Magnetic properties of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$

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Abstract. The magnetic properties of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ ferroborate with the competing Ho–Fe and Er–Fe exchange couplings have been experimentally and theoretically investigated. The measured magnetic characteristics ($M_\parallel$, $M_\perp$, $\chi_\parallel$, $\chi_\perp$) and observed features are interpreted using a single theoretical approach based on the molecular-field approximation and calculations within the crystal-field model of the rare-earth ion. Interpretation of the experimental data includes determination of the crystal-field parameters for Ho$^{3+}$ and Er$^{3+}$ ions in Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ and parameters of the Ho–Fe and Er–Fe exchange couplings.

1. Introduction

The rare-earth borates RM$_3$(BO$_3$)$_4$ (R = Y or La–Lu and M = Al, Sc, Cr, Fe and Ga) exhibit a vast diversity of magnetic, magnetoelectric, magnetoelastic and other physical properties [1, 2]. An increased interest in the borates RM$_3$(BO$_3$)$_4$ is due to the possibility of studying the substituted R$_{1-x}$R'$^x$Fe$_3$(BO$_3$)$_4$ compositions, where the presence of competing R–Fe and R'$^-$Fe exchange couplings can ensure the occurrence of spontaneous reorientation transitions [3-5]. The iron magnetic moments in HoFe$_3$(BO$_3$)$_4$ are antiferromagnetically ordered at $T_N \approx 38–39$ K and, with a decrease in temperature to $T_{SR} \approx 4.7–5$ K, lie in the $ab$ basal plane, similar to the magnetic moments of Ho$^{3+}$ ions [6]. At $T_{SR} \approx 4.7–5$ K, the spontaneous spin-reorientation transition occurs; as a result, the magnetic moments of the Ho- and Fe-subsystems align parallel to the $c$-axis. In ErFe$_3$(BO$_3$)$_4$ at $T < T_N \approx 38$ K, all magnetic moments lie in the $ab$ basal plane [1, 7]. In YFe$_3$(BO$_3$)$_4$ at $T < T_N \approx 37$ K, the magnetic moments of Fe lie in the basal plane $ab$ [1, 7]. Thus, the competition of contributions of the Ho-, Er- and Fe-subsystems to the magnetic anisotropy of Ho$_{1-x}$Er$_x$Fe$_3$(BO$_3$)$_4$ can result in the occurrence of the spontaneous and magnetic field-induced spin-reorientation transitions. These transitions were observed for Ho$_{1-x}$Nd$_x$Fe$_3$(BO$_3$)$_4$ [4, 8] and Nd$_{1-x}$Dy$_x$Fe$_3$(BO$_3$)$_4$ [3].

In this work the experimental and theoretical investigations of the field and temperature dependences of magnetization $M_\parallel(B)$ and the temperature dependences of the magnetic susceptibility $\chi_\parallel(T)$ of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ have been performed.
2. Experiment
The Ho_{0.9}Er_{0.1}Fe_{3}(BO_{3})_{4} single crystals were grown from fluxes based on bismuth trimolibdate 78 wt. 
% [Bi_{2}Mo_{3}O_{12} + 3B_{2}O_{3} + 0.45Ho_{2}O_{3} + 0.05Er_{2}O_{3}] + 22 wt. % Ho_{0.9}Er_{0.1}Fe_{3}(BO_{3})_{4} by the technique 
described in detail in [9, 10].

The magnetic measurements were performed on a Quantum Design Physical Property Measurement 
System in the temperature range of 3–300 K and magnetic fields of up to 9 T.

3. Theory
The magnetic properties of Ho_{0.9}Er_{0.1}Fe_{3}(BO_{3})_{4} crystals are determined both by the magnetic subsystems 
and by the interaction between them. The Fe-subsystem in this compound can be considered as 
consisting of two antiferromagnetic sublattices. The rare-earth subsystem (magnetized due to the f–d 
interaction) can also be represented as a superposition of two sublattices. In the calculations, we used 
a theoretical approach which has been applied for description of the magnetic properties of the RFe_{3}(BO_{3})_{4} 
(see e.g. [2, 5, 8]). This approach is based on a crystal-field model for the R-ion and on the molecular-
field approximation. Effective Hamiltonians describing the interaction of each Fe/Fe ion in the ith (i = 1, 
2) sublattice of the corresponding subsystem in the applied magnetic field B can be written as

