Evolution of superconductivity in Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$: Upper critical field measurements

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Measurements of the upper critical field $H_{c2}$ near $T_c$ of Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$ were performed by specific heat. A positive curvature in $H_{c2}$ versus $T$ was observed in samples and concentrations exhibiting two superconducting transitions. These results argue against this curvature being due to two-band superconductivity. The critical field slope $-dH_{c2}/dT$ suggests the existence of a crossover concentration $x_{cr} \approx 0.25$, below which there is a rapid suppression of effective electron mass with La-alloying. This crossover concentration was previously detected in the measurement of the discontinuity of $C/T$ at $T_c$.

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I. INTRODUCTION

PrOs$_4$Sb$_{12}$, a new heavy fermion superconductor, remains under extensive research scrutiny for a number of reasons. First of all, it clearly demonstrates that the heavy fermion state can exist in a system based on non-Kramers $f$-ions, such as Pr. This opens a possibility for novel electronic states and novel microscopic mechanisms leading to the heavy fermion behavior, which may also be applicable to many previously investigated systems, particularly those based on U. Secondly, several experimental observations suggest an unconventional superconductivity in this material. Both of these important aspects are the subjects of our investigation in which Pr is systematically replaced by La.

In our previous study$^2$ we have demonstrated the usefulness of this alloying method due to the fact that both end-compounds PrOs$_4$Sb$_{12}$ and LaOs$_4$Sb$_{12}$ exist in the same crystal structure with essentially identical lattice constants. This lack of a mismatch of the lattice parameters provides a unique opportunity to study the superconducting state that is normally very sensitive to local stresses resulting from a distribution of interatomic distances. Also, the fact that both end-compounds are superconducting with $T_c$’s differing only by a factor of 2 allows one to compare physical properties in closely related superconductors of which one is conventional while the other is probably not. Among experimental evidences suggesting the unconventional character of superconductivity in PrOs$_4$Sb$_{12}$ are the temperature dependence of the specific heat,$^3$ spontaneous appearance of a static magnetic field bellow the superconducting transition,$^3$ penetration depth measurements$^3$, and presence of two superconducting transitions in the specific heat$^2,6,7,8,9,10$ and thermal conductivity measurements$^2$. However, neither of the interpretations of these observations is well established and some of them remain controversial. In particular, there is no consensus whether the two transitions in the specific heat coexist or whether they occur at different parts of the sample at different temperatures.

Equally controversial is the origin of the heavy fermion behavior in PrOs$_4$Sb$_{12}$. Theoretical scenarios most often considered include the quadrupolar Kondo effect$^{12}$, inelastic scattering by low-energy crystal field levels,$^{13,14}$ rattling motion$^{15}$ of rare-earth ions, and fluctuations of the quadrupolar order parameter. However, a conventional superconductivity and lack of mass enhancement in LaOs$_4$Sb$_{12}$ indicate an importance of $f$-electrons. The first two models on the other hand, being single impurity type models, seem to be inconsistent with the results of the specific heat study of Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$. The $C/T$ (specific heat divided by temperature) discontinuity at $T_c$, which is proportional to $m^*$, is strongly suppressed by the La doping.$^2$ At the same time, the relevant single-impurity parameters, such as the crystalline electric field (CEF) spectrum and interatomic separations, are essentially unaltered by the alloying. However, since the $C/T$ discontinuity at $T_c$ depends also on a coupling strength, we have searched for additional evidences of the collective nature of the heavy fermion state in PrOs$_4$Sb$_{12}$. Here we report the results for the upper critical field of Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$ studied by specific heat.
II. EXPERIMENTAL

Samples used in this investigation were single crystals grown by the Sb-flux method. All specific heat measurements were performed on individual single crystals. These crystals were between 1 and 5 mg.

