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Electron spin resonance of Gd^{3+} and Nd^{3+} in $LuInA_4$ $(A=Cu,Ni)$

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Low-temperature (1.6 $K\lesssim T\lesssim 60$ K) data of electron spin resonance for Gd^{3+} and Nd^{3+} diluted in $LuInA_4$ $(A=Cu,Ni)$ compounds are presented. The results are interpreted in terms of a density of states at the Fermi level built up of a single *s* band for the Cu-based system and a multiple (*s* and *d*) bands for the Ni-based system. The susceptibility and specific heat data show negligible electron-electron exchange enhancement for both compounds. For the Cu-based system the exchange interaction between the rare-earth (Gd^{3+} and Nd^{3+}) local moment and the conduction electrons depends on the conduction-electron wave vector.
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I. INTRODUCTION

Electron spin resonance (ESR) of rare-earths (RE) impurities in metallic hosts has been widely used to study (i) the exchange interaction between the impurity localized magnetic moment and the conduction electrons, (ii) band-structures effects of the host metal, (iii) crystal-field effects, (iv) hyperfine interactions, (v) highly correlated electron systems, and (vi) superconductivity of the host metal.[1]

The exchange interaction experienced by a RE ion impurity in transition metals[2,3] and intermetallic compounds[1,4] varies in sign and magnitude depending on the transition-metal ion.[1] Because of the stability of the Gd^{3+} and Nd^{3+} ions $4f$ shell, the negative exchange integral is not associated with a covalent mixing mechanism.[5] It has been suggested that a negative effective exchange for RE impurities in some *d*-band compounds is due to the lack of orthogonality between the $4f$ and *d*-orbitals of the neighbor sites.[5,6] The purpose of this paper is to show that ESR of Gd^{3+} and Nd^{3+} in the $LuInA_4$ $(A=Cu,Ni)$ compounds can provide a means to probe the band structure of these systems. We showed that the ESR data of Gd^{3+} in $LuInCu_4$ (Ref. 7) and $YInCu_4$ (Ref. 8) could be explained in terms of a single *s* electronic-band contribution to the density of states at the Fermi level. Alternatively, we will show here that the ESR data of Gd^{3+} and Nd^{3+} in $LuInNi_4$ cannot be explained with a single band. We propose that the contribution of *s* and *d* electronic bands to the density of states at the Fermi level is required to explain the data.

II. EXPERIMENT

Single crystals of $Lu_{1-x}RE_xInA_4$ $(RE=Gd,Nd;$ $A=Cu,Ni;$ $0.0005\leqslant x\leqslant 0.005$ nominal) of cubic $AuBe_5$ (C15b, F43m)-type structure[9] were grown from a flux of excess InCu by the method described elsewhere.[10] The crystals were of cubiclike shape with typical sizes of $4\times 3\times 1$ mm^3. The ESR experiments were carried out in a Varian *E* line and a Bruker ELEXSYS *X*-band spectrometers, using a liquid-helium tail dewar (1.6–4.15 K) and a helium gas flux (4–60 K) adapted to a room-temperature TE_{102} cavity. Dysonian lineshapes[11] with $A/B\approx 2.2(2)$ were always observed. These line shapes are characteristic of localized magnetic moments in a metallic host with a skin depth smaller than the size of the samples. In order to increase the ESR signal to noise ratio, powdered crystals were used in most of the ESR measurements. Experiments conducted in single crystals did not show any anisotropy that could be attributed to crystal-field effects. Susceptibility measurements were made in a Quantum Design dc superconducting quantum interference device (SQUID) magnetometer. Specific heat measurements were performed in a small-mass calorimeter system that employs a quasiadiabatic thermal relaxation technique.[12] Samples used here ranged from 50 to 150 mg.

III. EXPERIMENTAL RESULTS

Figure 1 shows the specific heat for the $LuInNi_4$ compound in the temperature range of 2 $K\lesssim T\lesssim 20$ K. In the low temperature region, C/T increases linearly with T^2 as seen in the inset of Fig. 1. The fitting parameters, γ and β, obtained from these data are given in Table I. The Debye temperature, θ_D, is given in Table II.

