Direct observation of a magnetically ordered state in $YbCu_2Si_2$ under high pressure

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The intermetallic compound $YbCu_2Si_2$ is a well-known nonmagnetic (NM) Yb intermediate-valent compound with a Yb valence of 2.9 at ambient pressure and 300 K. In the present work we have investigated the effect of high pressure on the ground state properties of $YbCu_2Si_2$ on both microscopic and macroscopic levels by using the ^{170}Yb Mössbauer effect, electrical resistance, and x-ray diffraction techniques, respectively. High-pressure x-ray diffraction data indicate that the lattice structure of $YbCu_2Si_2$ is stable up to 22.2 GPa. The value of the bulk modulus $[B_0=168(10)\ \mathrm{GPa}^{-1}]$ is found to be close to the value expected for trivalent RCu_2Si_2 compounds. The pressure dependence of the electrical resistance reveals evidence for a pressure-induced magnetic order for $p \geqslant 8$ GPa. From our Mössbauer data, we conclude a crossover from the NM to a magnetically ordered state of localized Yb moments for $p \geqslant 8$ GPa and below 2 K. The pressure-induced change of the electric quadrupole splitting indicates that this transition is accompanied by a valence change towards the Yb^{3+} state. [S0163-1829(99)01729-4]

I. INTRODUCTION

For the last two decades Ce and Yb intermetallic compounds have attracted much attention because of their peculiar electronic and magnetic properties due to the close proximity of their f level from the Fermi energy. The properties of these compounds are dominated by essentially two characteristic energies scales T_K and T_{RKKY} (the Kondo effect and the RKKY interaction, respectively) which are both related to the hybridization strength between the $4f$ and conduction electrons.[1]

When $T_K \gg T_{RKKY}$ the ground state is nonmagnetic and the compound can even be in an intermediate valence regime; i.e., the f count is noninteger. As opposed to this, when $T_{RKKY} \gg T_K$ the compound can exhibit magnetic order. The most interesting situation occurs when the balance between both competing energy scales makes the compound close to a magnetic instability where heavy-fermion behavior coexists with either superconductivity and/or magnetic ordering.

Among Yb-based intermetallic compounds tetragonal $YbCu_2Si_2$ ($ThCr_2Si_2$-type structure) has attracted continuous interest as being in an intermediate-valent state, the valency $v \simeq 2.9$ at 300 K,[2] with a moderately high value of the linear specific heat coefficient $\gamma \simeq 135\ \mathrm{mJ\,mol^{-1}\,K^{-2}}$.[3] Neither electrical resistivity nor susceptibility measurements at ambient pressure showed any sign of magnetic ordering down to 0.4 K.[3] Since pressure is expected to favor the trivalent $4f^{13}$ state ($v=3$), there have been many attempts to observe pressure-induced valence changes and ultimately magnetic ordering in $YbCu_2Si_2$.

Regarding the effect of pressure on the valence state of Yb, first pressure (up to 1.2 GPa) experiments were carried out using dc magnetic susceptibility (χ) measurements at room temperature. They showed that χ increases with applied pressure pointing to a valence increase.[4] More conclusive were the inelastic neutron scattering experiments performed up to 1.7 GPa at 300 K.[5] They indicated that application of $p=1.7$ GPa induces a valence shift $\Delta v = 0.055$, i.e., $v=2.96$ (300 K, 1.7 GPa) together with a weak reduction of the overall crystal field splitting.

In order to find evidence for a pressure-induced magnetic order several high-pressure electrical resistivity measurements have been performed. Earlier high-pressure electrical resistivity measurements up to 8 GPa at temperatures between 5 and 300 K failed to detect any sign of magnetic ordering.[1,6] A set of experiments with extended pressure ranges (up to 25 GPa) gave some evidence for a pressure-induced magnetic order at $p>8$ GPa.[7–10] This finding was supported by recent thermopower data.[11]

Despite these experimental efforts, there is no direct evidence for such a pressure-induced magnetic order. Also nothing is known about the nature of the pressure-induced magnetic state, e.g., the value of Yb magnetic moment and the type of magnetic ordering are still missing information.

In order to shed more light on the nature of the ground state of $YbCu_2Si_2$ we have investigated the effect of pressure on both microscopic and macroscopic levels by using ^{170}Yb Mössbauer effect, electrical resistance, and x-ray diffraction techniques, respectively.