Before discussing results for the critical fields we comment on the ac-susceptibility data shown in Fig. 1. The ac-susceptibility measurements were performed in the Earth’s magnetic field and ac field of approximately 0.1 Oe. No significant differences in these results were detected for two different frequencies used, 27 and 273 Hz. The ac-susceptibility, arbitrarily normalized, is shown versus reduced temperature, $T/T_c$, where $T_c$ for this purpose was determined by the diamagnetic onset. The results of the ac-susceptibility are sample dependent, similarly to the discussed next specific heat. For $x = 0$ we show the data for two crystals, of which the crystal No. 2 clearly has two steps in the ac-susceptibility, while crystal No. 1 seems to have one step only. However, both samples have wide transitions. The transition for the crystal with a single step is even wider than the combined transition of crystal No. 2. All previously reported ac-susceptibility curves for $x = 0$ also showed large widths, typically 0.2 K. The temperature separation of the two steps in the susceptibility, 0.13-0.14 K, is approximately equal to the temperature separation of the two peaks in the specific heat. These results strongly argue for the inhomogeneous coexistence of two superconducting phases in PrOs$_4$Sb$_{12}$. Substitution of small amounts of La for Pr (5%) does not eliminate the two steps in the susceptibility but makes the separation of the two steps and the overall width of the transition smaller. In general, the ac-susceptibilities for samples in the range $x = 0.2$ to 0.8 are similar. There is a slight increase of the width of the transition (on the reduced temperature scale) between $x = 0.6$ and 0.8, followed by a dramatic reduction for LaOs$_4$Sb$_{12}$. The reduction of the width of the transition in the ac-susceptibility with small values of $x$ correlates with our previous observation of the decrease of the width of the superconducting anomaly in the specific heat between $x = 0$ and a crossover concentration $x_{cr} \approx 0.2 - 0.3$. The specific heat implied the disappearance of the second superconducting transition near $x_{cr}$.

These wide transitions in the ac-susceptibility of PrOs$_4$Sb$_{12}$ cannot be explained by very small $H_{c1}$ (field at which magnetic flux is completely expelled) in comparison with $H_{c2}$ (onset of diamagnetism). Based on the reported $dH_{c1}/dT$ slope we expect the width of the transition to be less than 20 mK. The origin of possible inhomogeneities is unclear. Sharp transitions observed in LaOs$_4$Sb$_{12}$ samples argue against structural defects since La and Pr are chemically similar. Thus, these inhomogeneities seem to be associated with 4f-electrons of Pr. One plausible scenario is a mixture of two electronic configurations of Pr, $4f^1$ and $4f^2$. The analysis of the high temperature magnetic susceptibility by various groups lead to the effective high temperature paramagnetic moment between 3.2 and 3.6 $\mu_B$/Pr. (The expected moments are 2.54 and 3.59 $\mu_B$ for $4f^1$ and $4f^2$ configurations, respectively.) Small size of crystals and sample holder contribution could lead to these discrepancies. Therefore, we have synthesized a large 50 mg crystal, which was placed between two long concentric and homogeneous tubes such that no background subtraction was needed to accurately determine the magnetization due to the crystal. The effective magnetic moment measured for this crystal at temperatures between 200 and 350 K is equal to the expected value for the $4f^2$ configuration. Also, the $L_{111}$ absorption and inelastic neutron scattering results argue for the average Pr valence very close to 3. Another possible scenario for the existence of inhomogeneities is due to the closeness of the system to a long range antiferro-quadrupolar order. Clusters with a short-range-order would have different superconducting parameters than the remaining part of the sample. The discussed next magnetic response of the specific heat shows that these two plausible superconducting phases might not be independent; there seems to be some coupling between them.

