153\Eu{\textsuperscript{153}}} and 69\Ga{\textsuperscript{69,71}} Zero-Field NMR Study of Antiferromagnetic State in EuGa\textsubscript{4}

Mamoru Yogi\textsuperscript{1,}, Saiori Nakamura\textsuperscript{1,}, Nonoka Higa\textsuperscript{1,}, Haruo Niki\textsuperscript{1,}, Yusuke Hirose\textsuperscript{2,}, Yoshichika O\textsuperscript{\textsuperscript{N}}uki\textsuperscript{2,}, and Hisatomo Harima\textsuperscript{3}

\textsuperscript{1}Department of Physics and Earth Sciences, Faculty of Science, University of the Ryukyus, Okinawa 903-0213, Japan
\textsuperscript{2}Graduate School of Science, Osaka University, Toyonaka 560-0043, Japan
\textsuperscript{3}Graduate School of Science, Kobe University, Nada-ku, Kobe 657-8501, Japan

We report 153\Eu and 69\Ga zero-field NMR under a zero magnetic field on the antiferromagnetic state of EuGa\textsubscript{4} with $T_N \approx 16$ K. We have successfully observed a 153\Eu zero-field NMR signal with well-resolved nuclear quadrupole splitting in the antiferromagnetic state of EuGa\textsubscript{4}. 69\Ga zero-field NMR spectra were also observed below $T_N$. The internal field and nuclear quadrupole frequency are estimated from a simulation of the spectra by the exact diagonalization of the nuclear spin Hamiltonian matrix. The asymmetrically split zero-field NMR spectra were explained by considering a configuration of the magnetic moments of Eu\textsuperscript{3+} lying in the basal $ab$-plane. The temperature dependence of the internal field, which is proportional to the sublattice magnetization, can be explained by the Brillouin function with $J = S = 7/2$.

KEYWORDS: europium compound, EuGa\textsubscript{4}, antiferromagnetism, zero-field NMR

Fig. 1. (Color online) (a) Crystal structure of EuGa\textsubscript{4}. (b) Magnetic moments configuration assuming the antiferromagnetic structure type-I (see text).

Strong electron correlations in rare-earth-based compounds induce various interesting physical phenomena such as heavy-electron behavior, multipole order, unconventional superconductivity, spin or valence quantum critical fluctuations, and non-Fermi liquid behavior.\textsuperscript{1–3} Eu is a rare-earth element known to have two kinds of valence states: Eu\textsuperscript{2+} (4$f^7$) and Eu\textsuperscript{3+} (4$f^6$). The divalent Eu state is magnetic ($J = S = 7/2$, $L = 0$), where $J$ is the total angular momentum, $S$ is the spin angular momentum, and $L$ is the orbital angular momentum. Therefore, the compounds with divalent Eu ions tend to order magnetically, following the Ruderman-Kittel-Kasuya-Yosida (RKKY) interaction. In contrast, the trivalent Eu state is nonmagnetic ($J = 0$, $S = L = 3$). Note that the valence of Eu in some compounds is changed by temperature, magnetic field, and pressure.\textsuperscript{4–7} A mixed valence state is also an interesting feature of the compound.

In this study, we focus on the Eu intermetallic compound EuGa\textsubscript{4}, which crystallizes in the BaAl\textsubscript{4}-type tetragonal structure (space group: No.139, $I4/mmm$) shown in Fig. 1(a).\textsuperscript{8,9} Eu atoms occupy the corners and the center of the body-centered lattice with local symmetry ($4/mmm$ in Hermann-Mauguin notation or International notation). As for Ga atoms, they have two crystallographically inequivalent sites, denoted Ga1 (4$m_2$) and Ga2 (4$mm$). A divalent Eu state was demonstrated from an isotropic behavior of the magnetic susceptibility in the paramagnetic state with the effective magnetic moment $\mu_{\text{eff}} \approx 7.86 \mu_B/$Eu.\textsuperscript{9} This is close to a divalent value of 7.94 $\mu_B/$Eu. Antiferromagnetic (AFM) order was confirmed from the measurements of resistivity, magnetic susceptibility, specific heat, and thermoelectric power below a Néel temperature $T_N \approx 16$ K.\textsuperscript{8,9} In the AFM state, the magnetic susceptibility for $H \parallel [100]$ and $[110]$ decreases with decreasing temperature, while the susceptibility for $H \parallel [001]$ is almost unchanged; thus, the magnetic moments are considered to lie in the $ab$-plane.\textsuperscript{9} Moreover, a recent neutron scattering experiment has clarified that the AFM structure is of type-I.\textsuperscript{10} The possible magnetic moment configuration in the AFM state is described by the arrows in Fig. 1(b). In addition, the possible emergence of the charge density wave (CDW) order was also reported from the measurements of electrical resistivity and thermoelectric power under pressure.\textsuperscript{9}