\[ \mathcal{H}_i(R) = \mathcal{H}^R_{i} - g_s \mu_B J^R_i \left[ B + \lambda \mathbf{M}^R_i \right], \]  

\[ \mathcal{H}_i(Fe) = -g_s \mu_B J^R_i \left[ B + \lambda \mathbf{M}^{Fe}_i + (1-x) \lambda^{Ho}_i \mathbf{m}^{Ho}_i + x \lambda^{Er}_i \mathbf{m}^{Er}_i \right], \]

where \( \mathcal{H}^R_{i} \) is the crystal-field Hamiltonian (see e.g. [8]), \( g_s \) is the Lande factor, \( J^R_i \) is the operator of the 
angular moment of the R-ion, \( g_s = 2 \) is the g-value, \( S_i \) is the operator of the spin moment of an iron ion, \( \lambda^R_i < 0 \) and \( \lambda = 0 \) (including intrachain \( \lambda^R_1 < 0 \) and interchain \( \lambda^R_2 < 0 \)) are the molecular constants of the 
antiferromagnetic interactions R–Fe and Fe–Fe, respectively. The magnetic moments of the ith iron 
\( \mathbf{M}^{Fe}_i \) and rare-earth \( \mathbf{m}^R_i \) sublattices per formula unit are defined as

\[ \mathbf{M}^{Fe}_i = 3g_s \mu_B \langle S^R_i \rangle, \quad \mathbf{m}^R_i = g_s \mu_B \langle J^R_i \rangle. \]  

In order to calculate the magnitudes and orientations of magnetic moments in the Fe- and R-
subsystems, it is necessary to solve a self-consistent problem based on Hamiltonians (1, 2) under the condition of minimum for the corresponding thermodynamic potential (see e.g. [5, 8]). Then, it is 
possible to determine the regions of stability of various magnetic phases, the critical fields for the phase 
transitions, the magnetization curves, magnetic susceptibilities, etc.

The magnetization and magnetic susceptibility of the compound (per f.u.) are defined as

\[ \mathbf{M} = \frac{1}{2} \sum_{i=1}^{2} \left( \mathbf{M}^{Fe}_i + (1-x) \mathbf{m}^{Ho}_i + x \mathbf{m}^{Er}_i \right), \]  

\[ \chi_k = \chi^{Fe}_k + (1-x) \chi^{Ho}_k + x \chi^{Er}_k, \quad k = a, b, c. \]

4. Results and discussion
In order to determine the parameters of the crystal-field we have used the experimental data on the 
temperature dependence of the magnetic susceptibility \( \chi_{Ho}(T) \), the field and temperature dependences of 
the magnetization \( M_{Ho}(B, T) \) and existing information on the structure of the ground multiplet of Ho\(^{3+}\) 
ion in HoFe_{3}(BO_{3})_{4} and Er\(^{3+}\) ion in ErFe_{3}(BO_{3})_{4}. The initial values of the crystal-field parameters, from 
which the procedure of minimization of the corresponding target function was started, were selected 
from the values available for ferroborates: HoFe_{3}(BO_{3})_{4} [11], ErFe_{3}(BO_{3})_{4} [11] and Ho_{0.5}Nd_{0.5}Fe_{3}(BO_{3})_{4} 
[8]. The best agreement is achieved for the following set of the crystal-field parameters \( (B^k_y, \text{ in cm}^{-1}) \):
\begin{align*}
B_6^5 &= 450, \quad B_0^4 = -1550, \quad B_3^3 = 860, \\
B_6^6 &= 340, \quad B_0^5 = 150, \quad B_6^6 = 425.
\end{align*}

The set of parameters (6) corresponds to the energies of the lower levels of the ground multiplet of the Ho\(^{3+}\) and Er\(^{3+}\) ions in Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) that are given in table 1 for \(B = 0\). These energies are given for \(T > T_N\) and with allowance for the \(f-d\) interaction at \(T = 2\) K < \(T_N\) (the inclined (INC) state, see below). It can be seen that the inclusion of the \(f-d\) interaction at \(T < T_N\) results in the removal of the degeneracy of the lower levels.

