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1993 J. Phys.: Condens. Matter 5 7709

(http://iopscience.iop.org/0953-8984/5/41/018)

J. Phys.: Condens. Matter 5 (1993) 7709–7718. Printed in the UK

Flux pinning, specific heat and magnetic properties of the laves phase superconductor $CeRu_2$

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Received 16 April 1993, in final form 26 July 1993

Abstract. A sharp transition from reversible behaviour to irreversible behaviour in the superconducting mixed state of single-crystal samples of $CeRu_2$ is described. Magnetization, AC susceptibility, resistivity and specific heat measurements under an applied field are reported. The results are discussed in relation to conventional models and those of more novel superconductivity, such as the Fulde–Ferrel state.

1. Introduction

$CeRu_2$ stands out from the class of 'conventional superconductors' in that it exhibits extreme type II behaviour (Ginsburg–Landau parameter, $\kappa = 16$), whilst permitting the possibility of sample preparation in a metallurgically clean state. It is also unusual in that alloying with magnetic ions is not detrimental to the superconductivity, and such substitution of magnetic ions has largely motivated previous investigations [1,4], which have in particular focused attention on the question of the co-existence of magnetic order and superconductivity. In this paper we concentrate, rather, on the pure compound $CeRu_2$ itself.

The arrangement of the paper is the following. Sections 2 and 3 give details of the sample preparation and summarize the measurement techniques. Contrary to previous investigations we find that for our sample the susceptibility in the normal state, described in section 4, can be interpreted solely in terms of a temperature-independent response. The more substantial discussion, given in section 5, concerns both AC and DC magnetic measurements made in the superconducting state, where an abrupt change from reversible to irreversible behaviour, concurrent with a peak in the magnetization hysteresis curve is reported. Beyond the immediate physical interest of this observation it is interesting to note that a somewhat similar effect has previously been seen in the heavy-fermion superconductor UPt_3 [5] and recent measurements on another heavy fermion material UPd_2Al_3 [6], also allude to a transition in behaviour with a similar variation in field against temperature. It is widely believed that UPt_3 has an unconventional superconducting state, with a multiple-component order parameter [7]. The nature of the superconducting state in $CeRu_2$ is then of special relevance and in section 6 we describe our specific heat measurements. These include measurements made in magnetic fields, which are essential to the determination of the electron-quasi-particle mass as well as to the interpretation of the superconducting state. The results are discussed in terms of conventional and less conventional models for the superconductivity in section 7.

0953-8984/93/417709+10$07.50

It should also be mentioned that the achievement of an enhanced irreversibility near the upper critical field H_{c2} as seen here has a more general technological relevance to the production of large magnetic fields.

2. Samples

The various measurements described were made on three different pieces of the same single crystal. The smallest piece was used to determine the resistivity and the intermediate-sized piece was used to determine the specific heat measured under an applied field. The largest piece of mass 33 mg was used for the magnetic measurements and also for additional zero-field measurements of the specific heat down to 330 mK. It was also checked that the magnetization versus applied field curve of the intermediate-sized piece was identical to the largest sample at a representative temperature of 4.5 K.

The pieces of crystal were carefully spark-cut from an ingot prepared in an induction furnace with a cold crucible under UHV. The starting materials were of the highest obtainable purity; the Ru powder (Johnson Matthey Ltd) was first melted under UHV and the Ce (Ames Laboratories) was additionally zone refined. $CeRu_2$ forms peritectically, and the largest crystals were obtained from quenching an apparently totally molten composition of 38% Ce with a subsequent anneal at 900 °C for 24 hours. Electron microprobe and optical examination showed the location of the crystals to be well separated from a region of the ingot which contained an additional Ce-rich phase. The width of the superconducting transition in the specific heat measurements was 50 mK, which demonstrates the good homogeneity of the crystals. The resistance ratio between 273 K and just above T_c was 15.

