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Anisotropy, disorder, and superconductivity in CeCu_2Si_2 under high pressure

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Abstract

Resistivity measurements were carried out up to 8 GPa on single-crystal and polycrystalline samples of CeCu_2Si_2 from differing sources in the homogeneity range. The anisotropic response to current direction and small uniaxial stresses was explored, taking advantage of the quasi-hydrostatic environment of the Bridgman anvil cell. It was found that both the superconducting transition temperature T_c and the normal state properties are very sensitive to uniaxial stress, which leads to a shift of the valence instability pressure P_v and a small but significant change in T_c for different orientations with respect to the tetragonal c -axis. The coexistence of superconductivity and residual resistivity close to the Ioffe–Regel limit around 5 GPa provides a compelling argument for the existence of a valence fluctuation mediated pairing interaction at high pressure in CeCu_2Si_2 .

1. Introduction

It has recently been proposed that a new type of superconductivity exists in the heavy fermion compound CeCu_2Si_2 under high pressure [1–5]. In this compound, the superconducting transition temperature T_c is enhanced near 3 GPa from its ambient pressure value of 0.7 K to around 2.5 K. The proposed pairing mechanism at high pressure is based on the exchange of critical valence fluctuations close to a (nearly) first order valence transition of the Ce ion at a pressure $P_v \simeq 4.5$ GPa. At ambient pressure, in contrast, critical magnetic fluctuations are believed to mediate the superconductivity. The magnetically ordered state, which in pure CeCu_2Si_2 competes with the superconducting state is thought to disappear at a magnetic quantum critical point at a small positive pressure P_c of approximately 0.1 GPa [6]. This quantum critical point is masked by the superconducting state in pure

samples, but can be directly observed by substituting Ge for Si which leads to a suppression of superconductivity [4, 7].

The necessary background to this work can be found in [3], which provides the context for the results and discussion reported below, and should be read in conjunction with this paper. The most important point to remember is that there is a second critical pressure P_v where the occupation number n_f of the electronic f orbitals on the Ce atom changes abruptly. This can be thought of as analogous to the Ce α/γ transition, though with the critical endpoint at very low, or perhaps negative temperature (the latter leading to a crossover rather than a first order transition). The system goes from a Kondo regime ($n_f \simeq 1$) to that with characteristics of an intermediate valence state (where $n_f < 1$). The enhancement of T_c near P_v , mediated by critical valence fluctuations, was predicted by an extended Anderson model including an extra Coulomb repulsion term U_{cf} between the conduction c and f electrons [1, 2]. A series of anomalies in the normal state electronic properties was also predicted, and these have been observed to occur around this pressure. They include a large enhancement of the residual resistivity ρ_0 , an enhancement of the Sommerfeld coefficient γ , and a resistivity linear in temperature, $(\rho - \rho_0) \propto T$ [2, 3, 8]. This simple model is surprisingly successful in describing the observed behaviour in CeCu_2Si_2 , and can help to provide the basis for a more complete description of the behaviour of related systems. However, there are further experimental facts beyond its scope, which may prove useful guidance towards a more complete understanding of the microscopic physics involved.

The superconducting and magnetic properties of CeCu_2Si_2 at ambient pressure are highly sensitive to small changes in composition and disorder [9–12], but there has been little systematic investigation into similar effects at high pressure. The ground state at ambient pressure depends strongly on the exact stoichiometry of the sample, giving so-called types A, A/S and S, where the magnetically ordered ‘A phase’ competes with the superconducting state ‘S’. Ishikawa [13] recently proposed a further subdivision of CeCu_2Si_2 properties, into so-called ‘low T_c ’ and ‘high T_c ’ types, with differing signs in the pressure dependence of the superconducting transition temperature T_c . In addition, the isostructural compound CePd_2Si_2 has been shown to be extremely sensitive to uniaxial stress under pressure. Dramatic variations in T_c in CePd_2Si_2 result from a change in crystalline orientation with respect to small non-hydrostatic components in a Bridgman anvil pressure cell with a quasi-hydrostatic steatite medium [14].

With these facts in mind, we wished to systematically explore, via resistance measurements under pressure, how the electronic properties around P_v depended on the sample, and on the pressure conditions; especially to see how valence fluctuation mediated superconductivity in CeCu_2Si_2 is affected by the presence of disorder and uniaxial stress. The results previously obtained in a hydrostatic helium pressure medium [3] provide a baseline for comparison.

