichiometric heavy fermion superconductor that displays a double superconducting transition is UPt₃. For single crystals of this compound, annealing can be an important factor in observing the double transition in specific heat. The results of an annealing study on a batch of PrOs₄Sb₁₂ crystals are shown in Fig. 1. The two data sets fall neatly on top of each other, which argues against any kind of inhomogeneous mass redistribution. Unlike UPt₃, however, the double transition in PrOs₄Sb₁₂ shows no indication of becoming more resolved due to annealing. It is still important to note that the double transition does not degrade with annealing, as might be expected if this feature were not an intrinsic effect of PrOs₄Sb₁₂.

Figure 2 displays the evolution of the double superconducting transition with magnetic fields for PrOs₄Sb₁₂. The double transition is clearly visible in the zero field data, while by 1 T the transition has been suppressed below 1 K. As the magnetic field is increased, the first transition appears to lose its sharpness, until by 0.75 T it is either nonexistent, or indistinguishable within the strong downturn of the Schottky anomaly. The inset to Fig. 2 shows the data after subtracting the 1 T curve, which further emphasizes the initial jumps. This evolution of the transition with magnetic field has been reported by several other groups, with mostly different results. Vollmer et al. found that the double transition disappears by 0.4 T, while the double transition observed by Grube et al. persists up to 0.6 T, similar to this work. The data reported by Measson et al. are noticeably different, as two clear transitions were visible up to ~2 T. All results, except those of Vollmer et al., were from experiments performed on one single crystal of PrOs₄Sb₁₂: the experiments of Measson et al. relied on an ac calorimetry method instead of the semi-adiabatic method utilized by Vollmer et al., Grube et al., and this work. In UPt₃, applying a magnetic field and lowering the temperature causes the two transitions to merge near ~0.5 T and ~0.4 K, depending on the field orientation, giving rise to an additional phase transition within the superconducting state. While several experiments provide evidence for multiple phases in the superconducting state of PrOs₄Sb₁₂, none of these phases can as yet be associated with the double superconducting transition.

Specific heat divided by temperature C(T)/T data for Pr(0.05)Sb₁₂ with 0 ≤ x ≤ 0.05 are displayed in Fig. 3. The normal state data below 10 K were fitted with an equation that includes electronic, lattice, and CEF contributions, with the results listed in Table I. The quantity r is a scaling factor for the Schottky anomaly, as discussed previously. This suppression of the Schottky anomaly could result from an energy dispersion due to Pr-Pr interactions or hybridization between the Pr f-electrons and ligand states, which results in entropy being transferred to the conduction electrons, thereby increasing the electronic specific heat coefficient γ. The errors for the parameters were determined by varying the Debye temperature θ₀ by ±10 K from its best fit value, as described previously. Only results from a Γ1 ground state fit are presented here; we were able to accurately use the simpler cubic CEF equations since these measurements were only performed in zero field. As can be seen from Table I, the splitting between the ground state and first excited state, δ, increases monotonically with increasing x, as expected. The electronic specific heat coefficient γ, on the other hand, displays a maximum at x = 0.02. The Debye temperature θ₀ is also enhanced for x = 0.01 and x = 0.02 compared to the neighboring concentrations.

The superconducting transitions in Fig. 4 are plotted as C(01)x(T)/T, where C(01)x is the electronic specific heat after the lattice and Schottky terms were subtracted. Structure in the superconducting transition is clearly apparent for x = 0.01 and x = 0.02, and also appears to be present for x = 0.04. In light of these data, the rounded structure of the x = 0.05 transition published previously might also hide a double transition, but it is too broad to tell for sure. The transitions for 0 ≤ x ≤ 0.04 were analyzed using an entropy conserving construction for both transitions. The upper and lower transitions and their respective specific heat jumps are presented in Table II. It is interesting to note that the lower transition has the largest jump for PrOs₄Sb₁₂, while for 0.01 ≤ x ≤ 0.04, the upper transition displays a larger jump. This is shown graphically in Fig. 5, which allows us to speculate that the upper transition jump and the overall transition jump may converge near x = 0.05. Also listed in Table II is the ratio ΔC/γTc for the full transition. The value for x = 0 of 1.76 is enhanced over the BCS value of 1.43, suggesting strong-coupling superconductivity. The results for the upper and lower transitions are not tabulated, although they can be easily calculated from the other supplied data. The upper and lower transitions have ΔC/γTc values of 0.86 and 1.12, respectively. As x increases, ΔC/γTc drops sharply, due to the strong increase in γ and the smaller decrease in Tc, to values well below 1.43, even for the full transitions.

