Si-NMR study of antiferromagnetic heavy-fermion compounds $CePd_2Si_2$ and $CeRh_2Si_2$

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(Received 27 January 1998)

We report Si-NMR studies on the magnetic property of pressure-induced superconductors $CePd_2Si_2$ and $CeRh_2Si_2$, which exhibit antiferromagnetic (AF) order with the Néel temperature $T_N = 10$ K and 36 K at ambient pressure, respectively. The NMR results in $CePd_2Si_2$ are consistent with those obtained from the previous neutron-diffraction (ND) experiments. On the other hand, the NMR study in oriented powder $CeRh_2Si_2$ has revealed that the spectrum splits into two peaks due to the onset of the AF order with the wave vector of $\boldsymbol{q}_1 = (1/2\,1/2\,0)$ below $T_{N1} = 36$ K and each peak splits further into two peaks below $T_{N2} = 25$ K due to the formation of AF domains with $\boldsymbol{q}_2 = (1/2\,1/2\,1/2)$. The saturation moment $M_{AF}(\mathrm{NMR}) = 0.36$ and $0.22\mu_B$ estimated from NMR are significantly smaller than $M_{AF}(\mathrm{ND}) = 1.86$ and $1.69\mu_B$ from ND. From this discrepancy in the sizes of M_{AF}, it is proposed that a correlation time in fluctuations of *f*-electron moments is longer than the characteristic time of observation for thermal neutrons but shorter than that for NMR. This probe dependence of M_{AF} was observed in the uranium heavy-fermion (HF) compounds UPt_3 and URu_2Si_2. From the temperature dependence of the nuclear spin-lattice relaxation rate $1/T_1$ in the paramagnetic state, the Kondo temperature T_K in $CeRh_2Si_2$ is estimated to be around ~ 100 K, much higher than $T_K \sim 12$ K in $CePd_2Si_2$. The $1/T_1$'s in both the compounds decrease markedly below T_N, followed by a $T_1T =$ constant behavior far below T_N. From the latter result, it is suggested that low-lying excitations at low temperatures are dominated by quasiparticle excitations in the AF ordered state in $CePd_2Si_2$ and $CeRh_2Si_2$. [S0163-1829(98)00837-6]

I. INTRODUCTION

A series of Ce-based compounds with the $ThCr_2Si_2$-type structure possess various types of ground states, including superconductivity at ambient pressure, pressure-induced superconductivity near an antiferromagnetic (AF) to nonmagnetic phase boundary, long-range magnetic order, and a nonmagnetic phase with magnetic correlations.[1] A parameter controlling physical properties is believed to be a strength in the hybridization between conduction electrons and f electrons (c-f hybridization). A competition between the Ruderman-Kittel-Kasuya-Yosida (RKKY) and the Kondo interaction, both of which originate from the c-f hybridization, governs what types of ground states are realized.[2] In the case of weak hybridization, the RKKY interaction is dominant, leading to a long-range magnetic order. In contrast, in the case of strong hybridization, the Kondo interaction is dominant, leading to a nonmagnetic valence fluctuating state. For heavy-fermion (HF) states in the case of intermediate c-f hybridization, superconductivity or itinerant magnetism with tiny magnetic moments was reported so far. Pressure-induced AF to a nonmagnetic phase transition is the focus of recent experiments since non-Fermi-liquid behaviors deviating from the canonical Fermi-liquid concept are observed[3] and superconductivity is realized close to such a phase boundary.[4]