2. Experimental methods

Resistivity measurements under pressure on a total of six CeCu_2Si_2 samples from two different sources are reported below. There were two polycrystalline samples, of type ‘low T_c ’ and ‘high T_c ’, respectively labelled 50 and 57 and prepared by Ishikawa’s group by a levitation method, which had already been rather well characterized as previously reported [13, 22]. The sample labels 50 and 57 are identical to those given in the references. In these previous studies, we found no significant variation of lattice parameters with composition, although their physical properties, particularly the superconducting ones, differed considerably [13]. The remaining four samples were A/S type single crystals from the same original source crystal prepared at the MPI Dresden. The latter crystals were grown in an Al_2O_3 crucible by a modified Bridgman

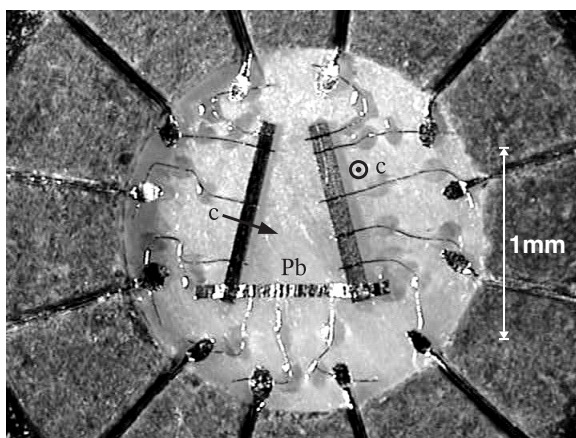


Figure 1. Cell 2, containing two A/S samples cut from neighbouring positions on the same source crystal. They are oriented with their c -axes parallel and perpendicular to the cell axis, and the current flows along the a -direction.

technique using Cu excess as flux medium [15]. X-ray powder diffraction confirmed the tetragonal ThCr_2Si_2 structure with lattice parameters $a = 4.099 \text{ \AA}$ and $c = 9.922 \text{ \AA}$. Specific heat measurements display a large mean-field-like anomaly at $T_A = 0.7 \text{ K}$ followed by a large peak at $T_c = 0.5 \text{ K}$. According to the strong difference in their magnetic field dependence, these anomalies can be attributed to the transition into the magnetic A-phase and into the superconducting state, respectively. The large size of the anomalies proves that both transitions are bulk ones.

The properties of the single crystals and polycrystals are controlled by the ratio between Ce-, Si- and Cu-content in the starting melt. The relation between composition and physical properties has been well established by different groups [9–13]. Unfortunately, the corresponding changes in structural parameters are very small, at the accuracy limits of x-ray and microprobe techniques, which has so far prevented the establishment of a reliable direct correlation between structural parameters and physical properties.

Two different pressure runs are reported in this paper, referred to below as cells 1 (containing four samples from all sources) and 2 (with two A/S samples) using a Bridgman anvil cell technique with a quasi-hydrostatic steatite solid pressure medium. Pressure gradients are believed to be less than 5% of the total at the highest pressures, determined from the width of the superconducting transition of Pb manometer. The first cell (1) contained the two polycrystals 50 and 57 and two A/S type single crystals, oriented with the measurement current parallel to the c - and a -axes, and labelled ‘A/S $I \parallel c$ ’ and ‘A/S $I \parallel a$ ’ respectively. The current direction was perpendicular to the axis of the pressure cell, and we believe, though unfortunately are not certain, that the c -axis of sample ‘A/S $I \parallel a$ ’ was oriented parallel to the cell axis.

Any uniaxial stresses due to an imperfectly hydrostatic pressure medium are likely to be aligned with the cell axis, and the second pressure cell (2), figure 1, was designed to exploit this systematically. The cell contained two samples, cut from neighbouring positions on the same A/S type single-crystal source. They were both oriented so that the current flowed along the a direction, while the c -axis was oriented parallel and perpendicular to the cell axis. The samples have been labelled ($\sigma \parallel c$) and ($\sigma \perp c$) respectively. Multiple contacts on each sample enabled the resistivity within different regions of the same sample to be compared. As they shared their longest side in the original source crystal, variations in composition along the length of one sample should be comparable to that between samples.

