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Quadrupole and lattice effects of orbitally degenerate 4f-electron systems

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Abstract

The quadrupole orderings and lattice effects in 4f-electron systems with orbital degeneracy in rare earth compounds have been investigated by ultrasonic and thermal expansion measurements. A ferroquadrupole (FQ) O_{xy} -type ordering associated with a trigonal distortion in HoB₆ with a Γ_5 ground state is presented. The trigonal distortion in ordered phase IV Ce_xLa_{1-x}B₆ ($x = 0.75$ and 0.70) with a Γ_8 ground state indicates O_{xy} -type FQ ordering in phase IV. In both systems, HoB₆ and Ce_xLa_{1-x}B₆, C_{44} anomalies are observed. In tetragonal HoB₂C₂, an ordered phase IV appears near a tetra-critical point, where the antiferroquadrupole phase II and antiferromagnetic phase III compete with each other. Noticeable ultrasonic attenuation and softening of elastic constants in phase IV of HoB₂C₂ due to quadrupole fluctuation with a slow relaxation time $\tau \sim 7 \times 10^{-9}$ s are found.

1. Introduction

The localized 4f-electron system of rare earth compounds has orbital as well as spin degrees of freedom. Therefore intersite interaction of the electric quadrupole gives rise to ferroquadrupole (FQ) or antiferroquadrupole (AFQ) ordering in addition to the magnetic orderings due to the magnetic exchange interaction or RKKY interaction. The 4f electronic ground state with orbital degeneracy in particular favours quadrupole orderings or multipole orderings at low temperatures [1, 2]. Because the direct product of Γ_8 is reduced as $\Gamma_8 \otimes \Gamma_8 = \Gamma_1 \oplus \Gamma_2 \oplus \Gamma_3 \oplus 2\Gamma_4 \oplus 2\Gamma_5$, three dipoles J_x, J_y, J_z , five quadrupoles $O_2^0, O_2^2, O_{yz}, O_{zx}, O_{xy}$

and seven octupoles $T_{xyz}, T_x^\alpha, T_y^\alpha, T_z^\alpha, T_x^\beta, T_y^\beta, T_z^\beta$ are expected for the order parameter of the Γ_8 quartet system [3]. The intermetallic compounds CeAg [4] and Ce₃Pd₂₀Ge₆ [5] with the Γ_8 quartet are known as the FQ ordering associated with O_2^0 accompanied by a structural change from the cubic class to the tetragonal class. A rare earth hexaboride DyB₆ with a Γ_8 ground state also shows FQ ordering of O_{yz}, O_{zx}, O_{xy} with a trigonal distortion [6]. CeB₆ and the substituted system Ce_xLa_{1-x}B₆ with a Γ_8 ground state show AFQ ordering of O_{yz}, O_{zx}, O_{xy} in addition to antiferromagnetic ordering [7]. Competitive phenomena associated with the Kondo effect and the intersite quadrupole and magnetic interaction in Ce_{1-x}La_xB₆ have been proposed [8]. The direct product $\Gamma_5 \otimes \Gamma_5 = \Gamma_1 \oplus \Gamma_3 \oplus \Gamma_4 \oplus \Gamma_5$ means that the Γ_5 triplet has three dipoles and five quadrupoles. HoB₆ with a Γ_5 ground state is known as a typical example of FQ ordering of O_{yz}, O_{zx}, O_{xy} with trigonal distortion [9]. The Γ_3 doublet possesses the two quadrupoles O_2^0, O_2^2 and the octupole T_{xyz} . PrPb₃ with a Γ_3 ground state is known as the AFQ ordering of O_2^0 at $T_Q = 0.4$ K [10]. The screening effect of the non-magnetic Γ_3 state by conduction electrons in Pr- or U-based compounds is a current topic for the non-Fermi liquid state [11].

2. Experimental details

Because the quadrupole of the 4f electron couples to the elastic strain, the ultrasound and thermal expansion are important probes to examine quadrupole effects in electronic systems with orbitally degenerate ground states [1, 2]. In the present paper we show our recent experimental results for HoB₆, Ce_xLa_{1-x}B₆, HoB₂C₂ obtained with ultrasonic and dilatometric measurements at Niigata University. The sound velocity v was measured by a phase comparator using double balanced mixers. The capacitance dilatometer, based on a three-terminal method, was made of oxygen-free copper. A sample was mounted by phosphorus bronze bellows with a spring constant $k = 3.6$ kgf mm⁻¹. The recovery force of the bellows expanded by 1 mm leads to an external pressure $P = 3.5 \times 10^{-3}$ GPa for a cross-sectional area of 10 mm², typical values in the present experiment. A ³He-evaporation refrigerator cooling down to 600 mK for dilatometric measurements was inserted into a superconducting magnet.