There is also some sample dependence of the two superconducting anomalies in the specific heat of the pure PrOs$_4$Sb$_{12}$ and of their response to small magnetic fields. Let us consider the zero field data first. Crystal No. 1, for which a wide transition in the ac-susceptibility is shown in Fig. 1, (most upper panel) has rather well defined anomalies in $C/T$ at temperatures $T_{c1}$ and $T_{c2}$. The method of extracting $T_{c1}$ and $T_{c2}$ for the purpose of critical fields’ analysis is illustrated in Fig. 2.) Another investigated crystal from the same batch exhibited almost identical zero-field specific heat. On the other hand crystal No. 2 from a different batch, that showed two steps in the ac-susceptibility, has a sharp peak at $T_{c2}$ and only a small shoulder that might correspond to a transition at $T_{c1}$. This strong sample dependence can be inferred from previously published specific heat data, as well. However, according to the majority of published specific heat measurements, the anomaly at $T_{c2}$ is more pronounced than that at $T_{c1}$. We have also observed different response of the two PrOs$_4$Sb$_{12}$ crystals to small magnetic fields. Magnetic fields were applied approximately in the (100) direction for all crystals investigated. For crystal No.1 magnetic field suppresses the height of the anomaly at $T_{c2}$ stronger than at $T_{c1}$, such that only the high temperature transition at $T_{c1}$ can be clearly seen in fields of the order 0.5 T. In particular, the negative slope in $C/T$ versus $T$ between $T_{c2}$ and $T_{c1}$ in $H = 0$ T becomes positive in the field of 0.5 T. On the other hand, in sample No. 2 the low temperature anomaly dominates at all fields studied up to 1.2 T. This last crystal has both zero and magnetic field specific heat similar to those presented by Frederick et al. Comparisons of the susceptibility and zero-field specific heat for the two crystals seems to indicate different vol-
ume fractions occupied by two superconducting phases, with crystal No. 1 having a relatively large fraction corresponding to the higher $T_c$ phase. However, the redistribution of the relative heights of the anomalies in crystal No. 1 by magnetic fields suggests some interdependence of the two superconducting phases.

Insets to Fig. 2 present the critical fields versus $T$ determined by the specific heat. For sample no. 2 we were not able to determine $T_{c1}$ and only one line of transitions, that for $T_{c2}$, is shown. In agreement with previous reports we find a positive curvature in $H_{c2}$ versus $T$ (near $T_c$) in sample no. 1. This positive curvature in $H_{c2}(T)$ for PrOs$_4$Sb$_{12}$ has been detected in various measurements, such as electrical resistivity, ac-heat capacity, magnetic susceptibility, and has been proposed to be a signature of the two-band superconductivity. Additional measurements of a part of this crystal showed that $H_{c2}$ between 0.3 and at least 0.6 T is linear in $T$. On the other hand, we do not find any curvature in $H_{c2}$ (extracted from the measurement of $T_{c2}$ versus $T$) near $T_{c2}$ in sample no. 2, within the resolution of this measurement.

A small, but detectable curvature, in $H_{c2}$ versus $T$ was observed again in some Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$ crystals with small amounts of Lanthanum, $x=0.02$ and 0.05. Figure 3 shows $C/T$ for one of our $x=0.05$ alloys in several representative fields. (Only one line of transitions could be clearly identified for all alloys with $x>0$. ) There might be some curvature in the $H_{c2}(T)$ for $x=0.1$ (Fig. 4), although our data are not sufficiently precise to resolve it. On the other hand, alloy $x=0.3$ (Fig. 5) and alloys with $x>0.3$ seem to have a conventional variation of $H_{c2}(T)$, without a positive curvature near $T_c$. The slope of $H_{c2}$ versus $T$ at $T_c$ is of great interest since it is related to the effective mass of Cooper pairs. The discussed plausible curvature makes the determination of $dH_{c2}/dT$ for the pure Pr compound and weakly doped alloys somewhat arbitrary. Furthermore, there is an important question whether $H_{c2}$ is linear in $T$ in the direct vicinity of $T_{c1}$ followed by a crossover regime to a linear dependence with a larger slope at lower temperatures or if there is a curvature at any temperature near $T_{c1}$. Our point by point specific heat measurement technique with limited resolution can not fully address this issue but seems to prefer the first scenario. For several small $x$-value alloys we were able to observe two linear regimes. Microwave surface impedance measurements by Broun et al. clearly identify two linear regimes in $\lambda^2$ versus $T$ near $T_c$. However, the relationship between our critical field measurement and reported penetration depth is not clear at present.

Therefore, in Fig. 5 we show $-dH_{c2}/dT$ calculated for sufficiently large fields, for which $H_{c2}$ is clearly linear in $T$ (for $H > 2000$ Oe for $x=0$ and $H > 1000$ for $x=0.02$ and 0.05), as closed symbols and the initial slope measured in fields smaller than 1000 Oe as open symbols. In agreement with previous studies this critical field slope for PrOs$_4$Sb$_{12}$, obtained in this case for fields larger than 0.5 kOe, is about $-2.1T/K$. $-dH_{c2}/dT$ decreases monotonically with $x$, but most of this reduction takes places for small values of $x$. Such a variation of the slope strongly implies that the effective mass of carriers is rapidly reduced by a small amount of La substituted for Pr.

