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Erbium luminescent probe in a $GdFe_3(BO_3)_4$ single crystal: phase transitions and temperature measurements

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In this study, we demonstrate a detection of three phase transitions in a GdFe₃(BO₃)₄ single crystal using the photoluminescence of Er^{3+} ions introduced in a small amount into the crystal. A first-order $R32 \rightarrow P3_121$ structural phase transition is seen as a sharp spectral line shift at $T_s = 127 \,\mathrm{K}$. Magnetic ordering of the iron subsystem at $T_N = 37 \,\mathrm{K}$ causes a splitting of Er^{3+} Kramers doublets and is detected by a splitting of spectral lines. A first-order spin-reorientation phase transition at $T_R = 9 \,\mathrm{K}$ manifests itself in a sharp change of the line splitting pattern. A comparison of the measured ratio of luminescence line intensities with the ratio of the Boltzmann populations of the involved levels is proposed to control the possible heating of the GdFe₃(BO₃)₄:Er³⁺ sample by exciting radiation.

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1. Introduction

The vast family of the rare-earth (RE) orthoborates $RM_3(BO_3)_4$, R=La, Pr–Er, Y and M = Al, Ga, Sc, Fe, Cr, is of great interest in terms of both solid state physics and practical applications. These compounds crystallize in the noncentrosymmetric trigonal structure of the natural mineral huntite. There are isolated chains of the M ions in the structure interconnected by RO_6 and BO_3 isolated units. In the case of magnetic M ions (Fe³⁺, Cr³⁺), quasi-one-dimensional magnets are realized. Aluminum borates $RAl_3(BO_3)_4$ have wide band gaps, good mechanical, thermal, and nonlinear optical properties, exhibit low quenching of RE luminescence and are widely used as media for different kinds of lasers, including minilasers on concentrated compounds [1] and self-frequency-doubling lasers [2–4]. Applications for the UV range are considered [5]. Recently, it was proposed to use several excited levels of the Gd³⁺ ion in YAl₃(BO₃)₄ (YAB) doped with Pr^{3+} and Gd³⁺ ions in the UV region of the spectrum to implement a Boltzmann thermometer operating from 30 to 800 K [6]. The UV region allowed detuning from background thermal radiation even at the highest temperatures. Gallium and scandium borates are less studied but also show good application potential [7,8].

The subfamily of RE iron borates was intensively studied all over the world, since L.N. Bezmaternykh and his coworkers in the Kirensky Institute of Physics in Krasnoyarsk managed to grow large single crystals of good optical quality. The RE iron borates possess diverse magnetic and magnetoelectric properties, depending on the particular R^{3+} ion [9–11]. Based on our high-resolution optical spectroscopy data on $RFe_3(BO_3)_4$, R=Pr, Nd, Sm, Eu, Tb, Dy, Ho, Er single crystals and his exchange-charge crystal-field model [12], B.Z. Malkin has developed a theoretical framework to explain magnetic, magnetoelectric, and optical properties of RE iron borates, taking into account the quasi-one-dimensionality of the magnetic subsystem of iron [13–22].

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Gadolinium iron borate GdFe₃(BO₃)₄ undergoes a first-order structural phase transition at $T_s = 174 \text{ K}$ [23] from the trigonal R32 structure to the also trigonal but less symmetric P3₁21 structure [24], it orders into an easy-plane antiferromagnetic structure at $T_{\rm N} = 37 \text{ K}$ [23, 25–27] and finally is subjected to a first-order spin-reorientation phase transition at $T_{\rm R} = 9 \text{ K}$ [25–27]. These phase transitions were detected by various methods, including Raman scattering [28] and spectroscopy of Nd³⁺ [25] and Er³⁺ [29] probes introduced into a GdFe₃(BO₃)₄ crystal, which has no intrinsic spectrum (of the Gd³⁺ ion) up to the UV region. It is worth mentioning that a light-induced absorption switching was observed in a GdFe₃(BO₃)₄:Nd³⁺ single crystal held at the temperature close to $T_{\rm R}$ [30].

