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Crystal-field study in rare-earth-doped semiconducting YBiPt

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Electron spin resonance (ESR) and magnetic-susceptibility experiments in the rare-earth-doped (R =Nd, Er, and Yb) cubic semiconducting YBiPt allow estimates of the fourth (A_4) and sixth (A_6) order crystal-field parameters for this compound. It is found that these parameters are of the same order for all the R studied. On the other hand, no crystal-field effects were found for the Gd^{3+} doped single-crystal system. Consistent with the small gap semiconducting character of the YBiPt intermetallic compound, a Dysonian ESR line shape with no g shift and Korringa broadening was observed. [S0163-1829(99)10925-1]

I. INTRODUCTION

The series of intermetallic compounds $R\text{BiPt}$ (R =rare earths Nd-Lu) have recently attracted great attention due to their interesting properties.¹ Most of these materials are narrow gap semiconductors ($\Delta=0.1\text{--}0.01$ eV) with a gradual evolution towards semimetallic behavior as R is varied through the rare-earth series.² YbBiPt is the heavy fermion system with the largest linear specific-heat coefficient, $\gamma=8$ J/mol K². The $R\text{BiPt}$ series forms in the cubic AgAsMg structure which can be viewed as three face-centered sublattices placed at (0,0,0), (1/4,1/4,1/4), and (3/4,3/4,3/4). In this series, the rare earths order antiferromagnetically (T_N) below 9 K and present negative paramagnetic temperatures $|\theta|\leq 44$ K.^{2,3} The systematic change from semiconducting, for the lighter R , to metallic compounds, for the heavier R , is attributed to the decrease in the unit-cell volume as the R series is traversed.^{2,3} Hence it is expected that the strength of the crystal-field potential plays an important role on the physical properties of these materials. The YBiPt compound belongs to this family and shows a semiconducting behavior with a lattice parameter close to that of the Tb-based compound, i.e., near the middle of the R series.^{2,3}

This work aims to study the crystal-field effects (CFE) in the $Y_{1-x}R_x\text{BiPt}$ (R =Nd, Gd, Er, Yb, and $0.002\leq x\leq 0.10$) compounds. By means of electron spin resonance (ESR) and magnetic-susceptibility experiments, it was possible to estimate the fourth (A_4) and sixth (A_6) order cubic crystal-field parameters (CFP) in the $Y_{1-x}R_x\text{BiPt}$ (R =Nd, Er, Yb, and $0.002\leq x\leq 0.10$) systems. We found that the overall crystal-field splitting is of the order of 100–200 K, of the same magnitude as the semiconducting gap (Δ) of these materials. In a previous report on ESR of Er^{3+} in YBiPt, we extracted the CFP from the analysis of the ESR spectra of the Er^{3+}

$\Gamma_8^{(3)}$ ground state.⁴ The CFP reported are in agreement with the values obtained in this work.

II. EXPERIMENT

Single crystalline samples of the $Y_{1-x}R_x\text{BiPt}$ (R =Nd, Gd, Er, Yb, and $0.002\leq x\leq 0.10$) compounds were grown from the melt in Bi flux as described previously.² Typical crystal sizes were $2\times 2\times 2$ mm³. The structure and phase purity were checked by x-ray powder diffraction and the crystals orientation determined by the usual Laue method. The ESR experiments were carried out in conventional Varian and Bruker ESR spectrometers using a TE₁₀₂ room-temperature cavity. The sample temperature was varied using a helium gas flux temperature controller and a quartz tail dewar for liquid-helium bath experiments. To increase the ESR signal-to-noise ratio, the temperature dependence of the spectra was taken in powdered samples. In order to look for anisotropic effects, single crystalline samples were used. Magnetization measurements have been taken in a Quantum Design dc superconducting quantum interference device MPMS-5 magnetometer. Specific-heat measurements were performed in a small-mass calorimeter system that employs a quasiadiabatic thermal relaxation technique.⁵ Samples employed ranged in mass from 50 to 150 mg.

