

ESR of Gd^{3+} in the intermediate-valence $YbInCu_4$ and its reference compound $YInCu_4$

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Electron-spin-resonance (ESR) experiments on Gd^{3+} in the intermediate-valence phase ($T < T_v$) of $YbInCu_4$ and in its reference compound $YInCu_4$ are interpreted in terms of an enhanced density of states at the Fermi level for the Yb-based compound. The Korringa rate and g shift measured in ESR and the susceptibility data allowed us to extract the electron-electron exchange enhancement factor α for the Yb-based compound. The exchange interaction between the Gd^{3+} local moments and the conduction electrons ($c-e$) is $c-e$ wave-vector dependent in both compounds. [S0163-1829(97)00701-7]

I. INTRODUCTION

Many rare-earth intermetallic compounds present interesting physical properties associated with the hybridization between localized f -electron states and conduction electrons ($c-e$). This has motivated experimentalists and theoreticians for the last several decades to study strongly correlated electron phenomena in intermediate-valence (IV) and heavy-fermion (HF) systems.¹ The Ce- and Yb-based compounds are particularly well suited for these studies, since the $4f$ shell of Ce and Yb can contribute, at most, one electron or hole to the conduction band, respectively, simplifying the theoretical analysis. The cubic $AuBe_5$ ($C15b, F43m$)-type structure² of the $YbInCu_4$ compound, is particularly interesting due to the first-order isostructural volume expansion phase transition found at $T_v \approx 50$ K.³ Extensive studies⁴ of susceptibility, specific heat, resistivity, Yb's Mossbauer, lattice parameter, L_{III} x-ray absorption, and NMR (Refs. 5 and 6) are consistent with a 0.45% volume change² and a Yb valence change from $z \approx 2.9$ at high temperatures to $z \approx 2.8$ at lower temperature.⁴ This property characterizes this compound as an intermediate f -ion valence system at low temperatures. To further study the electronic properties of this compound, we have measured the low-temperature electron-spin resonance (ESR) of Gd^{3+} in $YbInCu_4$ ($T \lesssim 50$ K) and in its reference compound $YInCu_4$ ($T \lesssim 100$ K). Susceptibility and specific-heat measurements were also performed.

While our work was in progress, a paper by Altshuler *et al.*⁷ on Gd^{3+} in $YbInCu_4$ was published. They have analyzed their ESR data by focusing on the exchange coupling between the Gd^{3+} local moment and the "quasilocalized Yb^{3+} moment." Thus, their analysis is mainly concentrated on the high-temperature data ($T > 50$ K). Their results resemble those found for Gd^{3+} impurities in a stable-valence weak-magnetic metallic^{8,9} or insulator^{10,11} hosts. In such

compounds the observed temperature-dependent g shifts and rapid broadening of the linewidths are associated, respectively, with the temperature dependence of the host magnetic susceptibility and with the relaxation via excited crystal-field levels.⁸⁻¹¹ The magnetic moment of the Yb^{3+} ions, for the $YbInCu_4$ compound at $T \geq T_v$, is well defined. For $T \geq T_v$ the compound behaves as a magnetic host with well established crystal-field levels for the free Yb^{3+} ion ground state $^2F_{7/2}$.^{4,12} Thus, exchange and/or direct magnetic interactions result in a shift and broadening of the Gd^{3+} resonance. Therefore, an analysis of ESR data in a magnetic host should be carried out keeping in mind that a shift and a broadening of the resonance may have a variety of origins. Although our data in $YbInCu_4$ agree with those of Altshuler *et al.*,⁷ the aim of our work and analysis has been focused on the temperature region where the ESR of Gd^{3+} actually probes the IV state of $YbInCu_4$ ($T < T_v$). In this case it is important to use a reference compound that allows the extraction of the various intrinsic parameters. For this reason, we have also measured the ESR and susceptibility of Gd^{3+} in $YInCu_4$ and specific heat of $YInCu_4$.