^{170}Yb Mössbauer spectroscopy offers the possibility to determine the electric quadrupole and magnetic hyperfine interactions. From the quadrupole splitting $E_Q = eQV_{zz}$, where V_{zz} is the electric field gradient (EFG) and Q the quadrupole moment of the excited $I_{ex}=2$ state, one can gain information

about the pressure-induced valence changes. The determination of the effective magnetic field B_{eff} allows one to evaluate the pressure dependence of the Yb magnetic moment $\mu_{Yb}(p)$ and of the ordering temperature $T_0(p)$ by measuring $B_{eff}(T)$ at different pressures. Measurements of the resistance $R(T,p)$ permit one not only to determine $T_0(p)$ but also to study corresponding variations of the crystalline electric field and electron correlation effects which are known to be important for the ground states properties of correlated electron systems. The x-ray diffraction experiments were performed with the aim to investigate the structural stability of $YbCu_2Si_2$ under high pressure and to provide the volume dependence of the relevant physical parameters.

II. EXPERIMENT

Polycrystalline single-phase samples of $YbCu_2Si_2$ were prepared by resistance heating of stoichiometric amounts of the elements in an evacuated tantalum crucible.[7] Their quality was checked by x-ray diffraction.

Measurements of the electrical resistance have been performed using the diamond anvil cell (DAC) technique[12] between 1.8 and 300 K up to 21 GPa. Pressure was determined by measuring the pressure-induced shift of the R_1 fluorescence line of ruby.

The pressure- and temperature-dependent variation of the lattice parameters was measured at the energy-dispersive x-ray diffraction station for high pressure at LURE, Orsay[13] (300 K measurements), and at HASYLAB, Hamburg[14] (variable temperature measurements). Silicon (300 K) oil and solid argon (28 K$\leqslant T<$300 K) were used as pressure transmitting medium in the DAC at pressures up to 22.2 GPa.

The ^{170}Yb high-pressure Mössbauer effect experiments were performed at temperatures ranging from 1.8 to 80 K and up to 8.9 GPa in a Chester-Jones-type setup with B_4C anvils. Related technical details are described elsewhere.[15] A superconducting lead manometer was employed in the pressure cell for *in situ* measurements of the pressure. The $^{170}TmB_{12}$ source ($\sim$40 mCi on a 2 mm active diameter) and the absorber were kept at the same temperature during the measurements.

III. RESULTS AND DISCUSSION

A. Volume dependence of the lattice parameters

Figure 1 shows typical energy dispersive diffraction patterns recorded at 300 K at pressures up to 22.2 GPa. All peaks besides the Yb fluorescence lines and escape peaks from the sample can be indexed according to the tetragonal $ThCr_2Si_2$-type structure. There is no evidence for any structural phase transition up to 22.2 GPa. The pressure dependence of the lattice parameters and of the unit cell volume are presented in Figs. 2 and 3, respectively. A smooth decrease of both *a* and *c* parameters is observed upon increasing pressure with a slightly faster decrease of the *a* parameter. Diffraction patterns recorded at 16 GPa at different temperatures from 300 down to 28 K indicate that $YbCu_2Si_2$ retains the tetragonal structure at low temperatures. The fit of the pressure-volume relationship (Fig. 3) to a Murnaghan's equation of state $V(p)=V_0[1+(B_0'/B_0)p]^{-1/B_0'}$, leads to a bulk modulus $B_0=168(10)$ GPa, $B_0'\approx 0.8$, and V_0 $=152.6$ Å^3. The value of B_0 is comparable to x-ray results for related Yb compounds.[16–18] Our x-ray-determined B_0 value differs considerably from the one estimated from Brillouin scattering, an indirect method which does not imply any pressure experiment.[19]

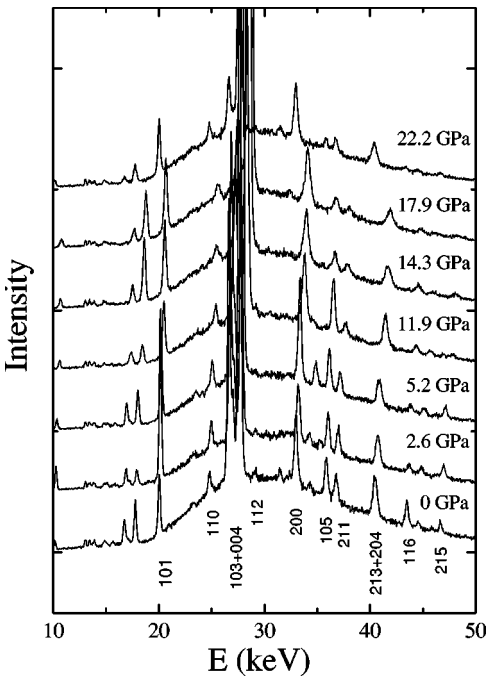

FIG. 1. Room temperature energy dispersive diffraction patterns of $YbCu_2Si_2$ obtained at different pressures up to 22.2 GPa. The diffraction data were collected at a scattering angle of 6.49°. Unmarked peaks represent escape lines and Yb fluorescence lines.