Figure 2 gives the magnetic susceptibility data for some of the $Lu_{1-x}RE_xInNi_4$ $(RE=Gd,Nd)$ crystals used in ESR experiments, corrected for the compound core diamagnetism. Using the effective magnetic moments, $\mu_{eff}=7.94$ μ_B and

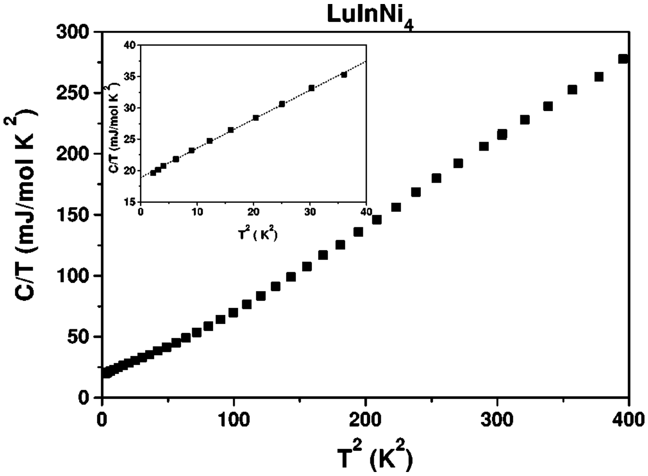

FIG. 1. Specific heat (C/T) as a function of T^2 for LuInNi$_4$. The inset shows the low temperature T^2 dependence of C/T. The dashed line is the best fit to $C/T=\gamma+\beta T^2$. The parameters γ and β are given in Table I.

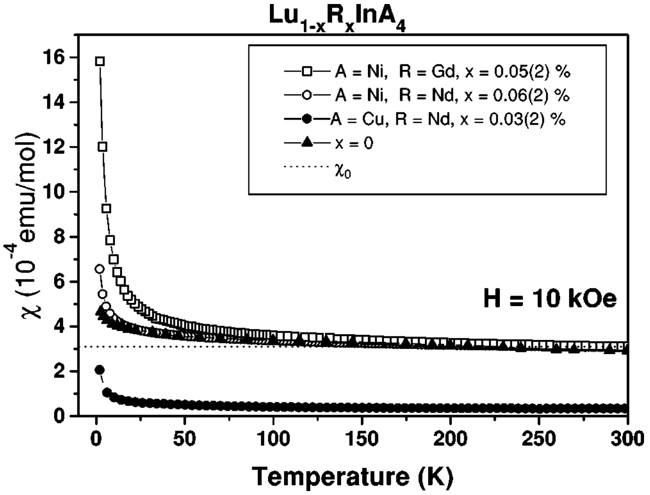

FIG. 2. Temperature dependence of the measured magnetic susceptibility at 10 kOe, for 0.16(5)% of Gd^{3+} and 0.06(2)% of Nd^{3+} in LuInNi$_4$, 0.03(2)% of Nd^{3+} in LuInCu$_4$ and pure LuInNi$_4$. The dashed line is the calculated Pauli susceptibility χ_0, with $\eta(E_F)=3.9(1)$ states/eV mol-spin for LuInNi$_4$.

3.62 μ_B for Gd^{3+} and Nd^{3+}, respectively, the Gd and Nd concentrations were estimated and their values are given in Table I. Also, the concentration of the Gd^{3+} natural impurities in LuInNi$_4$ was estimated and is given in Table I.

Figures 3 and 4 show the ESR powder spectra for ~0.2% of Gd^{3+} and ~0.05% of Nd^{3+} diluted in LuInA_4 (A=Cu,Ni) at $T=1.6$ K, respectively. The g values and linewidths were obtained from the fitting of the resonances to the appropriate admixture of absorption and dispersion lorentzian derivatives.[13] The solid lines are the best fit to the observed resonances and the extracted ESR parameters are presented in Table I. The inset of Fig. 4 shows the Nd^{3+} resonances corresponding to the various Nd isotopes and also the Gd^{3+} natural impurities resonance. The g-value for the ^{140}Nd ($I=0$) isotope is close to the g value of a Γ_6 Kramers doublet ground state ($g=2.667$). This indicates that the RE are in a site of cubic symmetry.[14] Table I gives the hyperfine constants ^{143}A and ^{145}A corresponding for the ^{143}Nd ($I=7/2$) and ^{145}Nd ($I=7/2$) isotopes, extracted from the measured spectra using the Breit-Rabi formula.[14]

Figures 5 and 6 show the temperature dependence of the linewidth for the ~0.2% of Gd^{3+} and ~0.05% of Nd^{3+} diluted in LuInA_4 (A=Cu,Ni), respectively. The linear dependence of the linewidth was fitted to the expression $\Delta H=a+bT$. Within the accuracy of the measurements, the g values are found to be temperature independent. The b and g parameters are independent of the Gd and Nd concentration. The values are presented in Table I.