The evolution of the double transition in Pr(0.05)Sb₁₂ is qualitatively similar to that seen in U(Pr₁₋ₓPdₓ)₃, which displays a double transition down to x = 0.003, but with no superconductivity observed at all for x > 0.004 above 1 K. The lower transition is very small in U(Pr₁₋ₓPdₓ)₃ for x = 0.003, but still may be present for higher concentrations at temperatures lower than what was measured. Nevertheless, the specific heat jump at the lower transition in both series of compounds decreases in magnitude faster than the jump for the upper transition. An interesting discrepancy between the two substituted series is that in U(Pr₁₋ₓPdₓ)₃, the difference between the two superconducting transitions, ΔTc, increases with increasing x, while in Pr(0.05)Sb₁₂ the separation between the transitions remains nearly constant. Unfortunately, the smaller amount of research on PrOs₄Sb₁₂ makes it difficult to determine which simi-
larities and differences between Pr(\text{Os}_{1-x}\text{Ru}_x)_4\text{Sb}_{12} and U(\text{Pt}_{1-x}\text{Pd}_x)_3 are meaningful. It should be noted that a double superconducting transition has been reported for Pr\text{La}_x\text{Os}_4\text{Sb}_{12} with \(x = 0.02\) and \(x = 0.05\), and the lower transition also appears to be the one suppressed with \text{La} substitution.\text{La} substitution.

Figure 5 displays fits to the data below \(T_c\) for both power law (appropriate for energy gaps with nodes) and exponential (appropriate for isotropic energy gaps) functions, a continuation of the fits performed previously. The new fits for \(x = 0.01, 0.02, \) and \(0.04\) were all made between \(\sim 0.6\) K (the base temperature of the measurement) and 1.15 K. Neither fit is better than the other within the fit range, but the phenomenological extrapolation of the fits to higher temperatures suggests that there may be a fundamental difference between the superconductivity in Pr\text{Os}_4\text{Sb}_{12} and the Ru-doped materials, even with only 1\% Ru. A specific heat study of U\text{Th}_x\text{Be}_{13} deep in the superconducting state revealed a similar evolution from power-law to BCS-like behavior with increasing \(x\), separated at \(x \approx 0.02\). The parameters from fits to the Pr\text{Os}_{1-x}\text{Ru}_x\text{Sb}_4 superconducting state data are listed in Table II. A rough estimate for the coupling strength can be obtained by calculating \(2\Delta c/T_c\) (with \(\Delta c\) in units of K), which is approximately 4.5 for the values of \(x\) studied in this work. This enhancement over the BCS estimate of 3.52 is consistent with other measurements on Pr\text{Os}_4\text{Sb}_{12} which indicate a strong-coupling energy gap.