3. HoB₆

Heavy rare earth hexaboride HoB₆ with a cubic CaB₆-type structure is a typical material showing FQ ordering at $T_Q = 6.1$ K. The specific heat [12] and ultrasonic measurements [13] show that the 4f electronic state of HoB₆ has a Γ_5 ground state, which possesses three dipoles and five quadrupoles. Because of the quadrupole–strain interaction in HoB₆, the FQ ordering at $T_Q = 6.1$ K is accompanied by a lattice distortion from a cubic class to a trigonal one. AFM ordering occurs successively at $T_N = 5.6$ K [6, 12, 13]. Figure 1(a) shows the temperature dependence of the transverse elastic constant C_{44} of HoB₆ in magnetic fields for a magnetic easy axis of $H \parallel [111]$. C_{44} shows a huge softening of about 70% from 200 K down to 6.4 K in zero field [13]. The coupling of the quadrupoles O_{yz}, O_{zx}, O_{xy} to the elastic strains $\varepsilon_{yz}, \varepsilon_{zx}, \varepsilon_{xy}$ is described as $H_{QS} = -g_{\Gamma_5}\{O_{yz}\varepsilon_{yz} + O_{zx}\varepsilon_{zx} + O_{xy}\varepsilon_{xy}\}$ [1, 2]. This coupling gives rise to a temperature dependence of $C_{44} = C_{44}^0(T - T_C^0)/(T - \Theta)$, which explains well the elastic softening of C_{44} in zero field of figure 1(a) with $T_C^0 = 5.45$ K and $\Theta = 2.10$ K. The applied magnetic field suppresses the softening of C_{44} and shifts the minimum point to higher temperatures. A broadening of the minimum point in a higher field of 8 T indicates smearing out of the FQ transition. We show the magnetic phase diagram of HoB₆ in the inset of figure 1(a). In increasing magnetic field, the AFM transition temperature T_N shifts to

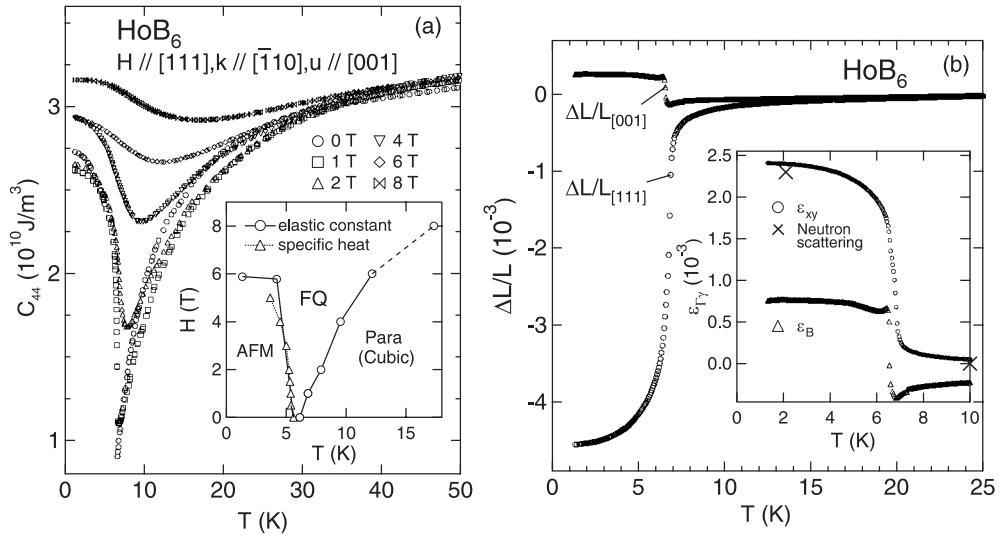


Figure 1. (a) Temperature dependence of the elastic constant C_{44} in HoB_6 under magnetic fields along the [111] direction. The inset shows the magnetic phase diagram of HoB_6 . Circles are obtained from elastic anomalies of C_{44} under magnetic fields along the [111] direction. Triangles are obtained from specific heat under magnetic fields along the [110] direction. (b) Thermal expansions $\Delta L/L_{[001]}$ and $\Delta L/L_{[111]}$ of HoB_6 . The inset shows the temperature dependence of the volume strain ε_B and the trigonal strain ε_{xy} of HoB_6 .

lower temperatures, while the FQ transition temperature T_Q shifts to higher temperatures. The increase of T_Q in magnetic fields is a common feature in materials showing FQ ordering [5].

