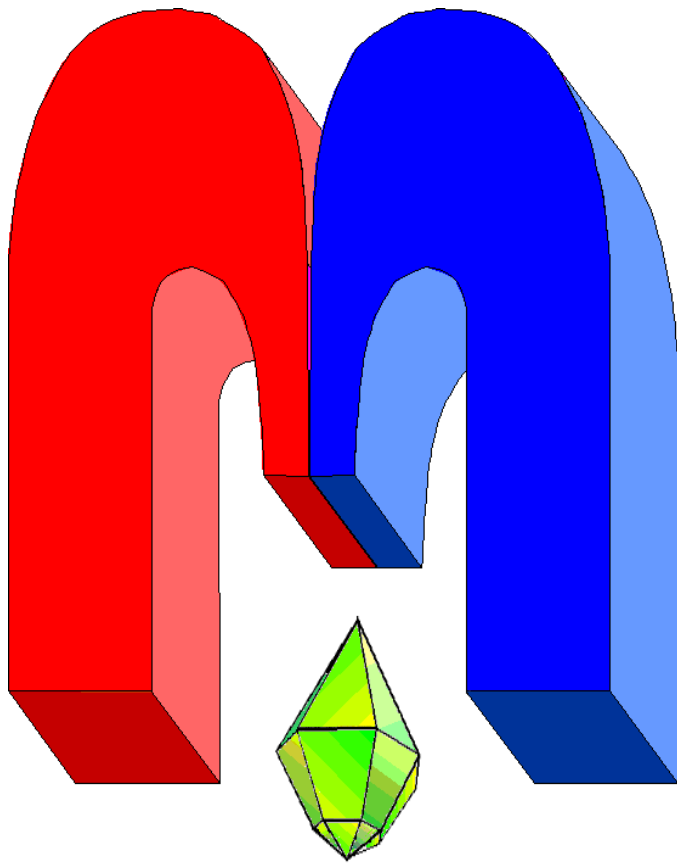


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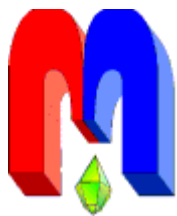
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‡ Dedicated to Professor Boris Z. Malkin on the occasion of his 85th birthday

Magnetization of LiErF₄ dipolar magnet in monocrystalline and polycrystalline form at low temperatures[†]

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This work presents the results of experimental and theoretical studies of the magnetic properties of the LiErF₄ single crystal and powder samples at low temperatures and applied field range of 0-9 T. The magnetization was calculated in the framework of the exchange-charge model taking into account dipole-dipole and electron-deformation interactions, with the calculation of the electron-deformation parameters. The theoretical analysis presents quantitative agreement in the temperature range of 2-300 K with the magnetization measurements of the LiErF₄ samples.

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Keywords: magnetization, magnetoelastic interaction

1. Introduction

Concentrated rare-earth–lithium double fluorides (also tetrafluorides, LiREF₄, RE = Gd–Yb) have gained attention as model objects in physics of dipolar magnetism, quantum phase transitions and crystal field effects on rare-earth 3+ ions (e. g. [1–9]). Besides, their unique magnetic and optical properties are important for practical applications as optical, magnetic and magneto-optical materials, e. g., in low-temperature adiabatic demagnetization refrigeration [10–14], in high-power Faraday devices [15, 16], in photonics and quantum electronics [17–19].

All LiREF₄ materials share the same, scheelite CaWO₄ type, *I*4₁/*a* symmetry with RE³⁺ ions in two magnetoequivalent positions in the unit cell [1, 20–22]. Rare-earth ions form a structure similar to a diamond crystal lattice [23], which makes rare-earth–lithium double fluorides a model object of order by disorder phenomena study [5, 24]. The significant influence of magnetic fields on the structure of the crystal lattice of double fluorides of rare earths has been observed earlier [25–28], and this effect appears only in strong magnetic fields ($B > 1$ T). The study of magnetic properties of LiREF₄ single crystals is an important problem for the development of the theory of magnetoelastic effects in magnetically concentrated crystals containing rare-earth ions [28].