$CePd_2Si_2$ and $CeRh_2Si_2$ show the AF order at $T_N \sim 10$ K and 36 K, respectively.[5] In $CePd_2Si_2$, the neutron-diffraction (ND) experiments clarified that the AF spin structure has a wave vector of $\boldsymbol{q} = (1/2\,1/2\,0)$ and its saturation moment is $M_{AF} \sim 0.7\mu_B$. It was reported that the AF order is suppressed by an application of pressure, $P_c \sim 2.5$ GPa,[6–9] which is much smaller than $P_c \sim 7.6$ GPa for $CeCu_2Ge_2$.[4] Remarkably, an onset of superconductivity has been found below $T_c \sim 0.3$ K under a pressure of 2.7 GPa.[7] In $CeRh_2Si_2$, the previous ND (Refs. 5 and 10) experiments revealed that there exist two AF phases with the wave vector of $\boldsymbol{q}_1 = (1/2\,1/2\,0)$ below $T_{N1} \sim 36$ K and $\boldsymbol{q}_2 = (1/2\,1/2\,1/2)$ below $T_{N2} \sim 25$ K.[11,12] An application of $P_c \sim 0.9$ GPa, which is much lower than $P_c \sim 2.5$ GPa in $CePd_2Si_2$, is reported to be enough to destroy the AF order with $T_{N1} = 36$ K completely.[6,9,13,14] Pressure-induced superconductivity was reported in a range of 0.5–1.6 GPa where the superconducting transition temperature reaches a maximum value of $T_c \sim 0.35$ K, although the subsequent experiments have not yet confirmed the onset of superconductivity.[13] $CePd_2Si_2$ and $CeRh_2Si_2$ are thus suitable systems for the investigation of magnetic and electronic properties in the vicinity of the AF magnetic to nonmagnetic phase boundary.

In this paper we report the Si-NMR studies of both compounds at ambient pressure. $CePd_2Si_2$ is a conventional AF magnet with $T_N = 10$ K and a Kondo temperature $T_K \sim 12$ K. By contrast, the magnetic properties in $CeRh_2Si_2$ are found to be anomalous. T_K is estimated to be as high as 100 K, whereas T_N has a record high value of $T_{N1} = 36$ K in cerium-based HF AF compounds. Below $T_{N2} = 25$ K, the two AF domains coexist with different wave vectors and saturation moments. A remarkable finding is that the sizes in $M_{AF}(\mathrm{NMR})$ obtained from NMR are much smaller than $M_{AF}(\mathrm{ND})$ from ND. This suggests that a correlation time of fluctuations in f-electron moments is longer than the characteristic time of observation for thermal neutrons but shorter than that for NMR.

II. EXPERIMENTAL PROCEDURES

Polycrystal ingot samples of $CePd_2Si_2$, $CeRh_2Si_2$, $LaPd_2Si_2$, and $LaRh_2Si_2$ were prepared by an argon-arc fur-

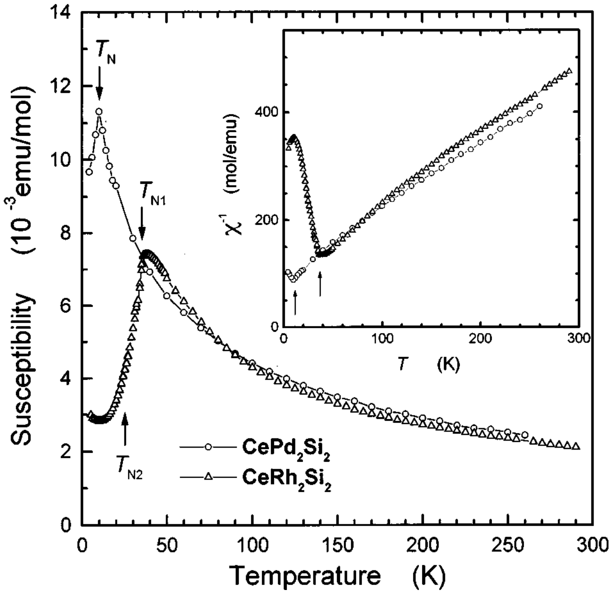

FIG. 1. Temperature dependence of the magnetic susceptibilities, $\chi(T)$ of $CePd_2Si_2$ and $CeRh_2Si_2$. Inset shows the inverse susceptibilities, $\chi^{-1}(T)$.