NMR measurement is a powerful technique for investigating magnetic properties from the microscopic viewpoint. The nuclear properties of both Eu and Ga nuclei are listed in Table I.\textsuperscript{11} The NMR of both nuclei is basically possible in EuGa\textsubscript{4}. However, a ligand nucleus is usually used in pulse NMR experiments on rare-earth-based magnetic compounds because the relaxation time of rare-earth nuclei is too short for observing the NMR signal. As for divalent Eu compounds, to the best of our knowledge, the signal of the Eu nucleus is only observed in a magnetically ordered state of Eu chalcogenide compounds EuX ($X = O, S, Se$, and Te).\textsuperscript{12–15} In this circum-

\textsuperscript{1}myogi@sci.u-ryukyu.ac.jp
ments causes the level splitting of the nuclear spin states. Then, the observation of NMR. However, if there is a large internal field and the nuclear magnetic moments in the magnetically ordered state, the Zeeman interaction between the internal field and the nuclear magnetic moment is called the zero-field NMR (ZFNMR).

The NMR spectra were obtained by sweeping the frequency and integrating the spin-echo signal intensity step by step.

The numerical calculation of the NMR resonance frequencies by the exact diagonalization of the nuclear spin Hamiltonian matrix explains well the peak positions of the spectrum. From this calculation, an internal field at the Eu nucleus $H_{\text{int}}^\text{Eu} = 27.08$ T and $\nu_Q = 30.5$ MHz are obtained at 4.2 K; the $H_{\text{int}}^\text{Eu}$ obtained is close to the value of 26.6 T determined by Mössbauer spectroscopy. A broad tail of each peak indicates a microscopic inhomogeneity of the electric state at the Eu site. This asymmetric shape of the spectrum can be explained by considering a small distribution of the EFG. Figure 2(b) shows a calculated spectrum assuming a log-normal distribution of $\nu_Q$, as shown in the inset of Fig. 2. The calculation well explains the observed $^{153}$Eu-ZFNMR spectrum. We infer two possible origins of this distribution. One is an intrinsic origin. An anomaly related to the CDW order was reported from the thermoelectric power measurement at ambient pressure. Therefore, a short range order would occur at ambient pressure, causing a small distribution of the EFG. The other is an extrinsic origin. The powdering of the crystal might cause microscopic distortions to the sample, causing a distribution of the EFG.

Eu has two isotopes, $^{151}$Eu and $^{153}$Eu, as listed in Table I. Using $H_{\text{int}}^\text{Eu} = 27.08$ T, we infer that the $^{151}$Eu-ZFNMR spectrum appears at approximately 270 ~ 300 MHz. However, we could not observe the signal. This is because the relaxation time of $^{151}$Eu is shorter than that of $^{153}$Eu. If a magnetic fluctuation is a predominant relaxation process, the relaxation rate is proportional to $\gamma^2$. Therefore, the relaxation time of $^{151}$Eu