Here, we explore a possibility to detect the mentioned phase transitions using the luminescence of the Er^{3+} probe in a $\text{GdFe}_3(\text{BO}_3)_4:\text{Er}^{3+}$ single crystal. The second goal of this study is the Boltzmann ratiometric luminescent thermometry with Er^{3+} ion in $\text{GdFe}_3(\text{BO}_3)_4$.

2. Experimental details

Two Er-doped single crystals were studied: $GdFe_3(BO_3)_4$: Er (1 at. %) and $GdFe_3(BO_3)_4$: Er (0.6 at. %). The crystals were grown on seeds from the solution-melts on the base of Bi₂Mo₃O₁₂, as described in Ref. [31]. Big transparent single crystals were green in color and had a good optical quality. Polished plane-parallel plates with the c axis lying in the plane were prepared. Temperature-dependent photoluminescence (PL) spectra excited by the radiation of multimode diode lasers (635 nm or 808 nm, with the spectral width of about 3 nm) were measured at the resolution of up to $0.2\,\mathrm{cm}^{-1}$ in the spectral range of the ${}^{4}\mathrm{I}_{13/2} \rightarrow {}^{4}\mathrm{I}_{15/2}$ optical transition of Er^{3+} (6200 - 6700 cm⁻¹ or 1.61 - 1.49 μ m) and the temperature interval from 3.5 to 290 K, using a Fourier spectrometer Bruker 125 HR and a closed-cycle helium cryostat Sumitomo SRP096. The crystal was cooled in steps of 0.5 K (5 K) in the range of temperatures $3.5 \text{ K} \rightarrow 50 \text{ K}$ $(50 \text{ K} \rightarrow 290 \text{ K})$. To reduce the thermal load on the sample, filters were used to attenuate the laser radiation, a double polished cold screen with small holes for radiation input and output was installed. The sample was glued with silver paste to the copper finger of the cryostat and additionally tightly wrapped with a sheet of metallic indium. Temperature was measured using a calibrated LakeShore DT-670 diode temperature sensor mounted in close proximity to the sample, controlled and recorded by a LakeShore Model 335 temperature PID controller. Temperature control was provided with an accuracy of ± 0.05 K.

3. Luminescent detection of phase transitions in a $GdFe_3(BO_3)_4$: Er^{3+} crystal

Figure 1a shows a part of the luminescence spectrum at the ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ transition of Er^{3+} in $GdFe_{3}(BO_{3})_{4}:Er^{3+}$. The scheme of crystal-field levels and optical transitions, which explains the lines' identification, is given in Fig. 1b.

The color intensity maps of Fig. 2 display the evolution of the spectrum with temperature. Abrupt lines' shift at 127 K (see Fig. 2a) is caused by a first-order structural phase transition $R32 \rightarrow P3_121$. The temperature of this transition in RE iron borates $RFe_3(BO_3)_4$ synthesized by solid state reaction varies linearly with the ionic radius r of the R^{3+} ion, $T_s = T_s(r)$, from $\sim 450 \text{ K}$ for $\text{ErFe}_3(BO_3)_4$ [$r(\text{Er}^{3+}) = 0.89 \text{ Å}$] to 88 K for $\text{EuFe}_3(BO_3)_4$ [$r(\text{Eu}^{3+}) = 0.947 \text{ Å}$], whereas $\text{SmFe}_3(BO_3)_4$ and $RFe_3(BO_3)_4$ with $r(R^{3+}) > r(\text{Sm}^{3+})$ remain in the R32 phase down to the lowest temperatures. $\text{GdFe}_3(\text{BO}_3)_4$ has $T_s = 174 \text{ K}$ [23]. Lower T_s temperatures observed for single crystals grown by solution-melt technique are due to incorporation of large Bi^{3+} ions from the $\text{Bi}_2\text{Mo}_3\text{O}_{12}$ flux into the crystal lattice during the growth process [32–36], which results in a $\text{Gd}_{1-x}\text{Bi}_x\text{Fe}_3(\text{BO}_3)_4$ compound with an increased lattice constant [33]. The estimate of the



Figure 1. a) PL spectrum of GdFe₃(BO₃)₄:Er³⁺ (0.6 at. %) in the region of the ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ transition of Er³⁺. $T = 50 \text{ K}, \lambda_{\text{ex}} = 808 \text{ nm}$; b) level diagram explaining the identification of luminescence lines.