nace. Stoichiometric quantities of Ce (3N), La (3N), Pd (3N), Rh (3N), and Si (5N) were melted in an argon atmosphere with zirconium getters, followed by annealing in vacuum for 4–5 days at 1000 °C in $CePd_2Si_2$ and $LaPd_2Si_2$, and at 800 °C in $CeRh_2Si_2$ and $LaRh_2Si_2$. The x-ray diffraction confirmed that all the samples are of a single phase with the $ThCr_2Si_2$-type structure. The magnetic susceptibility was measured at 1 kOe by using a superconducting quantum interference device magnetometer. The temperature (T) dependence of susceptibilities, $\chi(T)$ in $CePd_2Si_2$ and $CeRh_2Si_2$, are indicated in Fig. 1. $\chi(T)$ has a sharp cusp at 10 and 36 K for $CePd_2Si_2$ and $CeRh_2Si_2$, respectively, pointing to an onset of AF order. From the respective Curie-Weiss behavior in $\chi(T)$ above 60 and 100 K in $CePd_2Si_2$ and $CeRh_2Si_2$, an effective paramagnetic moment $\mu_{\rm eff}$ is estimated to be 2.61 and $2.58\mu_B$, which is close to $2.54\mu_B$ for the trivalent Ce^{3+} state. These results are consistent with the previous results.[15,16] The samples were crushed into powder with a size smaller than $\sim 38 \mu$m for NMR measurements. The Si NMR was carried out by a conventional phase-coherent laboratory-built pulsed NMR spectrometer. A field-swept Si-NMR spectrum was obtained by using a boxcar integrator at a constant frequency of 11.1 and 25.14 MHz for $CePd_2Si_2$ and $CeRh_2Si_2$ in a T range of 1.4–300 K, respectively. The nuclear spin-lattice relaxation time T_1 was measured by the saturation recovery method.

III. RESULTS AND DISCUSSIONS

A. NMR spectrum, Knight shift, and internal field

1. $CePd_2Si_2$

Figure 2(a) indicates the T dependence of the Si-NMR spectrum above $T_N = 10$ K for partially-oriented powder $CePd_2Si_2$, where the [110] direction is parallel to an external field H_0. As compared with the spectrum of the unoriented powder sample at 11.4 K shown in the inset of Fig. 2(a), the

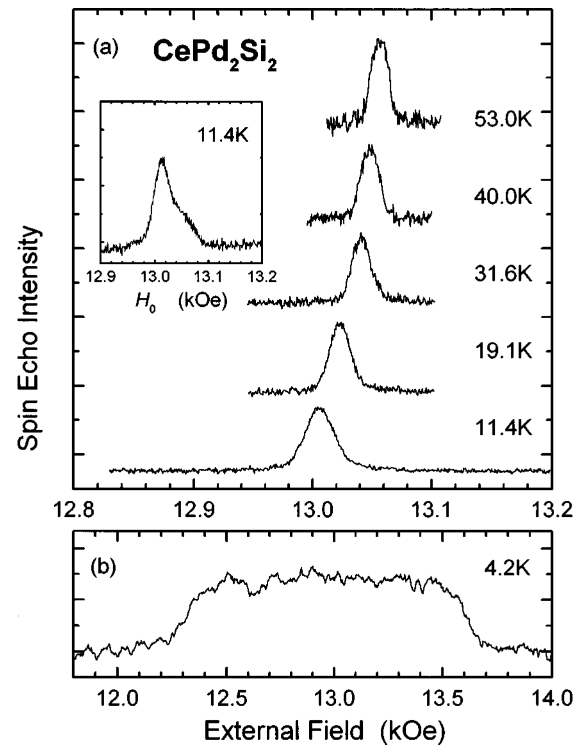

FIG. 2. The Si-NMR spectra of oriented powder $CePd_2Si_2$ with the [110] direction parallel to the external field (a) above and (b) below T_N. Inset shows the NMR spectrum for the unoriented powdered sample.

NMR linewidth in the oriented powder is one-third narrower than that in the unoriented one. This allows us to measure the Knight shift precisely. Figure 3 shows the T dependence of the Knight shift $K_{ab}(T)$ parallel to [110]. The inset of Fig. 3 indicates the $K_{ab}(T)$ vs $\chi_{ab}(T)$ plot with the temperature as an implicit parameter. The susceptibility, χ_{ab} parallel to

