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Evidence for unconventional superconductivity in single crystals of the antiferromagnetic heavy-electron compound URu₂Si₂

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Invited paper.

Specific heat C(T) and upper critical field $H_{c2}(T)$ measurements have been performed on two single crystal specimens (denoted A and B) of the antiferromagnetic heavy-electron superconductor URu_2Si_2 . Specific heat measurements on both single crystals reveal two distinct jumps, indicative of two superconducting phases. This is reminiscent of the antiferromagnetic heavy-electron compound UPt_3 in which multiple superconducting transitions have been observed and attributed to coupled antiferromagnetic and multicomponent superconducting order parameters, although two superconducting phases associated with two different states of the crystal cannot be ruled out. The relative magnitudes of the two specific heat jumps observed in crystal B suggest that the two superconducting phases occupy nearly equal volume fractions of the crystal. At temperatures below the lower jump, the specific heat of both crystals can be described by $C_s(T) = \gamma_s(0)T + AT^3$ with $\gamma_s(0) \approx 0.5\gamma_n(0)$, where $\gamma_n(0)$ is the value of the normal-state electronic specific heat coefficient $\gamma_n(T)$, extrapolated to T = 0. The two critical temperatures inferred from the specific heat jumps in crystal B, measured in magnetic fields H between 0 and 15 kOe applied parallel to the c-axis, have a similar dependence on H. Resistive measurements of $H_{c2}(T)$ on specimens from crystal A between 0 and 60 kOe reveal a kink near 2 kOe for $H \parallel c$ and strong positive curvature in $H_{c2}(T)$ below \sim 3 kOe for $H \parallel a$.

1. Introduction

The heavy-electron superconductors $CeCu_2Si_2$ [1], UBe_{13} [2], UPt_3 [3], and URu_2Si_2 [4-6] have attracted considerable attention because of the possibility that they exhibit an unconventional type of superconductivity involving novel types of pairing and/or pairing mechanisms [7]. Especially interesting are the three U heavy-electron superconductors. Although UBe_{13} , UPt_3 , and URu_2Si_2 have different crystal structures (cubic, hexagonal, and tetragonal, respectively) and electron effective masses m^* ($\sim 300m_e$ [8], $\sim 200m_e$ [9], and $\sim 25m_e$ [5], respectively), common physical characteristics appear to be emerging which illustrate the intimate relationship between superconductivity and magnetism and may

provide a basis for developing a fundamental understanding of superconductivity in these remarkable materials. These characteristics include the occurrence of antiferromagnetic ordering with a small magnetic moment μ of a few hundredths of a Bohr magneton below a Néel temperature $T_{\rm N} \approx 10 T_{\rm c}$, where $T_{\rm c}$ is the superconducting critical temperature, and the destruction of superconductivity accompanied by the appearance of magnetic order with a much larger magnetic moment of several tenths of a Bohr magneton upon substituting another element. Evidence for antiferromagnetic ordering in UBe₁₃ $(T_c \approx 0.9 \text{ K})$ at $T_N \approx 8.8 \text{ K}$, based upon thermal expansion measurements, was recently reported [10], but remains to be confirmed by μSR or neutron scattering measurements. Neutron scat-

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tering experiments [11] on UPt₃ ($T_c \approx 0.5 \, \text{K}$) indicate antiferromagnetic ordering below $T_N \approx 5.5 \, \text{K}$ with $\mu \approx 0.02 \, \mu_\text{B}/\text{U}$ -atom, while various measurements [4–6, 12–14] on URu₂Si₂ ($T_c \approx 1.4 \, \text{K}$) reveal antiferromagnetic ordering with $T_N \approx 17.5 \, \text{K}$ and $\mu \approx 0.03 \, \mu_\text{B}/\text{U}$ -atom. Superconductivity is suppressed and replaced by localized moment antiferromagnetism upon substitution of Th for U or Pd for Pt [15–17] in UPt₃ and Rh for Ru [18] in URu₂Si₂, and local moment ferromagnetism with $\mu \approx (0.4-0.6) \, \mu_\text{B}/\text{U}$ -atom upon substitution of Tc or Re for Ru [19] in URu₂Si₂.