LiErF₄ is an XY antiferromagnet with Neel temperature $T_N = 373(5)$ mK [29], easy plane is orthogonal to *c* axis. The energy spectrum of the Er³⁺ ion in the concentrated erbium tetrafluoride is shown in Table 2. The ground multiplet is ⁴I_{15/2}, its ground state is a Kramers doublet of Γ_{78} symmetry [4]. The *g*-factors have the values of $g_{\parallel} = 3.173(3)$, $g_{\perp} = 8.105(12)$ [30]. The linear magnetostriction of LiErF₄ single crystal in the field range of 0-3 T applied along the [100] axis has the order of $\Delta l/l \sim 10^{-4}$ [26]. A giant magnetocaloric effect was observed below the temperature of 2 K [31]. The neutron spectroscopy and specific heat study at low and ultra-low (sub-kelvin) temperatures was carried by Kraemer et al. [29], the magnetic phase diagram

[†]This paper is dedicated to Professor Boris Z. Malkin, who made a significant contribution to the field of magnetic radio spectroscopy in Kazan University, on the occasion of his 85th birthday.

was plotted and this system was found to have a huge interest for a research of a quantum phase transition. However, the magnetization of LiErF_4 single crystal as a function of the magnetic field, especially in strong magnetic fields (up to 5 T) and in the paramagnetic state at low temperatures (down to 2 K), has not been investigated as of now.

2. Experimental details

Single crystal LiErF_4 samples were grown in Kazan Federal University by the Bridgman-Stockbarger method. The resistive heater and thermal insulators were made of high-purity graphite. The crystals were grown at high vacuum (10^{-3} Pa) with a pulling rate of 1 mm/h using graphite crucibles for [100]-oriented boules. Single crystals were grown from compounds of 50 mol% ErF_3 (99.99 %) and 50 mol% LiF (99.99 %) at 850°C . These parameters are consistent with the growing conditions selected by Thoma et al. [21]. Powder LiErF_4 samples were synthesized in Kazan Federal University by sintering powders of fluorides taken in proportions according to the phase diagrams [21, 32] in the same oven as the single crystal samples. Powders of LiF (99.99 %) and ErF_3 (99.99 %) were mixed in an agate mortar and dried in the vacuum of $4 \cdot 10^{-3}$ Pa for 2 h at 150°C in a vitreous carbon crucible to prevent the hydrolysis. The synthesis was continued in the same crucible at 600°C in an atmosphere of high purity Ar (99.98 %) for 18 h. X-ray diffraction measurements were performed on a *Bruker D8 ADVANCE* diffractometer using $\text{Cu K}\alpha$ radiation with a wavelength $\lambda = 1.5418 \text{ \AA}$ to assess the crystallinity of the samples and for orientation of the single crystal samples. The comparison of the experimental powder LiErF_4 sample diffractogram and calculated one (Fig. 1) indicates no crystalline phases other than erbium–lithium double fluoride.

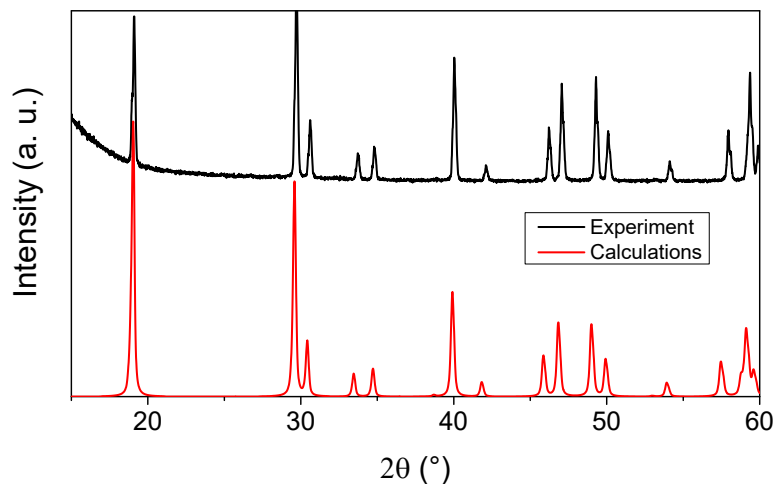


Figure 1. Experimental (top) and calculated (bottom) diffractograms of the powder LiErF_4 sample. Calculations were performed using *MAUD* software [33] with data taken from [20].

