Quasiparticle Excitations and Evidence for Superconducting Double Transitions in Monocrystalline $U_{0.97}^{\text{Th}}_{0.03}^{\text{Be}}_{13}$

Yusei Shimizu, Shunichiro Kitaoka, Shota Nakamura, and Toshiro Sakakibara
Institute for Solid State Physics, University of Tokyo, Kashiwa, Chiba 277-8581, Japan.

Dai Aoki, Yoshiya Homma, and Ai Nakamura
Institute for Materials Research (IMR), Tohoku University, Oarai, Ibaraki 311-1313, Japan.

Kazushige Machida
Department of Physics, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan.
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Superconducting (SC) gap symmetry and magnetic response of cubic $U_{0.97}^{\text{Th}}_{0.03}^{\text{Be}}_{13}$ are studied by means of high-precision heat-capacity and dc magnetization measurements using a single crystal, in order to address the long-standing question of its second phase transition at $T_{c2}$ in the SC state below $T_{c1}$. The absence (presence) of an anomaly at $T_{c2}$ in the field-cooling (zero-field-cooling) magnetization indicates that this transition is between two different SC states. There is a qualitative difference in the field variation of the transition temperatures; $T_{c2}(H)$ is isotropic whereas $T_{c1}(H)$ exhibits a weak anisotropy between [001] and [111] directions. In the low temperature phase below $T_{c2}(H)$, the angle-resolved heat-capacity $C(T, H, \phi)$ reveals that the gap is fully opened over the Fermi surface, narrowing down the possible gap symmetry.

The nature of superconductivity in heavy-fermion compounds is of primary importance because an unconventional pairing mechanism is generally expected to occur due to strong electron correlation between heavy quasiparticles. The discovery of heavy-fermion superconductivity in $U$Be$_{13}$ [1] triggered exploration of unconventional pairing mechanism in 5$f$ actinide compounds, and subsequently two uranium compounds, UPt$_3$ [2] and $U$Ru$_2$Si$_2$ [3–4], were found to show superconductivity. These $U$-based heavy-fermion superconductors have attracted considerable interest because of their unusual superconducting (SC) and normal-state properties. Among these, superconductivity in $U$Be$_{13}$ is highly enigmatic: it emerges from a strongly non-Fermi-liquid state with a large resistivity ($\rho \sim 150 \mu\Omega$cm). Also unusual is the temperature variation of the upper critical field $H_{c2}$: an enormous initial slope $-dH_{c2}/dT$$_{c2}$ $\sim 42$ T/K and an apparent absence of a Pauli paramagnetic limiting at low temperatures [5]. Extensive studies have been made to elucidate the SC gap symmetry [6,7], with an expectation of an odd-parity pairing in this compound [8–11]. Recently, it has been found quite unexpectedly that nodal quasiparticle excitations in $U$Be$_{13}$ are absent as revealed by low-$T$ angle-resolved heat-capacity measurements for a single crystalline sample [12].

A long-standing mystery regarding $U$Be$_{13}$ is the occurrence of a second phase transition in the SC state when a small amount of Th is substituted for $U$ [Fig. 1(a)] [13,14]. It has been reported that there exist four phases (A, B, C, and D) in its SC state, according to the previous $\mu$SR [16] and thermal-expansion [24] experiments using polycrystalline samples. The SC transition temperature $T_{c}$ is non-monotonic as a function of the Th concentration $x$ in $U_{1-x}^{\text{Th}}_{x}^{\text{Be}}_{13}$, and exhibits a sharp minimum near $x = 0.02$. Further doping of Th results in an increase of the bulk SC transition temperature ($T_{c1}$), reaching a local maximum at $x \sim 0.03$ [13]. Below $T_{c1}$, another phase transition accompanied by a large heat-capacity jump occurs at $T_{c2}$ in a narrow range of $0.019 < x < 0.045$ [14,16]. Interestingly, only for this $x$ region, weak magnetic correlations have been observed in zero-field $\mu$SR measurements [16]. The previous thermal-expansion study [24] claimed that the low-temperature (“$T_{c}$”) anomaly appearing below $T_{c}$ for $0 \leq x < 0.02$, which can be connected to the “$B^*$” anomaly” observed in pure $U$Be$_{13}$ [24,26,27], is a precursor of the transition at $T_{c2}$. Up to present, the true nature of the transition at $T_{c2}$ remains controversial [17,18]. Two different scenarios have been discussed so far on the $T_{c2}$ transition: (i) an additional SC transition that breaks time-reversal symmetry [19], and (ii) the occurrence of an antiferromagnetic ordering that coexists with the SC state [20,21]. Indeed, although it has been reported that the NMR spin-relaxation rate [6], heat capacity [22], and muon Knight shift [23], show unusual temperature dependence in the SC state, little is known concerning the gap structure in $U_{1-x}^{\text{Th}}_{x}^{\text{Be}}_{13}$ due to the lack of information about the anisotropy of its quasiparticle excitations in magnetic fields.