III. RESULTS AND ANALYSIS

Figure 1 shows the temperature dependence of the specific heat of YBiPt. A linear fit of the data to C/T vs T^2 yields a very small Sommerfeld coefficient ($\gamma\leq 0.1$ mJ/mol K). Therefore the electronic contribution to the heat capacity in this compound is negligible, i.e., the density of states at the Fermi level is very small.

Figure 2 shows the temperature dependence of the suscep-

FIG. 1. Specific heat (C/T) as a function of T^2 for YBiPt. 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 $\gamma \leq 0.1$ mJ/mol K² and $\beta = 0.79(7)$ mJ/mol K⁴.

tibility of $Y_{1-x}Gd_xBiPt$ ($x=0.002$, nominal concentration and $x=0$) single crystals measured at $H=0.5$ T. The data for the Gd-doped samples, corrected for the core diamagnetism, were fitted to a Curie-Weiss law. Assuming $7.94\mu_B/Gd$, the Gd concentration was estimated to be $\approx 0.21\%$, close to the nominal value.

Figure 3 shows the Gd^{3+} ESR powder spectra observed in $Y_{1-x}Gd_xBiPt$ at $T=280$ K and $T=7$ K for $x \approx 0.002$. The resonance shows the usual Dysonian line shape,⁶ characteristic of metallic particles of dimensions larger than the skin depth. The continuous lines in Fig. 3 are the best fit of the experimental spectra to a Lorentzian admixture of absorption and dispersion derivatives.⁷ From these fits, the linewidth ΔH and g value of the resonances are obtained. Several samples of Gd concentrations, between 0.2 and 0.5%, were measured. The temperature dependence of the Gd^{3+} ESR linewidth was fitted to the formula $\Delta H = a + bT$. Within the accuracy of the measurements, b and the g value were found to be concentration independent. The residual linewidth a increases with the concentration of Gd. The g value was found to be temperature independent in all cases. Table I

FIG. 2. Temperature dependence of the susceptibility of $Y_{1-x}Gd_xBiPt$ ($x \approx 0.002$, nominal concentration and $x=0$) single crystals measured at $H=0.5$ T.

FIG. 3. ESR spectra of Gd^{3+} in $Y_{1-x}Gd_xBiPt$ ($x \approx 0.0021$) at $T=7$ K and $T=280$ K. The thermal broadening of the linewidth was found to be $b \leq 0.05$ Oe/K and $g = 1.993(4)$.

gives the experimental parameters obtained for the most diluted samples.

The very small value found for $b \leq 0.05$ Oe/K and a g value close to those in insulators [$g = 1.993(5)$] indicates that the Gd^{3+} localized magnetic moment is basically not coupled to the conduction electrons.⁸ This is expected because (i) electronic structure calculations for YBiPt show a gap of about 0.08 eV at the Fermi level³ and (ii) from the specific-heat measurements (see Fig. 1) a very small density of states at the Fermi level is derived [$\eta(E_F) \leq 0.02$ states/eV mol spin].

ESR experiments in single crystals of $Y_{1-x}Gd_xBiPt$ did not show CFE at any temperature, neither a splitting of the line nor a linewidth anisotropy. Since the measured linewidth at 1.6 K was found to be $\Delta H \approx 40$ Oe, an upper limit for the fourth-order CFP $|b_4|$ can be estimated, ≤ 1 Oe. That is consistent with the trends already observed in low carrier density metallic pnictides⁹ and narrow gap semiconductors,¹⁰ where the b and b_4 parameters are small.