The magnetization of the powder samples was measured on a vibrating sample magnetometer *VSM* of *Physical Properties Measurement System (PPMS®)*, automated low-temperature and magnet system (*Quantum Design*), which allows DC magnetic measurements in the applied field range of 0-9 T and temperature range of 2-300 K (Kazan, Russia). The temperature dependencies of the magnetization of single crystals in the temperature range of 2-300 K and the dependencies of the magnetization on the magnetic field in the interval of 0-5 T applied along and perpendicular to the *c*-axis were measured with a DC-SQUID magnetometer *MPSM-2 (Quantum Design;*

Kanazawa, Japan). Ball-shaped LiErF₄ samples of 2 mm diameter were used in the single-crystal magnetization measurements.

3. Theoretical analysis

In the presence of an applied magnetic field \mathbf{B} , we write the Hamiltonian of a single Er³⁺ ion operating in the space of the ground multiplet states in the following form:

$$\hat{\mathcal{H}} = \hat{\mathcal{H}}_{\text{cf}} + \hat{\mathcal{H}}_Z + \sum_{\alpha\beta} \hat{V}'_{\alpha\beta} e_{\alpha\beta} + \sum_{\alpha,s} \hat{V}''_{\alpha}(s) w_{\alpha}(s). \quad (1)$$

$\hat{\mathcal{H}}_{\text{cf}}$ is the crystal field Hamiltonian

$$\begin{aligned} \hat{\mathcal{H}}_{\text{cf}} = & B_2^0 \hat{O}_2^0 + B_4^0 \hat{O}_4^0 + B_4^4 \hat{O}_4^4 + B_4^{-4} \hat{O}_4^{-4} \\ & + B_6^0 \hat{O}_6^0 + B_6^4 \hat{O}_6^4 + B_6^{-4} \hat{O}_6^{-4}, \end{aligned} \quad (2)$$

determined in the crystallographic system of coordinates by the set of seven crystal field parameters B_p^k (see Table 1), \hat{O}_p^k are the Stevens equivalent operators [22].

Table 1. Crystal field parameters for LiErF₄ (in cm⁻¹) [34].

B_2^0	B_4^0	B_6^0	B_4^4	B_4^{-4}	B_6^4	B_6^{-4}
190	-80	-2.3	-771	-667	-363	-222

$\hat{\mathcal{H}}_Z$ is the electronic Zeeman energy

$$\hat{\mathcal{H}}_Z = \mu_B \sum_j \mathbf{B}_{\text{loc}} \left(\hat{\mathbf{l}}_j + 2\hat{\mathbf{s}}_j \right), \quad (3)$$

where μ_B is the Bohr magneton, $\hat{\mathbf{l}}_j$ and $\hat{\mathbf{s}}_j$ are operators of electronic orbital and spin moments and the sum is taken over 4*f*-electrons. The magnetic dipole interaction between the Er³⁺ ions is taken into account by the introduction of a local magnetic field:

$$\mathbf{B}_{\text{loc}}(s) = \mathbf{B} + \sum_{s'} \left[\hat{Q}(s, s') - \frac{4\pi}{3v} N_m \delta_{ss'} \hat{\mathbf{l}} \right] \mathbf{M}(s'), \quad (4)$$

defined by the applied magnetic field \mathbf{B} , the lattice sums $\hat{Q}(s, s')$ calculated by the Ewald method ($Q_{xx}(s, s) = Q_{yy}(s, s) = 1.1702 \text{ cm}^{-3}$, $Q_{xx}(s_1, s_2) = Q_{yy}(s_1, s_2) = 0.4332 \text{ cm}^{-3}$, $Q_{zz}(s, s) = 0.6595 \text{ cm}^{-3}$, $Q_{zz}(s_1, s_2) = 2.1335 \text{ cm}^{-3}$, $s_1 \neq s_2$), the volume $v = 0.5a^2c$ of the unit cell, the demagnetization factor N_m and the magnetic moment $\mathbf{M}(s')$ of the ion in the s' -th sublattice. As the LiErF₄ is a dipolar magnetic, the exchange interaction has negligible effect, thus it has not been included.