In order to resolve the controversy regarding the second transition at $T_{c2}$, and to uncover its gap symmetry, in this Letter we report the results of high-precision heat-capacity and dc magnetization measurements on $U_{0.97}^{\text{Th}}_{0.03}^{\text{Be}}_{13}$. Single-crystalline $U_{0.97}^{\text{Th}}_{0.03}^{\text{Be}}_{13}$ samples were obtained using a tetra-arc furnace; the ingot was remelted several times and then quenched. By this procedure, we have succeeded in obtaining small monocrystalline samples with no additional heat treatment as confirmed by sharp X-ray Laue spots in Fig. 1(b). Heat capacity ($C$) was measured at low temperatures down to 60 mK by means of a standard quasi-adiabatic heat-pulse method in a $^3$He-$^4$He dilution refrigerator. Field-orientation dependences $C(H, \phi)$ were obtained under rotating magnetic fields in the (110) crystal plane that includes the [001], [111], and [110] axes, using a $5 \times 3 \times 3$T vector magnet. We define the angle $\phi$ measured from the [001] direction. Dc magnetiza-
tion measurements were performed along the [110] axis down to $T \sim 0.28$ K for the same single crystal using a capacitive Faraday magnetometer [28] installed in a $^3$He refrigerator. A magnetic-field gradient of 9 T/m was applied to the sample, independently of the central field at the sample position.

Figure 1(c) shows $C(T)/T$ curves measured at zero and various fields up to 5 T applied along [001] and [111] axes. At zero field, two prominent jumps occur at $T_{c1} \sim 0.56$ K and $T_{c2} \sim 0.41$ K, where the transition temperatures [red circles in Fig. 1(a)] are determined by transforming the broadened transitions into idealized sharp ones by an equal-areas construction. The results are in agreement with the previous reports [13, 14]. With increasing field, both transitions shift to lower temperature, getting closer to each other [13, 25]. Above 3.5 T, the two transitions become so close to each other and are difficult to resolve separately. There is a notable feature in the anisotropy of $C(T)$ in magnetic fields. At low fields below $\sim 1.75$ T, the shifts of the two transition temperatures are almost isotropic. At higher fields above 2.5 T, however, the $T_{c1}(H)$ becomes slightly anisotropic, $T_{c1}(H||[001]) > T_{c1}(H||[111])$, while $T_{c2}(H)$ remains isotropic. In general, an anisotropy of $T_{c1}(H)$ and $H_{c2}$ results from those of SC gap function and/or Fermi velocity. If the double transitions come from two inhomogeneous SC states with the same gap symmetry, they should show the same anisotropic (or isotropic) field response. Our experimental results exclude such an extrinsic possibility. Thus the difference between field anisotropy in $T_{c1}(H)$ and $T_{c2}(H)$ is an essential effect which strongly suggests that the order parameters of these two phases have qualitatively different field-orientation dependencies.