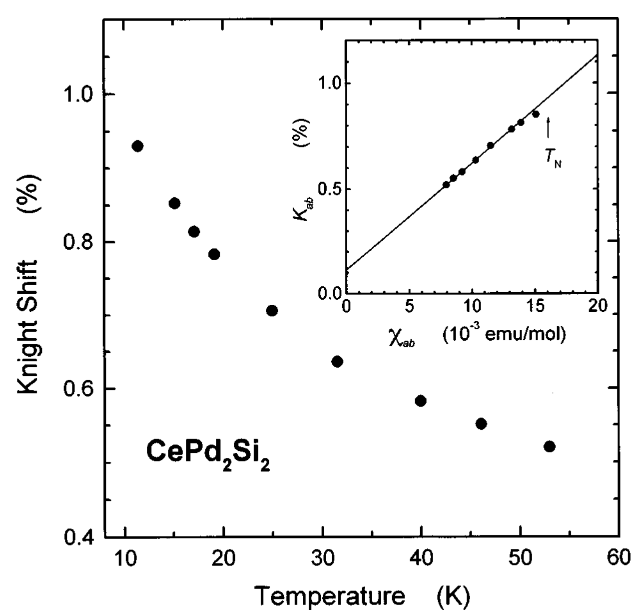

FIG. 3. Temperature dependence of the Knight shift K_{ab} parallel to the [110] direction. Inset shows the $K_{ab}(T)$ vs $\chi_{ab}(T)$ plot for $CePd_2Si_2$. Solid line indicates a best fit to the data with $A_{\rm hf} = 2.84$ kOe/μ_B.

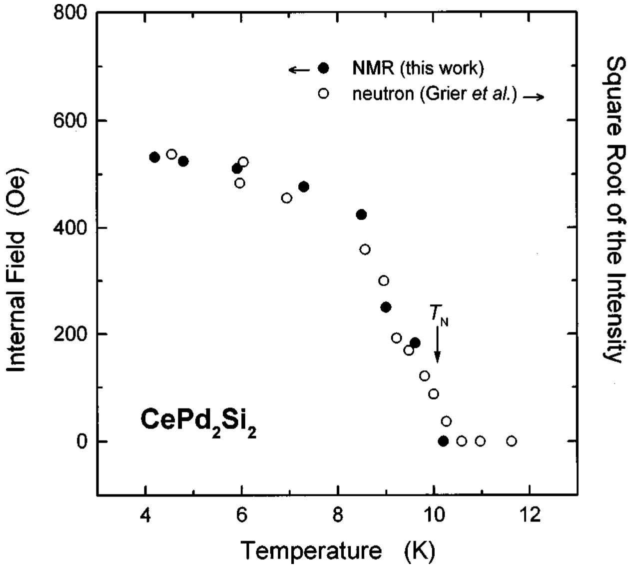

FIG. 4. Temperature dependence of the half width at half maximum (HWHM) in the Si-NMR spectrum of $CePd_2Si_2$ below T_N. HWHM is proportional to the internal field H_{int}. Open circle shows the square root of the neutron magnetic scattering intensity (Ref. 5).

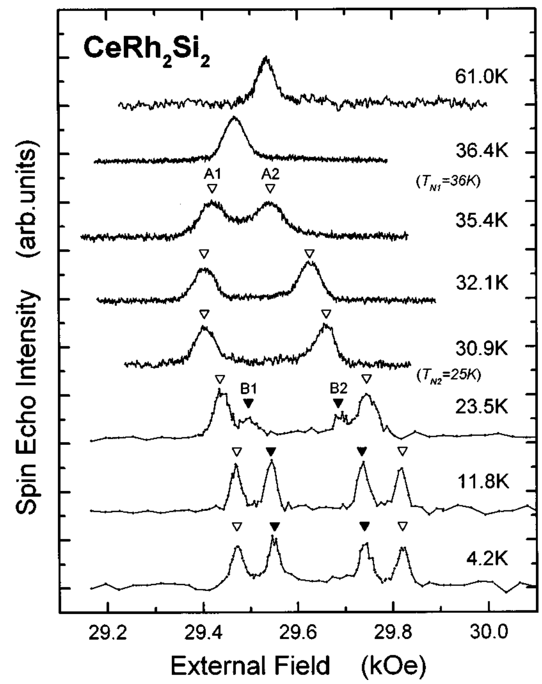

FIG. 5. The Si-NMR spectra of $CeRh_2Si_2$ at $f=25.14$ MHz with the c axis parallel to the external field.

[110] in the single crystal was used.[15] K_{ab} is nearly proportional to χ_{ab}. From a linear fit to the data, as drawn by solid line, a hyperfine-coupling constant $A_{hf}=2.84$ kOe/μ_B is obtained from the relation of $K_{ab}=(A_{hf}/N\mu_B)\chi_{ab}$, where N is the Avogadro's number and μ_B is the Bohr magneton.