II. EXPERIMENTAL DETAILS

Single crystals of $(Yb,Y)_{1-x}Gd_xInCu_4$ ($0.0005 \leq x \leq 0.002$ nominal) were grown from a flux of excess $InCu$ by the method described elsewhere.¹³ The crystals were of cubiclike shape with typical sizes of $4 \times 3 \times 1$ mm³. For the high-temperature ESR measurements, powdered crystals were used in order to increase the ESR signal-to-noise ratio. The ESR experiments were carried out in a Varian E-line X-band spectrometer, using a liquid-helium tail dewar (1.7–4.15 K) and a helium gas flux (7–100 K) adapted to a room-temperature TE₁₀₂ cavity. The susceptibility measurements

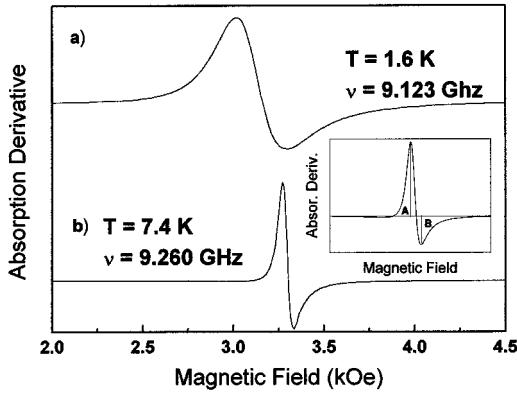


FIG. 1. ESR powder spectra of (a) 0.18% of Gd^{3+} in YbInCu_4 and (b) 0.25% of Gd^{3+} in YInCu_4 . The inset shows the definition of A and B for a Dysonian line shape.

were made in a Quantum Design dc superconducting quantum interference device magnetometer. Specific-heat measurements were performed in a small-mass calorimeter system that employs a quasiadiabatic thermal relaxation technique.¹⁴ Samples employed here ranged in mass from 45 to 145 mg.

III. EXPERIMENTAL RESULTS

Figure 1 shows the ESR powder spectra of Gd^{3+} ($\approx 0.2\%$) in YbInCu_4 and YInCu_4 measured at $T \leq 7$ K. Typical Dysonian line shapes¹⁵ with $A/B \approx 2.2(2)$ were observed. This type of line shape is characteristic of localized magnetic moments in a lattice with a skin depth smaller than the size of the sample particles. For YbInCu_4 the Gd^{3+} resonance shows a decreasing A/B ratio as the temperature increases, in agreement with the increase of the resistivity observed for this compound.^{4,16} The g value and linewidth were obtained using the method of Peter *et al.*¹⁷ Figure 2 gives the temperature dependence of the linewidth for both compounds. For the Yb-based system for $T > 30$ K, an increase in the broadening of the linewidth and of the g value were observed. A departure from a linear broadening is seen in Fig. 2 and is in agreement with recently published data on

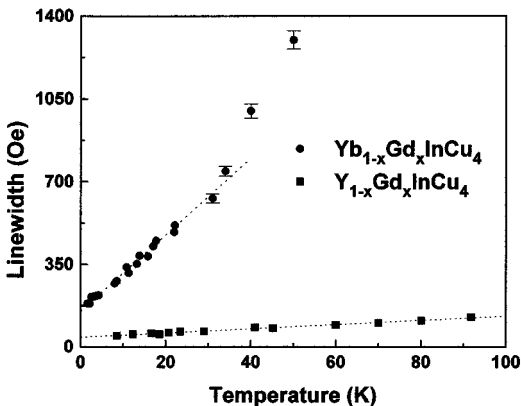


FIG. 2. Temperature dependence of the ESR linewidth for 0.18% of Gd^{3+} in YbInCu_4 (circles) and 0.25% of Gd^{3+} in YInCu_4 (squares). The dashed line is the best fit to $\Delta H = a + bT$.