As it has been already demonstrated, the superconductivity of PrOs$_4$Sb$_{12}$ can be considered in the clean limit. The value of the mean free path $l_0$ is between 1000 and 2000 Å. This lower limit is obtained from our resistivity measurement ($\rho_0$ approximately 5 $\mu\Omega$-cm) while the upper limit from the Dingle temperature. Thus, the mean free path is significantly larger than the reported coherence length $\xi_0$ of 116 Å calculated from the critical field slope. Our best estimate of $\xi_0$, 112 Å, is in excellent agreement with that value, considering uncertainties of the measurements. We expect the mean free path for LaOs$_4$Sb$_{12}$ to be of the same order as that for PrOs$_4$Sb$_{12}$. Using the published $\rho_0$ of approximately 3 $\mu\Omega$-cm we arrive at $l \approx 1600$ Å. The coherence length for LaOs$_4$Sb$_{12}$ is much larger than that for PrOs$_4$Sb$_{12}$. Assuming the validity of the clean limit formulae, $\xi_0 \approx 970$ Å, in agreement with the approximation of Sugawara et al. The corresponding Fermi velocities are 1.52 $10^6$ and 5.25 $10^6$ cm/s for the Pr and La compound, respectively. Since the lattice constants of both compounds are almost identical and Pr has the same number of valence electrons as La, the Fermi wave vectors should be equal. Therefore, the ratio of Fermi velocities should be equal to the ratio of effective masses or electronic specific heat coefficients. There is some distribution for reported $\gamma$ values of LaOs$_4$Sb$_{12}$. Using the largest reported value of 59 mJ/K$^2$ and the derived ratio of Fermi velocities we arrive at 200 mJ/K$^2$ as the estimate of $\gamma$ for PrOs$_4$Sb$_{12}$. This estimate is in contrast to the discontinuity in $\Delta C/T_c/\gamma$, which falls between 800 - 1000 mJ/K$^2$ (depending on sample and analysis method), suggesting an enhancement of $\Delta C/T_c/\gamma$ due to collective many-body effects beyond mass enhancement.

In the clean-limit of superconductivity, which appears to be the appropriate limit for Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$ alloys, the effective mass depends on $\sqrt{-dH_{c2}/dT/T_c}$. Figure 7 shows this latter quantity versus concentration $x$. Again, this figure suggests that the greatest reduction of the effective mass takes place for small values of $x(<0.2-0.3)$. Interestingly, when $\sqrt{-dH_{c2}/dT/T_c}$ is extrapolated from high and intermediate values of $x$ to $x=0$, the obtained value is close to the one calculated from the initial slope of $dH_{c2}/dT$ for PrOs$_4$Sb$_{12}$. This observation provides an indirect support to our hypothesis that there are two linear regimes in $H_{c2}$ versus $T$ in at least some crystals of the undoped Pr compound.

In the inset to Fig. 7 we present the overall discontinuity of the specific heat divided by $T_c$. This latter quantity can also be used as an estimate of the effective electron mass, $m^*$, particularly if the coupling strength were constant. Note a close resemblance of the concentration dependence of the two sets of data, $\sqrt{-dH_{c2}/dT/T_c}$ and $\Delta C/T_c$. Clearly, $x_{cr} \approx 0.2 - 0.3$ is a crossover concentration between a rapid reduction of these quantities.
up upon the La-doping and regime corresponding to moderate or small changes with $x$. The critical field slope (Fig. 5 and 6) implies that there is still significant reduction of $m^*$ with $x$ for $x > 0.3$, but the main change takes place for $x < x_{cr}$. A crossover concentration for $m^*$ seems to exist also in Pr(Os$_{1-x}$Ru$_x$)$_4$Sb$_{12}$ series. Its $x_{cr}$ is much smaller, of about 0.06 only. However, the Ru-alloying is expected to have much more drastic effects on f-electrons of Pr than the La-doping. The hybridization parameters are directly altered by the Ru-alloying and the CEF energies are increased.