Figure 1(b) shows the thermal expansion $\Delta L/L_{[001]}$ of HoB_6 along the [001] direction and $\Delta L/L_{[111]}$ along the [111] direction in zero magnetic field. The thermal expansion along the [001] direction increases to $\Delta L/L_{[001]} = 2.5 \times 10^{-4}$ at 1.5 K far below T_Q , while the thermal expansion along [111] shrinks appreciably: $\Delta L/L_{[111]} = -4.5 \times 10^{-3}$. The thermal expansions along the [001] and [111] directions are written as $\Delta L/L_{[001]} = \frac{1}{3}\varepsilon_B + \frac{1}{\sqrt{3}}\varepsilon_u$ and $\Delta L/L_{[111]} = \frac{1}{3}\varepsilon_B + \frac{2}{3}(\varepsilon_{yz} + \varepsilon_{zx} + \varepsilon_{xy})$, respectively. In the case of the cubic-trigonal transition in HoB_6 , it is expected that the tetragonal distortion is irrelevant ($\varepsilon_u = 0$) and the trigonal distortion is characterized by $\varepsilon_{yz} = \varepsilon_{zx} = \varepsilon_{xy} \neq 0$. The inset of figure 1(b) shows the temperature dependence of the spontaneous strains ε_{xy} corresponding to a tiny distortion from $\alpha = 90^\circ$ of a cubic class to $\alpha = 90^\circ + 2\theta$ of a trigonal class and volume expansion ε_B below T_Q . The spontaneous strain $\varepsilon_{xy} = 2.4 \times 10^{-3}$ is well consistent with $\varepsilon_{xy} = \sin \theta = 2.3 \times 10^{-3}$ obtained by $\theta = 0.132^\circ$ at 2.1 K in the neutron scattering experiments [14]. Considering that the trigonal distortion is proportional to the quadrupole moment as $\langle \varepsilon_{xy} \rangle = Ng_{\Gamma_5} \langle O_{xy} \rangle / C_{44}^0$, we conclude that the order parameter of the FQ ordering in HoB_6 below $T_Q = 6.1$ K is $O_{yz} = O_{zx} = O_{xy} \neq 0$.

4. $\text{Ce}_x\text{La}_{1-x}\text{B}_6$

The Kondo compound CeB_6 and dilute compound $\text{Ce}_x\text{La}_{1-x}\text{B}_6$ with a cubic CaB_6 -type structure have a Γ_8 quartet ground state, which possesses three dipoles, five quadrupoles and seven octupoles. Therefore these compounds exhibit interesting low-temperature properties due to competition among the magnetic and quadrupole intersite interactions. Substitution

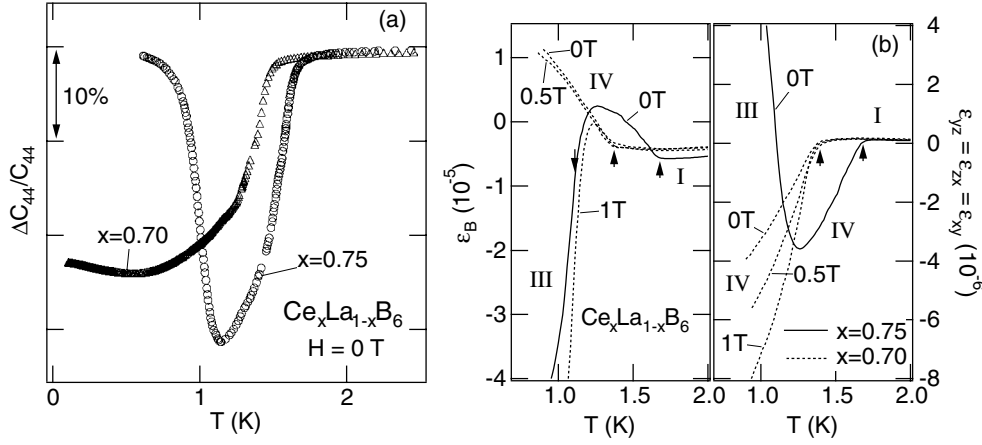


Figure 2. (a) Temperature dependence of elastic constant C_{44} in $Ce_xLa_{1-x}B_6$ ($x = 0.75$ and 0.70). (b) Temperature dependence of the volume strain ε_B and the trigonal strain ε_{xy} of $Ce_xLa_{1-x}B_6$ ($x = 0.75$ and 0.70).