Table 1. Data of Eu and Ga isotopes: nuclear spins $I$, nuclear quadrupole moments $Q$, and natural abundances N.A.\(^{11}\)

|        | $I$  | $\gamma I/2\pi$ (MHz/T) | $Q$ ($10^{-20}$cm$^2$) | N.A. (%) |
|--------|------|--------------------------|------------------------|---------|
| $^{151}$Eu | 5/2  | 10.5854                  | 90.3                   | 47.81   |
| $^{151}$Eu | 5/2  | 4.6744                   | 241.2                  | 52.19   |
| $^{69}$Ga | 3/2  | 10.2475                  | 17.1                   | 60.108  |
| $^{71}$Ga | 3/2  | 13.0204                  | 10.7                   | 39.892  |

![Fig. 2.](image-url) (Color online) (a) $^{153}$Eu-ZFNMR spectrum at $T = 4.2$ K. (b) Calculated spectrum for $^{153}$Eu in the case of $H_{\text{int}}^\text{Eu} \perp V_{zz}$ assuming a log-normal distribution of $\nu_Q$ as shown in the inset.

Figure 2(a) shows the $^{153}$Eu-ZFNMR spectrum at $T = 4.2$ K. $^{153}$Eu has a nuclear spin $I = 5/2$; therefore, five split peaks were observed when there is a finite electric field gradient (EFG). Note that the frequency separations between neighboring peaks are different, which gives us important information on the direction of the internal field. The Eu site in EuGa$_4$ has a tetragonal local symmetry (4/mmm); hence, the EFG becomes axially symmetric, namely, the asymmetry parameter of the EFG becomes zero ($\eta = 0$). In addition, the main principal axis of the EFG, denoted $V_{zz}$, is parallel to the c-axis. Thus, the nuclear spin Hamiltonian in the AFM state is given by

$$H = -\gamma I \cdot H_{\text{int}} + \frac{\hbar \nu_Q}{6} \left[ 3J_z^2 - I^2 \right].$$

The first term of the Hamiltonian represents the Zeeman interaction between the nuclear magnetic moment $\mu_n = \gamma I$ and the internal field $H_{\text{int}}$, where $\gamma I$ is the nuclear gyromagnetic ratio and $I$ is the nuclear spin. The second term in the Hamiltonian represents the nuclear quadrupole interaction between the EFG and the nuclear quadrupole moment $Q$. Here, $\nu_Q$ is the nuclear quadrupole frequency defined by $\nu_Q = 3eQV_{zz}/2(2I - 1)\hbar$. If $H_{\text{int}} \parallel V_{zz}$ and the Zeeman interaction is larger than the nuclear quadrupole interaction, the separations between peaks become the same. On the other hand, if $H_{\text{int}} \perp V_{zz}$, the separations of the peak become inequivalent, which is consistent with the observed $^{153}$Eu-ZFNMR spectrum, indicating ordered magnetic moments lying in the $ab$-plane. This is in good agreement with the results of magnetic susceptibility and neutron diffraction experiments.

The numerical calculation of the NMR resonance frequencies by the exact diagonalization of the nuclear spin Hamiltonian matrix explains well the peak positions of the spectrum. From this calculation, an internal field at the Eu nucleus $H_{\text{int}}^\text{Eu} = 27.08$ T and $\nu_Q = 30.5$ MHz are obtained at 4.2 K; the $H_{\text{int}}^\text{Eu}$ obtained is close to the value of 26.6 T determined by Mössbauer spectroscopy. A broad tail of each peak indicates a microscopic inhomogeneity of the electric state at the Eu site. This asymmetric shape of the spectrum can be explained by considering a small distribution of the EFG. Figure 2(b) shows a calculated spectrum assuming a log-normal distribution of $\nu_Q$, as shown in the inset of Fig. 2. The calculation well explains the observed $^{153}$Eu-ZFNMR spectrum. We infer two possible origins of this distribution. One is an intrinsic origin. An anomaly related to the CDW order was reported from the thermoelectric power measurement at ambient pressure. Therefore, a short range order would occur at ambient pressure, causing a small distribution of the EFG. The other is an extrinsic origin. The powdering of the crystal might cause microscopic distortions to the sample, causing a distribution of the EFG.