A double superconducting transition is one of the clear indications of unconventional superconductivity, and possibly a multi-component superconducting order parameter. Studies of \text{UPt}_3 strongly indicate that the lower transition in this compound arises from the onset of a new order parameter (presumably antiferromagnetic)\text{antiferromagnetic} and the double transition in Pr\text{Os}_4\text{Sb}_{12} is not, as yet, as well studied or understood. Time reversal symmetry breaking (TRSB) has been observed in Pr\text{Os}_4\text{Sb}_{12} through measurements of muon spin rotation (\(\mu\text{SR}\)) in zero field, which exhibit a spontaneous magnetic moment arising in the superconducting state. It is difficult to tell from the published data whether the moment arises due to the upper or the lower superconducting transition. In \text{UPt}_3, a spontaneous moment in \(\mu\text{SR}\) was reported below the lower superconducting transition, but this result has not proven to be reproducible via \(\mu\text{SR}\) measurements.\text{antiferromagnetic}

However, ultrasound, vortex, and flux flow measurements have revealed features below the lower superconducting transition associated with additional superconducting order parameters and, possibly, with TRSB. A magnetic moment has been observed to arise in \(\mu\text{SR}\) measurements below the lower superconducting transition of U\text{Th}_x\text{Be}_{13} with \(0.02 \leq x \leq 0.04\), also suggesting TRSB. Measurements of vortex motion in U\text{Th}_x\text{Be}_{13} with \(x = 0.0275\) below the lower superconducting transition indicate a sharp drop in vortex creep rates, very similar to that seen in \text{UPt}_3. It therefore would be very useful to perform these measurements on low \(x\) samples of Pr\text{Os}_{1-x}\text{Ru}_x\text{Sb}_{12} in order to determine if the TRSB and unconventional superconductivity truly track the double superconducting transition, as these present measurements suggest.

### IV. SUMMARY

In summary, we have investigated the double superconducting transition in Pr\text{Os}_4\text{Sb}_{12} through specific heat measurements in more detail than previously reported. Annealing the crystals did not appear to affect the features in the specific heat near \(T_c\). In a magnetic field, the transitions rapidly broaden in temperature and are suppressed by 1 T. Samples of Pr\text{Os}_{1-x}\text{Ru}_x\text{Sb}_{12} with smaller Ru concentrations than previously measured displayed double superconducting transitions in \(C(T)\) up to \(x = 0.04\). Power law and exponential fits to \(C(T)\) below \(T_c\) also suggest that the superconducting state for \(x = 0\) is different than that for \(x \geq 0.01\). The behavior of the double superconducting transition for Pr\text{Os}_{1-x}\text{Ru}_x\text{Sb}_{12} appears to only share qualitative characteristics with the superconducting heavy fermion systems U(\text{Pt}_{1-x}\text{Pd}_x)_3 and U\text{Th}_x\text{Be}_{13}.

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FIG. 1: Specific heat divided by temperature $C/T$ versus $T$ below 10 K for an unannealed set of single crystals of PrOs$\mathrm{Sb}_{12}$ (open circles) and the same sample annealed for 5 days at 500 °C (solid line). Inset: close-up of the double superconducting transition. There is almost no difference between the two data sets.

FIG. 2: Specific heat divided by temperature $C/T$ versus $T$ between 1 and 2 K in magnetic fields up to 1 T for an individual single crystal sample of PrOs$\mathrm{Sb}_{12}$. The data for fields below 1 T have been truncated below the superconducting transitions for clarity. Inset: $\Delta C/T$ versus $T$, where $\Delta C = C(H, T) - C(H = 1 T, T)$. 
TABLE I: Normal state physical properties of samples of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$, determined from specific heat data. The parameter $\delta$ is the splitting between the ground state and the first excited state in the Schottky anomaly, $r$ is the scaling factor for the Schottky anomaly (as described in the text), $\gamma$ is the estimated electronic specific heat coefficient, and $\Theta_D$ is the estimated Debye temperature. The errors in the parameters were determined by allowing $\Theta_D$ to vary by $\pm$10 K within the fits (see text and Ref. 16 for details).