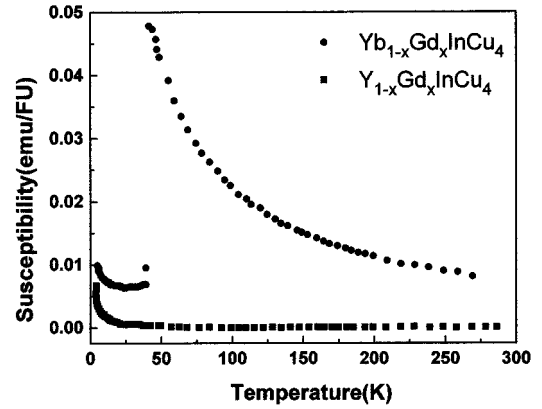


FIG. 3. Magnetic susceptibility as a function of temperature, measured at 1 kOe for 0.18% of Gd^{3+} in YbInCu_4 (circles) and 0.25% of Gd^{3+} in YInCu_4 (squares).

$\text{Yb}_{1-x}\text{Gd}_x\text{InCu}_4$.⁷ The linear dependence of the linewidth was fitted to the expression $\Delta H = a + bT$, with $a = 150(20)$ Oe, $b = 16(1)$ Oe/K and $a = 42(5)$ Oe, $b = 0.9(1)$ Oe/K for Gd^{3+} in YbInCu_4 and YInCu_4 , respectively. Within the accuracy of the measurements, the g values were found to be temperature independent for $T < 30$ K. The measured low-temperature ($T < 7$ K) g values were 2.073(8) and 2.004(2) for the Yb- and Y-based compounds, respectively. For the low concentration samples ($\approx 0.05\%$) we measured similar values, within our experimental error. In single crystals the Gd^{3+} resonance did not show crystal-field features, i.e., fine structure and/or anisotropic linewidth.

Figure 3 shows the magnetic susceptibility, corrected for the compound core-diamagnetism, for the samples used in our ESR experiments. From the low-temperature tail ($T > 45$ K) we estimate the Gd concentration to be 0.18(2)% and 0.25(2)% for the $\text{Yb}_{1-x}\text{Gd}_x\text{InCu}_4$ and $\text{Y}_{1-x}\text{Gd}_x\text{InCu}_4$ compounds, respectively. From the high-temperature ($T > 50$ K) susceptibility of the $\text{Yb}_{1-x}\text{Gd}_x\text{InCu}_4$ we obtain $4.24(10)\mu_B/\text{Yb}$, in good agreement with previous reports,³⁻⁶ and close to the $4.54\mu_B/\text{Yb}$ expected for $\text{Yb}^{3+}(4f^{13}, ^2F_{7/2})$. From the low-temperature data ($T < 45$ K) a temperature-independent contribution of $6(1) \times 10^{-3}$ emu/FU was estimated, in agreement with previous measurements.³⁻⁶

In Fig. 4 we present specific-heat measurements for the YInCu_4 compound in the temperature range between 2 K $< T < 20$ K. The low-temperature C/T data increase linearly with T^2 as seen in the inset of Fig. 4. The fitting parameters obtained from these data are $\gamma = 1.63(6)$ mJ/mol K^2 and $\beta = 0.327(2)$ mJ/mol K^4 . A Debye temperature $\theta_D = 330(5)$ K is also obtained.

In Table I we summarize the experimental parameters derived in the present work, and by other groups, for the YbInCu_4 and YInCu_4 compounds.

