Comparative photoemission studies on the superconducting gap of the filled skutterudite superconductors $LaPt_4Ge_{12}$ and $PrPt_4Ge_{12}$

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We performed a comparative study of the superconducting gap in the new filled skutterudite superconductors $LaPt_4Ge_{12}$ and $PrPt_4Ge_{12}$ using high-resolution photoemission spectroscopy. We succeeded in observing spectral changes across T_c that reflect the opening of the superconducting gap in both compounds and also in observing a noticeable difference in their respective superconducting spectral shapes near the Fermi level, pointing toward a more complex superconducting gap structure in $PrPt_4Ge_{12}$. In addition, we found that the two-gap model is more suitable for describing the superconducting-state spectrum of $PrPt_4Ge_{12}$ than the single-isotropic-gap and single-anisotropic-gap models, which suggests an explanation that multiband effects may possibly induce the anomalous superconducting properties of $PrPt_4Ge_{12}$.

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I. INTRODUCTION

Filled skutterudite compounds MT_4X_{12} (where M is a rare-earth or alkaline-earth metals, T is a transition metal, and X is usually a pnicogen) have a variety of physical properties that depend on the particular combination of M, T, and X.[1,2] Within this unique structure, the hybridization between the $4f$ electrons of an M atom and conduction electrons are tuned by certain atomic configurations. This leads to remarkable physical properties even in Pr-based filled skutterudites in which the Pr $4f$ electrons are considered to be more localized than those of Ce. In particular, for $PrOs_4Sb_{12}$, heavy fermion and exotic superconducting behavior has been observed[3] and the quadrupole degrees of freedom have been suggested to play a role,[2] in contrast to the conventional superconductivity of $LaOs_4Sb_{12}$.

$PrPt_4Ge_{12}$ is a newly synthesized filled skutterudite compound with a Pt-Ge framework[4–7] (Fig. 1) with a superconducting transition temperature T_c of 7.9 K,[5] which is unexpectedly high among similar Pr-based superconductors. The specific heat jump at T_c and the superconducting gap values of $PrPt_4Ge_{12}$ are larger than those of its non-$4f$ counterpart $LaPt_4Ge_{12}$ ($T_c = 8.3$ K); the values are close to the mean-field Bardeen-Cooper-Schrieffer (BCS) values, which indicates strong-coupling superconductivity of $PrPt_4Ge_{12}$. Electronic specific heat and muon-spin rotation (μSR) measurements down to very low temperatures suggested the presence of pointlike nodes in the superconducting energy gap.[8] Previous comparative zero- and longitudinal-field μSR experiments on $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$ reported spontaneous magnetization with a temperature variation resembling that of the superfluid density below T_c only for $PrPt_4Ge_{12}$, implying time-reversal symmetry breaking in $PrPt_4Ge_{12}$.[9] While these studies have suggested unconventional superconductivity of $PrPt_4Ge_{12}$, very recent studies of the ^{73}Ge-nuclear quadrupole resonance (NQR) showed a coherence peak just below T_c, indicating that the superconductivity is accounted for in terms of the conventional BCS regime.[10] In order to understand the anomalous superconducting properties of $PrPt_4Ge_{12}$, it is crucial to directly observe the superconducting gap.

In this study, we have performed high-resolution photoemission spectroscopy (PES) of $PrPt_4Ge_{12}$, as well as $LaPt_4Ge_{12}$, in order to elucidate the superconducting gap structures. We identified a difference in the superconducting spectral shape near the Fermi level (E_F) between $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$, which provides spectroscopic evidence for a difference in the superconducting gap structure. While spectral analysis using an isotropic Dynes function could reproduce the superconducting-state spectrum of $LaPt_4Ge_{12}$ with a gap size that is consistent with the thermodynamic measurements, it could not reproduce that of $PrPt_4Ge_{12}$, suggesting $PrPt_4Ge_{12}$ has a more complex gap structure. The superconducting-state spectrum of $PrPt_4Ge_{12}$ could not be fitted well with an anisotropic Dynes function used for explaining μSR and NMR results; however, a weighted sum of two Dynes functions could describe the experimental data, suggesting the need to consider Fermi surface sheet dependence of the superconducting gap to understand the anomalous superconducting properties.

