Tuning the electronic hybridization in the heavy fermion cage compound YbFe$_2$Zn$_{20}$ with Cd doping

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Abstract

The tuning of the electronic properties of heavy fermion compounds by chemical substitution provides excellent opportunities for further understanding the physics of hybridized ions in crystal lattices. Here we present an investigation on the effects of Cd doping in flux-grown single crystals of the complex intermetallic cage compound YbFe$_2$Zn$_{20}$, which has been described as a heavy fermion with a Sommerfeld coefficient of 535 mJ mol$^{-1}$·K$^{-2}$. The substitution of Cd for Zn disturbs the system by expanding the unit cell and, in this case, the size of the Zn cages that surround the Yb and Fe. With an increasing amount of Cd, the hybridization between the Yb 4f electrons and the conduction electrons is weakened, as shown by a decrease in the Sommerfeld coefficient, which should be accompanied by a valence shift of the Yb$^{3+}$ due to the negative chemical pressure effect. This scenario is also supported by the low temperature DC magnetic susceptibility, which is gradually suppressed and shows an increment of the Kondo temperature, based on a shift to higher temperatures of the characteristic broad susceptibility peak. Furthermore, the DC resistivity decreases with the isoelectronic substitution of Cd for Zn, contrary to expectations in an increasingly disordered system, and implying that the valence shift is not related to charge carrier doping. The combined results demonstrate the excellent complementarity between positive physical pressure and negative chemical pressure, and point to a rich playground for exploring the physics and chemistry of strongly correlated electron systems in the general family of Zn$_{20}$ compounds, despite their structural complexity.

Keywords: valence shift, heavy fermions, chemical pressure

(Some figures may appear in colour only in the online journal)
These phenomena not only modify the transport but also the magnetic behavior at low temperatures, suppressing the susceptibility of the high-temperature paramagnetic state \[1\]. Further manifestations appear in heat capacity measurements from which the estimated Sommerfeld coefficient, which accounts for the total electronic density of states, takes an unusually high value that is associated with electron effective mass renormalization.

A few years ago, Torikachvili et al. characterized six new Yb-based heavy fermion intermetallic compounds \[8\] in the YbT₂Zn₂₀ family (T = Fe, Co, Ru, Rh, Os and Ir) with Sommerfeld coefficients \(\gamma > 400 \text{ mJ mol}^{-1} \cdot \text{K}^{-2}\). The RT₂Zn₂₀ family adopts a complex cubic Ce₃Cr₂Al₂₀-type structure with the space group \(Fd\bar{3}m\) \[9\]. R and T atoms occupy unique crystallographic sites (Wyckoff positions 8a and 16d, respectively) and the Zn atoms occupy three different crystallographic sites (96g, 48f and 16c). An important feature in this structure is that the R and T atoms are each surrounded by Zn ions as nearest neighbors, in the form of cages (16 and 12 Zn atoms respectively) that leave a shortest R–R spacing of \(r \approx 6 \text{ Å}\), as exemplified in figure 1.

The large cages made of numerous Zn atoms have the effect of isolating the rare earths from each other, and result in only weak crystal electric field (CEF) effects \[10\]. Studies on compounds of this family with non-hybridizing ions (R = Y, Gd) have demonstrated that the electronic bands near the Fermi level created by the transition metal and the Zn₂₀ complex are by their own merit already rich in physical phenomena. For example, radically different magnetic behavior is observed in the case of YCo₂Zn₂₀, which shows Pauli-like paramagnetic behavior (DC magnetic susceptibility almost independent of the temperature) compared to YFe₂Zn₂₀, reported as a compound close to the Stoner limit \[11\] in which the DC magnetic susceptibility is strongly dependent on the temperature. These different 'Fermi seas', in which the d-band filling is very important, also drive the general behavior of the equivalent compounds containing magnetic rare earths. As a striking example, one can find low-temperature antiferromagnetic order (\(T_N \approx 5.7 \text{ K}\)) in GdCo₂Zn₂₀ \[11\], contrasting with high-temperature ferromagnetic order (\(T_C \approx 86 \text{ K}\)) in GdFe₂Zn₂₀ \[12\].