3. Methods

The DC magnetic measurements were made with a commercial Quantum Design Co. SQUID magnetometer where the magnetization is determined from the change of linked magnetic flux as the sample is raised through three superconducting coils (turns ratio $-1{:}2{:}-1$), with automated measurement only possible for this single direction of sample displacement. It turns out that measurements with this instrument as a function of applied field do not give the true full hysteresis curve of the sample and the interpretation of our measurements is now discussed with reference to the data shown in figure 1(*a*). The striking feature of figure 1(*a*) is the pronounced dip in magnetization seen in the superconducting mixed state at $\mu_0 H = 1.3$ T; it is particularly surprising that this is apparently seen for both increasing and decreasing the applied field. This last observation can however be easily understood as an artifact of the measurement technique if it is taken into account that the magnetic field is not perfectly homogeneous and that the sample is always exposed to a small increase of field as it is raised ($\Delta H/H \simeq 3 \times 10^{-3}$). For most measurements this small increase of field would have little consequence since the fractional change of magnetization induced would be small, of the order $\Delta M/M = \Delta H/H$. However, for the case of a superconductor with strong flux pinning the fractional change in magnetization $\Delta M/M$ can be large, with a change of magnetization of up to $\Delta M = -\Delta H$ ($\simeq 3000$ A m^{-1} at the field corresponding to the anomaly in figure 1(*a*)). In a measurement the recorded magnetization therefore corresponds to a value along the increasing-field part of a minor hysteresis loop. Such a minor hysteresis loop is shown as the solid line in the schematic insert of figure 1(*d*). Since experimentally, the magnetization found is not discernibly different for a different field history prior to starting the measurement, the increase of field as the sample is moved is sufficient that the magnetization always reaches the lower bound of the true full hysteresis curve (the dotted line in the figure) after only a short part of the total sample displacement, independent of the starting value of M. The measured magnetization in the experimental curve of figure 1(a) is then interpreted as being close to the increasing-field part of the full hysteresis loop. While it has been stated that automatic measurements were not possible while the sample is lowered, these measurements can be made using a less accurate manual method with the same apparatus and would be expected to give the magnetization at the other extremum of the minor hysteresis loop (the sample would be exposed to a decrease of field when it moves in this direction). At fields below where the anomalous dip in magnetization is seen, the SQUID output when the sample is lowered is not discernibly different from that obtained in the automatic measurements when the sample is raised, which indicates that the minor hysteresis loop is small for fields in this regime. In contrast, for a field corresponding to the dip in measured magnetization the SQUID output when the sample is lowered is very different from the measurement when the sample was raised, indicative of a significant hysteresis. The full hysteresis curve that is suggested in the light of these complementary measurements is given schematically in figure 1(d). The hysteretic behaviour shown in this curve is further confirmed by AC susceptibility measurements, described in section 5.

All the magnetic measurements were made with a [100] axis of the crystal ($CeRu_2$ has the C15 cubic structure) oriented approximately parallel to the applied field direction. The AC susceptibility measurements were carried out using the mutual inductance technique in a dilution refrigerator. The data are presented relative to measurements above the superconducting transition to eliminate a small background response as a function of field, which was also seen with the sample removed.

The specific heat under applied field data were measured by a relaxation method, and involved averaging cooling and heating data so that the measurement is independent of the value of the heat leak. Additional zero-field measurements down to 330 mK were made for increasing temperature, with apparatus employing a SQUID sensor to null the temperature difference between the sample and its surroundings.

4. Normal state magnetism

In the normal state the susceptibility was deduced from DC measurements which show a linear magnetic response with field (up to the maximum applied field of 5.5 T) between the superconducting transition temperature, 6.15 K and room temperature. Below T_c, but above the upper critical field, the same magnetic response is recovered which corresponds to a susceptibility (defined as M/H and in SI units) of 2.2×10^{-4}. This result differs from previous measurements [8, 9] where an additional Curie–Weiss contribution was found at low temperatures. Such a contribution may be attributed to metallurgically inferior samples, and might originate from a substantial quantity of the Ce having the Ce^{3+} magnetic form or the presence of magnetic impurities.