Figure 4 presents the Nd^{3+} ESR powder spectra observed in $Y_{1-x}Nd_xBiPt$ at $T=4.2$ K for $x \approx 0.002$. The spectra show a main line corresponding to the $^{140}Nd^{3+}$ ($I=0$) isotope and the hyperfine lines due to the $^{143}Nd^{3+}$ ($I=7/2$) and $^{145}Nd^{3+}$ ($I=7/2$) isotopes. The small line seen at 3400 Oe is associated to natural impurities of Gd^{3+} , probably present in Y. The positions of the hyperfine satellites were determined experimentally by taking the second derivative of the spectra. Table I gives the hyperfine constants $A(^{143}Nd)$ and $A(^{145}Nd)$ corresponding to the ^{143}Nd and ^{145}Nd isotopes as extracted from the observed spectra using the Breit-Rabi formula.¹¹ The g value and hyperfine constants given in Table I indicate that the Nd^{3+} ion is in a cubic site environment and that its ground state is a Γ_6 Kramers doublet.¹² These results were found to be concentration independent in the range $0.002 \leq x \leq 0.10$. Thus we may neglect the exchange coupling between the rare earths in the analysis of the susceptibility data. Within the accuracy of our measurements the linewidth and g value were temperature independent. That is consistent with the ESR results obtained for Gd^{3+} in YBiPt.

It is reasonable to assume that the cubic CFP, A_4 and A_6 , at the R site in $Y_{1-x}R_xBiPt$ ($R=Yb, Nd, Er$) would not be strongly affected by the R impurities. Therefore, the ratio

TABLE I. Experimental parameters for $R:\text{YBiPt}$.

	g	a Oe	b Oe/K	^{143}A Oe	^{145}A Oe	W K	x
Gd:YBiPt	1.993(4)	40(3)	≤ 0.05				
Nd:YBiPt	2.63(1)	70(5)	≤ 0.1	213(5)	133(5)	2.0(5)	0.15(5)
Yb:YBiPt						-3.9(7)	-0.61(8)
Er:YBiPt						$\approx -0.17^a$	$\approx 0.271^a$
PrBiPt						$\approx -1.79^a$	$\approx -0.20^a$
YbBiPt						$\approx -3.5^a$	$\approx -0.60^a$

^aSee Refs. 4, 16, and 17.

A_4/A_6 should remain basically the same for all R . The analysis of ESR data for the Er^{3+} in YBiPt (Ref. 4) show a $\Gamma_8^{(3)}$ ground state with Lea, Leask, and Wolf (LLW) (Ref. 13) parameters, $x=0.271$ and $W=-0.17$ K. Taking into account the ratios $\langle r^4 \rangle / \langle r^6 \rangle$ for Er^{3+} , Yb^{3+} and Nd^{3+} ,¹⁴ values of $x \approx -0.69$ for Yb^{3+} , and $x \approx 0.30$ for Nd^{3+} in YBiPt are obtained, from which a Γ_6 for Nd^{3+} , and a Γ_7 for Yb^{3+} as ground states are predicted. The Γ_6 ground state for Nd^{3+} is consistent with ESR data reported here (see Fig. 4).

Figures 5 and 6 show the temperature and field dependence of the inverse magnetic susceptibility $\chi^{-1}(T, H=1, 5 \text{ T}) - \chi_\infty^{-1}$, for the $\text{Y}_{0.9}\text{Yb}_{0.1}\text{BiPt}$ and $\text{Y}_{0.9}\text{Nd}_{0.1}\text{BiPt}$ single crystals. χ_∞^{-1} is the free ion value of the inverse susceptibility shown in the insets of these figures. Assuming the nominal concentration for the Yb^{3+} and Nd^{3+} doped samples, the expected high-temperature limit of the Curie law is obtained, $\mu_{\text{eff}} = 4.5(2)\mu_B$ and $\mu_{\text{eff}} = 3.65(20)\mu_B$ for Yb^{3+} and Nd^{3+} , respectively. The solid lines are the best fit to the data using the Hamiltonian

$$\mathcal{H} = B_4[O_4^0 + 5O_4^4] + B_6[O_6^0 - 21O_6^4] + g_J\mu_B \mathbf{H} \cdot \mathbf{J} \quad (1)$$

that includes the cubic crystal-field and Zeeman terms. The B_n and O_n^m are the fourth and sixth order CFP and Stevens equivalent operators, respectively. $B_n = A_n \langle r^n \rangle \theta_n$, g_J is the Landé factor and μ_B is the Bohr magneton.¹³ Diagonalizing