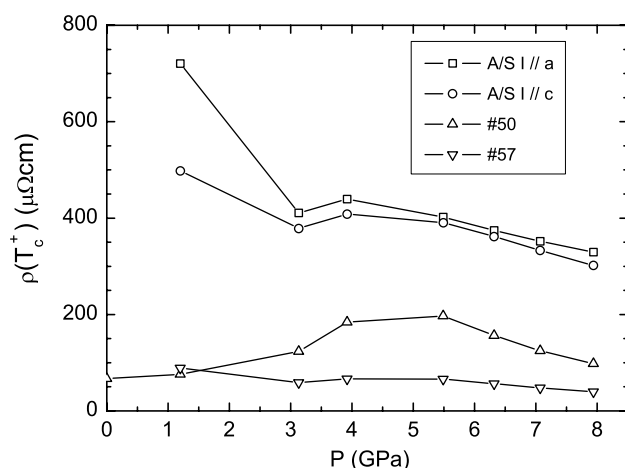


Figure 2. Normal state resistivity at T_c in cell 1, showing the enhancement of ρ_0 at P_v . Note that only sample 50 had full four-point contacts, so the others contain a monotonically decreasing additional contribution to the resistance, but the sample contribution is visible in all.

3. Results

The resistivity was first measured at $P = 0$; excellent agreement was found with previous results. Apart from the superconducting transition temperature T_c , several features of the normal state resistivity under pressure will be highlighted. These are the residual resistivity ρ_0 and A coefficient of the temperature dependence in the power law behaviour $\rho = \rho_0 + AT^n$ of the normal state at low temperature, and the position of the two crystal-field split resistivity maxima T_1^{\max} and T_2^{\max} . All of these show distinctive features at the valence instability pressure P_v .

The resistance has been normalized by the geometric factor of each sample measured on construction of the cell, which usually gives an absolute value within 10% for proper four-point measurements. Contrary to cell 2, which provided straightforward results, the gasket of cell 1 formed under too high a load, and due to migration of the contact wires, full four-point resistance measurements were possible in only one of the samples (no 50). The resistance measured for the other samples contained an additional series contribution, of varying size. It would be possible to subtract this, for example assuming a linear additional term and/or an adjustment of the geometric factor used to obtain the absolute resistivity, but the results obtained will then depend on these assumptions. We should add that obtaining even semi-quantitative values for the absolute resistivity at such high pressures is a challenging task, therefore we make no apology for trying to extract the maximum amount of information from what might be seen as an imperfect experiment.

In figure 2, one can easily see the enhancement of the residual resistivity at P_v in all samples in cell 1, superimposed in three cases on a monotonically decreasing additional series resistance. It is clear that the enhancement of the residual resistivity in sample 50 (the sample with four electrical contacts) is much larger than in the others, and reaches almost 200 $\mu\Omega\text{cm}$ close to 5 GPa. This sample also has a small negative magnetoresistance at 5.5 GPa, around 0.5% at 8 T and 4.2 K, in contrast to the other samples, which show a positive magnetoresistance. This is typical of CeCu_2Si_2 samples with very high residual resistivities at P_v , and perhaps due to the Zeeman shift of the f -level ϵ_f , moving the system away from the valence instability.

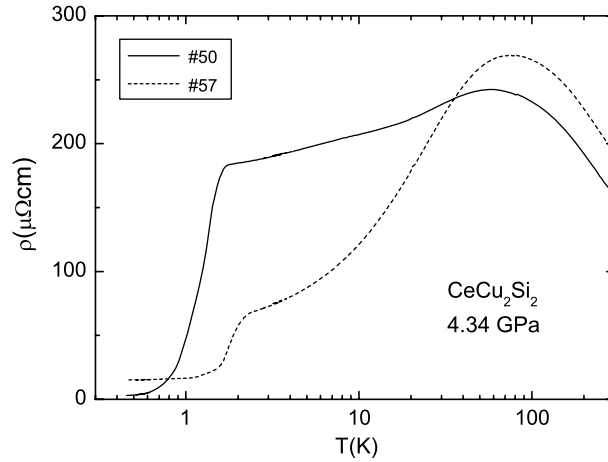


Figure 3. Resistivity of the ‘low T_c ’ (no 50) and ‘high T_c ’ (no 57, where ρ includes some additional contact resistance) samples in cell 1 at 4.34 GPa. Note the extremely large residual resistivity of no 50, yet a nearly complete superconducting transition is observed.