In the case of GdCo₂Zn₂₀, we have recently reported that the Ruderman–Kittel–Kasuya–Yosida (RKKY) interaction can adequately explain the antiferromagnetic ordering of this compound \[13\], as is expected in this diluted rare-earth system without electron–electron correlations. For GdFe₂Zn₂₀, however, a model that can quantitatively explain the ferromagnetic order with such high \(T_C\) is still an open question \[11, 14\].

In the case of Yb-based compounds \[15\], the strong influence of the environment in which this hybridizing rare earth is embedded is also notable, such as the two heavy fermion compounds YbFe₂Zn₂₀ with \(\gamma \approx 520 \text{ mJ mol}^{-1} \cdot \text{K}^{-2}\) and YbCo₂Zn₂₀ with \(\gamma \approx 7900 \text{ mJ mol}^{-1} \cdot \text{K}^{-2}\) \[8, 16\].

It is well known that the substitution of one element for another in a compound is one of the most versatile and powerful tools by which the materials scientist can disturb the system in a controlled manner and subsequently track the evolution of its properties. One recent example in this family is the substitution of Al for Zn in the GdFe₂Zn₂₀ compound, which can be understood as a combination of positive chemical pressure plus electron doping \[17\], leading to a drastic reduction in ferromagnetic behavior. Chemical substitutions in the Yb-based members have not been reported yet, but hydrostatic pressures up to 8.23 GPa have been applied in the YbFe₂Zn₂₀ compound \[18\], driving the characteristic Kondo temperature \(T_K\) to lower temperatures and showing the loss of Fermi liquid behavior. A critical pressure of 9.8 GPa was inferred in order to reach the quantum critical point in this system, so it is expected to move towards the Yb\(^{3+}\) valence. Conversely, in the case of the YbCo₂Zn₂₀ compound \[19\], it has been suggested that the magnetic ordering transition occurs at a lower pressure, around 1 GPa. In order to attempt negative chemical pressure without electronic doping in this heavy fermion system, we have performed Cd doping in single crystals of YbFe₂Zn₂₀. Small amounts of Cd substitution for Zn do indeed expand the crystal lattice, as shown by x-ray diffraction, but more importantly, also produce notorious changes in the magnetic, thermodynamic and transport properties, complementing previous studies applying positive (physical) pressure. A Yb valence-shift scenario is then suggested as an explanation for the observed physical changes.

2. Experimental details

Single crystalline samples of YbFe₂Zn₂₀\(_{1-x}\)Cd\(_x\) (0 ≤ \(x\) ≤ 1.4) were grown from excess Zn using the standard self-flux method \[20, 21\]. The constituent elements were 99.99% Yb (Ames), 99.9% Co, 99.9% Fe, 99.9999% Zn and 99.9999% Cd (Alfa-Aesar). The initial ratios of the starting elements were \(1 : 2 : 47 : yy\) for the pseudo-quaternary system \(\text{Yb} : \text{Fe} : \text{Zn} : \text{Cd}\). The elemental mixtures were sealed in an evacuated quartz ampoule and heated in a box furnace. The crystals were grown by slowly cooling the melt between 1100 °C and 600 °C over 100h. At 600 °C the ampoules were removed from the furnace, inverted and placed in a centrifuge to spin off the excess flux. The separated crystals are typically polyhedral, ~3 mm or larger.

The effective Cd concentrations \(x\) of all of our samples were estimated using energy dispersive x-ray spectroscopy (EDS)...
The Cu-Kα powder x-ray diffraction pattern of YbFe2Zn20–xCd x (0 ≤ x ≤ 1.4). The left inset shows the evolution of the lattice parameter showing an expansion of the unit cell, also shown in the right inset with a shift to low angles of the diffraction pattern as a function of the Cd amount.