A key question, then, is whether the second transition at $T_{c2}$ is a SC transition into a different gap symmetry. To address this question, we performed precise dc magnetization [$M(T)$] measurements across the double transitions. Figure 2 shows the temperature dependence of $M(T)$ measured at 1.5 T together with the $C(T)/T$ data for the same field on the same sample. FC and ZFC denote the data taken in the field-cooling and zero-field-cooling protocols, respectively. The FC-ZFC branching occurs below $\sim 0.5$ K close to $T_{c1}$ at this field, indicating the appearance of bulk superconductivity. We find a small but distinct kink in the ZFC data near $T_{c2}$, while no such anomaly can be seen in the FC curve. This fact implies a substantial change in the vortex pinning strength at this temperature, consistent with the previous vortex creep measurements [29, 30]. Regarding the possible origin of the enhanced vortex creep in the low-temperature regime, we find no signatures that can be ascribed to a magnetic transition in the FC curve near $T_{c2}$. Our magnetization data, therefore, strongly suggest that the transition at $T_{c2}$ is of a kind such that the SC order parameter changes. Indeed, it has been argued that such an enhancement of the vortex pinning occurs in a SC state with broken time reversal symmetry [30]. This conclusion is also consistent with the previous neutron scattering measurements [31] which show no evidence for magnetic ordering in $U_{0.965}$Th$_{0.035}$Be$_{13}$ down to 0.15 K.

Next we examine the magnetic-field dependence of the heat capacity and its anisotropy in more detail, whose behavior in low fields reflects quasiparticle excitations in the SC state and provides a hint for the gap symmetry [32, 34]. Figure 3(a) shows $C(H)/T$ measured at $T = 0.12, 0.18, 0.24, 0.30, 0.36,$
and 0.40 K for the cubic [001] and [111] directions, and the inset shows the enlarged $C(H)/T$ plot obtained at 0.08 K. Note that $C(H)$ below 1 T is quite linear to $H$ at the lowest temperature of 0.08 K. This behavior is in striking contrast with a convex upward $H$ dependence expected for nodal superconductors. Moreover, there is no anisotropy in $C(H)/T$ between $H \parallel [001]$ and [111] in low fields below ~2 T. The absence of the anisotropy is further demonstrated by angle-resolved $C(\phi)/T$ in Fig. 4(a), obtained in a field of 1 T rotated in the (110) crystal plane at $T = 0.08$, and 0.42 K, together with the result measured in the normal state at 0.60 K. The absence of any angular dependence in $C(\phi)/T$ in a low-$T$ low-$H$ region again excludes the possibility of a nodal-gap structure in which a characteristic angular oscillation should be expected in $C(\phi)/T$. The present $C(H, \phi)$ data thus indicate that nodal quasiparticles are absent in $U_{0.97}Th_{0.03}Be_{13}$, similarly to the behaviors observed in pure $UBe_{13}$.

At higher fields, double-step-like anomalies are observed in $C(H)/T$ at 0.42, 0.40, and 0.36 K [Fig. 3(a)]. Here the double transitions can be cleary defined by the differential data, $d[C(H)/T]/dH$, as shown in Figs. 3(b) and 3(c). The lower-field step occurs when the boundary $T_{c2}(H)$ is crossed, while the higher-field one corresponds to the transition at $T_{c1}(H)$, i.e., the upper critical field $H_{c2}(T) \equiv H_A$. Note that the position of the lower-field anomaly ($H_A^0$) is fully isotropic, whereas the higher-field one ($H_B^0$) shows an appreciable anisotropy, indicating that $H_{c2}$ becomes anisotropic: $H_A^0 \parallel [001] > H_B^0 \parallel [111].$ The anisotropy of $H_A^0$ becomes larger at lower temperatures. With decreasing $T$, both of the transition fields shift to higher fields, getting close to each other, and are difficult to discriminate below ~0.24 K [Fig. 3(a)]. These features of the transition fields are fully consistent with those observed for $T_{c1}(H)$ and $T_{c2}(H)$ shown in Fig. 1(c). Note that the isotropic behaviors in $C(H)/T$ as well as $T_{c2}(H)$ (Fig. 3) contrast starkly with the anisotropic behavior of $B^*$ anomaly found in pure $UBe_{13}$, suggesting that these phenomena may result from different origins. Figure 4(b) shows the $H$-$T$ phase diagram of $U_{0.97}Th_{0.03}Be_{13}$ determined from the present $C(T, H)$ measurements, where the two SC phases are denoted as A and B phases. The overall features of the phase diagram are essentially the same with those obtained previously.