B. Pressure effect on the electrical resistance

Figure 4 displays the temperature dependence (logarithmic scale) of the electrical resistance $R(T,p)$ normalized to its room temperature value $R(295\ \mathrm{K},p)$ for some selected pressures. The normalized resistance value increases continuously and exhibits a broad maximum which becomes more pronounced and shifted to lower temperature with increasing pressure ($0\leqslant p\leqslant 10$ GPa). The shape, below the maximum, of the resistance curves start to be modified above 7 GPa and a second bump shows up clearly at lower tem-

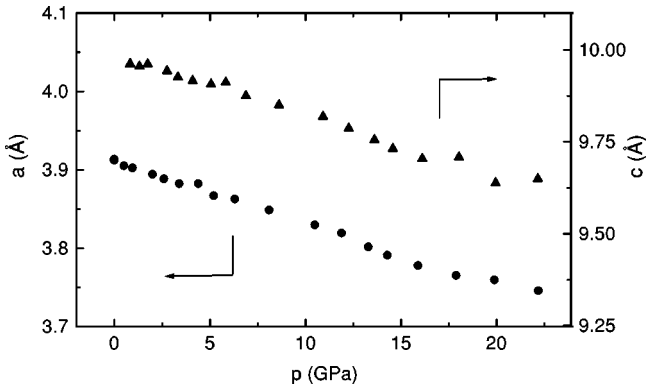

FIG. 2. Pressure dependence of the *a* and *c* lattice parameters of $YbCu_2Si_2$.

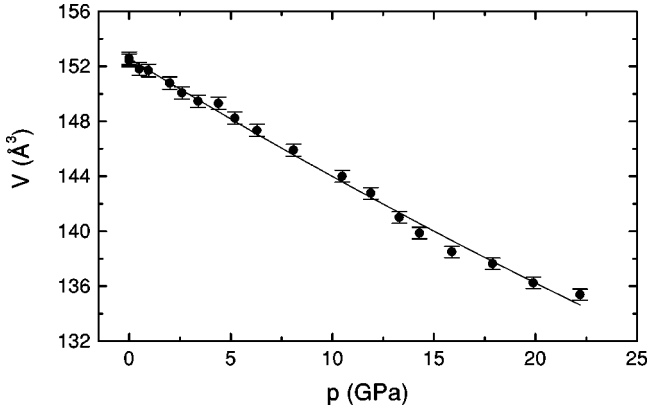

FIG. 3. Pressure variation of the unit cell volume of $YbCu_2Si_2$. The solid line is a fit to Murnahgan's equation of state (see text).

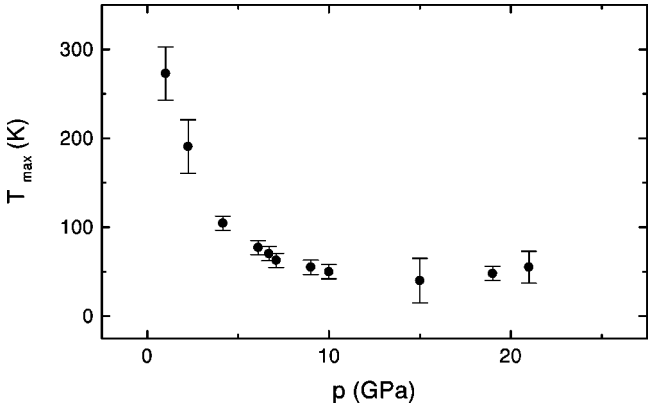

FIG. 5. Volume dependence of the high-temperature resistance maximum T_{max} for $YbCu_2Si_2$.

perature at $p \geqslant 15$ GPa. The pressure (volume) dependence of the high-temperature maximum, T_{max}, is shown in Fig. 5.