FIG. 4. ESR spectra of Nd^{3+} in $\text{Y}_{1-x}\text{Gd}_x\text{BiPt}$ ($x \approx 0.0020$) at $T=4.2$ K. The resonance Gd^{3+} natural impurities is also indicated. The vertical lines show the positions of the various hyperfine satellites appropriate for the ^{143}Nd and ^{145}Nd isotopes.

numerically the Hamiltonian we get the eigenvalues E_n and corresponding eigenfunctions that can be written as

$$|\phi_n\rangle = \sum_{M=-J}^J C_M^n |JM\rangle, \quad (2)$$

where the $|JM\rangle$ expand the manifold of angular momentum J . Hence the magnetic susceptibility is given by

$$\chi = \frac{g_J\mu_B \sum_n \exp\left(-\frac{E_n}{kT}\right) \sum_{M=-J}^J |C_M^n|^2 M}{H \cdot \sum_n \exp\left(-\frac{E_n}{kT}\right)}. \quad (3)$$

Defining the LLW parameters x and W by the equations

$$B_4F(4) = Wx, \quad (4)$$

$$B_6F(6) = W(1 - |x|), \quad (5)$$

FIG. 5. Temperature and field dependence of the inverse magnetic susceptibility $\chi^{-1}(T, H=1, 5 \text{ T}) - \chi_\infty^{-1}$ for the $\text{Y}_{0.9}\text{Yb}_{0.1}\text{BiPt}$ single crystal. χ_∞^{-1} is the free ion value of the inverse susceptibility shown in the inset. The solid lines are the best fit to the data of the calculated susceptibility including the Zeeman and LLW cubic crystal-field terms in the Hamiltonian. The Yb^{3+} crystal-field splitted ground state multiplet ($J=7/2$) is shown.

FIG. 6. Temperature and field dependence of the inverse magnetic susceptibility $\chi^{-1}(T, H=1 \text{ T}, 5 \text{ T}) - \chi_{\infty}^{-1}$ for the $\text{Y}_{0.9}\text{Nd}_{0.1}\text{BiPt}$ single crystal. χ_{∞}^{-1} is the free ion value of the inverse susceptibility shown in the inset. The solid lines are the best fit to the data of the calculated susceptibility including the Zeeman and LLW cubic crystal-field terms in the Hamiltonian. The Nd^{3+} crystal-field splitted ground state multiplet ($J=9/2$) is shown.

where $F(4)$ and $F(6)$ are scaling parameters appropriate for each J value, we perform a least-squares fitting of the susceptibility leaving x and W as adjustable parameters. The fittings for Yb^{3+} in YbBiPt lead to the LLW parameters $x = -0.61(8)$ and $W = -3.9(7)$. These parameters predict a Γ_7 ground state, a Γ_8 first excited state at $6(2)$ K, and a Γ_6 second excited state at $88(10)$ K (see Fig. 5). We did not observe the ESR line expected for the Kramer doublet Γ_7 ground state for Yb^{3+} . A possible explanation for it is that the YbBiPt is a heavy fermion with a semimetallic conductivity, thus the large coupling with conduction band should induce a large Korringa-like relaxation, even for rather low density of states in the Yb-doped YBiPt sample. Also, the proximity of the anisotropic Γ_8 first excited state at $6(2)$ K may contribute to a strong broadening of the resonance line. For Nd^{3+} in YBiPt , the fits lead to the LLW parameters $x = 0.15(5)$ and $W = 2.0(5)$, from which a Γ_6 ground state, a Γ_8 first excited state at $53(10)$ K, and a Γ_8 second excited state at $190(30)$ K are expected (see Fig. 6). The A_4 and A_6 CFP and crystal-field overall splitting, Δ_{cc} , for Yb^{3+} , Nd^{3+} , and Er^{3+} in YBiPt , are given in Table II. For comparison, the A_4 and A_6 CFP estimated from the level scheme for PrBiPt (Ref. 16) and YbBiPt (Ref. 17) are also given.