II. EXPERIMENT

Polycrystalline samples of $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$ were prepared by use of a conventional arc-melting method using a nonconsumable tungsten electrode in an atmosphere of high-purity argon (99.999%). The casted samples were repeatedly melted on a water-cooled copper hearth to ensure homogeneity. No contamination from the electrode or copper hearth was detected, and the weight loss during the process was negligible. The arc-melted samples were then annealed at 1073 K for 24 h under a vacuum pressure of 1×10^{-5} Pa. X-ray diffraction analysis confirmed that the samples consisted of either a single phase of $PrPt_4Ge_{12}$ or $LaPt_4Ge_{12}$. The T_c temperatures determined from magnetic susceptibility measurements were 7.9 and 8.2 K for $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$, respectively, with

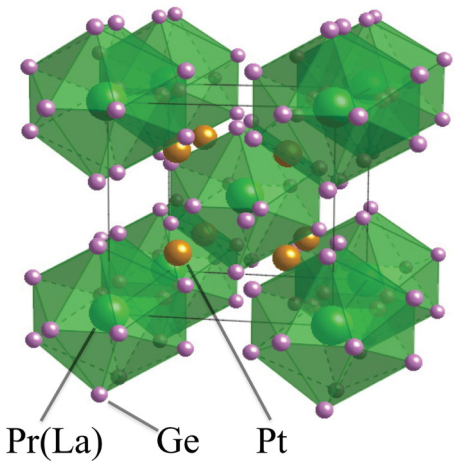

FIG. 1. (Color online) Crystal structure of Pr(La)Pt_4Ge_{12}.

the transition widths of 0.31 and 0.38 K that correspond to magnetization drops of between 10% and 90%.

High-resolution PES measurements of $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$ were performed using a newly constructed spectrometer with a hemispherical electronic energy analyzer (SCIENTA R4000) and a rare-gas discharge lamp (SPECS UVSL). The total energy resolution, using a Xe I (8.44 eV) resonance line, was set to 1.2 meV. The base pressure of the measurement chamber was below 7.5×10^{-9} Pa. Samples were fractured *in situ* to obtain clean surfaces. The E_F energies within an accuracy of ± 0.1 meV refer to those of gold films evaporated onto the sample surfaces. The sample temperature was measured with a Pt resistive sensor mounted close to the sample during the measurements. However, in order to accurately determine the temperatures in the PES measurement region, we derived the readings from the obtained analyses of temperature-dependent PES spectra analysis of the gold films using the known energy resolution of the spectrometer and the temperature-dependent Fermi-Dirac distribution function as a fitting parameter. We estimated the error in the temperature to be ± 1 K at most. Several PES measurements of a single sample taken at different machine times confirmed that the results are reproducible.

It should be noted that the 8.44-eV photon energy corresponds to a photoelectron escape depth of $\sim$10 nm for the states near E_F.[11] This is much longer than the escape depth of photoelectrons ($\sim$3 nm) used to obtain the dominant electronic structure of f electron materials in bulk[12] and is consistent with our observation of identical spectral shapes from several samples. Average grain sizes of the polycrystalline samples for both compounds determined from scanning electron microscope (SEM) measurements were $\sim 10\ \mu$m, which is larger than the coherence length of each compound [$LaPt_4Ge_{12}$, 13.2 nm (Ref. 13); $PrPt_4Ge_{12}$, 18.2 nm (Ref. 8)].