of a Ce atom by an La atom in $Ce_xLa_{1-x}B_6$ makes an appreciable modification to the magnetic phase diagram. The intersite quadrupole interaction favourable for the AFQ phase II is reduced much faster than the intersite magnetic interaction for the AFM phase III with decreasing Ce concentration x in $Ce_xLa_{1-x}B_6$ from the end material $x = 1.0$ (CeB_6). In the case of $x = 0.75$, a new phase IV has been found from ultrasonic, magnetization and resistivity measurements [15–17]. Magnetization measurement indicates an isotropic nature for phase IV [16]. The muon spin rotation (μ SR) experiment for $x = 0.70$ and neutron scattering for $x = 0.75$ have detected no sign of magnetic ordering in phase IV [18, 19]. In the case of $x = 0.70$, phase IV below $T_C = 1.4$ K is stable down to $T \sim 20$ mK in zero field [20]. As shown in figure 2(a), the elastic constant C_{44} exhibits a huge softening. For $x = 0.75$, C_{44} reduces considerably to 31% in phase IV between $T_C = 1.6$ K and $T_N = 1.1$ K. A sharp increase has been found at $T_N = 1.1$ K. For $x = 0.70$, C_{44} also shows a huge softening below around $T_C = 1.4$ K down to 20 mK, indicating a stable phase IV down to 0 K. It is noticeable that in both compounds the transverse C_{44} mode commonly shows huge softening in phase IV, while $(C_{11}-C_{12})/2$ shows an upturn only [15].

In order to determine the order parameter of phase IV, we have measured the thermal expansion along the [001] and [111] directions on $Ce_xLa_{1-x}B_6$ ($x = 0.75$ and 0.70) at low temperatures. The poor anomaly in $(C_{11}-C_{12})/2$ means that the Γ_3 -type quadrupoles O_2^0 and O_2^2 are free from long-range ordering in $Ce_xLa_{1-x}B_6$. We expect, therefore, that the tetragonal strain is zero ($\varepsilon_u = 0$) in phase IV. By using the results of the thermal expansion along the [001] and [111] directions in $Ce_xLa_{1-x}B_6$ ($x = 0.75$ and 0.70), we obtain the temperature dependence of the volume strain ε_B and the shear strain ε_{xy} in figure 2(b). Here we assume that the shear strains are equal, $\varepsilon_{yz} = \varepsilon_{zx} = \varepsilon_{xy}$, in phase IV. The volume strain shows an increase to $\varepsilon_B = 1.5 \times 10^{-5}$ at $T = 0.8$ K far below T_C in phase IV under zero field and under a field of 0.5 T. Appreciable deviation of ε_{xy} from zero in phase I to $\varepsilon_{xy} = 4 \times 10^{-6}$ in phase IV under zero field at $T = 0.8$ K far below T_C of $x = 0.70$ means a lattice distortion from a cubic class to a trigonal one. The spontaneous strain ε_{xy} increases with increasing magnetic field up to 1.0 T. The trigonal distortion associated with the spontaneous strain $\langle \varepsilon_{yz} \rangle = \langle \varepsilon_{zx} \rangle = \langle \varepsilon_{xy} \rangle \neq 0$ in phase IV is described in terms of the FQ ordering $\langle O_{yz} \rangle = \langle O_{zx} \rangle = \langle O_{xy} \rangle \neq 0$ as $\langle \varepsilon_{xy} \rangle = \{Ng_{\Gamma 5}/C_{44}^0\} \langle O_{xy} \rangle$. The present scenario of