Below T_N, the spectrum is markedly affected by the appearance of the internal field H_{int} at the Si sites, as seen in Fig. 2(b). The spectrum forms a rectangular shape characteristic of the AF powder pattern for $H_0 \gg H_{int}$. The orientation of the powder seems to be disturbed below T_N, associated with some change in the anisotropy of susceptibility. Figure 4 shows the T dependence of the half width at half maximum (HWHM) proportional to H_{int}. As seen in the figure, H_{int} increases to ~ 530 Oe at 4.2 K as the temperature decreases below T_N. It is evident that $H_{int}(T)$ is in good agreement with the T dependence of the square root of the ND intensity proportional to M_{AF}.[5] In this AF-spin structure with $\boldsymbol{q}=(1/2\,1/2\,0)$, the direction of M_{AF} is aligned along [110] and, hence, H_{int} is produced through the transferred hyperfine interaction with one Ce AF moment in the five nearest-neighbor Ce sites. This is because the contributions from the four Ce AF moments in the basal plane are canceled out at the Si sites. The isotropic transferred hyperfine-coupling constant H_{thf} per Ce $1\mu_B$ is estimated to be $A_{hf}/5 \sim 568$ Oe/μ_B. By using $H_{int}=532$ Oe, an estimate of its size at 4.2 K gives rise to $M_{AF}(\mathrm{NMR}) \sim 0.94\mu_B$ from the ratio of 532(Oe)/568(Oe/μ_B), which is somewhat larger than $M_{AF}(\mathrm{ND}) \sim 0.7\mu_B$.

2. $CeRh_2Si_2$

Figure 5 indicates the T dependence of the Si-NMR spectrum at 25.14 MHz for the oriented powder $CeRh_2Si_2$ with the c axis parallel to H_0. The NMR spectrum above T_N consists of a single peak. The Knight shift K_c increases upon cooling, in proportion to the susceptibility, as seen in Fig. 6. The inset displays the K_c vs χ_c plot with χ_c parallel to the c axis for the single crystal.[11] From a slope in the K_c vs χ_c plot, a hyperfine-coupling constant is deduced to be $A_{hf}=2.34$ kOe/μ_B.

As seen in Fig. 5, the spectrum below $T_{N1}=36$ K splits into two peaks ($A1$ and $A2$) due to the appearance of the first AF phase with $\boldsymbol{q}_1=(1/2\,1/2\,0)$. Below $T_{N2}=25$ K, where the secondary AF phase sets in, the two peaks ($B1$ and $B2$) newly appear, as seen in the figure. The intensities of $A1$ and $A2$ decrease rapidly below $T_{N2}=25$ K but remain about half volume. The intensities of $A1$, $A2$, $B1$, and $B2$ are

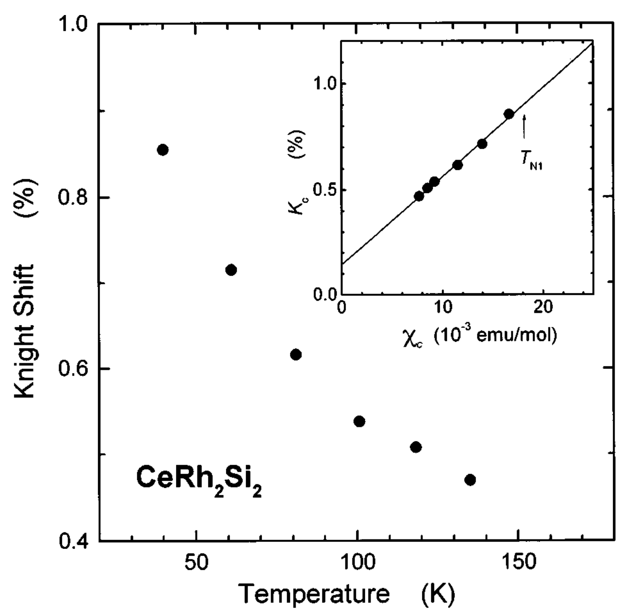

FIG. 6. Temperature dependence of the Knight shift K_c in $CeRh_2Si_2$. Inset shows $K_c(T)$ vs $\chi_c(T)$ plot. Solid line indicates a best fit to the data with $A_{hf\parallel}=2.34$ kOe/μ_B.