It is worthwhile at this stage to comment on the magnitude of the susceptibility. As a preliminary to this we discuss the electronic density of states (DOS) at the Fermi surface. The true DOS can be found most directly from quantum oscillatory measurements, but in the absence of such results an estimate from the linear coefficient of the specific heat is preferable to a determination from band-structure calculations, which, although they can describe correctly the Fermi-surface morphology, are notoriously unreliable in determining the DOSs for strongly correlated systems, as exemplified by studies of heavy fermion compounds [10]. The linear coefficient of the specific heat, 29 mJ K^{-2} (mole $Ce)^{-1}$ (see section 6), gives

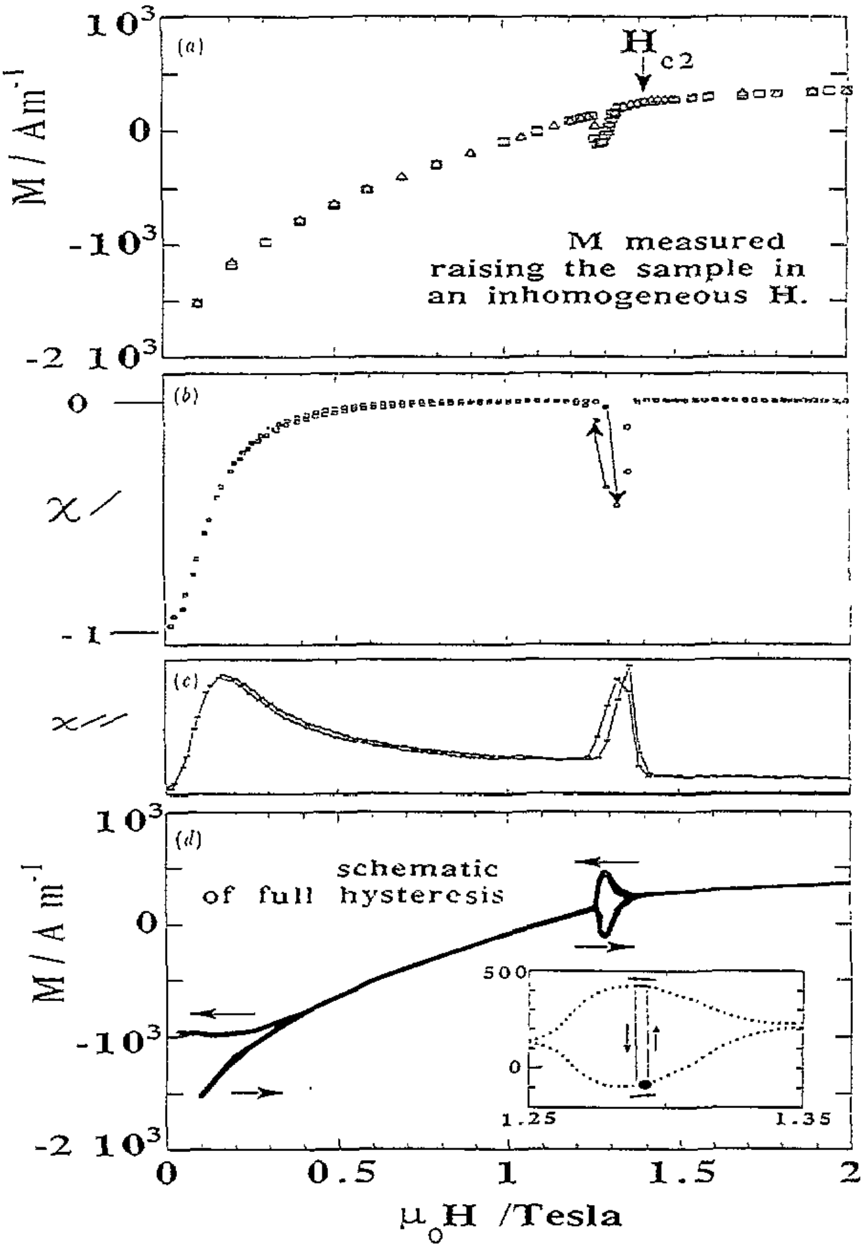

Figure 1. Magnetic measurements for the 33 mg single crystal of $CeRu_2$ at 5 K. (*a*) Magnetization measured on a commercial magnetometer against applied field. The squares/triangles denote that the applied field was changed in an increasing/decreasing sense prior to the start of each measurement. Since the applied field is not perfectly homogeneous the true full hysteresis curve is not obtained (see section 3). Figures (*b*) and (*c*) show the in-phase (χ') and dissipative (χ'') components of the AC susceptibility ($\mu_0 H_{AC} = 0.1$ mT, 127 Hz). The schematic full hysteresis curve inferred is given in figure (*d*) (see sections 3 and 5 of the main text). The insert of figure 1(*d*) shows a typical minor hysteresis loop (solid line) and the solid circle indicates the magnetization that would be recorded for a measurement in which the sample is moved in the direction of increasing field (corresponding to figure 1(*a*)); the dashed lines show the full hysteresis curve.

an enhancement of the DOS of the order 2.5 over the augmented plane wave calculation of Yanase [11].

In the absence of local paramagnetic moments, the various contributions to the susceptibility are the Pauli paramagnetism and Landau diamagnetism due to the conduction electrons, and the diamagnetic contribution of the ion cores. The contribution from interband effects will be assumed to be small. The diamagnetism of the ion cores is estimated from tabulated values [12] as $\chi_c = -2.5 \times 10^{-5}$, and the Landau diamagnetic contribution estimated as $\chi_L = -\mu_0\mu_B^2 < (m_e/m_b)^2 > \nu_0(0)/3 = -1.0 \times 10^{-5}$, where $\nu_0(0)$ is the non-interacting DOS from the band structure, and m_e and m_b are the free electron mass and band mass respectively. The Pauli contribution, χ_P, may then be expressed in terms of Fermi-liquid theory [13] as,

$$\chi - \chi_L - \chi_C \simeq \chi_P = \mu_0\mu_{\rm eff}^2\nu(0)/(1 + F_0^a) \tag{1}$$