The terms $\sum_{\alpha\beta} \hat{V}'_{\alpha\beta} e_{\alpha\beta} + \sum_{\alpha,s} \hat{V}''_{\alpha}(s) w_{\alpha}(s)$ in (1) correspond to linear interactions of the rare-earth ions with the homogeneous macro- and microdeformations, respectively, where \hat{e} is the deformation tensor and $w_{\alpha}(s)$ is the displacement of the s -th sublattice.

Table 2 shows the measured [4] and the calculated by diagonalizing the Hamiltonian (2) energy levels of the ground multiplet for LiErF₄ [35].

From the equilibrium conditions, we obtain the lattice macro-deformation induced by the external magnetic field [36]:

$$\hat{e}(\mathbf{B}) = -\frac{n}{v} \hat{S} \left[\langle \hat{V} \rangle_B - \langle \hat{V} \rangle_0 \right]. \quad (5)$$

Table 2. Energy (cm⁻¹) of the Stark sublevels of the ground multiplet of the Er³⁺ ion in LiErF₄.

	Experiment, [4]	Calculation, this work
Γ _{7,8}	0	0
Γ _{5,6}	18	17
Γ _{7,8}	29	22
Γ _{5,6}	57	57
Γ _{5,6}	256	245
Γ _{5,6}	294	289
Γ _{7,8}	321	316
Γ _{7,8}	346	343

Here \hat{S} is the compliance tensor of the lattice, $n = 2$ is the number of rare earth ions in the unit cell, and angular brackets $\langle \dots \rangle_B$, $\langle \dots \rangle_0$ indicate the quantum statistical averaging for $B \neq 0$ and $B = 0$, respectively, for the ion with the Hamiltonian $\hat{\mathcal{H}}_{cf} + \hat{\mathcal{H}}_Z$. Operator \hat{V} presents the renormalization of the operator \hat{V}' due to linear coupling of macro- and micro-deformations:

$$\hat{V} = \hat{V}' - \hat{b}\hat{a}^{-1}\hat{V}''.$$
 (6)

Here \hat{a} is the dynamic matrix of the lattice at the Brillouin zone centre, the tensors $\hat{b}_{\alpha\beta\gamma}(s)$ determine linear coupling between macro- and microdeformations.

Calculations are significantly simplified if the symmetry properties of the system are taken into account. Let us introduce linear combinations of components of the deformation tensor $e(A_g^1) = e_{zz}$, $e(A_g^2) = (e_{xx} + e_{yy})/2$, $e(B_g^1) = e_{xx} - e_{yy}$, $e(B_g^2) = e_{xy}$, $e_1(E_g) = e_{xz}$, $e_2(E_g) = e_{yz}$, which transform according to the irreducible representations $\Gamma = A_g, B_g, E_g$ of the factor group of the lattice. The magnetic field directed along the [001] axis induces only totally symmetric deformations A_g , whereas the field directed in the (001) plane induces totally symmetric and rhombic deformations B_g . In the basis of the symmetrized deformations, the operators in the electron–deformation interaction have the following form:

$$\hat{V}(A_g^\lambda) = a_{\lambda,2}^0 \hat{O}_2^0 + a_{\lambda,4}^0 \hat{O}_4^0 + a_{\lambda,4}^4 \hat{O}_4^4 + a_{\lambda,4}^{-4} \hat{\Omega}_4^4 + a_{\lambda,6}^0 \hat{O}_6^0 + a_{\lambda,6}^4 \hat{O}_6^4 + a_{\lambda,6}^{-4} \hat{\Omega}_6^4,$$
 (7)

$$\hat{V}(B_g^\lambda) = b_{\lambda,2}^2 \hat{O}_2^2 + b_{\lambda,2}^{-2} \hat{\Omega}_2^2 + b_{\lambda,4}^2 \hat{O}_4^2 + b_{\lambda,4}^{-2} \hat{\Omega}_4^2 + b_{\lambda,6}^2 \hat{O}_6^2 + b_{\lambda,6}^{-2} \hat{\Omega}_6^2 + b_{\lambda,6}^6 \hat{O}_6^6 + b_{\lambda,6}^{-6} \hat{\Omega}_6^6.$$
 (8)

The set of $a_{\lambda,p}^k$ and $b_{\lambda,p}^k$ parameters was calculated within the framework of the exchange-charge model [37] based on the crystal field [34] and EPR spectra [38] (Table 3).