One of the most striking recent developments is the apparent occurrence of two or more distinct superconducting phases in $(U_{1-x}Th_x)Be_{13}$ as a function of Th composition x [20] as revealed by high-pressure experiments [21] and in UPt_3 in the H-T plane [22-24]. The case of UPt₃ has been studied extensively and is the subject of several theoretical investigations in which the multiple superconducting transitions have been attributed to coupled antiferromagnetic and multicomponent superconducting order parameters [25, 26]. In this paper, we report specific heat and upper critical field measurements on single crystal specimens of the antiferromagnetic heavy-electron superconductor URu₂Si₂ which show features in the specific heat C(T, H) and upper critical field $H_{c2}(T)$ curves which are indicative of unconventional superconductivity.

2. Experimental details

Two single crystals of URu_2Si_2 (denoted A and B) were prepared from arc-cast polycrystal-line rods by zone-melting in a high vacuum. Laue X-ray diffraction analysis was used to orient the crystallites from which specimens for the specific heat and upper critical field measurements were cleaved and cut with a diamond wheel saw. Specific heat measurements as a function of temperature in magnetic fields up to 15 kOe applied parallel to the c-axis were made in a 3 He calorimeter using the heat-pulse technique. Resistive upper critical field $H_{c2}(T)$ measurements for two orientations of the applied

magnetic field H, $H\|c$ and $H\|a$, were made on two rectangular parallelepiped-shaped specimens from sample A in a ${}^{3}\text{He}{}^{-4}\text{He}$ dilution refrigerator in fields up to 60 kOe and temperatures down to 0.15 K using a four-wire bridge. The 16 Hz AC measuring currents of ~ 10 and $\sim 1 \text{ mA/cm}^2$ were directed along the a-axis for the measurements with $H\|c$ and $H\|a$, respectively.

Back-scattered Laue X-ray diffraction, electron microprobe analysis, and scanning electron microscopy SEM were performed on crystal B in order to establish that it is a single crystal of uniform composition. Crystal B was cleaved from the zone-melted rod; the c-axis, which is perpendicular to the shiny, layered surfaces of the cleaved ends, is oriented at an angle of $\sim 22^{\circ}$ with the axis of the rod, as verified by Laue diffraction patterns. Laue photographs were taken at numerous locations on the surface of the crystal including the cleared ends, and each displayed well-resolved diffraction spots. The mutual symmetry of the spots from different locations is consistent with constant crystallographic orientation everywhere on the surface. The preservation of the crystal structure over the entire surface strongly suggests that the sample is one single crystal throughout the bulk. SEM performed on a cleaved and polished surface of crystal B revealed no evidence of multiple or impurity phases, and no features down to the micron scale, indicating that the specimen is very uniform in morphology and chemical composition.

It has been suggested that Si deficiency could be responsible for the wide range of $T_{\rm c}$ values measured for different URu₂Si₂ specimens. Any Si inhomogeneity in one crystal might thereby create a range of $T_{\rm c}$ values which would be expected to broaden the superconducting jump(s) in the specific heat. We have measured the composition ratio of U:Ru:Si at several locations on each cleaved side of crystal B by electron microprobe analysis using a submicron beam size. The experimental uncertainty of this technique, obtained by repeated measurements at the same location, is estimated to be $\sim \pm 0.5\%$. On one side, the Si:U and Ru:U ratios

were both found to be constant within a standard deviation $\sigma = 0.5\%$. The other side showed a variation in the Si: U ratio of $\sigma \approx 2\%$, except for several locations near the edge of the cleaved face which deviated by at most $\approx 4\%$, while the Ru: U ratio was constant. We estimate the Si and Ru contents of the two sides to be within 2% of each other. The effect such a variation in Si content has on T_c remains to be established.