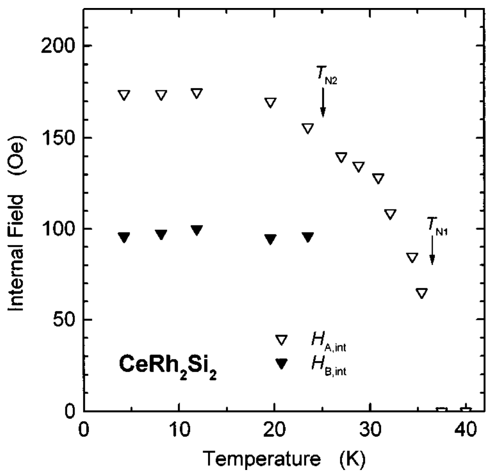

FIG. 7. Temperature dependence of the internal fields $H_{A,\,int}(T)$ and $H_{B,\,int}(T)$ that are obtained from the half value of separation in the A and B peaks in Fig. 5.

independent of the temperature below ~ 23 K where both the intensities for the A and B peaks are comparable to each other. Figure 7 shows the respective T dependence of $H_{A,\,int}(T)$ and $H_{B,\,int}(T)$, which are obtained from a half value of separation in the A and B peaks. $H_{A,\,int}=161$ Oe and $H_{B,\,int}=101$ Oe stay constant below 23 K, which means that both the M_{AF}'s are totally saturated just below $T_{N2}=25$ K.

The ND measurements revealed that the two independent magnetic reflections with $\boldsymbol{q}_1=(1/2\,1/2\,0)$ and $\boldsymbol{q}_2=(1/21/21/2)$ coexist at low temperatures, indicative of two possible magnetic structures.[5,10] One scenario is that two AF phases exist with different magnetic moments and AF wave vectors, $M_{q_1}=1.86\mu_B$ with $\boldsymbol{q}_1$ (denoted as the $\boldsymbol{q}_1$ domain) and $M_{q_2}=1.69\mu_B$ with $\boldsymbol{q}_2$ (denoted as the $\boldsymbol{q}_2$ domain). Another one is that the two magnetic structures are superimposed in the single magnetic phase, which yields the modulated structure with two kinds of moments, 2.52 and $0.12\mu_B$. For either case, the two Si sites should exist with different H_{int}. The ratio of the internal field $H_{A,int}/H_{B,int}=161/101\sim 1.59$, deduced from NMR is comparable to the ratio of M_{AF}, $M_{q_1}/M_{q_2}=1.86/1.69\sim 1.1$ for the former scenario, but far from $2.52/0.12\sim 21$ for the latter scenario. From the NMR spectrum combined with the ND results, it is concluded that the two AF domains coexist below T_{N2}. The comparable NMR intensities in the A and B peaks indicate that each domain occupies half volume.

In estimating M_{AF} for each domain, we notice that one Ce moment in the five nearest-neighbor Ce sites yields a dominant transferred hyperfine field, H_{thf}, at the Si sites. This is because a sum of H_{thf} at the Si sites arising from the four Ce moments in the basal plane becomes zero in $CeRh_2Si_2$ as well as in $CePd_2Si_2$. From the relation of $H_{thf}=A_{hf}/5=467$ Oe/μ_B with $A_{hf}=2.34$ kOe/μ_B, the M_{AF} in the $\boldsymbol{q}_1$ and $\boldsymbol{q}_2$ domains are estimated to be 0.36 and $0.22\mu_B$, respectively. The result is that $M_{AF}(\mathrm{NMR})=0.36$ and $0.22\mu_B$ are more significantly reduced than $M_{AF}(\mathrm{ND})=1.86$ and $1.69\mu_B$ in the $\boldsymbol{q}_1$ and $\boldsymbol{q}_2$ domain, respectively. This is in a striking contrast with the result in $CePd_2Si_2$ where $M_{AF}(\mathrm{NMR})\sim 0.9\mu_B$ is comparable with $M_{AF}(\mathrm{ND})\sim 0.7\mu_B$. The fact that M_{AF} is apparently probe dependent in $CeRh_2Si_2$ shows that the correlation time in fluctuations of f-electron moments is longer than the characteristic time of observation for thermal neutrons but shorter than for NMR.