III. RESULTS AND DISCUSSION

Figures 2(a) and 2(b) show high-resolution PES spectra near the E_F of $LaPt_4Ge_{12}$ and $PrPt_4Ge_{12}$, respectively, measured above and below T_c. For comparison, the normal-state spectrum of $LaPt_4Ge_{12}$ is also shown in Fig. 2(b). The normal-state spectra of the two compounds are nearly identical, suggesting

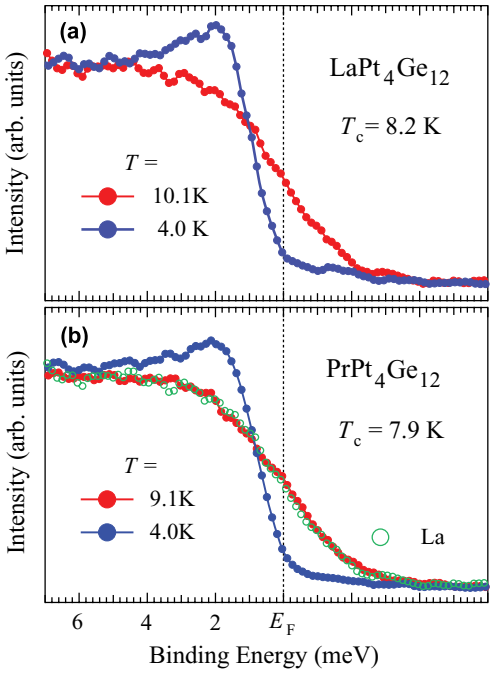

FIG. 2. (Color online) High-resolution photoemission spectra near E_F of (a) $LaPt_4Ge_{12}$ and (b) $PrPt_4Ge_{12}$ measured at normal (solid red circles) and superconducting (open blue circles) states. Normalized intensities were obtained by setting the average intensity of the normal-state spectra from 6 to 7 meV for each compound to 1. Normalization between normal- and superconducting-state spectra was performed with the integrated spectral intensity from a 10-meV binding energy to 10 meV above E_F. For comparison, the normal-state spectrum of $LaPt_4Ge_{12}$ is superimposed in Fig. 2(b) (open green circles).

negligible correlation effects in $PrPt_4Ge_{12}$, and a band picture may be a good starting point for describing the electronic structure. According to band structure calculations, the states near the E_F of $LaPt_4Ge_{12}$ and $PrPt_4Ge_{12}$ have a dominant Ge $4p$ character hybridized with Pt $5d$.[5] Resonant PES studies of $PrPt_4Ge_{12}$ do not show significant enhancement of the spectral intensity near E_F across the Pr $3d$-to-$4f$ threshold,[14] in contrast with similar studies of $PrFe_4P_{12}$, which is considered to be a heavy fermion material with significant hybridization of the conduction band and $4f$ electrons.[15] The spectrum at 4K (the superconducting state) of each compound shows a reduced intensity near the E_F region and a peak at 2 meV. These are typical superconducting-state spectrum features and reflect the opening of a superconducting gap. These results show the first direct observation of the superconducting gap in $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$. To elucidate the difference between $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$, we compared superconducting spectra normalized using the normal-state spectra intensity, as shown in Fig. 3. In the inset, we find that the intensity in the vicinity of the peak appears to be higher in the $PrPt_4Ge_{12}$ spectrum than in the $LaPt_4Ge_{12}$ spectrum. Near E_F, the spectral intensity of the leading edge region of $PrPt_4Ge_{12}$ is higher than that of $LaPt_4Ge_{12}$. As noted under Experiment, the T_c widths are nearly the same, indicating that extrinsic variation of T_c is not responsible for the spectral difference. In addition, we observed an identical spectral difference for