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at 4.2 K. The same value of the internal field and the relation becomes 5 ($\approx (151\gamma_n/153\gamma_n)^2$) times shorter than that of $^{153}$Eu. We used the shortest possible $\tau$ of 10 $\mu$s for $^{153}$Eu-ZFNMR, where $\tau$ is the time between the excitation pulse and the refoocusing pulse, implying that $\tau = 2 \mu$s is necessary for observing the $^{151}$Eu-ZFNMR signal. This is difficult for our present pulsed NMR spectrometer.

Next, we focus on the Ga-NMR. Ga has two isotopes, $^{69}$Ga and $^{71}$Ga, with a nuclear spin $I = 3/2$. Therefore, the spectrum consists of the center peak and two satellite peaks due to the nuclear quadrupole interaction for both Ga nuclei. Moreover, there are two independent Ga sites in EuGa$_4$ crystallographically; hence, four spectra are expected in the AFM state of EuGa$_4$. However, we found only two Ga-ZFNMR spectra at $T = 4.2$ K, as shown in Fig. 3. The local symmetries at both Ga sites give rise to an axially symmetric EFG with $V_{zz}$ parallel to the $c$-axis, as in the case of the Eu site. Thus, the nuclear spin Hamiltonian at both Ga sites can be written as eq. (1). The inequivalent separations between the center peak and two satellite peaks indicate that $V_{zz}^{Ga}$ is not parallel to $H_{int}^{Ga}$, which denotes the internal magnetic field at the Ga site. The numerical calculation of the NMR spectrum, assuming $V_{zz}^{Ga} \perp H_{int}^{Ga}$ and a log-normal distribution of $H_{int}^{Ga}$ as shown in Fig. 3(c), explains well the experimental results shown by solid lines in Figs. 3(a) and 3(b). Although the origin of the distribution of $H_{int}^{Ga}$ is not known, we speculate that this relates to the distribution of the EFG at the Eu site. From this calculation, $H_{int}^{Ga} \approx 3.03$ T and $69V_{Q}/71V_{Q} = 5.08(3.21)$ MHz are obtained at 4.2 K. The same value of the internal field and the relation $69V_{Q}/71V_{Q} \approx 69Q/71Q$ reveal that these spectra come from the same Ga site; namely, the spectra come from either the Ga1 or Ga2 site.

Fig. 3. (Color online) (a) and (b) $^{69,71}$Ga-ZFNMR spectrum at $T = 4.2$ K. Solid lines indicate a calculated spectrum for $^{69,71}$Ga in the case of $H_{int}^{Eu} \perp V_{zz}^{Ga}$ assuming a log-normal distribution of the internal field $H_{int}^{Ga}$, as shown in (c).

The magnetic dipole interaction from the four nearest-neighbor Eu moments shown in Fig. 4 with $\mu_{Eu} = 6.07\mu_B$ produces the magnetic dipole fields $H_{dip}^{Ga1} = (0.334, 0, 0)$ T and $H_{dip}^{Ga2} = (0.176, 0, 0)$ T for the Ga1 and Ga2 sites, respectively. This is one order of magnitude smaller than $H_{int}^{Ga}$, indicating that the transferred hyperfine field is dominant for $H_{int}^{Ga}$. Hence, we discuss the internal fields at both Ga sites, considering the short range transferred hyperfine interaction between the Ga nucleus and ordered moments on the four nearest-neighbor Eu sites. A similar analysis was applied to discuss the magnetic structure of BaFe$_2$As$_2$.\cite{17}

First, we focus on the internal field at the Ga1 site. The internal field can be written as the sum of contributions from each Eu site as

$$H_{int}^{Ga1} = \sum_i B_i \cdot m_i \ (i = xz, \bar{x}z, \bar{y}z, \text{and} \, \bar{y}z),$$