**Table 1.** Energies of the eight lower levels of the ground multiplets of the Ho\(^{3+}\) and Er\(^{3+}\) ions in Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) at \(B = 0\) that are split by the crystal field (parameters (6)) in the paramagnetic and ordered (with allowance for the \(f-d\) interaction) temperature ranges.

| \(R\) | \(T\) | \(\Delta = E_i - E_1\), cm\(^{-1}\) (\(i = 1\)-8) |
|---|---|---|
| Ho | \(T > T_N\) | 0, 0, 7.1, 20.3, 20.3, 62.7, 135, 192 |
| | 2 K < \(T_N\) | 0, 5.0, 16.8, 26.6, 30.1, 70, 141, 194 |
| Er | \(T > T_N\) | 0, 0, 28.3, 28.3, 77.3, 77.3, 129, 129 |
| | 2 K < \(T_N\) | 0, 5.1, 30.4, 31.8 77.9, 82.3, 130, 133.5 |

The calculated magnetic characteristics presented below in the figures were calculated for the parameters given in table 2, which also gives the parameters of HoFe\(_3\)(BO\(_3\))\(_4\) [11] and ErFe\(_3\)(BO\(_3\))\(_4\) [11] for comparison.

In the calculations, we also use the uniaxial anisotropy constants of iron (\(K_2^Fe = -2.7\) T·\(\mu_B\) and \(K_4^Fe = 0.83\) T·\(\mu_B\) at \(T = 4.2\) K) and the anisotropy constant of iron in the basal plane (\(K_{66}^Fe = -1.35 \cdot 10^{-2}\) T·\(\mu_B\) [12]). The \(K_2^Fe\) and \(K_4^Fe\) values are consistent with the corresponding uniaxial constants determined when describing the INC state in Pr\(_x\)Y\(_{1-x}\)Fe\(_3\)(BO\(_3\))\(_4\) [13] and Ho\(_{0.5}\)Nd\(_{0.5}\)Fe\(_3\)(BO\(_3\))\(_4\) [8].

To calculate the magnetic characteristics of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) when an external field is directed along or perpendicular to trigonal axis \(c\), we used the schemes of orientation of the magnetic moments of iron \(M_i^Fe\) and a rare-earth \(m_i^R\) subsystems shown in figure 1.

Figure 2 shows the experimental magnetization curves \(M_m(B)\) of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) obtained at \(T = 3\)–30 K in magnetic fields directed along the trigonal axis (B\(||c\)) (figure 2a) and in the basal plane (B\(||a\)) (figure 2b). The characters of magnetization of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) in the basal plane and along the trigonal axis differ weakly, which indicates a weakly anisotropic contribution of the rare-earth subsystem induced by the crystal-field of the holmium-erbium ferroborate. figure 3 shows the experimental and calculated curves \(M_m(B)\) of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) at \(T = 3\) K and for comparison \(M_m(B)\) curves of HoFe\(_3\)(BO\(_3\))\(_4\) [14] at \(T = 2\) K. As can be seen from figures 2, 3 there are no anomalies on the experimental and calculated magnetization curves \(M_m(B)\) of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) at \(T = 3\)–30 K, in contrast to the magnetization curve \(M_{HoFe}^c\) of HoFe\(_3\)(BO\(_3\))\(_4\) [14] at \(T = 2\) K, on which a jump of magnetization is clearly visible in the field \(B \approx 0.5\) T. The jump in curve \(M_{HoFe}^c\) is caused by spin-flop transition in the Fe-subsystem of HoFe\(_3\)(BO\(_3\))\(_4\) [11].

It can be seen from inset in figure 3 that the experimental curves \(M_{HoFe}^c\) (at \(B \approx 0.95\) T) and \(M_m(B)\) of Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) (at \(B \approx 0.2\) T) are characterized by magnetization jumps.