4. Results and discussion

The magnetization M (magnetic moment per unit cell) of the powder of particles distributed equiprobably with respect to the direction of the applied field \mathbf{B} can be calculated as follows:

$$M = \frac{2\mu_B g_L}{4\pi} \int_0^{2\pi} d\phi \int_0^\pi d\theta \sin\theta \frac{\text{Tr} \left[\hat{\mathbf{J}} \frac{\mathbf{B}}{|\mathbf{B}|} \exp(-\hat{\mathcal{H}}\beta) \right]}{\text{Tr} \left[\exp(-\hat{\mathcal{H}}\beta) \right]}.$$
 (9)

Here, g_L is the Landé factor, $\text{Tr}[\dots]$ is the trace of the matrix, $\beta = (k_B T)^{-1}$ is the inverse temperature.

Table 3. Parameters of the interaction of Er^{3+} ions with deformations of A_g and B_g symmetry used in the work (in cm^{-1}).

p	k	$a_{1,p}^k$	$a_{2,p}^k$	p	k	$b_{1,p}^k$	$b_{2,p}^k$
2	0	16	-885	2	2	2045	4114
4	0	305	357	2	-2	2305	-886
4	4	2129	3228	4	2	-854	-1554
4	-4	1710	3592	4	-2	1449	1423
6	0	107	-88	6	2	-135	-528
6	4	1158	1452	6	-2	-427	-523
6	-4	502	1368	6	6	-605	-927
				6	-6	-691	-1046

In a self-consistent field approximation, the following procedure has been used to calculate the magnetization. First, the matrix of the Hamiltonian (1) with $M = 0$, $\hat{e} = \hat{0}$ and $w_\alpha(s) = 0$ is diagonalized to calculate the values of the macro- and micro-deformations $\hat{e}(\mathbf{B})$, $w_\alpha(s)$ and the magnetization M . At the next step, the obtained values of M , $\hat{e}(\mathbf{B})$ and $w_\alpha(s)$ are substituted into the Hamiltonian and the calculations are repeated.

Field dependencies of the magnetization of the powder LiErF_4 sample calculated using formula (9) together with the experimental data at different temperatures are shown on the Fig. 2. The calculations used the value of demagnetization factor equal to $N_m = 1$.