IV. DISCUSSION

The fine structure in the ESR spectrum of the S -state ion Gd^{3+} is believed to be associated with the admixture of excited crystal-field-split configurational states into the ground S state mainly via spin-orbit coupling.⁹ Although it is not a general rule, in many materials it has been found that the Gd^{3+} fourth-order CFP b_4 is negative in insulators¹¹ and semiconductors of appreciable gap,¹⁵ but positive in metals,⁹ whereas, for materials of low carriers density this parameter was found to be very small.⁹ Therefore it could be expected that in the narrow gap semiconductor YBiPt $|b_4|$ would be small and the CFE of Gd^{3+} in YBiPt could be masked by the resonance residual linewidth. But we showed above that the A_4 and A_6 CFP obtained for the non- S -state ions, Er^{3+} , Nd^{3+} , and Yb^{3+} in YBiPt are of the same order of magnitude as those reported for rare earths in other cubic materials, where CFE were observed in the ESR spectra of Gd^{3+} .^{18–21} It suggests that CFE should be present in the ESR of Gd^{3+} -doped YBiPt , specially when considering the relative small Gd^{3+} ESR residual linewidth (≈ 40 Oe). Thus the absence of CFE in the ESR spectrum of Gd^{3+} in YBiPt is yet not understood. Thus a mechanism that may explain the observed “quenching” of CFE for the S state ion Gd^{3+} in small gap semiconductors and semimetals with low carrier density is still missing.

The A_4 and A_6 CFP found here for YBiPt are consistent with those obtained from specific-heat and susceptibility measurements in PrBiPt (Ref. 16) and neutron-diffraction experiments in YbBiPt (see Table II).¹⁷ Notice that the level scheme shown in Fig. 6 is very close to that obtained for YbBiPt .¹⁷ The small differences are probably associated with the different lattice parameter and with the metallic character of YbBiPt . Notice that the splitting of the quartet, claimed in Ref. 17, was not observed in any of our compounds. The sign and order of magnitude of the A_4 CFP is in agreement with that obtained from a point-charge model (PCM) assuming a tetrahedron of $-e$ charges sited at the Pt first near neighbors (see Table II). But the A_6 CFP turns out to be positive, contrary to the experimental value given in Table II. Besides, the addition of an octahedron of negative charges at the position of the Bi next near neighbors, increases the positive value of A_4 and A_6 . It is generally accepted that for cubic crystal-fields, due to the absence of second-order terms, the first nearest neighbors constitute the main contribution to the CFP. Therefore corrections to the PCM such as screening, size effects, and covalency need to be included to improve the calculated values of the CFP using a simple PCM.

TABLE II. Extracted parameters for $R:\text{YBiPt}$.

	A_4 K per a_0^{-4}	A_6 K per a_0^{-6}	A_4 (PCM) K per a_0^{-4}	A_6 (PCM) K per a_0^{-6}	Δ_{cc} K
PrBiPt	$\approx -3^a$	$\approx -1.2^a$	-11.60	0.11	$\approx 210^a$
Nd:YBiPt	-7(4)	-1.4(8)	-11.93	0.11	190(30)
Er:YBiPt	≈ -15	≈ -1	-13.24	0.13	85(20) ^a
Yb:YBiPt	-24(8)	-2.6(9)	-13.50	0.13	88(10)
YbBiPt	$\approx -21^a$	$\approx -2.4^a$	-13.50	0.13	$\approx 70^a$

^aSee Refs. 4, 16, and 17.

V. CONCLUSIONS

The CFP A_4 and A_6 in $Y_{1-x}R_x\text{BiPt}$, for the non-S-state ions, $R=\text{Nd}^{3+}$, Er^{3+} , and Yb^{3+} , were determined from ESR and magnetic susceptibility experiments. The obtained values were found to be in reasonable agreement with those measured by others in PrBiPt (Ref. 16) and YbBiPt .¹⁷ However, the PCM only accounts for the sign and value of the A_4 CFP. Surprisingly, CFE were not observed in the ESR spectrum of Gd^{3+} in YBiPt . An upper limit of about 1 Oe for the $|b_4|$ parameter is estimated from the residual ESR linewidth.

That result and those from others⁹ suggests that a small $|b_4|$ value is characteristic for narrow gap semiconductors and semimetals of low carrier densities.

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