3. Results and discussion

Displayed in fig. 1 are zero-field C versus T data between ~0.5 K and 2 K for the two URu₂Si₂ single-crystal specimens (denoted A and B). There are two features in the C(T) data for specimen A which suggest the presence of two superconducting transitions: one at $T_{c1} \approx$ 1.3 K which occupies most of the sample volume and another at $T_{c2} \approx 1.2 \,\mathrm{K}$, which occupies a small fraction of the sample volume. The C(T)data for sample B also exhibit two features which are indicative of two superconducting transitions, one at $T_{c1} \approx 1.3 \text{ K}$ and the other at $T_{c2} \approx 1.0 \text{ K}$, which occupy about equal fractions of the sample volume. There is no obvious feature in the C(T)data for sample B at $T \approx 1.2$ K. Taken at face value, the C(T) data in fig. 1 for samples A and B suggest the presence of two superconducting

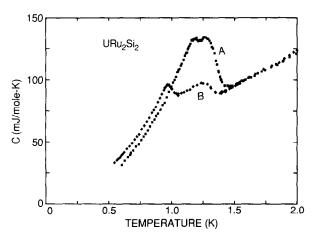


Fig. 1. Specific heat C vs. temperature T of two single-crystal specimens of URu_2Si_2 (denoted A and B) between 0.5 K and 2 K in zero applied magnetic field.

transitions in each sample which are separated by ~ 0.1 K in sample A and ~ 0.3 K in sample B. The volume fractions of the superconducting phases and the temperature interval between them appear to depend sensitively on the state of the samples (e.g., strains, impurities, disorder, etc.). This may explain why C(T) data [8] previously reported in polycrystalline samples of URu₂Si₂ generally display a broad feature indicative of a continuous distribution of T_c s in the range $\sim (1.0-1.4)$ K, rather than the sharper features at the multiple T_c s observed in the single crystals reported here. Presumably, the less strained and more highly ordered state of the single crystal specimens allows the multiple superconducting transitions to be resolved in the C(T) data. A similar situation was encountered for UPt₃, where specific heat measurements on polycrystalline samples revealed a broad feature indicative of a continuous distribution of T_c s extending over a temperature range spanning the two transitions later observed in single crystal and higher-quality polycrystalline specimens. For UPt₃, however, the temperature difference between the two superconducting transitions does not vary significantly among different samples from $\sim 60 \,\mathrm{mK}$.

Analysis of the C(T) data below $T_{\rm c2} \approx 1.2$ K for sample A and below $T_{\rm c2} \approx 1.0$ K for sample B revealed that the data could be described well by the expression

$$C_{s}(T) = \gamma_{s}(0)T + AT^{3}, \qquad (1)$$

down to ~0.5 K, the low-temperature limit of the present experiment. The best fit for sample B is shown in fig. 2 as a solid line below ~1.0 K where $\gamma_s(0) = 42 \, \text{mJ/mol K}^2$ and $A = 62.9 \, \text{mJ/mol K}^4$. Assuming eq. (1) holds to T = 0, the superconducting state entropy $S_s(T)$ was calculated by integrating $C_s(T)/T$ given by the fit of eq. (1) to the C(T) data from 0 to $T_m \approx 0.5 \, \text{K}$, the lower-temperature limit of the experiment, and the actual $C_s(T)$ data numerically from T_m to 1.4 K. The normal-state entropy $S_n(T)$ was evaluated by integrating $C_n(T)/T$ from 0 to 1.4 K using $C_n(T) = \gamma_n(T)T$ and a linear extrapolation of the C(T)/T data above $T_{c1} \approx 1.0 \, \text{m}$

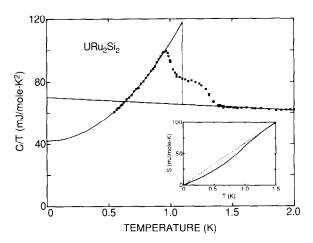


Fig. 2. Specific heat C/T vs. temperature T of a single-crystal specimen of URu_2Si_2 (sample B of fig. 1). C(T)/T for an ideal single superconducting transition with the same entropy as the data at T_{c1} is depicted by the solid lines. The solid line through the data below T_{c2} represents the best fit of $C_s(T)/T = \gamma_s(0) + AT^2$. The solid line through the data above T_{c1} represents the best fit of $C_n(T)/T = \gamma_n(0) - \alpha T$. The superconducting- and normal-state entropies $S_s(T)$ (solid line) and $S_n(T)$ (dotted line) are shown in the inset.