(2)

where $B_i$ is the hyperfine coupling tensor between the Ga1 and Eu sites, and $m_i$ is the ordered moment on the Eu$_i$ site. From the local symmetry of the Ga1 site, the components of $B_i$ can be written as

$$B_{xz} = \begin{pmatrix} B_{11} & 0 & B_{13} \\ 0 & B_{22} & 0 \\ B_{31} & 0 & B_{33} \end{pmatrix},$$

$$B_{\bar{x}z} = \begin{pmatrix} B_{11} & 0 & -B_{13} \\ 0 & B_{22} & 0 \\ -B_{31} & 0 & B_{33} \end{pmatrix},$$

$$B_{\bar{y}z} = \begin{pmatrix} B_{22} & 0 & 0 \\ 0 & B_{11} & -B_{13} \\ 0 & -B_{31} & B_{33} \end{pmatrix},$$

$$B_{\bar{y}z} = \begin{pmatrix} B_{22} & 0 & 0 \\ 0 & B_{11} & B_{13} \\ 0 & B_{31} & B_{33} \end{pmatrix}.$$

(3)

In the case of the type-I AFM structure shown in Fig. 4(a), the magnetic moments are

$$m_{xz} = m_{\bar{x}z} = -m_{\bar{y}z} = -m_{\bar{y}z} \equiv m_{ab}.$$

(4)
To include the possibility that the Eu moments are not parallel to the \( a \)-axis, we set \( m_{ab} = (m_a, m_b, 0) \). Therefore,
\[
H_{\text{int}}^{\text{Ga1}} = (B_{x}x + B_{y}y - B_{z}z) \cdot m_{ab}
\]
\[
= \begin{pmatrix}
2(B_{11} - B_{22}) & 0 & 0 \\
0 & 2(B_{22} - B_{11}) & 0 \\
0 & 0 & 0
\end{pmatrix}
\begin{pmatrix}
m_a \\
m_b \\
0
\end{pmatrix}
\]
\[
= 2(B_{11} - B_{22}) \begin{pmatrix}
m_a \\
-m_b \\
0
\end{pmatrix}.
\]  
(5)

Thus, the internal field at the Ga1 site appears in the \( ab \)-plane.

Next, we focus on the internal field at the Ga2 site. The magnetic moment configuration is shown in Fig. 4(b). The internal field at the Ga2 site is also written as eq. (2) with a different hyperfine coupling tensor denoted \( C_i \) (\( i = xy, \bar{xy}, xy, \bar{x}y \), and \( xy \)). From the local symmetry of the Ga2 site, the components of \( C_i \) can be written as
\[
C_{xy} = \begin{pmatrix}
C_{11} & C_{12} & C_{13} \\
C_{12} & C_{11} & C_{13} \\
C_{31} & C_{31} & C_{33}
\end{pmatrix},
\]
\[
C_{\bar{xy}} = \begin{pmatrix}
-C_{11} & -C_{12} & -C_{13} \\
-C_{12} & C_{11} & C_{13} \\
-C_{31} & C_{31} & C_{33}
\end{pmatrix},
\]
\[
C_{xy} = \begin{pmatrix}
C_{11} & -C_{12} & C_{13} \\
-C_{12} & C_{11} & C_{13} \\
C_{31} & -C_{31} & C_{33}
\end{pmatrix},
\]
\[
C_{\bar{x}y} = \begin{pmatrix}
C_{11} & C_{12} & -C_{13} \\
C_{12} & C_{11} & -C_{13} \\
-C_{31} & -C_{31} & C_{33}
\end{pmatrix}.
\]  
(6)

Following a similar procedure with the magnetic moments
\[
m_{xy} = m_{\bar{xy}} = m_{x\bar{y}} = m_{\bar{y}x} \equiv m_{ab},
\]  
(7)

we obtain
\[
H_{\text{int}}^{\text{Ga2}} = (C_{xy} + C_{\bar{xy}} + C_{xy} + C_{\bar{x}y}) \cdot m_{ab}
\]
\[
= \begin{pmatrix}
4C_{11} & 0 & 0 \\
0 & 4C_{11} & 0 \\
0 & 0 & 4C_{33}
\end{pmatrix}
\begin{pmatrix}
m_a \\
m_b \\
0
\end{pmatrix}
\]
\[
= 4C_{11} \begin{pmatrix}
m_a \\
m_b \\
0
\end{pmatrix}.
\]  
(8)

Thus, the internal field at the Ga2 site also appears in the \( ab \)-plane. This result does not change even if we consider the Eu magnetic moments located above and below the Ga2 site. The directions of the internal field at both Ga sites are in good agreement with the experimental result \( H_{\text{int}}^{\text{Ga1}} \perp c \)-axis. However, we could not clarify which site contributes to the Ga-ZFNMR signals.