Compared with results of previous investigations,[6–10] the T_{max} values appear to be sample dependent, probably due to the strong anisotropic character of $YbCu_2Si_2$. Here, one first observes a rapid decrease of T_{max} from about 270 K at ambient pressure followed by a progressive flattening with almost constant T_{max} values between 9 GPa and 19 GPa ($0.05 < 1 - V/V_0 < 0.10$). Above 20 GPa ($1 - V/V_0 \simeq 0.105$) T_{max} slightly increases. The negative pressure derivative of T_{max} for $p < 15$ GPa is opposite to that found in all nonmagnetic Ce compounds but similar to that observed in other Yb intermetallics.[20,21] It is thus tempting to assume that T_{max} scales with the Kondo temperature T_K and to deduce then an electronic Grüneisen parameter $\Omega = \partial \ln T_{max} / \partial \ln V \simeq -27$ which be estimated from the experimental value of $\partial T_{max} / \partial p \simeq -40$ K GPa^{-1} ($p < 2.3$ GPa) by using the measured bulk modulus [$B_0 = 168(10)$ GPa]. The value of Ω is comparable to the one reported for $YbCu_{4.5}$ ($\Omega = -27$),[20] but much smaller than the value reported recently for the nonmagnetic heavy-fermion Yb_2Ni_2Al ($\Omega = -165$).[18] The above statement is, however, somewhat questionable since the crystal field splitting (T_{CEF}) (Ref. 5) at ambient pressure compares to the value estimated for T_K and both characteristic energies decrease with increasing pressure. It definitely breaks down at higher pressures ($p > 15$ GPa) where T_{max} rises and a low-temperature maximum sets in. Here T_{max} is no longer linked to T_K but probably to the scattering with populated excited crystal field states.

Similar behavior was already observed in other Yb compounds.[20–22] The change of the physical origin of T_{max} is more clearly seen if the resistance data normalized to the resistance R_{max} at T_{max} are plotted versus T/T_{max} (Fig. 6). The observed deviation from a scaling law[1] (see Fig. 6) clearly indicates that T_K no longer plays a central role. Figure 6 shows that reasonable scaling appears for $p < 9$ GPa when $0.6 < T/T_{max} < 2$. This is a quite extended range of

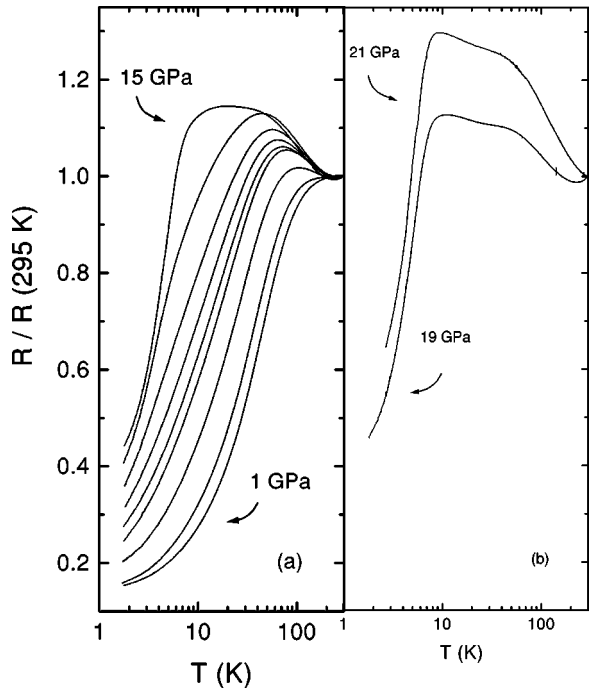

FIG. 4. Temperature dependence of the normalized electrical resistance [$R/R(295\ \mathrm{K})$] of $YbCu_2Si_2$ at some selected pressure values: (a) 1.0 GPa (lower curve), 2.3 GPa, 4.2 GPa, 6.1 GPa, 6.7 GPa, 7.1 GPa, 9.0 GPa, 10.0 GPa, and 15 GPa (upper curve); (b) 19 GPa and 21 GPa.

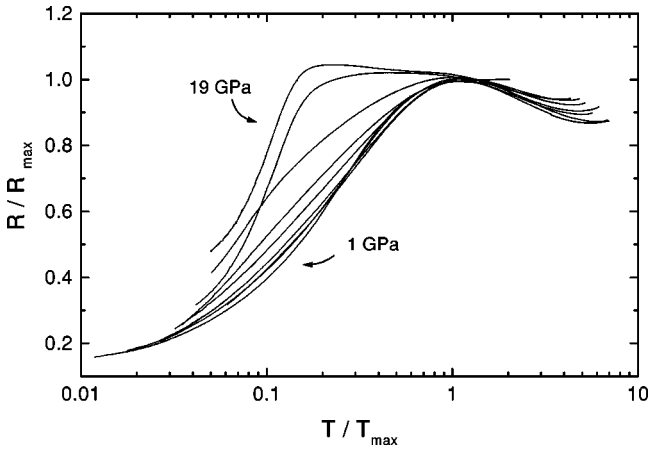

FIG. 6. Resistance vs temperature for $YbCu_2Si_2$ at different pressures plotted on normalized scales as $R/R_{max}(p)$ vs $T/T_{max}(p)$. The pressure increases from the bottom to the top: 1 GPa (lower curve), 2.3 GPa, 4.2 GPa, 6.1 GPa, 6.7 GPa, 7.1 GPa, 9.0 GPa, 10.0 GPa, 15 GPa, and 19 GPa (upper curve).