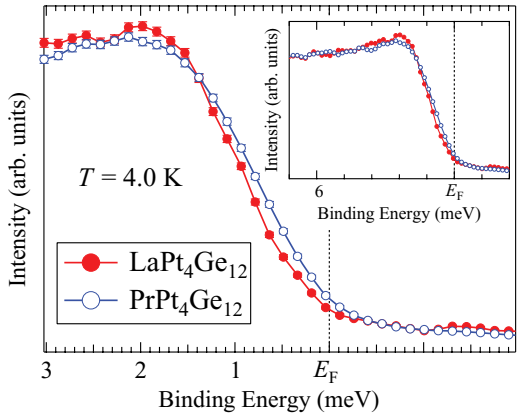

FIG. 3. (Color online) A comparison of the superconducting spectra of $LaPt_4Ge_{12}$ (solid red circles) and $PrPt_4Ge_{12}$ (open blue circles) normalized by the same procedures used for Fig. 2. Error bars show the statistical accuracy obtained from the square root of the photoelectron count. Note that the spectral intensity near the E_F region of $PrPt_4Ge_{12}$ is larger than that of $LaPt_4Ge_{12}$, indicating a difference in the superconducting gap structure. Inset shows the same spectra over a wider energy range.

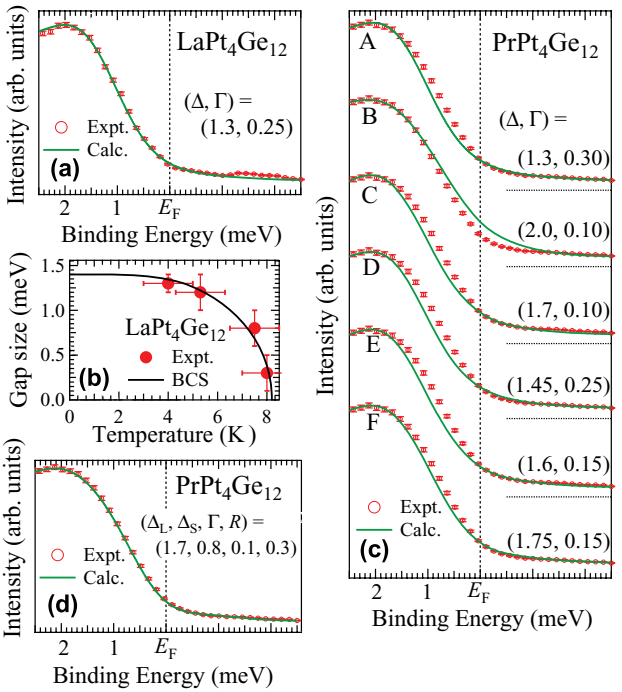

FIG. 4. (Color online) (a) Result of the fitting analysis of $LaPt_4Ge_{12}$ (solid green curve) compared with the experimental data at 4.0 K (open red circles). (b) Temperature dependence of the superconducting gap of $LaPt_4Ge_{12}$. Solid red circles indicate experimentally obtained temperature dependence of the superconducting gap, and the solid curve indicates the BCS relation[12] under conditions of $\Delta_0 = 1.4$ meV and $T_c = 8.3$ K. (c) Results of the fitting analysis of $PrPt_4Ge_{12}$ (solid green curve) using an isotropic Dynes function (A) and anisotropic Dynes functions (B, $\Delta_0|\sin\phi\sin\theta|$; C, $\Delta_0|\sin\theta|$; D, $\Delta_0(1-\sin^4\phi\sin^4\theta)$; E, $\Delta_0[1-(\sin^4\phi+\cos^4\phi)\sin^4\theta)$, F, $\Delta_0[1-3\cos^2\theta\sin^2\theta-3\sin^2\theta\cos^2\theta\sin^4\theta]^{1/2}$) compared with the experimental data at 4.0 K (open red circles). (d) Result of the fitting analysis of $PrPt_4Ge_{12}$ (solid green curve) using a weighted sum of two isotropic Dynes functions, compared with the experimental data at 4.0 K (open red circles). All the experimental spectra are the same those shown in Fig. 3. Error bars show the statistical accuracy obtained from the square root of the photoelectron count. The dotted horizontal lines in (c) are the zero lines for each data set.

several samples in different experimental runs. These facts indicate that the observed spectral difference reflects the difference in the superconducting electronic structures of the two compounds. The observation that the $PrPt_4Ge_{12}$ spectrum has a higher intensity near E_F compared to that of $LaPt_4Ge_{12}$ implies a more complicated gap structure in $PrPt_4Ge_{12}$ than in $LaPt_4Ge_{12}$. To discuss the superconducting gap structure of $PrPt_4Ge_{12}$ and $LaPt_4Ge_{12}$, we performed a numerical analysis of the experimental spectra using the Dynes function.