The present experiment thus provides strong evidence that $U_{0.97}Th_{0.03}Be_{13}$ exhibits double SC transitions with two different SC order parameters. Let us discuss possible SC gap symmetries in this system. A key experimental fact is that the SC gap is fully open over the Fermi surface in both the B and C phases, as suggested by the present and previous studies, respectively. This would imply either (i) the SC gap function itself to be nodeless, or (ii) the SC gap function to have nodes only in the directions in which the Fermi surface is missing. Regarding the latter, band calculations tell us that the Fermi surface is missing along the (111) direction, except...
for a tiny electron band [38, 39]. Given the fact that spontaneous magnetism is observed from zero-field μSR only below \( T_c \) in addition, it would be natural to assume that the B phase is a time-reversal-symmetry broken SC state. Under these constraints, two plausible scenarios can be proposed to explain the multiple SC phases in \( U_{1-x}Th_xBe_{13} \). One is to employ a degenerate order parameter belonging to higher dimensional representations of the \( O_h \) symmetry (degenerate scenario). The other is to assume two order parameters belonging to different representations of the \( O_h \) group, nearly degenerate to each other (accidental scenario) [19].

**Degenerate scenario:** The group theoretic classification of the gap functions under the cubic symmetry \( O_h \) has been given by several authors [12, 40, 42]. Among them, the two-dimensional odd-parity \( E_u \) state is a promising candidate for the order parameter which naturally explains the existing experimental data of both pure and Th-doped \( \text{UBe}_{13} \). The possibility of the odd-parity state has also been suggested from the \( \mu\text{SR} \) Knight shifts experiments [23]. As for the odd-parity \( E_u \) state, we have two basis functions, \( l_1(k) = \sqrt{3}(\hat{x}k_x - \hat{y}k_y) \) and \( l_2(k) = 2\hat{z}k_z - \hat{x}k_x - \hat{y}k_y \), and their combined state, \( d(k) = l_1 + l_2 = \hat{x}k_x + \hat{y}k_y + e^3 \hat{z}k_z \) with \( e = e^1 \frac{3}{2} \). The non-unitary state \( d(k) = l_1 + i l_2 \) has point nodes only along the \( \langle 111 \rangle \) direction, therefore, the nodal quasiparticle excitations can be missing considering the calculated Fermi surface [38, 39]. The condition of the occurrence of each two-dimensional SC state can be examined using the Ginzburg-Landau free energy density, \( F = \alpha(T)(|l_1|^2 + |l_2|^2) + \beta_1(|l_1|^2 + |l_2|^2)^2 + \beta_2(l_1^* l_2^* + l_1 l_2)^2 \) with \( \alpha(T) = \alpha_0(T_c - T) \), where \( \beta_1 > 0 \) is required for the stability. If \( \beta_2 > 0 \), the non-unitary state with the broken time-reversal symmetry becomes stable in lower \( T \) as a ground state (the B phase). With increasing temperature the degeneracy of the order parameters is lifted at \( T_c \) and one of them appears in the A phase \( (T_c < T < T_{c1}) \). Logiclly, the other one appears in the C phase by changing dopant \( x \). In pure \( \text{UBe}_{13} \) (the C phase), a nodeless gap function, i.e., \( l_2(k) = 2\hat{z}k_z - \hat{x}k_x - \hat{y}k_y \), which is a unitary state, is likely, explaining the absence of nodal quasiparticle excitations [12] without invoking the Fermi-surface topology.