The probe dependence of M_{AF} was reported in the uranium HF compounds UPt_3 (Refs. 17–19) and URu_2Si_2.[20–22] In these compounds the saturation moments are as small as $\sim 10^{-2}\mu_B$ from ND, but no indication of H_{int} from NMR was observed. The AF saturation moments in $CeRh_2Si_2$, which are two orders of magnitude larger than those in UPt_3 and URu_2Si_2, may make it easy to detect the reduced magnetic moments in $CeRh_2Si_2$ by NMR. Such a probe-dependent aspect of M_{AF} in $CeRh_2Si_2$ may be explored from the μSR experiment as well.

The recent ND result on the single crystal,[23] which is consistent with the previous ND results by Grier *et al.*,[5] has revealed that the $\boldsymbol{q}_1$ domain involves two structures with crystallographically equivalent wave numbers of $\boldsymbol{q}_1^+=(1/2\,1/2\,0)$ and $\boldsymbol{q}_1^-=(-1/2\,1/2\,0)$. Combined with the NMR results, they concluded that the three phases form a AF domain-superlattice structure with $1.42\mu_B$ for the $\boldsymbol{q}_1^+$ and $\boldsymbol{q}_1^-$ domains and $1.34\mu_B$ for the $\boldsymbol{q}_2$ domain. We stress that these values are significantly different from those obtained from NMR as well. In magnetic structures with wave vectors of $(1/2\,1/2\,C)$ in the body-centered lattice, the magnetic interaction is independent of C between the nearest-neighbor c planes and, hence, has a similar energy between each domain. This may be the main reason why the AF domain-superlattice structure is stabilized as the peculiar ground magnetic structure. Furthermore, due to the small energy difference between each domain, thermal and/or quantum fluctuations of each domain are likely sources for the estimate in M_{AF} being probe dependent in $CeRh_2Si_2$. This magnetic state may be responsible for the small critical pressure $P_c\sim 0.9$ GPa, regardless of the large value of T_{N1}.

B. Nuclear spin-lattice relaxation rate, $1/T_1$

Measurements of the nuclear spin-lattice relaxation rate $1/T_1$ were performed at $f=11.1$ MHz for $CePd_2Si_2$, $LaPd_2Si_2$, and $LaRh_2Si_2$. The $1/T_1$ in $CeRh_2Si_2$ was measured at $f=25.1$ MHz at the A1 peak in an entire temperature range. $1/T_1$ in all the compounds was uniquely determined with a single T_1 component. Figure 8 shows the T dependence of $1/T_1$ in $CePd_2Si_2$ (closed circles) and $CeRh_2Si_2$ (closed triangles) together with those in $LaPd_2Si_2$ (open circles) and $LaRh_2Si_2$ (open triangles). A $T_1T=$ constant behavior for $LaPd_2Si_2$ and $LaRh_2Si_2$ is observed with respective value of $(T_1T)^{-1}\sim 0.079$ $(\mathrm{K\cdot sec})^{-1}$ and 0.040 $(\mathrm{K\cdot sec})^{-1}$. The $1/T_1$ in $CeRh_2Si_2$ is in agreement with the previous results.[24]

In the high-temperature region, local spin fluctuations of $4f$ moments dominate the relaxation process, $1/T_1$ being nearly T independent above 12 and 100 K for $CePd_2Si_2$ and $CeRh_2Si_2$, respectively. Empirically, the temperature at which $1/T_1$ starts to decrease from the T-independent behavior corresponds to a Kondo temperature T_K, below which the systems enter a crossover regime towards the HF state. The T_K in $CePd_2Si_2$ is 12 K, which is in good agreement with $T_K\sim 10$ K determined from the quasielastic ND.[25,26] The fact that T_K is comparable with the T_N in $CePd_2Si_2$ implies that

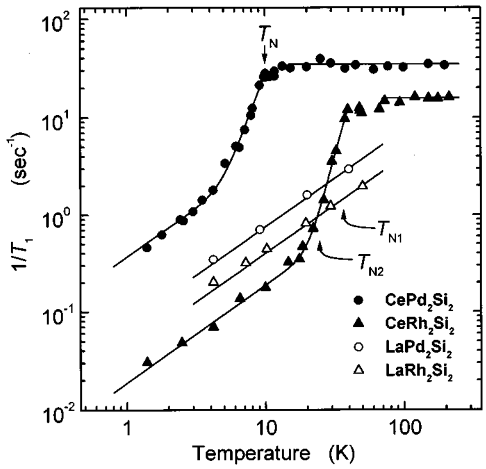

FIG. 8. Temperature dependence of the nuclear spin-lattice relaxation rate $1/T_1$ for $CePd_2Si_2$, $CeRh_2Si_2$, $LaPd_2Si_2$, and $LaRh_2Si_2$.