The temperature-dependent specific heat (Cp) was measured for all of our samples in a Quantum Design physical properties measurement system (PPMS) using the relaxation technique at zero field. The DC transport option of the same system is evolving towards a better Fermi liquid description. The Sommerfeld coefficients of the reference compounds YFe2Zn20 and LuFe2Zn20 [10] (γ ≈ 50 mJ mol−1 K−2) give an idea of the Yb effect in this system. Accordingly, the quasi-particle effective mass decreases from m∗ ≈ 764me (x = 0) to m∗ ≈ 607me (x = 1.4), from a simplified estimate using a parabolic heavy band with the keF of conventional metals like Au [22] (see table 1). The non-linearities make it difficult to extract the β parameter and Debye temperature ΘD from the standard method of fitting Cp/T = γ + βT2 in our results. In order to obtain a better estimation of the phonon contribution, in figure 3(b) we plot Cp − γT as a function of T3. Using ΘD ≈ (∆Cp/∆T)1/3 with ∆Cp = 6.02 × 10^23 mol−1 and Z = 184 as the number of atoms per unit cell, we obtain the corresponding Debye temperature for each concentration of Cd. A gradual decrease in ΘD as a function of the Cd amount is shown and summarized in table 1.

Figure 3 presents the T-dependence of the magnetic susceptibility of YbFe2Zn20–xCd x where a maximum is shown in all samples, which may be related to the screening of the localized moments by the CE or valence-intermediate behavior.

Figure 3. (a) The low-temperature behavior of Cp/T as a function of T3 for YbFe2Zn20–xCd x (0 ≤ x ≤ 1.4). (b) The estimation of the phonon contribution using a plot of Cp − γT as a function of T3.
This will be discussed further on. There is a progressive reduction in the maximum as a function of the Cd amount. A zoom at low temperatures, showing a shift to high temperatures of the position of the maximum as Cd increases.

This is seen at low temperatures, in agreement with previous studies [8, 18]. For the doped samples, the coherence peak broadens as the overall resistivity decreases strongly in the full range of temperatures. The inset shows the low-temperature behavior of the electrical resistivity as a function of $T^2$, showing Fermi liquid behavior $\rho = \rho_0 + AT^2$ at the lowest temperatures (solid lines).

4. Analysis and discussion

The combined set of figures and tables presented in the previous section make it evident that despite the limited amount of Cd ions that it was possible to introduce into the crystals (up to 7%), the substitutions resulted in significant changes in almost every measured physical property. Since Cd represents an isoelectronic substitution for Zn, it is reasonable to expect that the primary source of these changes is the crystalline lattice expansion demonstrated in figure 2.

Let us begin by discussing the specific heat results within the framework. The low-$T$ linear behavior of this framework. The low-$T$ linear behavior of $\gamma$ is nearly ferromagnetic, and very recent spectroscopic studies have detected small but discernible moments induced on the cage structure [11], which may be involved in the extra contribution to the effective moment. The obtained results are summarized in the table 2.

Figure 4 presents the $T$-dependent electrical resistivity data for our Cd-doped single crystals, which show metallic behavior. For the undoped sample a clear coherence shoulder is seen at low temperatures, in agreement with previous studies [8, 18]. For the doped samples, the coherence peak broadens as the overall resistivity decreases strongly in the full range of

Table 1. The Cd concentrations (x), Sommerfeld coefficient $\gamma$, quasiparticle effective mass $m^*$, beta parameter of the specific heat $\beta$, and Debye temperature $\Theta_D$ for the YbFe$_2$Zn$_{20-x}$Cd$_x$ system.

| Conc. | $\gamma$ | $m^*$ | $\beta$ | $\Theta_D$ |
|-------|---------|-------|--------|-----------|
| Cd    | (mJ mol$^{-1}$·K$^{-4}$) | (mJ mol$^{-1}$·K$^{-4}$) | (K) | (K) |
| 0     | 535(5)  | 764   | 2.99(1) | 494(5)    |
| 1.0   | 498(3)  | 712   | 3.32(1) | 475(5)    |
| 1.3   | 430(3)  | 614   | 3.94(1) | 452(5)    |
| 1.4   | 425(3)  | 607   | 4.10(1) | 442(5)    |

Table 2. The Cd concentrations (x), Pauli-like susceptibility ($\chi_0$), effective moment ($\mu_{\text{eff}}$) and magnetic susceptibility at $T = 0$ ($\chi(T = 0)$) for the YbFe$_2$Zn$_{20-x}$Cd$_x$ system.