IV. ANALYSIS AND DISCUSSION

Figure 1 shows the electronic contribution to the heat capacity in LuInNi$_4$. A Sommerfeld coefficient, $\gamma=19(1)$ mJ/mol-K^2, was obtained from it. In a free c-e gas model, this coefficient is given by $\gamma=(2/3)\pi^2k^2\eta(E_F)$. Then, for LuInNi$_4$, we calculate the density of states at the Fermi level $\eta(E_F)=3.9(1)$ states/eV mol-spin. From this density of states, we estimate an electronic spin susceptibility, $\chi_e=2\mu_B^2\eta(E_F)$, of $\approx 0.31\times10^{-3}$ emu/mol. This value is in good agreement with the susceptibility (corrected for the corediamagnetism) measured at high temperatures (see dashed line in Fig. 2). Thus, as in LuInCu$_4$[7] the Stoner's factor is negligible. Therefore, we conclude that electron-electron exchange enhancement is not important in LuInNi$_4$. The exchange interaction, $J_{fs}\mathbf{S}.\mathbf{s}$, between a localized $4f$ electron spin ($\mathbf{S}$) on the RE ion impurities and the c-e's spin ($\mathbf{s}$) of the host metal causes a g shift (Knight shift) (Ref. 15) and a linear thermal broadening of the ESR lines (Korringa rate).[16] Allowing for a $\mathbf{q}$-dependent exchange interaction,

TABLE I. Experimental parameters for (Gd, Nd): LuInA_4 (A=Cu,Ni).

	g	a	b	c	γ	β	^{143}A	^{145}A
		Oe	Oe/K	%	mJ/mol-K^2	mJ/mol-K^4	Oe	Oe
LuInCu$_4$					2.03(3)^a	0.41(2)^a		
LuInNi$_4$				0.009(7)^b	19(1)	0.46(2)		
Lu(Gd)InCu$_4$	2.003(3)^a	41(2)^a	0.9(1)	≈0.2				
Lu(Gd)InNi$_4$	1.980(2)	30(5)	6.0(8)	0.16(5)				
Lu(Nd)InCu$_4$	2.582(4)	52(5)	3.5(5)	0.06(5) and ~0.005^b			215(10)	130(8)
Lu(Nd)InNi$_4$	2.61(2)	93(10)	30(6)	0.03(5)				

[a] See Ref. 7.

[b] Gd^{3+} natural impurities concentration.

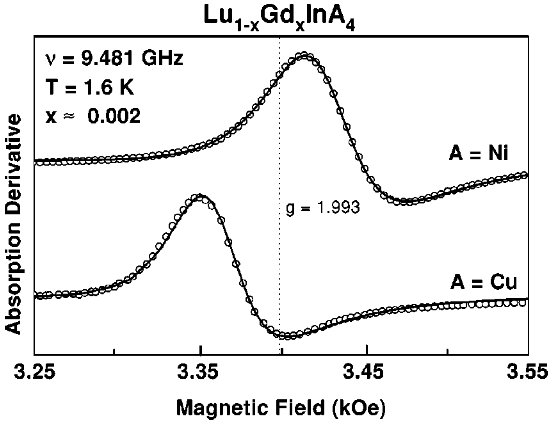

FIG. 3. ESR powder spectra of ~0.2% Gd^{3+} in LuInCu$_4$ and LuInNi$_4$ at $T=1.6$ K. The solid lines are the best fit of the resonance to a Dyson line shape.