where $\nu(0)$ is now the measured DOS and F_0^a is a parameter representing the spin anti-symmetric isotropic component of the interaction between the quasi-particles. It is not clear what value should be taken for the effective moment of the quasi-particles $\mu_{\rm eff}$, as this is sensitive to the orbital characters of the principal bands at the Fermi surface. However if a value $\mu_{\rm eff} = \mu_B$ (μ_B is the Bohr magneton) is taken, appropriate to the translationally invariant case, we find from the measured χ that the parameter F_0^a is -0.4 or equivalently that the Wilson–Sommerfield ratio is $\pi^2 k_B^2 \chi_P/3\gamma\mu_0\mu_B^2 = 1.7$. The ratio χ_P/γ is less than the magnitude typical of Ce intermediate valence materials (e.g. $CeSn_3$) but is similar to values for non-magnetic Ce heavy fermion compounds (e.g. $CeCu_2Si_2$ and $CeCu_6$) [14], although for $CeRu_2$ the enhancement of the DOS is too modest for classification as a heavy fermion compound.

5. The superconducting state

In general, the detailed characterization of the magnetic properties of the superconducting mixed state is highly involved. The following discussion therefore concentrates on the principal features of our data which are relatively insensitive to changes of, for example, the AC measuring field or the relaxation time between changing the applied field and the measurement of the susceptibility.

Figures 1(*b*) and 1(*c*) show the measured AC susceptibility. The in-phase AC susceptibility χ' is negative for fields near to H_{c2}, but it undergoes an abrupt change at a lower field, H^*, to near zero, slowly becoming negative only at much smaller fields. Negative values of χ' can be taken as indicative of pinning of the flux lattice, so that the flux lattice appears strongly pinned above H^*, with an abrupt transition to an unpinned regime below this field. This is consistent with the schematic full hysteresis curve given in figure 1(*d*) and with measurements made with a commercial DC SQUID magnetometer; the latter however require careful interpretation (given in section 3) when there is flux pinning because the presence of a small gradient in the applied field means that the true full hysteresis curve is not directly obtained. The out-of-phase component of the susceptibility, χ'', measures the energy dissipated principally due to viscous drag acting on the rapid vibration of the flux lines induced by the AC field, and cannot be related simply to the full hysteresis curve which is appropriate to longer time scales (minutes as opposed to hundredths of a second) where such fluid flux line motion will have relaxed.