1.4 K to estimate $\gamma_n(T)$ below T_{c1} ; i.e.,

$$\gamma_{\rm n}(T) = \gamma_{\rm n}(0) - \alpha T \ . \tag{2}$$

The best fit of $\gamma_n(T)$ is shown as a straight line in fig. 2, where $\gamma_n(0) = 70 \text{ mJ/mol K}^2$ and $\alpha = 4.3 \text{ mJ/mol K}^3$.

Shown in the inset of fig. 2 are the resultant $S_s(T)$ and $S_n(T)$ curves (represented by solid and dashed lines, respectively) for sample B, where the superconducting-state entropy S_s above T_{c1} is equal to the normal-state entropy S_n within 0.5%. This agreement reflects the accuracy of the fits. Analysis of the C(T) data for sample A yields the following parameters: $\gamma_s(0) = 33.4 \, \text{mJ/mol} \, \text{K}^2$, $A = 60.3 \, \text{mJ/mol} \, \text{K}^4$, $\gamma_n(0) = 75.4 \, \text{mJ/mol} \, \text{K}^2$, $\alpha = 7.7 \, \text{mJ/mol} \, \text{K}^3$, and above T_{c1} , S_s is equal to S_n within 4%.

The finite $\gamma_s(0)$ and the T^3 temperature dependence which characterize the superconductingstate specific heat of URu_2Si_2 should provide important clues to the nature of the unconventional superconductivity apparently displayed by this material. The finite $\gamma_s(0)$ presumably reflects the existence of a region on the Fermi surface over which the superconducting energy gap vanishes, which may be due to pair breaking by nonmagnetic impurities, while the T^3 temperature dependence may be associated with the distribution of nodes in the anisotropic energy gap over the Fermi surface. The fraction of electronic states involved in the superconductivity $f_s = [1 - \gamma_s(0)/\gamma_n(0)]$ is ~56% and ~40% for samples A and B, respectively. It is noteworthy that previous measurements [27] of C(T) under pressure on polycrystalline URu₂Si₂ samples yielded $C_s(T) = \gamma_s(0)T + BT^2$ below T_c , with $\gamma_{\rm s}(0) = 6.5 \,\mathrm{mJ/mol}\,\mathrm{K}^2$, which is probably an artifact of the distribution of T_c s in the polycrystalline material. This form does not provide a satisfactory description of the $C_{\rm s}(T)$ data for the URu₂Si₂ single crystal specimens reported here. The behavior of C(T) below T_{c2} in URu_2Si_2 single crystals is also different than in UPt₃ single crystals, where the specific heat in the superconducting state has been found to be of the form $C_{\rm s}(T) = \gamma_{\rm s}(0)T + BT^2$ [22].

Analysis of the specific heat near the critical temperatures $T_{\rm c1}$ and $T_{\rm c2}$ using the entropy-conserving construction shown in fig. 2 yields for sample B: $\Delta C(T_{\rm c})/[f_{\rm s}\gamma_{\rm n}(T_{\rm c})T_{\rm c}]=2.0$ where $T_{\rm c}$ is the temperature of the ideal single transition specific heat discontinuity.

Specific heat C versus T data for sample B, taken between ~ 0.5 and 2.0 K in magnetic fields of 0, 1, 5, 10 and 15 kOe applied parallel to the c-axis, are displayed in fig. 3 (the 1 kOe data have been omitted for clarity). The two specificheat jumps diminish, broaden, and shift to lower temperatures with increasing field. Shown in the inset of fig. 3 is the H-T phase diagram that results from plotting the field dependence of the peaks.