$J_{fs}(\mathbf{q})$,[8,17] but in the absence of conduction electron-electron exchange enhancement,[18,19] *bottleneck*, and *dynamic* effects, the g shift (Δg) and Korringa rate (b) can be written as[20]

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

and

$$b = \frac{d(\Delta H)}{dT} = \frac{\pi k}{g_i \mu_B}\left(g_i \frac{g_J - 1}{g_J}\right)^2 \langle J_{fs}^2(\mathbf{q})\rangle \eta^2(E_F), \tag{2}$$

where g_i is the ionic g factor measured in insulators ($g_i = 1.993$ (Ref. 21) for Gd^{3+} and $g_i = 2.63$ (Ref. 22) for Nd^{3+}), g_J is the Lande g factor ($g_J = 2$ for Gd^{3+} and $g_J = 8/11$ for Nd^{3+}). $J_{fs}(\mathbf{0})$ and $\langle J_{fs}^2(\mathbf{q})\rangle$ are the effective exchange parameters between the RE^{3+} local moment and the conduction electrons in the presence of conduction-electron momentum transfer.[17] The g shift measures the conduction-electrons polarization ($\mathbf{q}=0$) and the Korringa rate the conduction-electron momentum transfer ($0 \leqslant \mathbf{q} \leqslant 2k_F$), averaged over the Fermi surface.[17] Finally, $\eta(E_F)$ is the ''*bare*'' density of states for one spin direction at the Fermi surface, k is the Boltzman constant, and μ_B is the Bohr magneton.

In the analysis of the ESR data for Gd^{3+} and Nd^{3+} in LuInCu$_4$ the contribution from different conduction-electron bands can be neglected because the measured Korringa rates are much smaller than those expected from the measured g shifts [see Eq. (4) below].[23,24] Besides, Δg and b were found to be concentration independent, i.e., the RE^{3+} spin system is *unbottleneck* in LuInCu$_4$. Thus, by taking into consideration the $\mathbf{q}$ dependence of the exchange interaction only, Eqs. (1) and (2) may be combined to give:[8,19]

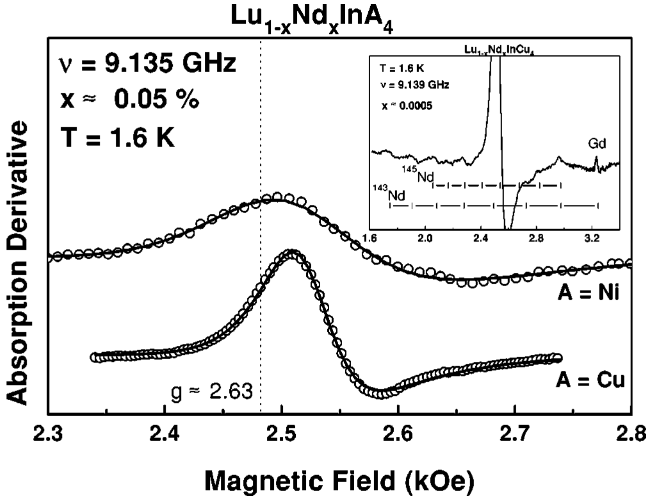

FIG. 4. ESR powder spectra of ~0.05% Nd^{3+} in LuInCu$_4$ and LuInNi$_4$ at $T=1.6$ K. The solid lines are the best fit of the resonance to a Dyson line shape. The inset shows the resonances for the various Nd^{3+} isotopes and the resonance of natural impurities of Gd^{3+}.

$$\frac{b}{(\Delta g)^2} = \frac{\pi k}{g_i \mu_B} \frac{\langle J_{fs}^2(\mathbf{q})\rangle}{J_{fs}^2(\mathbf{0})}. \tag{3}$$

In the case of the absence of a $\mathbf{q}$ dependence of the exchange interaction, Eq. (3) reduces to

$$\frac{b}{(\Delta g)^2} = \frac{\pi k}{g_i \mu_B}. \tag{4}$$

From the experimental values given in Table I, we observe that Eq. (4) does not hold for LuInCu$_4$. Therefore, a $\mathbf{q}$-dependent exchange interaction must be included. Using in Eqs. (1) and (2) the g factors (g_i and g_J) for Gd^{3+} and Nd^{3+}, $\pi k / g_i \mu_B$, and the values of Δg, b, and $\eta(E_F)$ given in Tables I and II, the exchange parameters between the local moment and the conduction-electrons for Gd^{3+} and Nd^{3+} in LuInCu$_4$ were estimated. Table II summarizes these

TABLE II. Derived parameters for (Gd, Nd): LuInA_4 (A=Cu,Ni).