The most striking feature of the curves is that there is a sharp change of behaviour at a field $H^*(T)$, determined consistently by both AC (taken as the onset of a negative susceptibility in increasing field) and DC techniques, and this is plotted as a function of temperature in figure 2. The magnitude of the anomaly immediately above $H^*(T)$ becomes much larger at lower temperatures. Above a temperature $T^* = 5.5$ K the region of irreversibility near H_{c2} no longer occurs and the $M(H)$ curve shows the more standard behaviour of a strongly paramagnetic superconductor [15]. For fields less than $H^*(T)$ the magnetization is remarkably reversible (χ' is small), an attribute usually only associated with high-quality samples of elemental superconductors. The small irreversibility in this regime below $H^*(T)$ becomes discernible only at small fields or at the lowest temperatures, consistent with the conventional idea of the strength of pinning reflecting the energy difference between a pinning site and the bulk. The measurements shown in figures 1(*b*) and 1(*c*) were quasi-static, made with the applied DC field fixed; AC susceptibility results obtained when the DC field was scanned are broadly similar to these static measurements but with a larger offset of $H^*(T)$ between increasing and decreasing the field.

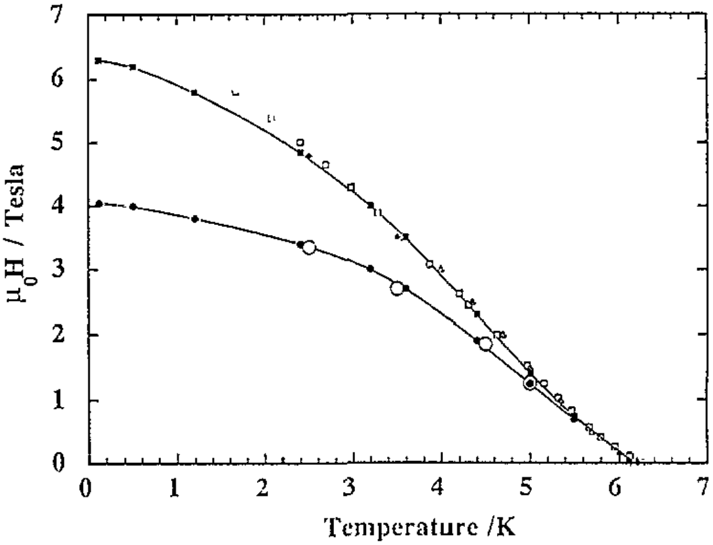

Figure 2. Magnetic phase diagram showing H_{c2} (closed squares from AC susceptibility, open squares from resistivity, triangles from specific heat, diamonds from DC magnetization), and $H^*(T)$, the field at which there is an abrupt change in reversibility (closed circles from AC susceptibility, open circles from DC magnetization). The solid lines are guides to the eye.

$M(H)$ was found to be continuous at H_{c2} at all temperatures. From the discontinuity in $\mathrm{d}M/\mathrm{d}H$ at H_{c2} the Ginzburg–Landau parameter κ_2 can be determined as

$$(\partial M/\partial H)_s - (\partial M/\partial H)_N = 1/\beta(2\kappa_2^2 - 1) \qquad (2)$$