the AF ordered state occurs before the HF state is fully established, and its nature is anticipated to hold a localized character rather than an itinerant character. On the other hand, the T_K in $CeRh_2Si_2$ is estimated to be as high as ~ 100 K, being much higher than $T_K \sim 33$ K deduced from the quasielastic ND.[26] The fact that T_K is much higher than the T_N in $CeRh_2Si_2$ means that the AF order must be in the itinerant regime, which makes magnetic interactions different from the conventional RKKY interaction.

The $1/T_1$'s in both the compounds drop rapidly below T_N without any critical divergence near T_N. Any anomaly in $1/T_1$ is not appreciable around $T_{N2} = 25$ K in $CeRh_2Si_2$. Experimentally, $1/T_1$ below T_N is well reproduced by the following expression as

$$(T_1T)^{-1} = A + B\exp(-E_g/k_BT),$$

as indicated by the solid line in Fig. 8. An exponential drop in $1/T_1$ may be due to a partial loss of low-lying excitations below T_N. The energy gap E_g in $CePd_2Si_2$ is estimated to be 2.37 meV, which is in good agreement with $E_g \sim 2.3$ meV in the spin-wave excitation spectrum probed by the inelastic ND.[25,27] The first term A is the quasiparticle contribution. The value of $(T_1T)^{-1} \sim 0.33$ $(\mathrm{sec}\cdot\mathrm{K})^{-1}$ in $CePd_2Si_2$ below 4 K is larger than $(T_1T)^{-1} \sim 0.079$ $(\mathrm{sec}\cdot\mathrm{K})^{-1}$ in $LaPd_2Si_2$. This suggests that a part of the f electrons that do not participate in the AF ordered moments may have an itinerant character to form the HF state even far below T_N. On the other hand, the value of $(T_1T)^{-1} \sim 0.0188(\mathrm{sec}\cdot\mathrm{K})^{-1}$ in $CeRh_2Si_2$ below 10 K is smaller than $(T_1T)^{-1} \sim 0.0402(\mathrm{sec}\cdot\mathrm{K})^{-1}$ in $LaRh_2Si_2$. This result indicates that the Fermi-liquid excitation below 10 K is considered to originate from the HF band, which is the consequence of the strong c-f hybridization, since $1/T_1$ cannot be explained by the sum of two relaxation contributions from itinerant $4f$ electrons and conduction electrons. The result of $1/T_1$ also suggests that the magnetic order in $CeRh_2Si_2$ occurs in the HF regime, consistent with above-mentioned experimental results of higher T_K and the small ordered moments.

IV. SUMMARY

The Si-NMR studies in $CePd_2Si_2$ and $CeRh_2Si_2$, which undergo the pressure-induced superconducting transition, have revealed differences in magnetic characteristics. The NMR results in $CePd_2Si_2$ are consistent with those obtained from ND as regarding the sizes in M_{AF}, T_K, and the gap in the spin-wave excitation spectrum.

In contrast, the NMR results in $CeRh_2Si_2$ are unconventional. Another splitting in the NMR spectrum at $T_{N2} = 25$ K was found, indicating a magnetic structure consisting of independent AF domains. A notable result is that $M_{AF}(\mathrm{NMR}) = 0.36$ and $0.22\mu_B$ are significantly smaller than $M_{AF}(\mathrm{ND}) = 1.86$ and $1.69\mu_B$. We have suggested that the correlation time in fluctuations of f-electron moments is longer than the characteristic time of observation for thermal neutrons, but shorter than that for NMR. Relevant to this, we propose that quantum spin fluctuations are responsible for this probe dependence of M_{AF}. A probable source for quantum spin fluctuations may originate from the fluctuations of each domain due to the small difference between the energies of each domain suggested from the recent ND experiments.[23]

ACKNOWLEDGMENTS

We wish to thank S. Kawarazaki, Y. Ōnuki, and Y. Miyako for helpful discussions, and T. C. Kobayashi and K. Amaya for DC-χ measurement. This work was supported by Grants-in-Aid for the Scientific Research on Priority Areas from the Ministry of Education, Science and Culture of Japan.

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