In figure 3, samples 50 and 57 are compared at 4.34 GPa, close to the maximum of ρ_0 . The raw data for sample 57 is shown, after normalization by a geometrical factor, to give a resistivity value. The small offset due to experimental difficulties is clear, but does not detract from the overall shape of the resistivity curve, which is typical of CeCu₂Si₂ at this pressure. In sample 50, however, there is evidently an enormously enhanced impurity contribution to the total resistivity, with a negative temperature dependence reminiscent of a Kondo impurity system, in addition to the usual scattering which increases up to $T^{\max} \sim T_K$. A negative slope becomes apparent at higher pressure where the A coefficient of the resistivity has collapsed and the impurity contribution dominates the resistivity. We should add that this sort of behaviour, where superconductivity coexists with very high residual resistivity, has also been observed in single crystals [16]. The existence of samples which superconduct at high pressure despite such enormous residual resistivities, of the order of the Ioffe–Regel limit (which is around $100 \mu\Omega \text{ cm}$ at ambient pressure), is strong evidence in favour of valence fluctuation mediated pairing. The common view is that unconventional superconductivity is incompatible with large electronic scattering rates. However, in this case both the superconductivity and the enhanced impurity scattering may share a common origin, as we believe that valence fluctuations are responsible for both the pairing interaction and the renormalization of impurity potentials [2, 8].

Figure 4 shows the onset temperature of the superconducting transition T_c^{onset} for the samples in cell 1. The most remarkable feature is that in three out of four samples the superconducting region extends up to a much higher pressure than seen in hydrostatic conditions. Only in sample ‘A/S $I \parallel a$ ’ was T_c^{onset} suppressed at high pressures similar to the result observed in the helium cell. The transitions at the highest pressures are partial, but there is a clear drop in resistance below T_c^{onset} . Very broad resistive transitions are intrinsic to CeCu₂Si₂ at high pressure, and have been identified as due to filamentary superconductivity rather than to pressure gradients [3]. The superconducting state at very high pressure may therefore be solely of a filamentary nature. The enhanced T_c^{onset} at very high pressure is a clear indication of the sensitivity of the high pressure superconducting state to anisotropy, either via the current direction, or anisotropic pressure conditions.

Cell 2 was designed to test the effect of anisotropic stress, in analogy to similar work on CePd₂Si₂ [14]. The two samples were aligned at right angles with respect to the cell axis.

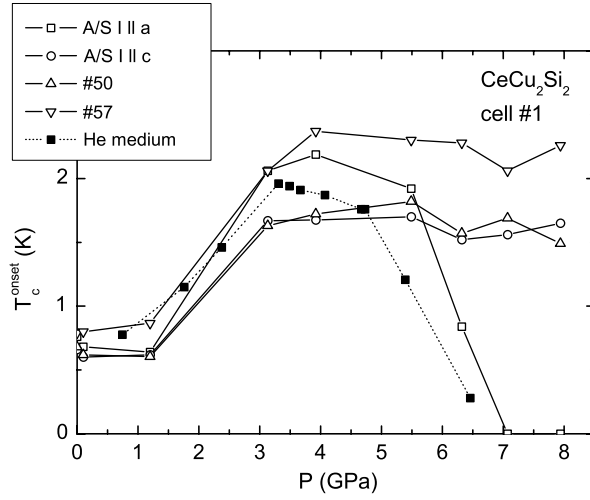


Figure 4. The superconducting pressure range is extended considerably in three out of four samples. Only sample ‘A/S // a’ showed the expected total disappearance of superconductivity above 7 GPa.

We might expect the slight uniaxial stress associated with the solid pressure medium to be oriented along this axis. The superconducting and normal state properties did indeed differ between the two samples in a way which corresponded to more than just a shift of the pressure scale, which might be expected if each sample was simply sampling part of an inhomogeneous pressure distribution. Figure 5 shows the onset T_c^{onset} , and completion $T_c^{R=0}$ temperatures of the resistive superconducting transition for samples ($\sigma \parallel c$) and ($\sigma \perp c$). $T_c^{R=0}$ has been previously identified with the bulk superconducting transition [3]. It is clear that the extension of T_c^{onset} above 6 GPa seen in cell 1 was not reproduced; indeed, both samples in cell 2 follow the pressure dependence of ‘A/S // a’ fairly closely. However, the difference in T_c between the two samples is an order of magnitude larger than within each. There is a slight enhancement of T_c in sample ($\sigma \parallel c$), and a very clear difference in the shape of the resistive transitions (see [17] for details). The lack of extension of the superconducting region to very high pressure in cell 2 does not entirely rule out uniaxial stress as the explanation for such behaviour in cell 1; the gasket instability in the latter probably leads to higher non-hydrostatic stresses inside the pressure cell.