The Dynes function is a modified BCS function that introduces phenomenological broadening effects into the BCS function and is known to describe a superconducting gap with isotropic s-wave symmetry very well.[16] The Dynes function is defined by $D(E,\Delta,\Gamma) = \mathrm{Re}\{(E-i\Gamma)/[(E-i\Gamma)^2-\Delta^2]^{1/2}\}$, where Δ is the superconducting gap size and Γ is the phenomenological broadening parameter. This parameter was originally introduced to represent finite-lifetime effects of the quasiparticles at the gap edge, but it is often used as a fitting parameter and, thus, includes other factors such as the superconducting gap variations and anisotropy. To fit a spectrum, the Dynes function is multiplied by a Fermi-Dirac function of the measured temperature and convolved with a Gaussian function corresponding to the experimental energy resolution. The fitting parameters are chosen to reproduce experimental spectra within a binding-energy region from the top of the quasiparticle peak to 5 meV above E_F. We found that the superconducting-state spectrum of $LaPt_4Ge_{12}$, shown in Fig. 4(a), is well reproduced using an isotropic Dynes function with a Δ and Γ of 1.3 meV and 0.25 meV, respectively. The magnitude of the superconducting gap as a function of temperature $\Delta(T)$ is plotted in Fig. 4(b). The gap size changes systematically with temperature, which is consistent with the BCS theory.[17] We estimate $\Delta(0)$ to be 1.4 meV by extrapolating the observed gap magnitude and assuming a temperature dependent gap. This corresponds to a reduced gap value $2\Delta(0)/k_BT_c$ of 3.9 ± 0.5, which is comparable to the mean-field BCS value $[2\Delta(0)/k_BT_c = 3.54]$. The reduced gap value also agrees well with the values determined from the specific heat measurements $[2\Delta(0)/k_BT_c = 3.88]$,[5] indicating that PES is a reliable experimental tool for studying the superconducting gap in this new filled skutterudite superconductor. The Γ value for the $LaPt_4Ge_{12}$ fit is larger than those for elemental metals[18] but comparable to that for $LaRu_4P_{12}$ ($T_c = 7.2$ K), where the value was discussed in relation to possible superconducting gap anisotropy.[19] Nonetheless, the nearly isotropic gap in the present study for $LaPt_4Ge_{12}$ agrees well with the nuclear magnetic resonance (NMR)[20] and NQR studies and also suggests that the absence of the Hebel-Slichter peak in the $LaPt_4Ge_{12}$ spectrum[10,20] is not due to the spatial distribution or variation in momentum space of superconducting gap.

For $PrPt_4Ge_{12}$, the Dynes function analysis was performed with an isotropic gap (model A) and with allowed anisotropic gaps (models B–F)[21] used to explain μSR and NQR data,[8,10] as shown in Fig. 4(c). We found that the isotropic function, model

A, does not reproduce well the experimental data of the peak and the leading edge region simultaneously. This is in sharp contrast to the reasonable fitting seen for $LaPt_4Ge_{12}$ but is consistent with the differences seen in the raw data (Fig. 2). Thus, the present study provides spectroscopic evidence for different superconducting gap structures in $LaPt_4Ge_{12}$ and $PrPt_4Ge_{12}$; there is a larger deviation from an isotropic superconducting gap in $PrPt_4Ge_{12}$ than in $LaPt_4Ge_{12}$. This is consistent with the reported superconducting property differences of the two compounds that emphasized the anomalous properties of $PrPt_4Ge_{12}$ (thermodynamic and magnetization measurements[8] and comparative μSR studies[9]), which allows us to conclude that the more complex gap structure in $PrPt_4Ge_{12}$ induces the anomalous superconducting properties.