The low-temperature magnetic state of substituted ferroborate Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) is unknown. Our extensive calculations of the magnetic phases that appear in Ho\(_{0.9}\)Er\(_{0.1}\)Fe\(_3\)(BO\(_3\))\(_4\) at various orientations of the magnetic moments of the Ho-, Er- and Fe-subsystems suggest the presence of a low-temperature
state that differs from both the EP and the EA states. An antiferromagnetic phase with the magnetic moments of iron deviating from axis $c$ through an angle $\theta \approx 65.5^\circ$ (at $T = 2$ K) appears; as a result, a cone of easy magnetization axes (inclined state) forms at $B = 0$ (see figure 1a). The possible magnetic field-induced intermediate states with a noncollinear antiferromagnetic structure result from the competition of the contributions of the Ho-, Er-, and Fe-subsystems to the total magnetic anisotropy of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ and Zeeman energy. The magnetic anisotropy of the Er- and Fe-subsystems stabilizes the EP magnetic structure. The Ho-subsystem stabilizes the EA state. As a result, at certain temperatures and fields, the iron magnetic moments can be oriented at angle $\theta$ to the $c$-axis. The possible implementation of the initial INC state in ferroborates was experimentally confirmed in the Pr$_x$Y$_{1-x}$Fe$_3$(BO$_3$)$_4$ compounds [15, 13].

Table 2. Parameters of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ and, for comparison, parameters of HoFe$_3$(BO$_3$)$_4$ [11] and ErFe$_3$(BO$_3$)$_4$ [11] (the intrachain Fe-Fe exchange field $B_{dd1}$, the interchain Fe-Fe exchange field $B_{dd2}$, and the $f$–$d$ exchange field $B_{fd}$ are the low-temperature exchange fields corresponding to the molecular constants $\lambda_1$, $\lambda_2$, and $\lambda_{fd}$, respectively; $\Delta_{fd}$ is the low-temperature splitting of the ground state of an R-ion due to the $f$–$d$ interaction in the following states: an inclined (INC) magnetic state, easy-axis (EA) state, and easy-plane (EP) state; $T_{SR}$ is the spin-reorientation transition temperature; $T_N$ is the Neel temperature; $\Theta$ is the paramagnetic Neel temperature for the iron subsystem; $M_0 = |M(T = 0, B = 0)| = 15 \mu_B$ is the magnetic moment of iron per formula unit).

| RFe$_3$(BO$_3$)$_4$ | R = Ho | R = Ho$_{0.9}$Er$_{0.1}$ | R = Er |
|------------------|--------|----------------------|-------|
| $B_{dd1} = \lambda_1 M_0$, T | 68 | 68 | 62.5 |
| $\lambda_1$, T/$\mu_B$ | $-4.53$ | $-4.53$ | $-4.16$ |
| $B_{dd2} = \lambda_2 M_0$, T | 26 | 26 | 26 |
| $\lambda_2$, T/$\mu_B$ | $-1.73$ | $-1.73$ | $-1.73$ |
| $B_{fd} = \lambda_{fd} M_0$, T | 3.49 | 3.1 (Ho) | 1.3 |
| $\lambda_{fd}$, T/$\mu_B$ | $-0.23$ | $-0.21$ (Ho) | $-0.09$ |
| $\Delta_{fd} = \mu_B |\lambda_{fd}|/M_0$ | $\sim 10.6$ (EA) | $\sim 9.7$ (EP) | $\sim 9.7$ (EP) |
| $T_{SR}$, K | $\sim 4.7$-5 | - | - |
| $\Theta$, $^\circ$ | 0 ($T < T_{SR}$) | $\sim 65.5$ | 90 |
| ($B = 0$) | 90 ($T > T_{SR}$) | ($T = 2$ K) | |
| $T_N$, K | $\sim 37.4$-39 | $\sim 37$ | $\sim 37.4$ |
| $\Theta$, K | $-210$ | $-165$ | $-145$ |

When the trigonal Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ crystal is magnetized in the basal $ab$ plane in weak fields, all the three possible domains with the antiferromagnetic axes oriented at an angle of $120^\circ$ to each other contribute to the magnetization (scheme c in figure 1). The $M_d(B)$ curves were calculated using the approach proposed in [12], where the magnetization processes occurring in easy-plane NdFe$_3$(BO$_3$)$_4$ were comprehensively studied with allowance for the possible existence of three types of domains. In the critical field $B_{SR} \approx 0.2$ T a spin-flop transition occurs in a domain with an antiferromagnetism axis along axis $a$ $L_0$ (see schemes c, d in figure 1) causing an anomaly of the $M_d(B)$ curve. This anomaly in curve $M_d(B)$ of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ exhibits smearing related to the presence of a real domain structure and a certain demagnetizing factor.
Figure 1. Schemes of orientations of the magnetic moments of the iron and rare-earth \( \mathbf{m} = \mathbf{m}_{0.9}^{\text{Ho}} + \mathbf{m}_{0.1}^{\text{Er}} \) sublattices used in the calculation of the magnetic characteristics for different temperature ranges and different directions of the external magnetic field: (a) \( B = 0 \) (inclined magnetic state), (b) \( B \parallel c \) (the \( ab \) plane is perpendicular to the figure plane), and (c, d) \( B \perp c \) (the axis \( c \) is perpendicular to the figure plane).