Resistive upper critical field $H_{\rm c2}$ versus temperature T data, with $H\|c$ and $H\|a$, for two ${\rm URu_2Si_2}$ single-crystal specimens cut from sample A are shown in figs. 4 and 5, respectively. The $H_{\rm c2}(T)$ data were deduced from measurements of the electrical resistivity as a function of temperature in various fixed magnetic fields, where $T_{\rm c}$ in each field was defined as the midpoint of the resistive superconducting transition

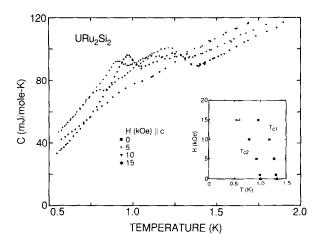


Fig. 3. Specific heat C vs. temperature T of a single-crystal specimen of URu_2Si_2 (sample B) between 0.5 K and 1.6 K in various magnetic fields H applied parallel to the c-axis. The dependence of T_{c1} and T_{c2} on H is displayed in the inset.

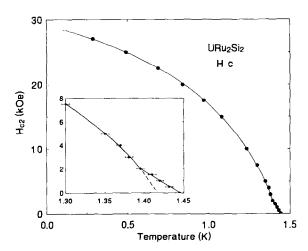


Fig. 4. Upper critical field H_{c2} vs. temperature T of a URu_2Si_2 single-crystal specimen from crystal A, measured resistively with the current I flowing along the a-axis and the magnetic field H applied parallel to the c-axis. Shown in the inset are $H_{c2}(T)$ data below 8 kOe which reveal the existence of a kink near 2 kOe. Solid and dashed lines are guides to the

curve. The sample measured with $H \parallel c$ displayed sharp superconducting transitions with a residual resistivity just above $T_{\rm c}$ of $\rho_0 \approx 26~\mu\Omega$ cm and a large zero-field $T_{\rm c} \approx 1.45~{\rm K}$. The sample measured with $H \parallel a$ exhibited somewhat broader

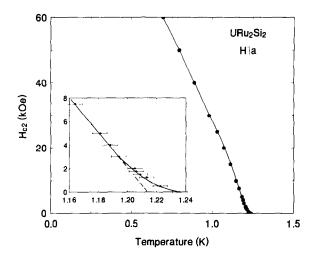


Fig. 5. Upper critical field $H_{\rm c2}$ vs. temperature T of a URu₂Si₂ single crystal specimen from crystal A, measured resistively with the current I flowing along the a-axis and the magnetic field H applied parallel to the c-axis. Shown in the inset are $H_{\rm c2}(T)$ data below 8 kOe which reveal the existence of a kink near 2 kOe. Solid and dashed lines are guides to the eye.

superconducting transitions, a residual resistivity $\rho_0 \approx 17 \, \mu\Omega$ cm, and a smaller $T_c \approx 1.24 \, \mathrm{K}$. For both specimens, the residual resistivity ratio $\rho(300 \, \mathrm{K})/\rho_0$ is ~ 20 . The difference in quality between the two crystals is probably due to the different locations in the zone-refined rod. The $H_{\rm c2}(T)$ data in figs. 4 and 5 reveal a large amount of anisotropy with a value $H_{\rm c2}^{\parallel a}/H_{\rm c2}^{\parallel c} \approx 2.5$ at 0.7 K. The magnitudes, general shapes and anisotropy of the $H_{\rm c2}(T)$ curves for $H \parallel c$ and $H \parallel a$ are similar to those reported previously [28].