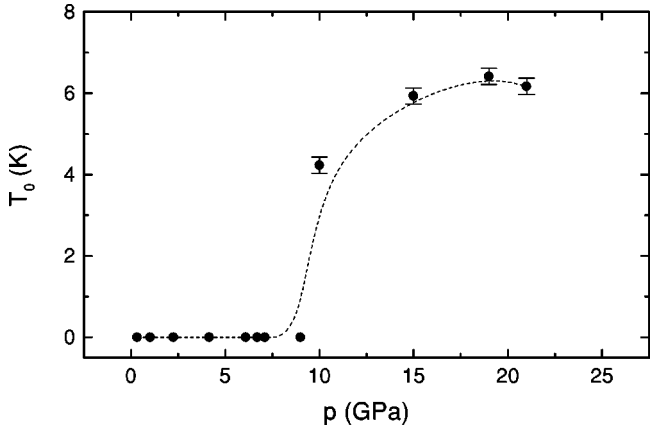

FIG. 7. Volume dependence of the pressure-induced magnetic ordering temperature (T_0) in $YbCu_2Si_2$ as deduced from the high-pressure resistance data.

temperature because T_{max} is rather large. The breakdown of scaling with increasing pressure in the high-temperature range ($T/T_{max}>1$) indicates that T_{CEF} plays the dominant role while at the lowest temperatures ($T/T_{max}<1$) it points to the growing importance of the RKKY interaction as shown in the measured curve at $p \geqslant 9$ GPa.

As already pointed out above (see Fig. 4) the resistance curves change their low-temperature shape for pressure $p > 7$ GPa. This behavior is attributed to the onset of magnetic ordering at T_0. Our measurements allow us to determine the pressure (volume) dependence of T_0 which was defined as the temperature where d^2R/dT^2 shows a minimum in the temperature range $T<10$ K (Fig. 7). The T_0 estimated as above are somewhat higher than those reported by Alami-Yadri *et al.*,[10] but both sets of data can be reconciliated if T_0 in Ref. 10 is defined as the temperature where the resistivity versus temperature derivative $d\rho/dT$, at $T < 10$ K, shows its first anomaly when decreasing the temperature. The critical pressure p_c where magnetic order is induced is found to be close to 8 GPa, in good agreement with the high-pressure Mössbauer results (see below). Figure 7 shows that $T_0(p)$ rises steeply before saturating at $p \geqslant 19$ GPa. This behavior mirrors the one observed in Ce compounds, where application of pressure suppresses magnetic order,[23] and results from a delicate balance between the Kondo and RKKY interactions.[24] Further insights that pressure induces a magnetically ordered state in $YbCu_2Si_2$ are provided by the T^3 dependence of the low-temperature resistance observed above the critical pressure as well as by thermopower data.[11]

C. High-pressure ^{170}Yb Mössbauer measurements

1. Electronic ground state at ambient pressure

The ambient pressure Mössbauer experiments were performed using a standard $YbCu_2Si_2$ absorber, i.e., outside the pressure cell, at 4.2 K and 1.8 K (Fig. 8). The spectra at both temperatures can be well fitted assuming the presence of an axial quadrupole interaction only with $eQV_{zz}=4.0(2)$ mm/s at 4.2 K, in accordance with previous results.[25] No sign of magnetic hyperfine splitting was observed down to 1.8 K. The principal component V_{zz} of the EFG was estimated to