where the geometric parameter β is taken as 1.16 appropriate to a triangular flux lattice. This determination is more reliable in the range $T^* < T < T_c$ where the magnetization curve below H_{c2} is reversible and gives the value $\kappa_2 \to \kappa = 16$ as $T \to T_c$, as already stated in the introduction. The dirty limit value of κ, determined from the normal state resistivity (here 4.5 $\mu\Omega$ cm, inferred from the measured resistance ratio and the absolute resistivity for polycrystalline materials [16]) and the linear coefficient of the specific heat is $\kappa_{\mathrm{dirty}} \simeq 2$, so that we must indeed be close to the intrinsic clean limit. Between 2.5 K and 6 K, the determination of H_{c2} from the resistive transition gives an estimate of H_{c2} identical to the field at the discontinuity of $\mathrm{d}M/\mathrm{d}H$ in the DC measurements, the field at which the AC dissipation drops to near zero and H_{c2} determined from the specific heat. The gradient $\mathrm{d}H_{c2}/\mathrm{d}T$ corresponds to a Ginzburg–Landau coherence length $\xi(0) = 61$ Å.

H_{c1}, estimated from the low-field maximum of the magnetization in the DC measurements, was determined to be of the order $H_{c2}/130$ for measurements in the temperature range 3.5–6 K. The value of κ implied by the Ginzburg–Landau relation

$$\kappa = \surd((H_{c2}/2H_{c1})\ln\kappa) \tag{3}$$

is thus in fair agreement with the estimate from the discontinuity in $\mathrm{d}M/\mathrm{d}H$.

Finally it should also be pointed out that the observation of anomalous magnetic behaviour has been previously reported for the case of a phase-impure sample of $CeRu_2$ [17]. In that work the nature of the anomaly was obscured by a uniformly large hysteresis, but the temperature dependence of a change in magnetic behaviour correlates well with the dependence found here for the change from reversible to irreversible behaviour. A weak peak effect in the magnetic hysteresis has also been previously noted for the related compound $LaRu_2$ [18] but the evolution with temperature was not reported.

6. Specific heat

The specific heat measured under different applied fields plotted as C/T against T^2 is shown in figure 3. It is clear that there is a significant decrease in C/T measured under applied field from that obtained by linearly extrapolating the zero-field gradient above T_c. This observation could be indicative of structure in the electronic density of states close to the Fermi level or of structure in the low-energy phonon DOS. The latter explanation is made plausible by a low Debye temperature and the fact that phonon anomalies give rise to similar effects in the A15 superconductors. The change in the entropy between absolute zero and T_c for the superconducting state is found to be equal to that for the normal state measured under field. This result supports the implicit assumption that the primary effect of the applied field is to suppress the superconducting transition without significantly modifying the underlying electron/phonon structures. The normal state ($H > H_{c2}$) data can be fitted to an expansion in odd powers of the temperature while imposing equality of the entropy with the superconducting state at the transition. The value of the T^3 coefficient, so obtained, corresponds to a Debye temperature of 140 K, which is much lower than the value 172 K obtained from the slope of C/T against T^2 above 7 K. A contribution from magnetic fluctuations of a magnitude sufficient to explain the observed temperature dependence of the specific heat is unlikely in the light of the measured susceptibility unless the fluctuations are of an antiferromagnetic nature, and the susceptibility in fields larger than H_{c2} also rules out anomalous features in the DOS.

The linear electronic coefficient of the specific heat is $\gamma = 29$ mJ K^{-2} (mole $CeRu_2)^{-1}$, which is less than half the value obtained by a naive extrapolation of zero-field measurements [19, 21] from above T_c. It is interesting that this value is in fact smaller than that reported for $LaRu_2$ [19], which has no f electrons, although comparison is complicated since $LaRu_2$ undergoes a structural transition and no longer has the C15 structure below 30 K.

The difference in C/T between measurements in the superconducting state and at high field give a clear T^3 dependence between T_c and 1.8 K, the lowest temperature for which measurements were made under field. The discontinuity in specific heat is $\Delta C = 2.0\gamma T_c$, a value characteristic of strong coupling. We also report zero-field measurements down to 330 mK. These results are not shown, but after subtraction of the phonon contribution (T^3