**Accidental scenario:** We briefly discuss the possibility of the accidental scenario, starting with the simplest and most symmetric \( A_{1u} \), namely \( d_{A_{1u}}(k) = \hat{x}k_x + \hat{y}k_y + \hat{z}k_z \) with an isotropic full gap as the C phase for \( x = 0 \). From \( x = 0.019 \) to \( x = 0.045 \), we consider the combined state of 1D representations, the above \( p \)-wave \( A_{1u} \) and \( f \)-wave \( A_{2u} \) with \( d_{A_{2u}}(k) = \hat{x}k_x(k_x^2 - k_y^2) + \hat{y}k_y(k_y^2 - k_z^2) + \hat{z}k_z(k_z^2 - k_x^2) \). The combined state of \( A_{1u} \) and \( A_{2u} \), namely, non-unitary \( d(k) = d_{A_{1u}} + i d_{A_{2u}} \) is nodeless irrespective of the Fermi-surface topology, although \( d_{A_{2u}} \) alone has point nodes along \( \langle 100 \rangle \) and \( \langle 111 \rangle \) directions. Thus nodeless \( A_{1u} \) and the \( A_{2u} + i A_{2u} \) states can explain the absence of nodal quasiparticles in pure and Th-doped \( \text{UBe}_{13} \), respectively [44]. Similarly, the other order parameters belonging to different irreducible representations are possible, e.g., \( A_{1u} + i E_u \), the determination of the two order parameters is not easy due to the arbitrariness of their combinations.

Finally, it is worth discussing the topology of the \( H-T \) phase diagram. In Fig. 4(b), it may appear that the lines of \( T_c(H) \) and \( T_c^2(H) \) merge into a single 2nd-order transition line in a high-field region. Such a case is, however, not allowed in the thermodynamic argument of the multicritical point [46, 47]. Instead, a crossing of the two 2nd-order transition lines at a tetra-critical point is possible [46]. This argument imposes the existence of another 2nd-order transition below \( H_c2 \) for \( T \lesssim 0.25 \text{ K} \), but no evidence for such a transition line has been obtained so far in our measurements as well as in previous thermal expansion studies [24]. It might be natural to consider an anti-crossing of the two 2nd-order transition lines [48]. The crossing of \( T_c(H) \) and \( T_c^2(H) \) in \( 0.017 \text{ Th}_{0.09} \text{Be}_{13} \) will be examined further in future studies.

To conclude, low-energy quasiparticle excitations and magnetcic response of \( \text{U}_{0.97} \text{Th}_{0.03} \text{Be}_{13} \) were studied by means of heat-capacity and dc magnetization measurements. The magnetization results evidence that the second transition at \( T_c \) is between two different SC states. Strikingly, the present \( C(T, H, \phi) \) data strongly suggest that the SC gap is fully open over the Fermi surface in \( \text{U}_{0.97} \text{Th}_{0.03} \text{Be}_{13} \), excluding a number of gap functions possible in the cubic symmetry. Our new thermodynamic results entirely overturn a widely believed idea that nodal quasiparticle excitations occur in the odd-parity SC state with broken time-reversal-symmetry. The absence (presence) of anisotropy for \( T_c^2 \) \( (T_c^1) \) in fields clearly demonstrates that the gap symmetry in the B phase \( (T < T_{c2}) \) is distinguished from that of the A phase \( (T_{c2} < T < T_{c1}) \). Moreover, the isotropic behavior of the \( T_{c2}(H) \) in \( U_{1-x} \text{Th}_{x} \text{Be}_{13} \) contrasts starkly to the anisotropic field response of \( B^\ast \) anomaly found in pure \( \text{UBe}_{13} \). These findings lead to a new channel to deepen its true nature of the ground state of \( U_{1-x} \text{Th}_{x} \text{Be}_{13} \), clarifying the origin of the unusual transition inside the SC phase.

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* Electronic address: yuseishimizu@imr.tohoku.ac.jp. Present address: Institute for Materials Research, Tohoku University, Oarai, Ibaraki, 311-1313, Japan.

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