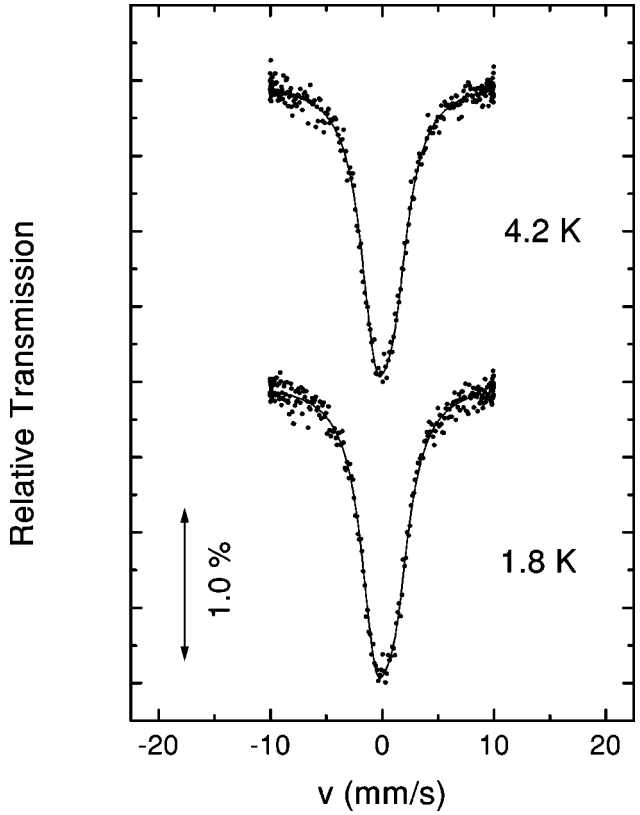

FIG. 8. ^{170}Yb Mössbauer spectra recorded at ambient pressure at 4.2 K and 1.8 K.

amount to $-5.3(3)\times10^{21}$ V/m^2 taking $Q=-2.11\times10^{-28}$ m^2 for the value of the quadrupole moment of the excited ($I_{ex}=2$) state. V_{zz} is made of three main components. There is in general a dominant contribution V_{zz}^{4f}, which originates from $4f$ electrons in a partially filled shell, a component V_{zz}^{latt} from the lattice charges, and a contribution arising from the aspherical distribution of the valence $6p$ and $5d$ electrons, $V_{zz}^{6p.5d}$. The sum of the last two contributions, $V_{zz}^{latt}+V_{zz}^{6p,5d}$, can be estimated from the quadrupole splitting data of ^{155}Gd in the isostructural $GdCu_2Si_2$ compound.[26,27] For the *S*-state ion Gd^{3+}, V_{zz}^{4f} vanishes and $V_{zz}=V_{zz}^{latt}+V_{zz}^{6p,5d}$. From the ^{155}Gd experimental results one deduces $(V_{zz}^{latt}+V_{zz}^{6p,5d})\simeq2.90\times10^{21}$ V/m^2. It follows that V_{zz}^{4f} in $YbCu_2Si_2$ amounts to $\simeq-8.2\times10^{21}$ V/m^2 at 4.2 K, i.e., $eQV_{zz}^{4f}\simeq6.2$ mm/s. As expected V_{zz}^{4f} is the major contribution to V_{zz} but the other components cannot be neglected; they represent about 35% of the $4f$ contribution. It is difficult to differentiate experimentally V_{zz}^{latt} and $V_{zz}^{6p.5d}$; nevertheless, it was shown from EFG computations based on band structure calculations for $GdCu_2Si_2$ that $V_{zz}^{6p.5d}=4.8\times10^{21}$ V/m^2 overcomes by a factor of about 2 (with opposite sign) the V_{zz}^{latt} contribution.[28]

V_{zz}^{4f} at 4.2 K and at ambient pressure is rather small; it represents only about 30% of the free-ion Yb^{3+} value. This value as well as its unusual temperature dependence[25,29] was explained straightforwardly by a theoretical model,[29,30] taking into account hybridization between $4f$ and conduction electron states together with crystal electric field effects. The results of this model agree well with the weak ($\Delta v\approx0.1$)

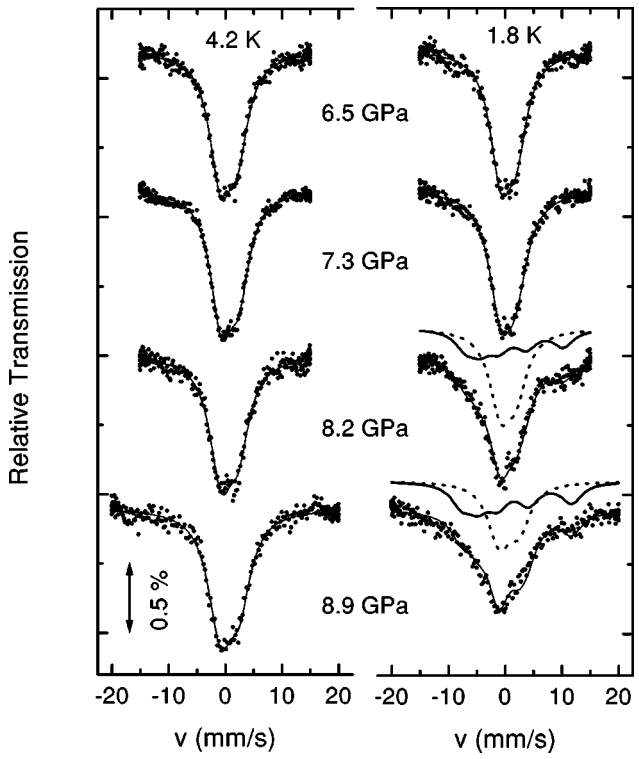

FIG. 9. Typical ^{170}Yb Mössbauer spectra recorded at 4.2 K and 1.8 K at different pressures. Mössbauer spectra in the magnetically ordered state were fitted by a superposition of a magnetic (solid line) and nonmagnetic (dashed line) component (see text).

temperature dependence of the Yb valence observed experimentally.[31]

2. Pressure-induced valence change

The Mössbauer spectra recorded at 4.2 K and 1.8 K at various pressures up to 8.9 GPa are shown in Fig. 9. Spectra at 8.9 GPa and at 4.2 K and 80 K are shown in Fig. 10.