Shown in the inset of fig. 4 are the $H_{c2}(T)$ data for $H \| c$ below 8 kOe which show a distinct kink at ~ 2 kOe. The error bars represent the uncertainty of T_c . The slope of the curve changes from $\mathrm{d}H_{c2}/\mathrm{d}T \approx -40\,\mathrm{kOe/K}$ below 2 kOe to $\approx -70\,\mathrm{kOe/K}$ for fields above 2 kOe. An extrapolation of the higher field data to zero field, as shown by the dashed line in the inset of fig. 4, yields a critical temperature which is $\sim 30\,\mathrm{mK}$ below T_c . It seems unlikely that the kink in the $H_{c2}(T)$ curve for $H \| c$ is related to a convergence of T_{c1} and T_{c2} at $\sim 2\,\mathrm{kOe}$, since this would require that T_{c2} increases rapidly with H below

3 kOe, in contrast to the parallel shift of T_{c1} and T_{c2} to lower temperatures with increasing H found for sample B from the measurements of C(T, H) (fig. 3). The inset of fig. 5 reveals a similar deviation from normal behavior below ~3 kOe in the $H_{c2}(T)$ curve for H||a|. In this case, the data display upward curvature below ~3 kOe instead of a distinct kink. At zero field, the initial slope is $dH_{e2}/dT \approx -37 \text{ kOe/K}$; above 3 kOe, H_{c2} is linear in T with a slope of $-150 \,\mathrm{kOe/K}$. This behavior of the $H_{c2}(T)$ curve is consistent with the positive curvature observed in previously reported $H_{c2}(T)$ data taken on single crystal specimens of URu₂Si₂ for H||c| and H||a, where the density of data was not sufficiently high to establish the possible existence of a kink for H||c| [28]. The positive curvature of $H_{c2}(T)$ in low fields for URu_2Si_2 has been explained in terms of the effect of coupled antiferromagnetic and multicomponent superconducting order parameters [29]. A similar situation was encountered for the heavy-electron superconductor UPt₃; whereas the original measurements of $H_{c2}(T)$ on single-crystal specimens for $H \parallel c$ and $H \perp c$ revealed a strong positive curvature near T_c [9], subsequent measurements established the existence of a kink in $H_{c2}(T)$ for $H \perp c$ [30, 31]. For both URu₂Si₂ and UPt₃, the kink in $H_{c2}(T)$ is observed for H in the direction of the antiferromagnetically aligned moments.

The double superconducting transitions revealed by the C(T, H) measurements and the positive curvature (along with the possible kink for H||c| in the $H_{c2}(T)$ data taken on the singlecrystal specimens of URu₂Si₂ reported herein may be associated with coupled antiferromagnetic and multicomponent superconductingorder parameters. However, it is difficult to rule out the possibility that the two superconducting phases observed in single-crystal specimens A and B are associated with two regions of the single crystal which are characterized by different compositions, states of strain, atomic order, etc. However, this seems unlikely on the basis of the sharpness of the superconducting transitions observed in the specific heat and the structural and chemical analysis we have performed on specimen B. More extensive experiments on these and other single-crystal URu₂Si₂ specimens are in progress in order to determine whether or not the multiple superconducting phases are intrinsic, and, if so, to test relevant theories of superconductivity in heavy-electron compounds.

Note added in proof

Subsequent to submitting this manuscript, we performed resistive upper critical field measurements on a rod cut from crystal B, with the field applied parallel to the c-axis. The results reveal a kink, similar to that for crystal A (see fig. 4), occurring at a field of 2 kOe. This specimen also displayed many unusual features including a superconducting transition with an unprecedentially high onset temperature of 2 K, $T_c(50\%) =$ 1.64 K, $\Delta T_c(10-90\%) = 0.38$ K, a residual resistivity ratio RRR of 60, and a residual resistivity $\rho_0 \approx 6 \,\mu\Omega$ cm. We remark that an anomaly has been seen in the specific heat of a polycrystalline sample of URu_2Si_2 at ~2.7 K [32], which the authors suggest could be due to rearrangement of the spin density wave. Very recently, we received a preprint [33] of a similar specific-heat study on single crystals of URu₂Si₂ in which the authors claim that the double transitions they observe are due to different unique crystal phases which are as yet unidentified.

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