For further investigation, we have calculated the nuclear quadrupole frequency based on the band calculation by a full potential linear augmented plane wave (FLAPW) method on the basis of a local density approximation (LDA) assuming paramagnetic and ferromagnetic states of EuGa4 without spin-orbit interaction. Here, we used the lattice parameter reported by Nakamura et al.\(^9\) The estimated values are listed in Table II. These values are not in good agreement with the experimental values, especially in the case of the \( ^{153}\text{Eu} \) nucleus. This disagreement probably comes from the lack of a spin-orbit interaction and/or a large Coulomb interaction in 4\( f \) electrons beyond the LDA in the present calculation. As for the Ga nucleus, \( ^{69}\text{LDA}(\text{Ga2}) \) for the ferromagnetic state is comparatively close to the experimentally obtained \( ^{69}\nu_Q \). Therefore, we speculate that the observed Ga-ZFNMR spectra are derived from the Ga2 site, and the internal field at the Ga1 site is close to zero, which may be caused by almost the same value of the hyperfine coupling tensor components \( B_{11} \) and \( B_{22} \). If this speculation is true, the \( ^{69}\text{Ga} \) nuclear quadrupole resonance signal at the Ga1 site should be observed at approximately 10 ~ 20 MHz considering the theoretical estimation of \( ^{69}\nu_Q^{\text{LDA}}(\text{Ga1}) \). However, we have not observed the signal until now, the reason for which is not clear.

Figures 5(a) and 5(b) show the temperature dependences of the internal fields at the Eu and Ga sites, respectively. In AFM materials, the internal field is proportional to the sublattice magnetization. The Eu\(^{2+}\) state is stable and the magnetic state is well explained by the local moment picture in EuGa4; thus, the temperature dependence of \( H_{\text{int}} \) is well explained by the Brillouin function with \( J = S = 7/2 \) and \( T_N = 16 \text{ K} \), as shown by the solid lines in Fig. 5.

In summary, we have carried out zero-field NMR measurement in an antiferromagnetically ordered state of EuGa4. The analysis of the \( ^{153}\text{Eu} \) and \( ^{69,71}\text{Ga} \) ZFNMR spectra with

| \( ^{153}\nu_Q^{(\text{Eu})} \) | \( ^{69}\nu_Q^{(\text{Ga1})} \) | \( ^{69}\nu_Q^{(\text{Ga2})} \) |
|-----------------|-----------------|-----------------|
| Calc. (PM) | 9.28 | 10.29 | 0.833 |
| Calc. (FM) | 10.9 | 11.5 | 2.8 |

| \( ^{153}\text{Eu} \) | \( ^{69}\text{Ga} \) | \( ^{71}\text{Ga} \) |
|-----------------|-----------------|-----------------|
| \( H_{\text{int}} \) (T) | \( H_{\text{int}}^{\text{Ga}} \) (T) | \( H_{\text{int}}^{\text{Ga}} \) (T) |
|-----------------|-----------------|-----------------|
| 25 | 5.0 | 5.0 |
| 20 | 10.0 | 10.0 |
| 15 | 15.0 | 15.0 |
| 10 | 20.0 | 20.0 |
| 5 | 30.0 | 30.0 |

Fig. 5. (Color online) Temperature dependence of the internal fields at the (a) Eu site and (b) Ga site. Solid lines indicate a calculation of the Brillouin function with \( J = S = 7/2 \).
well-resolved nuclear quadrupole splitting tells us that the magnetic moments lie in the $ab$-plane. The huge internal field at the Eu nucleus site and the temperature dependence of the internal fields at both Eu and Ga sites reveal that a well-localized $f$-electron-derived magnetic state is realized in EuGa$_4$.

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