	$\eta(E_F)$ states/eV mol-spin	θ_D K	$J_{fs}(\mathbf{0})$ meV	$\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}$ meV	J_{fd} meV
LuInCu$_4$	0.42(2)[a]	≈305[a]			
LuInNi$_4$	3.9(1)	≈295			
Lu(Gd)InCu$_4$			24(6)[a]	15(4)[a]	
Lu(Gd)InNi$_4$			37(10)	23(8)	−8(3)
Lu(Nd)InCu$_4$			115(40)	35(8)	
Lu(Nd)InNi$_4$			215(70)	65(20)	−20(8)

[a] See Ref. 7.

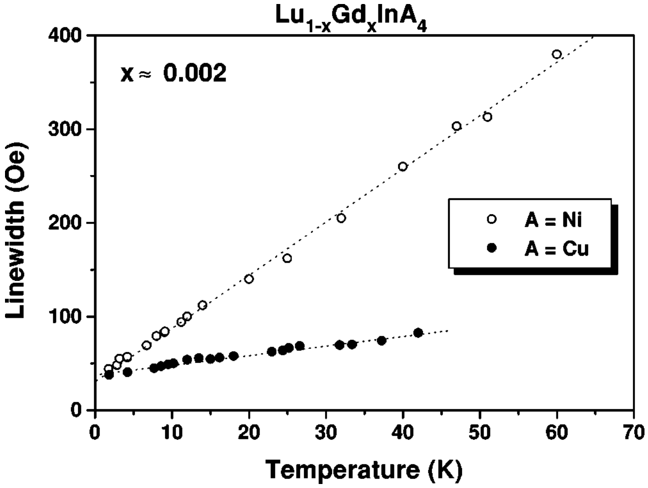

FIG. 5. Temperature dependence of the ESR linewidth for $\sim$0.2% of Gd^{3+} in $LuInCu_4$ and $LuInNi_4$. The dashed lines are the best fit to $\Delta H = a + bT$. Values of a and b are given in Table I.

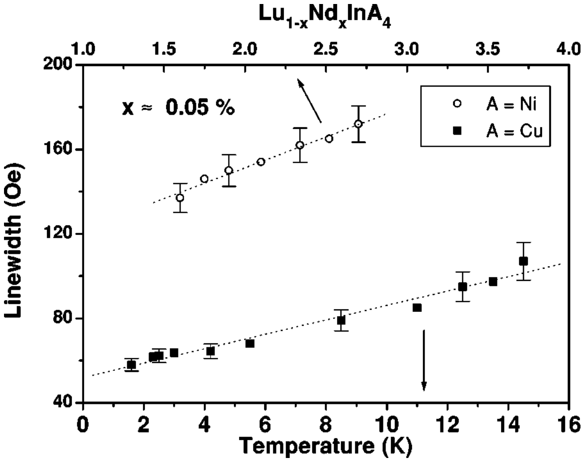

FIG. 6. Temperature dependence of the ESR linewidth for $\sim$0.05% of Nd^{3+} in $LuInCu_4$ and $LuInNi_4$. The dashed lines are the best fit to $\Delta H = a + bT$. Values of a and b are given in Table I.

parameters. Notice that the ratio, $\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}/J_{fs}(\mathbf{0})$, is different for each RE^{3+}. That suggest a different wave-vector dependence of the exchange interaction for each RE^{3+} in $LuInCu_4$.

To attempt to explain the ESR data of the Gd^{3+} and Nd^{3+} in $LuInNi_4$ we propose that contributions from s and d conduction-electron bands are relevant. The justification for this assertion are: (i) the measured Korringa rates are much larger than those expected from the measured g shifts [see Eq. (4) and Table I];[24,25] and (ii) the g shifts are negative for both Gd^{3+} and Nd^{3+} (see Figs. 3 and 4). Notice that in the case of a single-band model, due to the $g_J = 8/11$ value for Nd^{3+}, the g shifts for Gd^{3+} and Nd^{3+} are of opposite sign [see Eq. (1)]. In a two, s and d, band framework, Eqs. (1) and (2) can be rewritten as[25]

$$\Delta g = g_i \frac{g_J - 1}{g_J}[J_{fs}(\mathbf{0}) \quad \eta_s(E_F) + J_{fd}(\mathbf{0}) \quad \eta_d(E_F)], \qquad (5)$$