| Conc. | $\chi_0$ | $\mu_{\text{eff}}$ | $\chi(T = 0)$ |
|-------|---------|-------------------|---------------|
| Cd    | (emu mol$^{-1}$) | (\mu$_{\text{B}}$ f.u.$^{-1}$) | (emu mol$^{-1}$) |
| 0     | 0.0055(4) | 4.9(1)           | 0.074(2)      |
| 1.0   | 0.0037(4) | 5.1(1)           | 0.066(2)      |
| 1.3   | 0.0109(5) | 5.1(1)           | 0.060(2)      |
| 1.4   | 0.0086(4) | 5.1(1)           | 0.056(2)      |
4f electrons, a weaker f-CE coupling strength ($J_{ff}$), and eventually to a magnetically ordered ground state governed by the RKKY interaction. Conversely, 'negative pressures', as represented by the present Cd doping, allow the 4f$^{14}$ state to decrease in energy, towards 4f$^{13}$ and through the Fermi level, which increases $J_{eff}$ and favors a Kondo-screened Fermi liquid ground state while hybridization is present, then eventually leads to a non-magnetic (closed-shell) divalent ground state for Yb. In the whole intermediate valence range, the high degree of 4f-CE hybridization results in an enhanced density of states (DOS) around the Fermi level when compared to the end states (Yb$^{3+}$ or Yb$^{2+}$), so enhanced values of $\gamma$ are expected to eventually decrease in either direction towards the more typical values of common metals.

In terms of the phonon contribution to the specific heat, and regarding the Debye temperature $\Theta_D$ as that at which the RKKY interaction. Conversely, the highest frequency phonon modes are excited, figure 3(b) implies that Cd substitution for Zn decreases the stiffness of the cage system. In principle, the Cd atoms may enter any of the Zn sites (16c, 48f or 96g) and there should indeed be a disordered occupation of all three. However, for LaRu$_2$Zn$_{20}$ it was recently reported that the Zn ions at the 16c site exhibit low-energy, localized vibration modes (rattling) [25]. This is indicative of the ‘loose’ Zn ions at this site due to the presence of extra space and consequently broadened Madelung potentials. If so, the larger Cd atoms may be expected to occupy this site preferentially, and the small decrease in the value of $\Theta_D$ is due to the greater atomic mass of Cd. This preferential occupation of a single site may be partly behind the reasons why our crystal flux growths have shown a successful but limited effect in introducing Cd into the YbFe$_2$Zn$_{20}$, whose lattice parameter (figure 2) is significantly smaller than the $a = 14.4263(2)$ of LaRu$_2$Zn$_{20}$. High-resolution diffraction experiments will be required to confirm these expectations.

DC magnetic susceptibility measurements (figure 4) show a broad maximum that is typical of valence fluctuating systems, but can also be related to the Kondo physics [26]. Well-defined valence fluctuating systems are usually characterized by moderately large values of $\gamma$ (50–100 mJ mol$^{-1}$ K$^{-2}$) as in the cases of Yb$_2$TGe$_6$ [27] and Yb$_2$Ni$_{12}$As$_7$ [28]; Curie–Weiss behavior limited to much higher temperatures (CeSn$_3$ [29], CeRu$_2$ [30]); effective Yb valence values well shifted away from 3+ (Yb$_2$Ni$_{12}$As$_7$ [28], YbNiGe$_3$ [31]) and a maximum of the magnetic susceptibility at high temperatures associated with the spin-fluctuation temperature $T_{sd}$ [32], as in the case of CeRu$_2$ [30] and Yb$_2$Ni$_{12}$As$_7$ [28]. Conversely, well-defined heavy fermion systems have significantly higher values of $\gamma$ (YbNiSi$_3$ [33], YbT$_2$Zn$_{20}$ [8]), Curie–Weiss behavior extending to lower temperatures (YbNiSi$_3$ [33], Yb$_2$Ni$_3$P$_2$ [28], YbT$_2$Zn$_{20}$ [8]), Kondo-type interaction (Yb$_2$Ni$_3$P$_2$ [28]) present at low temperatures, and an effective Yb valence very close to 3+ (Yb$_2$Ni$_3$P$_2$ [28], YbNiSi$_3$ [31, 33]). The crossover region between these two regimes does not have a well-defined limit, so shifts in the maximum of the magnetic susceptibility for such crossover systems can be indicative of shifts in both the Kondo-type interaction ($T_K$) and the spin-fluctuation ($T_{sd}$). Microscopic techniques such as XAS are required in order to quantitatively follow effective Yb valence shifts as a function of Cd doping and temperature.