Figure 2. Temperature dependence of the PL spectrum presented as intensity color maps in the region of the ⁴I_{13/2} → ⁴I_{15/2} transition of Er³⁺ in a,b) GdFe₃(BO₃)₄:Er³⁺ (1 at. %), λ_{ex} = 635 nm; c) GdFe₃(BO₃)₄:Er³⁺ (0.6 at. %), λ_{ex} = 808 nm.

Bi content x using the relation for the "effective" ionic radius: $r_{\text{eff}} = (1 - x)r_{\text{Gd}} + x \sim r_{\text{Bi}}$ with $r_{\text{Gd}} = 0.938$, $r_{\text{Bi}} = 1.03$ Å [37] and the $T_s(r)$ dependence from [23] gives $x = 0.048 \pm 0.005$. This value can be compared with x = 0.05 obtained in [33] from X-ray diffraction data.

The magnetic-ordering phase transition at $T_{\rm N} = 37 \,\mathrm{K}$ is of a second order, it manifests itself by a smoothly growing spectral lines' splitting corresponding to the splitting of erbium Kramers doublets in an internal magnetic field created by the ordered magnetic moments of iron. The line $6484 \,\mathrm{cm}^{-1}$ (B \rightarrow 2) displayed in Figs. 2b and 2c splits into three components, which points Erbium luminescent probe in a $GdFe_3(BO_3)_4$ single crystal: phase transitions...

to an equal splitting of the initial and terminal levels of the optical transition. This splitting reaches $3 \,\mathrm{cm}^{-1}$ at the temperature of 10 K.

The reorientation of the iron magnetic moments from the *ab* plane to the trigonal *c* axis abruptly changes the splitting pattern, and for the considered line $6484 \,\mathrm{cm}^{-1}$ four components are clearly observed. Note that the temperatures of this first-order transition differ markedly for the 0.6 and 1 at. % Er-doped compounds: $T_{\rm R} = 9 \,\mathrm{K}$ (see Fig. 2c) and 5 K (Fig. 2b), respectively. The reason for this could be the easy-plane anisotropy of Er^{3+} in the huntite structure: $\mathrm{ErFe}_3(\mathrm{BO}_3)_4$ orders into an easy-plane antiferromgnetic structure. Thus, a growing amount of the erbium impurity lowers the $T_{\rm R}$ temperature.

4. Boltzmann ratiometric luminescent thermometry with Er^{3+} doped into $GdFe_3(BO_3)_4$

As was shown above, an Er^{3+} impurity ion is an excellent luminescent probe of structural and magnetic phase transitions in $\text{GdFe}_3(\text{BO}_3)_4$. In what follows, we show that it can also serve as an internal thermometer to control, in particular, a possible heating of the sample by an exciting laser radiation.



Figure 3. Temperature dependences of the ratios $LIR[(B \rightarrow 2)/(A \rightarrow 2)]$ of the integral intensities of the luminescence lines 6520 cm^{-1} [transition $B({}^{4}I_{13/2}) \rightarrow 2({}^{4}I_{15/2})]$ and 6484 cm^{-1} [transition $A({}^{4}I_{13/2}) \rightarrow 2({}^{4}I_{15/2})]$ for GdFe₃(BO₃)₄ doped with Er^{3+} a) 0.6 at. % and b) 1.0 at. % (symbols) and n_B/n_A of the populations of the B and A levels separated by an interval 36 cm⁻¹, assuming the Boltzmann distribution (dashed lines). The excitation wavelength was a) 808 nm and b) 635 nm.