IV. ANALYSIS AND DISCUSSION

In the simplest treatment of the exchange interaction, $J_{fs}\mathbf{S}\cdot\mathbf{s}$, between a localized $4f$ electron spin (\mathbf{S}) on a solute atom (Gd^{3+}) and the free $c-e$'s spin (\mathbf{s}) of the host metal, the ESR g shift (Knight shift)¹⁸ and the thermal broadening of the linewidth (Korringa rate),¹⁹ when “bottleneck” and

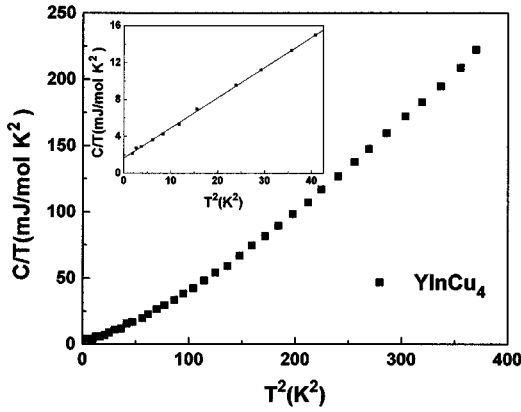


FIG. 4. Specific heat (C/T) as a function of T^2 for YInCu_4 . The inset shows the low temperature T^2 dependence of C/T . The solid line is the best fit to $C/T = \gamma + \beta T^2$, with $\gamma = 1.63(6) \text{ mJ/mol K}^2$ and $\beta = 0.327(2) \text{ mJ/mol K}^4$.

“dynamic” effects are not present,²⁰ can be written as

$$\Delta g = J_{fs} \eta(E_F), \quad (1)$$

and

$$\frac{d(\Delta H)}{dT} = \frac{\pi k}{g \mu_B} J_{fs}^2 \eta^2(E_F), \quad (2)$$

where J_{fs} is the effective exchange interaction between the Gd^{3+} local moment and the $c-e$ in the absence of $c-e$ momentum transfer,²¹ $\eta(E_F)$ is the “bare” density of states for one spin direction at the Fermi surface, k is the Boltzman constant, μ_B is the Bohr magneton, and g is the Gd^{3+} g value.

Equations (1) and (2) are normally used in the analysis of ESR data for highly diluted rare-earths magnetic moments in intermetallic compounds with appreciable residual resistivity, i.e., large $c-e$ spin-flip scattering. In our case the ESR parameters are found to be independent of the concentration.²⁰ Hence, it is expected that the following relation would hold:

$$\frac{d(\Delta H)}{dT} = \frac{\pi k}{g \mu_B} (\Delta g)^2. \quad (3)$$

Using the g value of Gd^{3+} in insulators as $1.993(2)$,²² ($\pi k/g \mu_B = 2.34 \times 10^4 \text{ Oe/K}$), the measured g shifts and the thermal broadening of the linewidths b for the Gd^{3+} resonance in YbInCu_4 and YInCu_4 , we found (i) that for YbInCu_4 , when replacing $\Delta g \approx 0.08(1)$ in Eq. (3), a thermal broadening of $b \approx 150(40) \text{ Oe/K}$ is obtained. That value is much larger than the one measured, $b \approx 16(1) \text{ Oe/K}$, and (ii)

for YInCu_4 , when replacing $\Delta g \approx 0.011(4)$ in Eq. (3), we calculated a thermal broadening of $b \approx 3(2) \text{ Oe/K}$. That value is also larger than the one measured, $b \approx 0.9(1) \text{ Oe/K}$. Therefore, we conclude that the approximations made in Eqs. (1) and (2) are not valid for either compound, and conduction electron-electron correlations^{23,24} and \mathbf{q} -dependent exchange interaction, $J_{fs}(\mathbf{q})$,²¹ must be considered in the analysis of our ESR data. $J_{fs}(\mathbf{q})$ is the Fourier transform of the spatially varying exchange. In our analysis we will only consider the contribution from a single $c-e$ band, because the measured thermal broadenings of the linewidths were found to be much smaller than those expected for the measured g shifts.^{25,26}