The normal state properties provide a much clearer picture of the effect of uniaxial stress on CeCu_2Si_2 under pressure. This can be most simply described as a shift of the valence instability P_v to higher pressure in sample ($\sigma \perp c$) than in ($\sigma \parallel c$). We have previously identified P_v with several anomalies in the normal state resistivity, including the following.

- Merging of the crystal field split Kondo resistivity maxima T_1^{max} and T_2^{max} .
- A maximum in the residual resistivity ρ_0 .
- A sudden change in the A coefficient of the resistivity $\rho = \rho_0 + AT^2$, and a change of the $A \propto (T_1^{\text{max}})^{-2}$ scaling.

The shift of P_v is evident in all three properties, as shown in figures 6 and 7. In figure 6(a), one can see that T_1^{max} , proportional to the Kondo temperature T_K , rises faster in sample ($\sigma \parallel c$) than in ($\sigma \perp c$). T_2^{max} , which reflects the crystal field splitting Δ_{CEF} , is difficult to distinguish above the lowest pressure, but it has been shown to remain more or less constant [18]. The two crystal-field split resistivity peaks merge into a single maximum at different pressures in

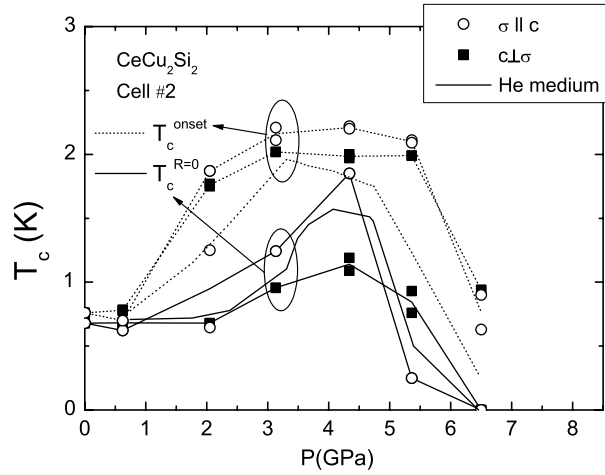


Figure 5. Onset T_c^{onset} (dotted lines), and completion $T_c^{R=0}$ (continuous lines) temperatures in cell 2 compared with helium cell results. Two results are shown for samples ($\sigma \parallel c$) (open circles) and ($\sigma \perp c$) (filled squares), taken from measurements using different contacts on the same sample. The differences in T_c between samples are significantly larger than local variations within a single sample.

the two samples, and this corresponds approximately to the ρ_0 maximum. The latter is shown in figure 6(b), where a Lorentzian peak can be fitted to the variation of ρ_0 . Note that the values of ρ_0 plotted in figure 6(b) are extracted from a power law fit to the resistivity above T_c , so the very low values at 0.6 GPa may be slightly anomalous. The presence of the A phase at ambient pressure also makes direct comparison difficult.

The shift of P_v determined above corresponds to a difference of around 0.5 GPa between ($\sigma \perp c$) and ($\sigma \parallel c$). This is confirmed when the A coefficient of the resistivity is examined (figure 7). The analysis is slightly complicated by the non-Fermi-liquid behaviour of the resistivity found in the entire region around P_c and P_v , where exponents $1 < n < 2$ are found in power law resistivity fits $\rho = \rho_0 + \dot{A}T^n$. However, in this case a reasonable comparison is still possible on a logarithmic scale, since there is a drop in \dot{A} of over an order of magnitude at P_v , and even without forcing a quadratic fit the variations in n can only contribute a maximum factor of two to the result. At higher pressure (above 6 GPa), the temperature dependent impurity scattering dominated the resistivity and made it impossible to obtain a reliable fit. The inset of figure 7, where \dot{A} is plotted against T_1^{max} , indicates the crossover from the strongly to weakly correlated branches of the Kadowaki–Woods plot [19, 20]. A comparison with the helium cell results from [3] (where a quadratic fit was forced) shows that the drop in \dot{A} around 5 GPa corresponds to the transition between the two regions where the expected $\dot{A} \propto (T_1^{\text{max}})^{-2}$ scaling is followed (indicated by solid lines). The result is clear. The two samples track each other up to 4.5 GPa, after which the drop in \dot{A} occurs more quickly in ($\sigma \parallel c$) than ($\sigma \perp c$), confirming that ($\sigma \parallel c$) has reached the valence transition at a lower pressure.