At 4.2 K and 1.8 K (up to 7.3 GPa) one observes only pure quadrupole split spectra with slightly broadened lines ($W=4.9$ mm/s vs 3.2 mm/s at $p=0$) and where eQV_{zz} has increased by a factor of 2 from 4 mm/s at ambient pressure to about 8 mm/s at 8.9 GPa at 4.2 K (Fig. 11). The line broadening could be attributed either to little nonhydrostaticity effects (the pressure gradient across the the sample is less than 10% at 8.9 GPa) or to relaxation effects as observed in other Yb intermetallics.[32,33]

The pressure dependence of eQV_{zz} has to be related to the volume dependence of the different contributions to V_{zz}. Whereas V_{zz}^{latt} is expected to have little variation, any pressure-induced valence shift towards $v=3$ will increase V_{zz}^{4f}. The pressure dependence of $V_{zz}^{6p,5d}$ is so far unknown and difficult to anticipate because it varies from one system to another. While it is strongly volume dependent in Gd metal[34] and in YbNiSn,[35] little pressure effects on the EFG were observed in some Gd intermetallics[34] and in Yb_2Ni_2Al.[18] Nevertheless, similar pressure enhancements of V_{zz} were observed in other intermediate-valent Yb compounds[32,33] and ascribed to valence changes. For example, the pressure-induced increase of eQV_{zz} in YbCuAl (Ref. 32) up to 5.4 GPa at 4.2 K, due to a minor change of the Yb valence from $\approx$2.8 to about 2.9, is more than a factor of 2. Thus, it may be concluded that the observed pressure dependence of V_{zz} is mainly due to the pressure-induced valence shift towards the Yb^{3+} state.

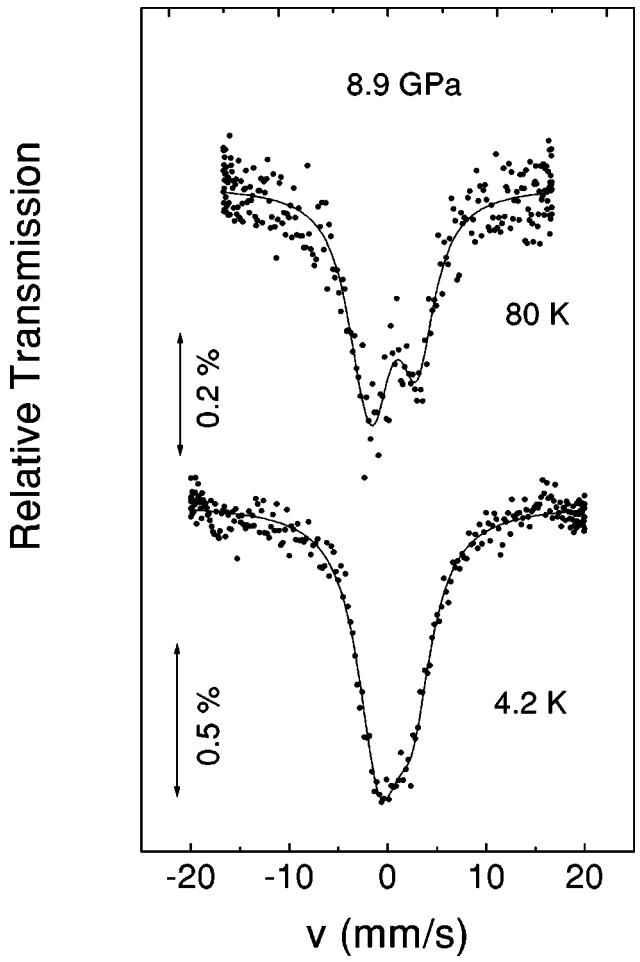

FIG. 10. ^{170}Yb Mössbauer spectra collected at 8.9 GPa and at 4.2 K and 80 K.