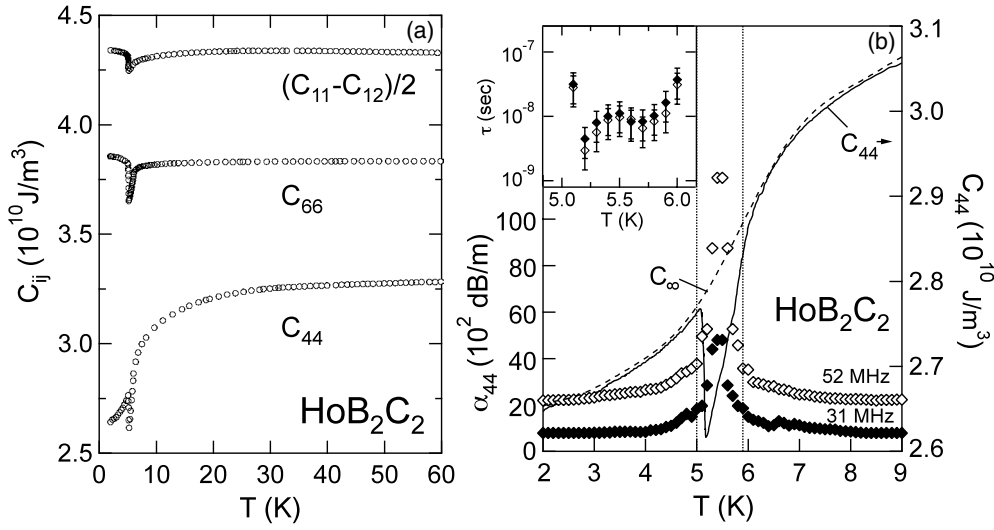


Figure 3. (a) Temperature dependence of the elastic constant $(C_{11}-C_{12})/2$, C_{44} , C_{66} in tetragonal HoB_2C_2 . (b) Ultrasonic attenuation coefficient α_{44} and elastic constant C_{44} across phase IV in HoB_2C_2 . Inset shows the quadrupole relaxation time τ in phase IV.

the FQ ordering is consistent with the non-magnetic properties in phase IV indicated by the neutron scattering and μSR measurements [18, 19].

It is remarkable that phase IV is located near the tetra-critical point, where the AFQ phase II and AFM phase III coexist. This means that the mutual competition between the AFQ interaction favouring phase II and the AFM interaction for phase III brings about phase IV with FQ ordering. It is also remarkable that the Kondo temperature $T_K \sim 1$ K is comparable to the transition temperatures due to both AFM and AFQ interactions [8].

5. HoB_2C_2

It has recently been found that the ternary compounds HoB_2C_2 [21] with a tetragonal structure of space group $P4/mbm$ (D_{4h}^5) [22] exhibit AFQ ordering at $T_{C2} = 5.0$ K in addition to AFM ordering at $T_{C1} = 5.9$ K. The resonant x-ray measurements confirmed the AFQ ordering in phase II of HoB_2C_2 and the isomorphous compound DyB_2C_2 [23–25]. The magnetic phase diagram of HoB_2C_2 under fields $H \parallel [110]$ shows that the phase boundary from the paramagnetic phase I to the AFQ phase II crosses the boundary to the AFM phase III at a tetra-critical point of field about 1.0 T. Such a reversal of the AFQ and AFM transition sequence is curious. It is of some interest that the magnetic intermediate state in HoB_2C_2 , named phase IV between $T_{C2} = 5.0$ K and $T_{C1} = 5.9$ K, disappears in a small magnetic field of 1.0 T. Detailed neutron diffraction experiments [26, 27] have indicated that phase IV is a short-range magnetically ordered state with an incommensurate AFM structure which is described by a propagation vector $\mathbf{k} = [1 + \delta, \delta, \delta']$ (with $\delta = 0.112$ and $\delta' = 0.04$). It should also be pointed out that the broad diffuse magnetic components around $\mathbf{k} = [1, 0, 0]$ are growing from above $T_{C1} = 5.9$ K and remain in phase IV. The order parameter of phase IV of HoB_2C_2 is not settled yet.

In the present paper, we have investigated the elastic properties of HoB_2C_2 by means of ultrasonic measurements. Figure 3(a) represents the temperature dependence of the elastic