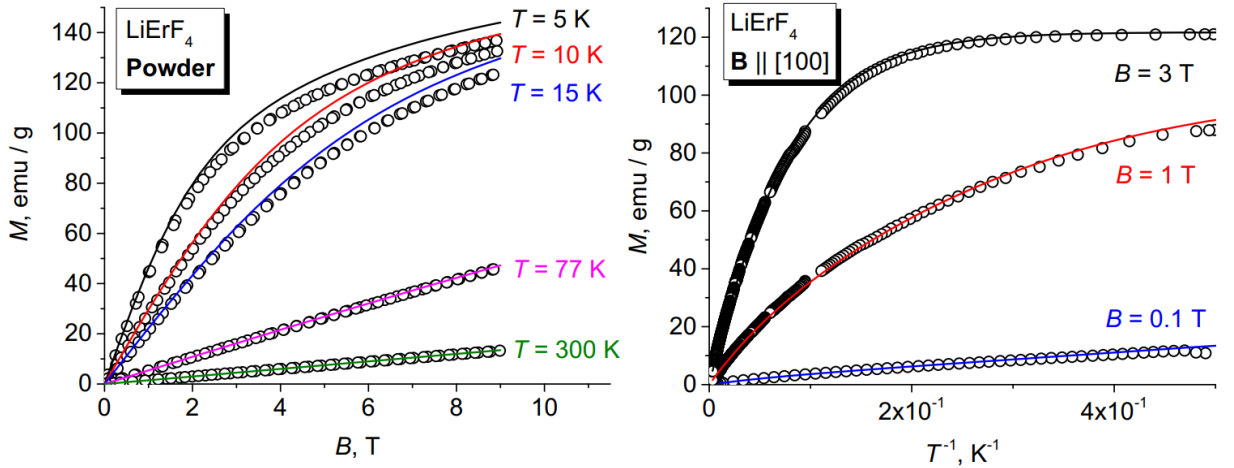


Figure 2. *Left:* Field dependencies of the magnetization of the powder LiErF_4 sample in an external magnetic field B at different temperatures. *Right:* Inverse temperature dependencies of the magnetization of the single crystal LiErF_4 sample in an external magnetic field $\mathbf{B} \parallel [100]$. Measurements by symbols and calculations by lines for both.

For a single crystal, the integration over all directions is unnecessary and therefore is omitted:

$$M = 2\mu_B g_L \frac{\text{Tr} \left[\hat{\mathbf{J}} \frac{\mathbf{B}}{|\mathbf{B}|} \exp(-\hat{\mathcal{H}}\beta) \right]}{\text{Tr} \left[\exp(-\hat{\mathcal{H}}\beta) \right]}. \quad (10)$$

Temperature dependencies of the magnetization of the single crystal LiErF_4 sample were measured in the external magnetic field \mathbf{B} applied along the $[100]$ direction (Fig. 2). Field-dependencies of the magnetization of the single crystal LiErF_4 sample were measured and calculated in the external magnetic field \mathbf{B} applied along the $[100]$ and the $[001]$ directions (Fig. 3).

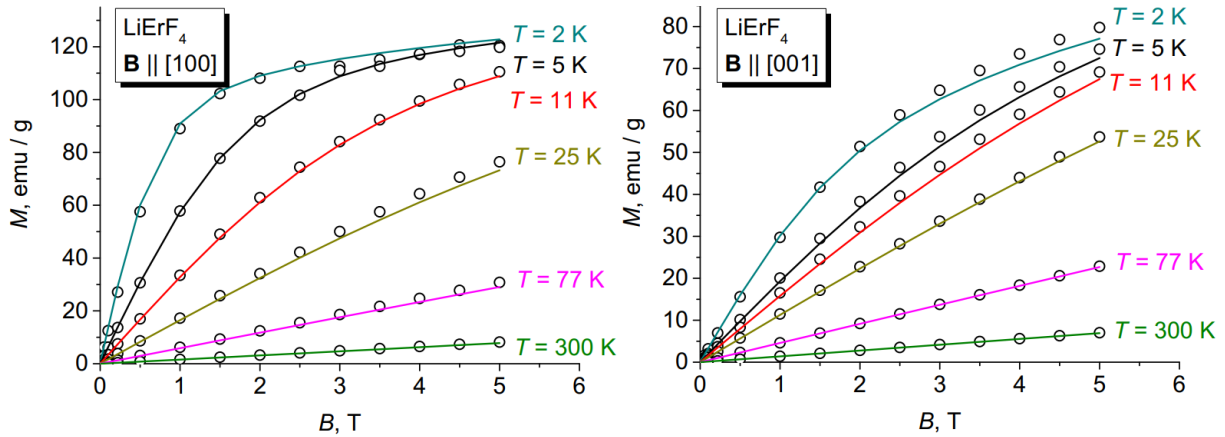


Figure 3. Field dependencies of the magnetization of the single crystal LiErF_4 sample in an external magnetic field $\mathbf{B} \parallel [100]$ (left) and $\mathbf{B} \parallel [001]$ (right), measured at different temperatures (symbols) and calculated ones (lines).

5. Conclusion

Magnetic properties of single crystal samples and polycrystal powder samples of LiErF_4 were measured and calculated taking into account the electron-deformation interaction. It has been shown that not only experimental data from this research, but also experimental Er^{3+} ion energy spectrum in the crystal field of LiErF_4 can be reproduced at low temperatures not only qualitatively, but also quantitatively, with a satisfactory accuracy in terms of a self-consistent theory that uses a unified set of parameters of the crystal field and electron-deformation interactions.

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