The temperature dependence of eQV_{zz} at 6.5 GPa and 8.9 GPa is shown in Fig. 11 together with the ambient pressure

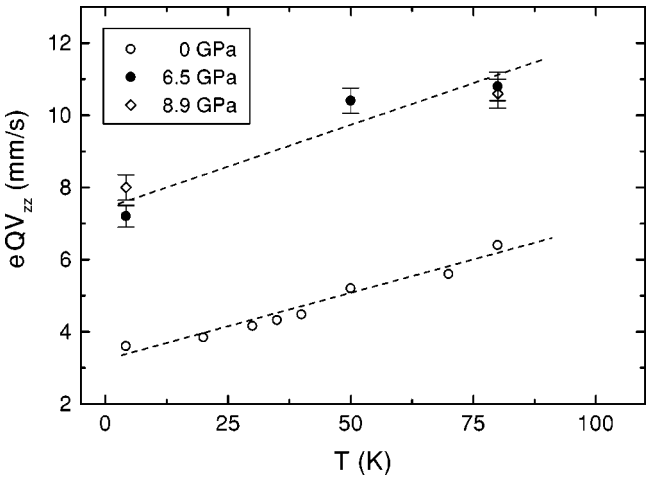

FIG. 11. Temperature dependence of eQV_{zz} at ambient pressure (○) as taken from Ref. 25 and at 6.5 GPa and 8.9 GPa (●, ◇, present work).

data of Ref. 25. Here we observe a much weaker dependence of eQV_{zz} at $p=8.9$ GPa compared to that at ambient pressure. This finding is consistent with a valence change towards Yb^{3+}. The fact that eQV_{zz} at high pressure increases with increasing temperature (instead of decreasing), as reported in YbCuAl (Ref. 32) and $YbPd_2Si_2$ (Ref. 33), could be explained by a temperature-dependent contribution of the $6p,5d$ electrons to eQV_{zz} in $YbCu_2Si_2$.

3. Pressure-induced magnetic ordering

For the spectra recorded at the highest pressures (8.2 GPa and 8.9 GPa) and at 1.8 K a strong change in their shape is evident (Fig. 9). These data provide the most clear sign of a pressure-induced magnetic ordering in $YbCu_2Si_2$. The analysis of the spectral shapes shows that they cannot be accounted for assuming either a single magnetic component or magnetic relaxation within an exchange-split-isolated Kramers doublet.[36] Best fits to the data were obtained with two subspectra corresponding to a well-separated magnetic and nonmagnetic component. During the least-squares fitting procedure which includes diagonalization of the combined magnetic and quadrupole Hamiltonian, the linewidth and the quadrupole splitting were fixed to their paramagnetic state values at 4.2 K. The hyperfine field B_{eff}, the angle Θ between V_{zz} and B_{eff}, and the relative spectral areas of both subspectra were kept as free parameters. For $p=8.9$ GPa the magnetic subspectrum which represents about 50% of the total spectral area can be approximated with $B_{eff}=127(4)$ T and $\Theta=21(5)°$. Both B_{eff} and the magnetic fraction increase with increasing pressure. The value of the Yb magnetic moment is estimated to amount to $\mu_{Yb}\simeq 1.25\mu_B$ at 8.9 GPa, using the relation $B_{eff}=C\mu_{Yb}$ where $C=102$ T/μ_B.[37] The value of the Yb magnetic moment is rather large and compares with those obtained in magnetically ordered Kondo-lattice Yb compouds, e.g., YbNiSn (Refs. 35 and 37) and YbPtAl (Ref. 38).

Although the ordering temperature T_0 cannot be estimated accurately from our Mössbauer data, the observed behavior is consistent with the T_0 values determined by resistance measurements (Fig. 7). The coexistence of a magnetic and nonmagnetic component may be ascribed to a first-order pressure-induced transition as reported recently for Yb_2Ni_2Al,[18] but one cannot at this stage disregard other explanations: the formation of magnetic clusters in the paramagnetic phase (Griffiths phase) (Ref. 39) for $p\geqslant 8.2$ GPa and $T\leqslant 1.8$ K. To clarify this last point ^{170}Yb Mössbauer measurements at lower temperatures and at higher pressures are highly desired.

IV. CONCLUSIONS

We have investigated the effect of pressure on the ground state properties of the nonmagnetic intermediate valent compound $YbCu_2Si_2$ using the x-ray diffraction, electrical resistance, and the ^{170}Yb Mössbauer effect techniques. We observe a pressure-induced change of the electric quadrupole splitting, indicating a valence shift of Yb towards the Yb^{3+} state. At $p\geqslant 8$ GPa and below 2 K we find a crossover from the nonmagnetic intermediate-valent state to a magnetically ordered state. The values of the magnetic hyperfine fields in the high-pressure phase indicate that the magnetic ordering is due to localized Yb magnetic moments.

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