| $x$ | $\delta$ (K) | $r$ (mJ/mol K$^2$) | $\gamma$ (K) | $\Theta_D$ (K) |
|-----|-------------|------------------|-------------|----------------|
| 0   | 7.36±0.04   | 0.56±0.01        | 586±33      | 211            |
| 0.01| 8.28±0.01   | 0.43±0.01        | 817±10      | 278            |
| 0.02| 8.43±0.01   | 0.35±0.01        | 844±12      | 271            |
| 0.04| 9.19±0.01   | 0.45±0.02        | 727±42      | 201            |
| 0.05| 10.2±0.01   | 0.39±0.01        | 775±25      | 224            |

TABLE II: Superconducting state physical properties of samples of Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$, determined from specific heat data. $T_c$ is the superconducting transition temperature, $\Delta C$ is the jump in $C(T)$ at $T_c$, $n$ is the exponent of the power-law fit below $T_c$, $\gamma^e_s$ is the electronic specific heat coefficient in the superconducting state ($\gamma^p_s = 0$ for the power law fits), and $\Delta_e$ is the parameter in the exponential fit below $T_c$ that is proportional to the energy gap. The errors in the parameters were determined by allowing $\Theta_D$ to vary by $\pm$10 K within the fits (see text and Ref. 16 for details), with the exception of $T_c$ and $\Delta C/T_c$ (the errors for $\Delta C/T_c$ are represented graphically in Fig. 4).

| $x$ | upper transition | lower transition | full transition |
|-----|------------------|------------------|-----------------|
|     | $T_{c1}$ (K)     | $\Delta C_{c1}$ (mJ/mol K$^2$) | $T_{c2}$ (K) | $\Delta C_{c2}$ (mJ/mol K$^2$) | $\Delta C/\gamma T_c$ (mJ/mol K$^2$) | $\Delta_e$ (K) |
| 0   | 1.84             | 565              | 1.73           | 681            | 1.76          | 2.27±0.01 | 9.12±1.1 | 3.97±0.07 |
| 0.01| 1.79             | 393              | 1.65           | 258            | 1.73          | 0.74      | 65.1±0.6 | 3.73±0.02 |
| 0.02| 1.77             | 345              | 1.62           | 249            | 1.71          | 0.65      | 82.1±0.5 | 3.93±0.01 |
| 0.04| 1.72             | 337              | 1.58           | 145            | 1.68          | 0.66      | 63.6±1.3 | 3.77±0.03 |
| 0.05| —                | —                | —              | —              | 1.63          | 0.42      | 55.2±0.7 | 3.79±0.01 |
FIG. 3: Specific heat divided by temperature $C/T$ versus $T$ below 10 K for Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ with $0 \leq x \leq 0.05$. Inset: superconducting transition temperature $T_c$ (filled circles, left axis) and the electronic specific heat coefficient $\gamma$ (filled squares, right axis) as a function of Ru concentration $x$. The vertical lines for $T_c$ delineate the upper and lower transitions for those concentrations which display double transitions.

FIG. 4: Comparison of double superconducting transitions in $C_{el}/T$, after lattice and Schottky anomaly terms corresponding to a $\Gamma_1$ ground state have been subtracted, for Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ with (a) $x = 0$, (b) $x = 0.01$, (c) $x = 0.02$, and (d) $x = 0.04$. The transitions were approximated with an entropy conserving construction.
FIG. 5: Specific heat jump at $T_c$, $\Delta C/T_c$, for PrOs$_4$Sb$_{12}$ as a function of Ru concentration $x$. The dashed lines represent speculations on the continued evolution of $\Delta C/T_c$.

FIG. 6: Comparison of exponential (dashed line) and power law (solid line) fits below $T_c$, after subtracting lattice and Schottky anomaly terms for a $\Gamma_1$ ground state, for PrOs$_{1-x}$Ru$_x$Sb$_{12}$ with (a) $x = 0$, (b) $x = 0.01$, (c) $x = 0.02$, and (d) $x = 0.04$. The $x = 0$ fits only extend up to $\sim 1.2$ K, while the $x = 0.01$, $x = 0.02$, and $x = 0.04$ fits only extend up to $\sim 1.15$ K, as described in the text.