As mentioned above the electronic contribution to the heat capacity for the YInCu_4 compound yields a $\gamma = 1.63(6) \text{ mJ/mol K}^2$. Assuming a free $c-e$ gas model for YInCu_4 , $\gamma = (2/3) \pi k^2 \eta(E_F)$, we calculate a density of states at the Fermi level, $\eta(E_F) = 0.34(2) \text{ states/eV mol spin}$. For this density of states, one would expect an electronic-spin susceptibility, $\chi_e = 2 \mu_B \eta(E_F)$, of $\approx 0.03 \times 10^{-3} \text{ emu/FU}$. That is of the order of the “background” susceptibility (corrected for the core diamagnetism) measured at high temperatures for this compound (see Fig. 3). Hence, one can assume that electron-electron correlations are not important in YInCu_4 . Taking into account only the wave-vector dependence of the exchange interaction, $J_{fs}(\mathbf{q})$,²¹ in Eqs. (1) and (2) the exchange parameters should be replaced by $J_{fs}(\mathbf{0})$ and $\langle J_{fs}^2(\mathbf{q}) \rangle$, respectively. At the Gd^{3+} site the g shift probes the $c-e$ polarization ($\mathbf{q} = 0$) and the Korringa rate the $c-e$ momentum transfer ($0 \leq \mathbf{q} \leq 2k_F$) averaged over the Fermi surface.²¹ Using $\eta(E_F) = 0.34(2) \text{ states/eV mol spin}$, $\Delta g = 0.011(4)$, and $b = 0.9(1) \text{ Oe/K}$, we found the exchange parameters between the Gd^{3+} local moment and the $c-e$ in YInCu_4 to be $J_{fs}(\mathbf{0}) = 32(10) \text{ meV}$ and $\langle J_{fs}^2(\mathbf{q}) \rangle^{1/2} = 18(5) \text{ meV}$.

For YbInCu_4 the low-temperature linear part of the heat capacity gives an electronic contribution of $\gamma = 50(5) \text{ mJ/mol K}^2$.^{4,6,16} For a free $c-e$ gas we obtained $\eta(E_F) = 10(1) \text{ states/eV mol spin}$, about 30 times larger than that found for YInCu_4 . For this density of states, we obtained a $c-e$ spin susceptibility, $\chi_e = 2 \mu_B \eta(E_F)$, of $\approx 0.7 \times 10^{-3} \text{ emu/FU}$. This is one order of magnitude smaller than the temperature-independent part of the susceptibility measured in this compound for $T < 45 \text{ K}$, $6(1) \times 10^{-3} \text{ emu/FU}$. This suggests that a strong electron-electron exchange enhancement contributes to the $c-e$ spin susceptibility in YbInCu_4 below its valence transition. It is known, that in the presence of such an electron-electron exchange enhancement, the host metal $c-e$ spin susceptibility can be approximated by^{23,24}

TABLE I. Experimental parameters for $\text{Gd}:(\text{Yb},\text{Y})\text{InCu}_4$.

	g	a Oe	b Oe/K	c %	γ mJ mol K ²	β mJ mol K ⁴
$\text{Yb}(\text{Gd})\text{InCu}_4$	2.073(8)	150(20)	16(1)	0.18(2)	50(5) ^a	0.33(4) ^a
$\text{Y}(\text{Gd})\text{InCu}_4$	2.004(2)	42(5)	0.9(1)	0.25(2)	1.63(6)	0.327(2)

^aSee Refs. 4, 6, and 16.

TABLE II. Extracted parameters for Gd:(Yb,Y)InCu₄.

	$\eta(E_F)$ states eV mol spin	θ_D K	$J_{fs}(\mathbf{0})$ meV	$\langle J_{fs}^2(\mathbf{q}) \rangle^{1/2}$ meV	$K(\alpha)$	α
Yb(Gd)InCu ₄	10(1) ^a	≈ 276 ^a	2.4(1.5)	1.3(1.0)	0.36(20)	0.7(2)
Y(Gd)InCu ₄	0.34(2)	330(5)	32(10)	18(5)	≈ 1	≈ 0

^aSee Refs. 4, 6, and 16.

$$\chi_e = 2\mu_B^2 \frac{\eta(E_F)}{1-\alpha}, \quad (4)$$

where α accounts for the electron-electron interaction, $(1-\alpha)^{-1}$ is the Stoner enhancement factor, and $\eta(E_F)$ is the *bare* density of states for one spin direction at the Fermi level obtained from specific-heat experiments. An upper limit for α of ≈ 0.9 is estimated assuming that the temperature-independent part of the susceptibility, $6(1) \times 10^{-3}$ emu/FU, is only due to an enhanced *c-e* spin susceptibility.