Figure 4 demonstrates the effects of Cd substitution in the system’s magnetic response. Increasing the Cd content results in a reduction of the magnetic susceptibility at $T = 0$ ($\chi(T = 0)$). At high temperatures, the system behaves as a paramagnet with an effective magnetic moment that comes mostly from the Yb 4f electrons, although the somewhat enhanced values in comparison to the Yb$^{3+}$ effective moment (see table 2) point to additional magnetic contributions from the Fe and/or Zn atoms [23]. At low temperatures, the screening of the magnetic moments by the Ce causes a suppression of the magnetic response, leaving a Pauli-like enhanced magnetic susceptibility at $\chi(T = 0)$. For comparison, elemental Pd is a transition metal with d-electrons that are on the verge of magnetism, and features $\chi(T = 0) = 0.75 \times 10^{-3}$ emu mol$^{-1}$ [1], which is still two orders of magnitude smaller than the obtained values summarized in table 2 for these heavy fermion compounds. In this limit we have an enhanced magnetic susceptibility $\chi(T = 0)$ that gives information about the electron correlations in the system, when compared with the Sommerfeld coefficient obtained in the specific heat measurements.

With this in mind, we calculate the Wilson ratios $R_W = \chi/\gamma$ and the results (see table 3) show that Cd substitution slightly reduces this parameter from 1.3 in the ternary to 1.1 in the sample with the highest amount of Cd, in agreement with a reduction of the electron–electron correlation from the point of view of the Landau parameter [1]. Thus, a shift in the direction from non-magnetic to magnetic response in the YbFe$_2$Zn$_{20}$–Cd$_x$ is supported by the DC magnetic susceptibility measurements, whose maximum value also decreases as a function of the Cd content.

We note that although there is a strong reduction in both $\chi(T = 0)$ and $\gamma$ with the Cd substitution, there is only a slight decrease in the Wilson ratio. This means that it is possible to observe a reduction of hybridization in both the specific heat and magnetic susceptibility measurements. Theoretical investigations of the pressure effects on Yb-based compounds [34] show that the linear coefficient of the specific heat and Pauli-like magnetic susceptibility increase with positive pressure, which is therefore consistent with the idea of negative pressure effects on Yb inside the Zn cage supported by our experimental results.

The inset of figure 4 highlights a concomitant rise in the temperature associated with the broad maximum in magnetic susceptibility, from 12 K for the ternary to 17 K for the highest

| Conc. | $\rho_0$ ($\mu\Omega \cdot \text{cm}$) | $A \times 10^{-1}$ | $R_W$ |
|-------|----------------|------------------|--------|
| $x$   | ($\mu\Omega \cdot \text{cm}$) | ($\mu\Omega \cdot \text{cm} \cdot \text{K}^{-2}$) |        |
| 0     | 8.4(3)          | 1.30(3)          | 1.3(1) |
| 1.0   | 7.1(2)          | 0.40(4)          | 1.1(1) |
| 1.3   | 2.0(2)          | 0.07(1)          | 1.1(1) |
| 1.4   | 1.0(2)          | 0.02(1)          | 1.1(1) |
Cd-doped sample. Since this broad maximum temperature is associated with the Kondo temperature of the system, we can state a priori that $T_K$ increases with Cd doping (in agreement with the Doniach diagram for negative pressure in the Yb ion \cite{24}), as will be shown further on.

The Kondo physics behind this varying hybridization system can also be discussed based on the transport measurements (figure 5). The Cd doping has a strong effect on the transport properties, as can be seen in the reduction of the residual resistivity $\rho_0$ in the slope of the linear regime at low temperatures in the $\rho$ versus $T^2$ plot (associated with the A parameter, see inset), and also in the behavior at high temperatures.