4. Discussion

Ishikawa attributed the differences at ambient pressure between ‘low T_c ’ and ‘high T_c ’ type CeCu₂Si₂ to different pairing symmetries [13]. Normal state and superconducting properties at ambient pressure differed considerably between the two categories of sample, which were located in adjacent regions in the homogeneity range [21, 22]. The ‘low T_c ’ samples were characterized by a slight excess of copper, and a higher residual resistivity. However, specific

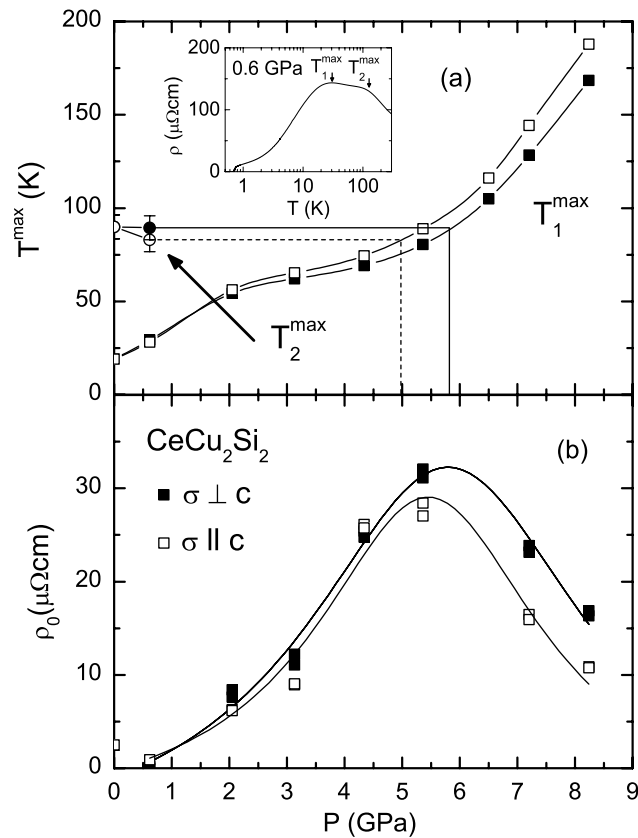


Figure 6. (a) The crystal field split Kondo resistivity maxima T_1^{\max} and T_2^{\max} (defined in inset) merge at a higher pressure in sample ($\sigma \perp c$) (filled symbols) than in ($\sigma \parallel c$) (empty symbols). The uncertainty on T_1^{\max} is smaller than the symbol size. (b) The residual resistivity maxima in the two samples corresponds approximately to the pressures determined in (a). P_v is therefore at a higher pressure in ($\sigma \perp c$) than in ($\sigma \parallel c$). The same symbols at a given pressure correspond to measurements from different contacts on the same sample.

heat measurements showed a more robust superconducting transition in the ‘low T_c ’ than the ‘high T_c ’ samples. Despite the difference in $\partial T_c / \partial P$ previously observed close to $P = 0$, the ‘low T_c ’ and ‘high T_c ’ samples both displayed the usual enhancement of T_c at high pressure. The most striking difference was seen in the residual resistivity, which reached huge levels in sample 50 around P_v . Given that both samples had very similar but not identical compositions, we might speculate on the origin of this. According to [13, 21, 22], there appeared to be no significant difference in crystal structure between the two categories of sample. The exact location of any atomic disorder may be highly significant, as the enhancement of impurity scattering is due to critical fluctuations [8, 23] and the effect may well depend significantly on the nature and location of the impurity itself, not only at P_v , but also P_c . The effect of disorder on T_c noted by Yuan *et al* [4] suggests that the initial reduction in T_c with pressure in the ‘low T_c ’ samples is due to such (possibly enhanced) impurity scattering, suppressing superconductivity between the two critical points.