and

$$b = \frac{d(\Delta H)}{dT} = \frac{\pi k}{g_i \mu_B}\left(g_i \frac{g_J - 1}{g_J}\right)^2 [\langle J_{fs}^2(\mathbf{q})\rangle \, \eta_s^2(E_F) + F_d \langle J_{fd}^2(\mathbf{q})\rangle \, \eta_d^2(E_F)], \qquad (6)$$

where F_d is the reduction core polarization factor, which depends of the orbital degeneracy of the d band at the Fermi Level.[26] For $LuInNi_4$, *bottleneck* and *dynamic* effects are not taken into account because the g shifts and Korringa rates are RE concentration independent, and no temperature dependence of the g shift was measured. In the absence of a band-structure calculation for $LuInNi_4$, we argue that its band structure will be similar to that of the isomorphous compound $LuInCu_4$.[27] Besides, we have not seen any magnetism (ESR and magnetization) that could be associated to $Ni^{2+}(3d^8)$ in $LuInNi_4$. Then, we assume that the contribution of the s band is the same in both compounds. The density of states associated to the Sommerfeld coefficient derived above may be written as $\eta_{tot}(E_F) = \eta_s(E_F) + \eta_d(E_F)$. Thus, we can extract the contribution of the d electrons to the density of states at the Fermi level in $LuInNi_4$. Using $\eta_s(E_F) = 0.42(2)$ states/eV-1 mol-spin,[7] one finds $\eta_d(E_F) = 3.48(12)$ states/eV-1 mol-spin. As found for isomorphous compounds,[7,8,28] we may expect the ratio $\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}/J_{fs}(\mathbf{0})$ to be the same in the Cu- and Ni-based compounds. To the best of our knowledge there is no calculation that take into consideration the $\mathbf{q}$ dependence of exchange interaction between localized spins and d-conduction electrons. So, we take J_{fd} to be $\mathbf{q}$-independent $[\langle J_{fd}^2(\mathbf{q})\rangle^{1/2} = J_{fd}(\mathbf{0}) = J_{fd}]$. If crystal-field splitting of the d electronic levels (e_g, t_{2g}) at the Fermi level are not included, the F_d factor in Eq. (6) may be shown to be 1/5.[26] Having made those assumptions, we derived the values for the parameters, $\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}$,$J_{fs}(\mathbf{0})$, and J_{fd}, for Gd^{3+} and Nd^{3+} in $LuInNi_4$ listed in Table II. Notice that the Gd^{3+} exchange parameters with the s-conduction electrons, $J_{fs}(\mathbf{0})$ and $\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}$, are comparable to those found in the isomorphous compounds $RE\mathrm{InCu}_4$ $(RE = \mathrm{Y,Lu})$.[7,8] Therefore, we feel confident about the assumption that the s-conduction electrons contribution to the density of states at the Fermi level are about the same in these isomorphous compounds. Nevertheless, this assumption may underestimates $\eta_s(E_F)$, and in turn, overestimates $\eta_d(E_F)$. Therefore, more precisely, the values extracted for the exchange parameters in $LuInNi_4$ are an upper limit for the exchange with the s electrons and a lower limit for the exchange with the d electrons.

V. CONCLUSIONS

The ESR data of Gd^{3+} and Nd^{3+} in $LuInCu_4$ are reasonably well described within a framework of: (i) a single s-band model with no electron-electron exchange enhancement, and (ii) a wave-vector dependent exchange interaction between the $4f$ localized magnetic moment and the conduction electrons, $J_{fs}(\mathbf{q})$. On the other hand, for the $LuInNi_4$ compound a two band model, s and d, with no electron-electron exchange enhancement can explain the ESR results. The d-electron band may be thought to be associated with the incomplete Ni electronic $3d$ shell.

It is interesting to note that for the Cu-based compounds, our results show that the $\mathbf{q}$ dependence of the exchange interaction, $J_{fs}(\mathbf{q})$, is RE dependent. The Nd^{3+} exchange parameters are systematically larger than those of Gd^{3+}. That is probably caused by the larger Nd^{3+} $4f$ shell radius. Again, the values given for the exchange parameters $\langle J_{fs}^2(\mathbf{q})\rangle^{1/2}$, $J_{fs}(\mathbf{0})$, and J_{fd} for the Ni-based systems should be taken with care.

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