Fitting results using models B–F and the experimental spectra at 4 K are also shown in Fig. 4(c). We found that the superconducting-state spectrum of $PrPt_4Ge_{12}$ is not well described using the anisotropic gap functions. Note that using a single Dynes function analysis can be considered to correspond to one isotropic Fermi surface sheet as a normal-state electronic structure. To consider effects beyond such this simple case, it may be valuable to assume multiple Fermi surface sheets that may assist the superconducting gap to have different magnitudes. As a first step, we used the weighted sum of two isotropic Dynes functions for large $D_L(E,\Delta_L,\Gamma)$ and small $D_S(E,\Delta_S,\Gamma)$ gaps, expressed as $D_{L+S} = RD_L(E,\Delta_L,\Gamma) + (1-R)D_S(E,\Delta_S,\Gamma)$, where R is the amplitude ratio of the larger gap to the total sum. We found that this model reproduces the experimental data very well with $(\Delta_L,\Delta_S,\Gamma,R) = (1.7, 0.8, 0.10, 0.3)$ in units of meV [Fig. 4(d)]. For simplicity, we adopt the same Γ value for both gaps. The use of two isotropic Dynes functions, however, does not necessarily mean two fully pronounced s-wave gaps, as we have found that in the present study the two Dynes functions with anisotropic gaps can also explain the data very well. If one considers the fact that the Dynes function does not take into account the underling density of states, strong correlations, or a number of other possible complicating factors, better reproducibility using two Dynes functions for $PrPt_4Ge_{12}$ is, at most, suggestive of an explanation. However, band structure calculations have predicted multiband crossing of the E_F of RPt_4Ge_{12} (R = Sr, Ba, and La)[22] and multiple Fermi surface sheets of $ThPt_4Ge_{12}$.[23] Existence of multiband crossings or multiple Fermi surface sheets are a condition for multigap superconductivity. An upturn of the upper critical field as a function of temperature for the higher T_c region, one of the characteristics of two-gap superconductivity,[24,25] is indeed observed in $PrPt_4Ge_{12}$. Improvement of energy resolution may help to distinguish full details of superconducting gap structure.

In $PrOs_4Sb_{12}$, multiband superconductivity has been proposed to explain several experiments.[26–30] The advantage of this approach is that it is able to explain previous reports of seemingly controversial superconducting gap structures by considering nodal and full gaps open on different Fermi surface sheets. Multiband superconductivity may also assist in understanding the previously observed superconducting properties of $PrPt_4Ge_{12}$. One interesting scenario is a band with a nearly isotropic gap that contributes to the presence of the Hebel-Slichter peak in NQR studies, while another band with large gap anisotropy is responsible for the line-node-like low-temperature phenomena. If one of the two bands is related to the presence of spontaneous magnetization in $PrPt_4Ge_{12}$ and vanishes with the substitution of La, it may explain the smooth variation in the spontaneous magnetization as a function of La concentration in $Pr_xLa_{1-x}Pt_4Ge_{12}$ observed in μSR measurements.[9] This study has suggested one possible explanation based our experimental data; to confirm the multiband superconductivity in $PrPt_4Ge_{12}$, further experimental studies are necessary.

IV. CONCLUSION

We have investigated the superconducting gap of $PrPt_4Ge_{12}$ in comparison with $LaPt_4Ge_{12}$ using high-resolution PES. In contrast to $LaPt_4Ge_{12}$, where the superconducting-state spectrum was well reproduced using an isotropic Dynes function, the $PrPt_4Ge_{12}$ spectrum was not well reproduced by either a single isotropic or anisotropic gap model. The weighted sum of two Dynes functions (whether isotropic or not) produced the best fit for the experimental spectrum. The present results have established that there is a difference in the superconducting gap structures of the two superconductors (a more complex superconducting gap in $PrPt_4Ge_{12}$), and a possible relationship between the multiband effects and the anomalous superconducting properties of $PrPt_4Ge_{12}$ was discussed. We hope the present results motivate further experimental and theoretical studies to understand the origin of the difference in the gap structures, which may further our understanding of Pr-based filled skutterudite superconductors.

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