constants C_{44} , C_{66} and $(C_{11}-C_{12})/2$ of the transverse ultrasonic modes in HoB_2C_2 with a tetragonal lattice. C_{44} shows the largest softening of 22%, compared with 5.5% for C_{66} and 2.4% for $(C_{11}-C_{12})/2$. The multiplet $J = 8$ of Ho^{3+} splits into 5A (singlet), 4B (singlet) and 4E (doublet) at C_{4h} point group symmetry in HoB_2C_2 . We have proposed a pseudo triplet model of 4f electronic ground states [28] consisting of a ground E doublet and an excited A singlet (or B singlet) at excited energy Δ to explain the softening of transverse modes in figure 3(a). Here we assume that Δ is equal to $T_{C2} = 5.0$ K. The released entropy $R \ln 3$ in specific heat measurements [29] up to $T_{C1} = 5.9$ K in HoB_2C_2 conforms to the present pseudo triplet model. The direct product of the E doublet in the point group C_{4h} is reduced as $E \otimes E = 2A \oplus 2B$. This means that the E doublet has the quadrupole moments of O_2^2 and O_{xy} in the diagonal part in matrices which are responsible for the $(C_{11}-C_{12})/2$ and C_{66} modes with B symmetry, respectively. On the other hand, the quadrupole moments O_{yz} , O_{zx} with E symmetry are responsible for the off-diagonal transition from the ground E doublet to the low-lying excited A singlet (or B singlet). A small excited energy $\Delta \sim 5$ K, however, gives rise to the softening of C_{44} proportional to reciprocal temperature. In the case, the strain susceptibility successfully describes the softening of all transverse modes of HoB_2C_2 .

An abrupt increase in the softening of all elastic modes and shrinkage of the ultrasonic echo in phase IV between $T_{C1} = 5.9$ K and $T_{C2} = 5.0$ K has also been found. Figure 3(b) represents the ultrasonic attenuation of the C_{44} mode as a function of temperature with constant frequencies of 31 and 52 MHz (left axis). Vertical lines indicate the phase boundaries of phase IV. The elastic constant C_{44} with a frequency of 8 MHz and assumed high-frequency limit C_∞ are also shown (right axis). The peak values of the attenuation coefficient α_{44} around 5.5 K in phase IV increase with increasing frequency of the ultrasonic wave as $\alpha_{44} = 50 \times 10^2 \text{ dB m}^{-1}$ at 31 MHz and $\alpha_{44} = 120 \times 10^2 \text{ dB m}^{-1}$ at 52 MHz. This is due to the slowing down of relaxation time of the quadrupole fluctuation in phase IV. The inset of figure 3(b) shows a temperature dependence of relaxation time τ which was calculated from the experimental result by employing a Debye-type dispersion formula as $\alpha(\omega) = \{(C_\infty - C_0)/2\rho v^3\}\omega^2\tau/(1 + \omega^2\tau^2)$. We obtain a very slow relaxation time $\tau = 7 \times 10^{-9}$ s in phase IV, which is much slower than the relaxation time of phase III and I, being a small attenuation. This result means that the slow fluctuation of the quadrupole moments O_{yz} , O_{zx} is very much enhanced in phase IV of HoB_2C_2 .

It is noted that the diffuse neutron scattering intensity in phase IV is related to the dipole fluctuation in phase IV. The remarkable enhancement of the quadrupole as well as the dipole fluctuation is the most important characteristic of phase IV. It is also noticeable that phase IV appears in the region where the AFQ phase II and the AFM phase II compete with each other. The origin of the fluctuation of both quadrupole and dipole in phase IV of HoB_2C_2 remains to be solved.

6. Conclusion

We have measured the elastic constants and thermal expansion in HoB_6 and $\text{Ce}_x\text{La}_{1-x}\text{B}_6$ with orbitally degenerate ground states. The elastic softening of the C_{44} mode around T_Q and trigonal distortion below T_Q in HoB_6 indicate FQ ordering of $\langle O_{yz} \rangle = \langle O_{zx} \rangle = \langle O_{xy} \rangle \neq 0$. In phase IV of $\text{Ce}_x\text{La}_{1-x}\text{B}_6$ ($x = 0.75$ and 0.70), the FQ ordering of $\langle O_{yz} \rangle = \langle O_{zx} \rangle = \langle O_{xy} \rangle \neq 0$ is deduced by the lattice distortion from a cubic class to a trigonal one. The mutual balance between AFQ interaction for phase II and AFM interaction for phase III in $\text{Ce}_x\text{La}_{1-x}\text{B}_6$ gives rise to phase IV with FQ ordering closing to the tetra-critical point. The elastic softening in the transverse mode of the tetragonal compound HoB_2C_2 suggests the pseudo triplet ground

state of the system. The quadrupole fluctuation with slow relaxation time $\tau = 7 \times 10^{-9}$ s deduced from the present ultrasonic attenuation measurement and the magnetic fluctuation by the neutron scattering are the most characteristic properties of phase IV in HoB₂C₂. The characterization of the order parameter of phase IV in HoB₂C₂ is still an open problem.

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