The reported data of the hydrostatic pressure in YbFe$_2$Zn$_{20}$ showed that residual resistivity increases as a function of pressure \cite{18} and is in agreement with the reduction of $\rho_0$ for negative pressures (see table 3). The inset of figure 5 shows that the A parameter, which is proportional to the density of states at the Fermi level \cite{35}, is reduced with Cd substitution, in agreement with the heat capacity and DC magnetic susceptibility.

In order to follow the evolution of the A parameter as a function of pressure, we must estimate the values of pressure that correspond to the expansion of the lattice parameter. This was achieved by adopting a representative value of 160 GPa for the bulk modulus of this family of compounds \cite{36} and using Murnaghan’s equation of state \cite{37}. Figure 6 shows the overall evolution of the A parameter from high positive physical pressures (adapted from \cite{18}) to our negative chemical pressure estimates. There is a very good match with the two independent techniques, and the trend demonstrates a complete recovery of the Fermi liquid behavior at negative pressures.

Lastly, we address the broadening of the coherence shoulder at low temperatures ($T \leq 25$ K) with the Cd substitution and a reduction of the slope of the linear regime at high temperatures in the main panel of figure 5 ($\rho$ versus $T$), associated with a general reduction of electron–phonon scattering in the entire temperature range.

The influence of the hybridized Yb ion in this unusual behavior can be better understood by comparing this magnetic system with its ‘non-magnetic’ reference compound LuFe$_2$Zn$_{20}$ (here we are neglecting any minor magnetic scattering from Fe and Zn). Figure 7 shows the magnetic contribution to the resistivity ($\rho_{\text{Yb}} - \rho_{\text{Lu}}$) of our YbFe$_2$Zn$_{20-x}$Cd$_x$ crystals, obtained by subtracting the resistivity of LuFe$_2$Zn$_{20}$ \cite{18}) from those in figure 5. The undoped magnetic system rapidly loses the coherent peak as Cd is introduced and approaches the behavior of the reference compound. As argued previously, the system is going in the direction of non-magnetic response, supporting the idea of a valence-shift of the Yb ion.

A final piece of valuable information can be extracted from these resistivity measurements. The Kadowaki–Woods ratio \cite{38} \((KWR = A/\gamma^2)\) for each Cd concentration has been calculated and summarized in table 4. Before the Kondo screening takes place, the ground state degeneracy of the Yb 4f levels due to crystalline electric field splitting plays an important role in the definition of the value of the Kondo temperature. In agreement with the generalized Kadowaki–Woods plot \cite{8, 35}, our obtained values suggest that in the ternary (YbFe$_2$Zn$_{20}$) the Yb ion displays $N = 6$ degeneracy in the ground state, but with Cd substitution the system goes toward $N = 8$ degeneracy. With this information, and using \cite{8} $T_K = (R \ln N)\gamma/\gamma$, we can estimate the evolution of the Kondo temperature, presented in table 4.

As previously anticipated by the position of the maximum in DC magnetic susceptibility (inset figure 4), the Kondo
temperature increases as a function of the Cd substitution. Once again, the Cd doping effect is seen to move the system towards a non-magnetic state, supporting the idea of a valence shift towards divalent Yb.

5. Conclusions

An Yb valence shift due to the negative chemical pressure effect resultant from the Cd doping of the heavy fermion YbFe 2 Zn 30 has been shown, supported by strong changes observed in XRD, heat capacity, magnetic susceptibility and resistivity measurements as a function of temperature on samples with an increasing Cd content (up to 7%). The negative chemical pressure drives the Kondo temperature of the system to higher values, and the regime of the Fermi liquid behavior is extended, which agrees with and complements previous work applying positive physical pressure. The results demonstrate the high efficiency of the Cd doping in this type of system, towards the tuning of the RKKY and the Kondo interaction strengths. We expect that this work will provide key reference elements to help understand the behavior of related heavy fermions in the same family, such as YbCo 2 Zn 30, in which much stronger 4f-CE hybridization is found.

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