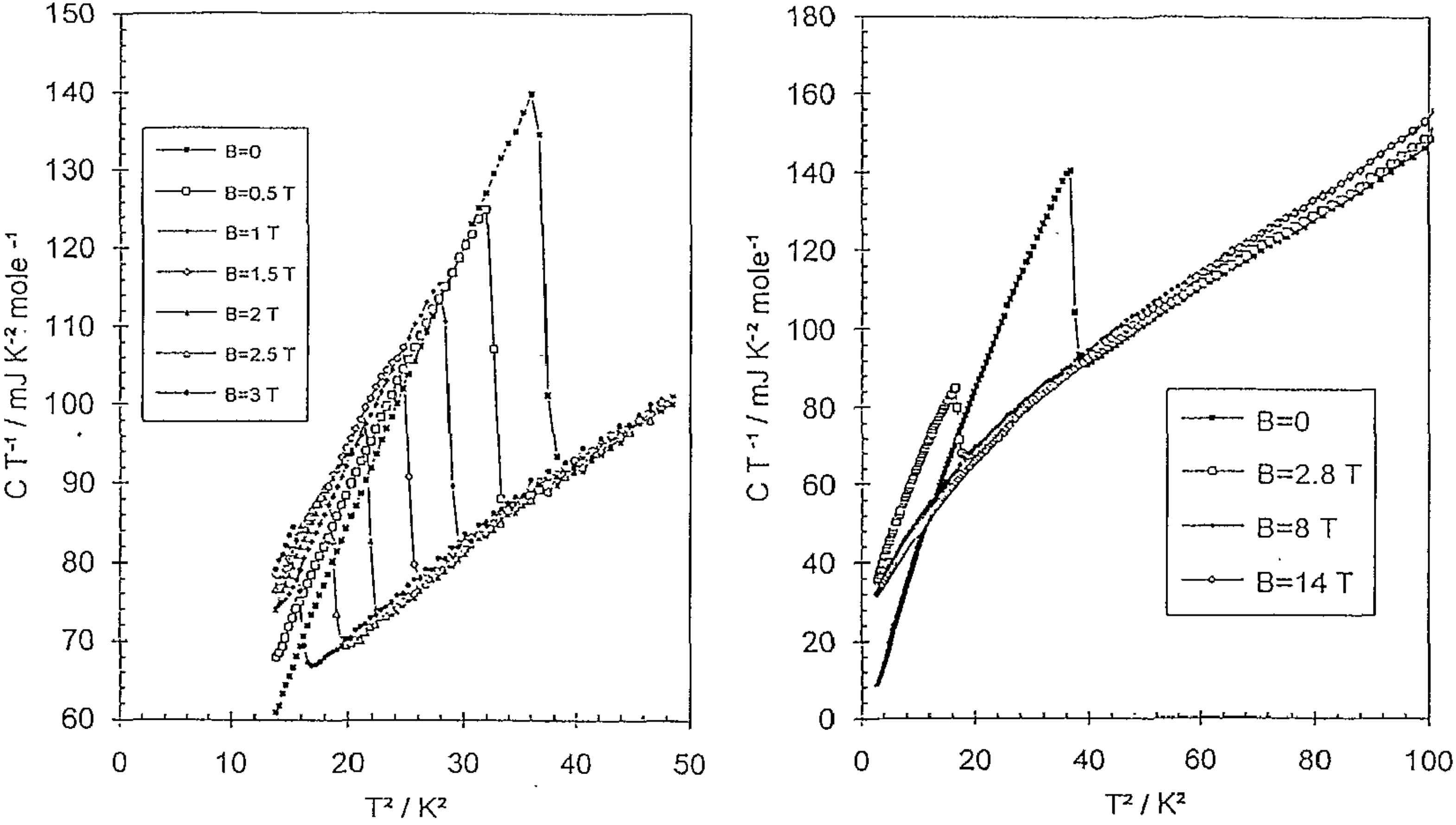

Figure 3. The total specific heat of $CeRu_2$ plotted as C/T against T^2 for various fields.