The A/S type CeCu_2Si_2 shows a similar enhancement of T_c at high pressure to the sample measured in hydrostatic conditions. All other samples, including those of other types, have shown the enhancement of T_c when pressurized to over 3 GPa. Regarding both the properties

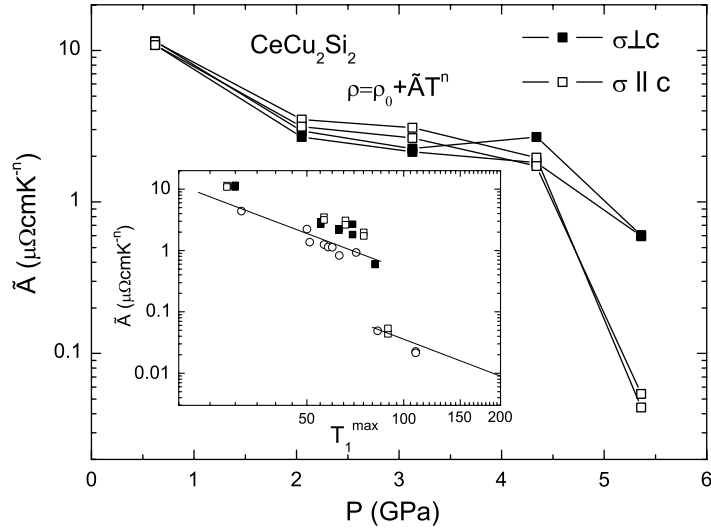


Figure 7. There is a drop in \tilde{A} of more than an order of magnitude at the valence transition, at different pressures for each sample. The inset shows the corresponding change of the $\tilde{A} \propto (T_1^{\text{max}})^{-2}$ scaling expected due to the Kadowaki–Woods relation (solid lines). He cell results (open circles) using a quadratic fit are included in the inset.

at ambient pressure and around P_v , the evidence so far seems to suggest only that the $A \rightarrow A/S \rightarrow S$ series represents a slight shift of the pressure scale. Thus any individual CeCu₂Si₂ sample can be classified by two or maybe three variables, corresponding to the aforementioned pressure shift, and to the impurity concentration (and perhaps also to the nature of the disorder).

Regarding the effect of uniaxial stress on CeCu₂Si₂, Monthoux and Lonzarich have shown that a more anisotropic structure leads to an increase in T_c for both magnetic and density fluctuation mediated pairing [24]. In the CeMIn₅ (where M is Co, Rh, or In) systems, and related Pu compounds, it has also been shown that T_c is strongly dependent on the ratio of the tetragonal lattice parameters c/a [25]. While the exact effect of the quasi-hydrostatic medium is hard to quantify, our observations are not inconsistent with these scenarios. This does not necessarily help to distinguish between spin and valence fluctuation mediated superconductivity, but it provides a constraint which must be satisfied by any more complete theoretical model. Further avenues for exploration from both a theoretical and experimental point of view come from the highly anisotropic resistivity, including some evidence that T_c , as defined by zero resistivity, may depend on current direction!

One clear result not so far mentioned is that the height of the resistivity peak at T_1^{max} is significantly larger for $I \parallel a$ than $I \parallel c$. This is not the case for the magnitude of $\rho(T_2^{\text{max}})$, reflecting the higher symmetry of the higher-lying crystal field split f states. For example, at ambient pressure the ratio $(\rho \parallel c)/(\rho \parallel a)$ is about 0.57 around 18 K and 0.95 near 100 K. The effect of the CEF on the anisotropy of the resistivity in CeCu₂Si₂ was analysed in [26].

Finally, we would like to emphasize again the importance of the coexistence of superconductivity and very high residual resistivity shown in figure 3. This is perhaps the most compelling evidence so far for a novel pairing mechanism in CeCu₂Si₂ at high pressure.

5. Conclusions

Resistivity measurements were carried out at high pressure on several different CeCu₂Si₂ samples, in the presence of small non-hydrostatic stress components. All samples showed

an enhancement of superconductivity around 2–3 GPa, and also an enhancement of residual resistivity with a maximum around 5 GPa. Complete resistive superconducting transitions can coexist with residual resistivities of the order of 150–200 $\mu\Omega$ cm around the valence instability close to 5 GPa. Individual CeCu₂Si₂ samples, usually classified at ambient pressure by the presence or otherwise of an ordered ‘A’ phase and/or superconductivity, belong to the same continuum, which can be traversed by pressure and/or Ge substitution. Disorder can also be used to classify samples, and the enhancement of residual resistivity under pressure probes this. It may be a fruitful avenue for future research to examine the effect of changing the nature of the impurities. Uniaxial stress was found to have a significant effect on both the normal and superconducting properties around the valence instability, which can be summarized by a shift of the valence instability pressure P_v .

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