Figure 2. Experimental magnetization curves \( M_{\mathbf{a},\mathbf{c}}(B) \) of \( \text{Ho}_{0.9}\text{Er}_{0.1}\text{Fe}_3(\text{BO}_3)_4 \) for \( \mathbf{B}\parallel \mathbf{c} \) (a) and \( \mathbf{B}\parallel \mathbf{a} \) (b) at \( T = 3–30 \) K.
Figure 3. Experimental (symbols) and calculated (lines) magnetization curves $M_{a,c}(B)$ of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ at $T = 3$ K. $M_{a,c}^{HoFe}$ - magnetization curve of HoFe$_3$(BO$_3$)$_4$ [14] at $T = 2$ K. Inset: magnetization curves $M_a(B)$ in fields up to 1 T.

Figure 4. Experimental (symbols) and calculated (lines) magnetization curves $M_{a,c}(B)$ at $T = 10$ K.

Figure 5 shows the experimental and calculated temperature dependences of the initial magnetic susceptibilities $\chi_{a,c}(T)$ of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$. The weakly anisotropic behavior of the $\chi_{a,c}(T)$ curves is clearly visible. As follows from the inset to figure 5 the experimental magnetic susceptibility $\chi_{c}^{HoFe}(T)$ of HoFe$_3$(BO$_3$)$_4$ [4] near $T_{SR} = 4.5$ K decreases sharply, that can be explained by the existence of the spin-reorientation transition from the EP state (at $T > T_{SR}$) to EA state (at $T < T_{SR}$). The experimental investigation of the curves $\chi_{a,c}(T)$ showed the absence of a spin-reorientation transition at $T < 3$ K. The calculation of the temperature dependences $\chi_{a,c}(T)$ of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ with the determined parameters made it possible to establish the absence of a spin-reorientation transition at $T < 3$ K.

The Schottky-type anomaly in the experimental $\chi_{a,c}(T)$ curves near $T = 3.5$ K is related to the redistribution of the lower levels populations for the ground multiplet of the Ho$^{3+}$ ion in Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ and a correct calculation of the magnetization processes reproduces this effect.
Thus, the absence of anomalies in the magnetic characteristics of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$, associated with the spin-orientation transition, is due to the EP contribution of the Er$_{0.1}$-subsystem stabilizes the EP state Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ till lower temperatures than in HoFe$_3$(BO$_3$)$_4$.

5. Conclusion
We experimentally and theoretically investigated the magnetic properties of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ with the competing Ho–Fe and Er–Fe exchange couplings and obtained a good agreement between the theory and experiment for the entire set of the measured characteristics. The single theoretical approach allowed us to determine the important parameters by comparing the calculated data with the experimental results.

During magnetization along axis $a$ in a field of about 0.2 T, a spin-flop transition in one of the three possible domains resulting from trigonal symmetry causes the shape of the $M_a(B)$ curves that is characteristic of a phase transition smeared under real domain structure conditions.

The experimental and theoretical investigation of the temperature dependences of the magnetic susceptibility $\chi_{a,c}(T)$ showed the absence of a spin-reorienation transition in Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ with the decrease in temperature to 3 K. The Schottky-type anomaly in the experimental $\chi_{a,c}(T)$ curves near $T = 3.5$ K is related to the redistribution of the lower levels populations for the ground multiplet of the Ho$^{3+}$ ion in Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ and a correct calculation of the magnetization processes reproduces this effect.

The absence of anomalies in the magnetic characteristics of Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$, associated with the spin-orientation transition, is due to the EP contribution of the Er$_{0.1}$-subsystem stabilizes the EP state Ho$_{0.9}$Er$_{0.1}$Fe$_3$(BO$_3$)$_4$ till lower temperatures than in HoFe$_3$(BO$_3$)$_4$.

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