terms and higher) obtained in the above fit to the normal state, the temperature dependence of the specific heat at low temperature (0.33–2.5 K) is found to be very well modelled by a small linear contribution plus an exponential dependence,

$$C = a\mathrm{e}^{-b/T} \qquad a = 10\gamma T_c \qquad b = 1.6T_c. \tag{4}$$

The small linear contribution (linear coefficient=0.5 mJ K^{-2} (mole CeRu$_2$)$^{-1}$) is of the same magnitude as our confidence limit in estimating the addenda, but might also indicate pair breaking. The parameters a and b are typical of strong coupling materials and correlate with the magnitude of $\Delta C/\gamma T_c$ [22]. Taken together with the observation that power laws fail to fit the data adequately in this temperature range, this provides strong evidence that the symmetry of the superconductivity is conventional, but with the caveat that the interpretation is still reliant on an extrapolation of the high-field measurements to lower temperatures.

7. Discussion

The sharp increase in irreversibility at high fields above H^* is somewhat reminiscent of the so-called 'peak effect' seen for metallic alloys with significant inclusions of a second phase [23, 24]. Such an effect can originate from a change in the balance between local pinning force gradients and the elastic coefficient for deforming the flux lines associated with pinning by the inclusions. To explain the absence of such an effect above T^* a proximity-effect induced normal-superconducting transition of the pinning regions might be invoked [25]. Such a transition might then be argued to give a small discontinuity in the specific heat at $T(H^*)$: an uncritical analysis of our specific heat data (section 6) does provide a hint of such a transition, but it should be stressed that this is at the limit of resolution. The scope of this mechanism to explain the results for CeRu$_2$ is however restricted by electron-microprobe examination of similar crystals, originally adjacent to those used. No inclusions were detected and the crystals were found to be homogeneous and when they were ground, gave phase-pure x-ray powder patterns. There however remains the possibility that such an effect can be associated with defects, which would have to have influence over distances of several coherence lengths. This explaination cannot be definitely excluded, although it might seem unlikely since it would have to be consistent with the sharp transition at T_c, and further would require a somewhat contrived scenario to explain the temperature dependence of $H^*(T)$. The role of surface damage to an observation of a peak effect [15] would seem improbable here as the crystals were extracted by spark-cutting under conditions to ensure minimal damage.

The gross form of the $B(T)$ dependence of the transition between irreversibility and reversible behaviour warrants comparison to a transition to a Fulde–Ferrell state [26, 27], in which the superconducting state is modulated. The hysteresis in the position of the transition for increasing and decreasing the field in magnetic measurements and slight evidence of an anomaly in the specific heat at the transition provide palpable evidence that the change to irreversibility could be associated with a genuine first-order thermodynamic transition. However there are differences when a more exact comparison is made with existing theoretical predictions. In particular, despite being an extreme type II clean superconductor the upper critical field is well below the usual paramagnetic limit, which must be exceeded in the existing theory to give such a state.

There remains the possibility of some form of magnetic transition possibly relating to a localization of electronic structure induced by the superconducting gap opening [17]. This possibility will be better clarified by forthcoming polarized neutron scattering measurements.

As already stated in the introduction, a similar abrupt change in magnetic reversibility in the superconducting mixed state might also be associated with observations of anomalies in mechanical resonance [28] and ultrasonic attenuation measurements [29] for UPt_3. Recent magnetostriction measurements for another heavy fermion material UPd_2Al_3 [6] have also provided evidence of a first-order transition within the mixed state. Theoretical attempts to explain these results in the case of UPt_3 have often presumed a multiple-component order parameter, such as required for a transition to double-quantized flux-lines [30].

From our measurements of the specific heat it appears that the superconducting state of $CeRu_2$ can be interpreted in terms of a single-component order parameter in the context of the strong coupling BCS model. That we have then demonstrated a transition in behaviour in the superconducting mixed state with a very similar dispersion to the transitions seen in the above materials indicates that a multiple-component order parameter is not essential to produce such phenomena. Most importantly, more detailed work is desirable on all these materials to study the importance of metallurgical defects to such transitions. Should the transitions indeed be found to be intrinsic, existing theoretical models such as that for the Fulde–Ferrel state seem inadequate, and we hope that this work will also motivate the study of other mechanisms, perhaps relating to the coupling of the order parameter to elastic or magnetic deformations.

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