Consider this positive curvature or two slopes in $H_{c2}$ versus $T$ of PrOs$_4$Sb$_{12}$ in the framework of the two-band superconductivity model. A smaller initial slope seems to suggest that the transition at $T_{c1}$ is dominated by lighter quasiparticles. Heavier electrons contribute mainly to the lower temperature transition. However, our results suggest that the ratio of the effective masses of the two bands is of the order 2, only. This could explain why the two discontinuities in $C(T)$ at $T_{c1}$ and $T_{c2}$ in some of the reported results are of similar magnitude. Furthermore, the La-alloying data indicate that the change of slope in $H_{c2}$ versus $T$, near $T_c$, correlates with the existence of two superconducting transitions in the specific heat. I.e., alloys with $x < x_{cr}$ in addition to showing enhanced $\Delta C/T_c$, usually exhibit a curvature or two different slopes in $H_{c2}$ versus $x$ and wide or two transitions in the specific heat. This correlation between the existence of the curvature in $H_{c2}$ versus $x$, argued to be due to two-band superconductivity, and presence of two superconducting transitions is unexpected.

III. SUMMARY AND ACKNOWLEDGEMENTS

The change of the slope in $H_{c2}$ versus $T$ near $T_c$, previously ascribed to two-band superconductivity, seems to be characteristic of samples and concentrations that exhibit two superconducting anomalies. This correla-
tion is clearly inconsistent with the proposed Josephson coupling of the two bands.4,10 In this latter case, only the higher temperature transition should be observed. On the other hand, if we consider the No. 1 crystal of PrOs$_4$Sb$_{12}$ only, for which we clearly observe the curvature in $H_{c2}$ versus $T$, the magnetic field response could be understood if the coupling between the two order parameters was brought about by external magnetic fields.

The reported temperature variation of the upper critical field in Pr$_{1-x}$La$_x$Os$_4$Sb$_{12}$, obtained from specific heat measurements, implies the existence of a crossover concentration, $x_{cr} \sim 0.25$, at which both the rate at which $-dH_{c2}/dT$ varies with $x$ and the character of the temperature variation of $H_{c2}$ change. The same crossover concentration was previously identified in $\Delta C/T_c$. The positive curvature (or change in slope) in $H_{c2}$ versus $T$ persists in some crystals with small values of $x$, possibly to $x_{cr}$.

Finally, our results suggest strong sample dependence of superconducting properties of PrOs$_4$Sb$_{12}$. The ac-susceptibility seems to indicate the presence of inhomogeneities that are difficult to account for considering sharp transitions in the isostructural and chemically almost identical LaOs$_4$Sb$_{12}$. On the other hand, assuming homogeneous samples, the upper superconducting transition in sample No. 2 might be related to the phase transition with an order higher than 2.22 In such a case, strong sample dependence and ac-susceptibility mimicking a two-step transition can be expected. A higher order phase transition (such as the third order) would be very susceptible to impurities and imperfections leading to the $2^{nd}$ order transition in sufficiently imperfect crystals. The expulsion of magnetic flux would be weak upon

FIG. 4: $C/T$ versus $T$, near $T_c$, for $x = 0.1$ in small magnetic fields. The inset shows $H_{c2}$ versus $T$.

FIG. 5: $C/T$ versus $T$, near $T_c$, for $x = 0.3$ in magnetic fields. The inset shows $H_{c2}$ versus $T$.

FIG. 6: $-dH_{c2}/dT$ versus $x$. Open circles for $x < 0.1$ correspond to $-dH_{c2}/dT$ measured directly at $T_c$. See text for details.

FIG. 7: $\sqrt(-dH_{c2}/dT/T_c)$ versus $x$. Open circles denote $\sqrt(-dH_{c2}/dT/T_c)$ at $T_c$ (see text for details). The dashed lines are guides to the eye. The inset represents the discontinuity of $C/T$ at $T_c$ and $T_e$ (identified by the maxima in $C/T$) versus $x$. 

lowering temperature from $T_{c1}$ to $T_{c2}$ followed by a much more rapid expulsion below the lower superconducting transition. Also, this scenario could account for different response to magnetic field of two crystals exhibiting different order phase transitions at $T_{c1}$. However, the possibility of the 3rd order phase transition remains speculative since few materials exhibiting phase transitions with an order higher than 2 were reported.\textsuperscript{22}

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