One of the methods to determine the temperature is based on measuring the lines' intensity ratio $LIR(T) = I_i/I_j$ of two luminescent lines originating from two energy levels, *i* and *j*, of a luminescent center, separated by an energy interval $E_i - E_j = \Delta E$ [38]. Equilibrium populations of the levels obey the Boltzmann distribution: $n_i(T)/n_j(T) = \exp(-\Delta E/k_{\rm B}T)$, and thus

$$LIR(T) = \frac{I_i(T)}{I_j(T)} = \frac{W_i n_i(T)}{W_j n_j(T)} = K e^{-\Delta E/k_{\rm B}T},$$
(1)

where $K = W_i/W_j$ is the temperature-independent ratio of probabilities. It is worth noting that, in general, the fulfilment of Eq. (1) should be checked, as both the Boltzmann distribution and the independence of K on temperature can fail [39].

In Fig. 3, we compare the ratio of Boltzmann populations n_B/n_A of the two crystal-field levels of Er^{3+} in $\text{GdFe}_3(\text{BO}_3)_4$: Er^{3+} , B and A, separated by the energy interval 36 cm⁻¹ and the line intensity ratio of the two luminescent lines $B\rightarrow 2$ and $A\rightarrow 2$ with their initial levels B and A, respectively, for the two excitation wavelengths, $\lambda_{ex} = 635 \text{ nm}$ and $\lambda_{ex} = 808 \text{ nm}$. While in the case of the $\lambda_{ex} = 808 \text{ nm}$ these quantities are well proportional one to another (excluding the temperature regions near the phase transitions at $T_s = 127 \text{ K}$ and $T_N = 37 \text{ K}$), a noticeable deviation below of about 30 K is observed in the case of $\lambda_{ex} = 635 \text{ nm}$. This is, evidently, due to a heating of the sample by laser radiation.

The wavelength $\lambda_{ex} = 808 \text{ nm}$ effectively excites the lowest level of the ${}^{4}I_{9/2}$ crystal-field multiplet. A subsequent relaxation to the ${}^{4}I_{13/2}$ multiplet is followed by the strong luminescent transitions in the region of 1.5 μ m. On the contrary, $\lambda_{ex} = 635 \text{ nm}$ falls into weak vibronic sidebands of the ${}^{4}I_{15/2} \rightarrow {}^{4}F_{9/2}$ transition of Er^{3+} , so a higher laser power is required to excite luminescence and, additionally, a longer nonradiative relaxation path leads to the initial level of the luminescence. All this leads to greater heating of the sample by laser radiation in the case of $\lambda_{ex} = 635 \text{ nm}$. Analysis of the data presented in Fig. 3b shows that instead of nominal 5 K at the spin-reorientation transition in a $\text{GdFe}_3(\text{BO}_3)_4:\text{Er}^{3+}(1 \text{ at. \%})$ crystal a real temperature at the sample is $9 \pm 0.2 \text{ K}$, the same as for the $\text{GdFe}_3(\text{BO}_3)_4:\text{Er}^{3+}(0.6 \text{ at. \%})$ crystal.

5. Conclusions

On the example of a GdFe₃(BO₃)₄ crystal doped with Er^{3+} , we have demonstrated a detection of structural and magnetic phase transitions by the method of rare-earth luminescent probe, with a simultaneous control of the temperature using the same luminescent probe. An internal Boltzmann ratiometric luminescent thermometer was realized on the two luminescence lines in the ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ transition of Er^{3+} originating from two erbium crystal-field levels separated by a gap of 36 cm⁻¹. Comparison of the measured line intensity ratios in the cases of different excitation wavelengths, 808 and 635 nm, and the relevant Boltzmann population ratio has shown that the excitation 635 nm heats the sample when a nominal temperature is below of about 35 K. The heating reaches 2 K at 15 K and grows to 4 K at 5 K. The heating does not exceed 0.5 K at 10 K if the excitation 808 nm is used.

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