In the presence of electron-electron exchange enhancement and a \mathbf{q} dependence of the exchange interaction, $J_{fs}(\mathbf{q})$, the g shift [Eq. (1)] and the thermal broadening of the linewidth [Eq. (2)] may be rewritten as^{20,27}

$$\Delta g = J_{fs}(\mathbf{0}) \frac{\eta(E_F)}{1-\alpha}, \quad (5)$$

and

$$\frac{d(\Delta H)}{dT} = \frac{\pi k}{g\mu_B} \langle J_{fs}^2(\mathbf{q}) \rangle \eta^2(E_F) \frac{K(\alpha)}{(1-\alpha)^2}, \quad (6)$$

where $K(\alpha)$ is the Korringa exchange enhancement factor.^{28,29} Then Eq. (3) becomes

$$\frac{d(\Delta H)}{dT} = \frac{\pi k}{g\mu_B} \frac{\langle J_{fs}^2(\mathbf{q}) \rangle}{J_{fs}^2(\mathbf{0})} (\Delta g)^2 K(\alpha). \quad (7)$$

Using $\Delta g = 0.08(1)$, $b = 16(1)$ Oe/K, and assuming that the wave-vector dependence of the exchange interaction in YbInCu₄ is the same as that of its isomorphic compound YInCu₄, i.e., [$\langle J_{fs}^2(\mathbf{q}) \rangle / J_{fs}^2(\mathbf{0}) \rangle \approx 0.31(15)$], we calculated $K(\alpha) \approx 0.36(20)$ from Eq. (7). From the work of Shaw and Warren²⁹ this value corresponds to $\alpha \approx 0.7(2)$, which is compatible with the upper limit of ≈ 0.9 estimated from the enhancement of the *c-e* spin susceptibility. Then, using

$\eta(E_F) = 10(1)$ states/eV mol spin, $\alpha \approx 0.7(2)$, and Eqs. (5) and (6) we obtain, $J_{fs}(\mathbf{0}) \approx 2.4(1.5)$ meV and $\langle J_{fs}^2(\mathbf{q}) \rangle^{1/2} \approx 1.3(1.0)$ meV for the exchange parameters between Gd³⁺ and the *c-e* in YbInCu₄. These values are at least ten times smaller than their respective values found for Gd³⁺ in YInCu₄, suggesting a much higher *c-e* localization for the Yb than for the Y-based compound. That is consistent with a large $\eta(E_F)$ associated with a ‘‘narrow’’ band at the Fermi level in IV and HF systems. Table II summarizes the parameters obtained in the present work for Gd³⁺ in (Yb,Y)InCu₄.

V. CONCLUSIONS

The ESR data presented for Gd³⁺ in the IV phase of YbInCu₄ ($T < T_v$) show that the large density of states at the Fermi level, characteristic of an IV and HF system, results in a g shift and a Korringa rate larger than those found in its reference compound YInCu₄. Our results also indicate that the high density of states of an IV system perturb the g shift and the Korringa rate in a different way. We found that these parameters are strongly dependent on the electronic properties of the chosen compound, such as electron-electron exchange interaction,²⁷ wave-vector dependence of the exchange interaction, $J_{fs}(\mathbf{q})$,²¹ band structure,²⁶ and the local-moment *c-e* relaxation effects.²⁰

In summary, our results show that ESR experiments can be used to monitor the high density of states in some of the highly correlated electron IV and HF systems. However, one should be aware that this property may not be observable for other strongly correlated electron systems when using the ESR technique.^{30–33}

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