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Spectroscopic and magneto-optical investigations of spin-reorientation phase transition in $\text{TbFe}_3(\text{BO}_3)_4$

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Spectroscopic and magneto-optical investigations of spin-reorientation transition induced by a magnetic field in antiferromagnetic terbium ferrobaborate $\text{TbFe}_3(\text{BO}_3)_4$ were performed. In the phase-transition region, a magnetic intermediate state with a periodic alternation of domains of initial and spin-flopped magnetic phases appears. As a result, a strong light scattering by the crystal takes place. It is shown that the main mechanism of light scattering is related to an appearance of the large Faraday rotation in a high-field magnetic phase. Visual polarization investigations of the domain structure in the intermediate state were carried out. The energy of the interphase boundary between the antiferromagnetic and high-field magnetic phases was calculated. The existence domain of the intermediate magnetic state was determined in the H - T coordinates.

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I. INTRODUCTION

Rare-earth ferrobaborates with the general formula $\text{RFe}_3(\text{BO}_3)_4$ (R—rare-earth element) have attracted attention because of diversity of magnetic structures and phase transitions realized in them due to the interaction of two magnetic subsystems: rare-earth and iron subsystem. Depending on the choice of rare-earth ion, they can be easy-axis or easy-plane antiferromagnets in the magnetically ordered state, and, in some compounds, the spontaneous spin-reorientation phase transition between two states takes place.¹

At room temperature ferrobaborates have a trigonal huntite crystal structure (the space group $R\bar{3}2$).^{2,3} The main elements of this structure are spiral chains of edgesharing FeO_6 octahedra elongated along the c axis. Three neighboring chains of the octahedra are connected with each other through prisms RO_6 and triangular groups BO_3 . At much lower temperatures some ferrobaborates undergo a transition into the lower symmetry phase with the structure $P3_121$.³ In this phase, as distinct from the high-temperature one, two non-equivalent iron positions appear. As regards rare-earth ions, they still occupy positions of one type but the symmetry of these positions is lowering from D_3 to C_2 .

In the terbium ferrobaborate $\text{TbFe}_3(\text{BO}_3)_4$, the structural phase transition takes place at 192 K.^{4,5} In $\text{TbFe}_3(\text{BO}_3)_4$, the antiferromagnetic ordering of iron subsystem with an orientation of Fe^{3+} magnetic moments along the three-fold axis C_3 occurs at $T_N = 40$ K. At the same time the antiferromagnetic ordering, caused by the interaction with the iron subsystem, takes also place in the terbium subsystem with magnetic moments parallel to the same axis.^{5,6} The magnetic moment of the terbium ground state is $8.6 \mu_B$, and the Landé factor is 17.8.^{5,6}

From the investigation of absorption spectra in the infrared region (${}^7F_6 \rightarrow {}^7F_2$ transition in Tb^{3+}), as the temperature

decreases below T_N , the splitting of some lines, equal to 32 cm^{-1} at 5 K, has been found.⁷ The authors have attributed this value to the splitting of the Tb^{3+} ground quasidoublet in the effective magnetic field resulted from the magnetic ordering of the iron subsystem.

As known from the magnetization measurements,^{5,6} when the magnetic field is oriented along the easy axis C_3 , the spin-reorientation phase transition of the first order is induced in the terbium ferrobaborate ($H_c = 35$ kOe at $T = 4.2$ K). In the iron subsystem, a spin-flop transition takes place, and magnetic moments of both terbium sublattices orient along the field direction.

New information about the interaction between magnetic subsystems of iron and terbium and about their behavior at the spin-reorientation phase transition can be obtained from investigation of the optical absorption spectra in $\text{TbFe}_3(\text{BO}_3)_4$ in magnetic fields. In the region of the field-induced phase transition of the first order, generally, a magnetic intermediate state with a periodic alternation of the initial antiferromagnetic phase and high-field one occurs.^{8,9} In this case, besides the spectroscopic method, the magneto-optical method of investigation (the most direct method of investigation of the magnetic intermediate state) can turn out to be useful.^{10,11} The aim of this work was the spectroscopic and magneto-optical investigation of the spin-reorientation phase transition in the terbium ferrobaborate $\text{TbFe}_3(\text{BO}_3)_4$.

II. EXPERIMENT

The single crystals of $\text{TbFe}_3(\text{BO}_3)_4$ were grown by the solution-melt technique described in Ref. 12. The samples had a shape of plane-parallel plates with the thickness of 1 and 0.11 mm, oriented perpendicular to the three-fold axis.

In order to investigate absorption spectra, the grating spectrograph DFS-13 was used. The light detection was

performed by a linear array of light-sensitive diodes being part of a multichannel optical analyzer. The absorption spectral resolution was about 0.35 cm^{-1} . The magnetic field was created by a superconducting coil. The light scattering in the phase-transition region was investigated under illumination of a sample using monochromatic light obtained from a grating monochromator, and also under illumination of a helium-neon laser ($\lambda = 633 \text{ nm}$). The registration of scattered light was performed by PMT. In order to plot the dependence of optical signal directly on the magnetic field, we used a multichannel analog-to-digital converter, to which the signal proportional to the PMT voltage and to the current in the solenoid was fed. The converted signals were acquired by a computer in a real-time mode.

Investigations of inhomogeneous magnetic states, formed in the phase-transition region, were carried out by means of visual polarization observations, as well as by measuring field dependences of rotation of the polarization plane. The optical scheme of polarizing microscope was used for the visual polarization observations. The field dependences of the light-polarization plane were performed by means of a modulation technique with the modulation of light in the polarization plane and with locked-in detection. In these measurements, the helium-neon laser was used as a light source.

III. RESULTS AND THEIR DISCUSSION

A. The absorption spectra of $\text{TbFe}_3(\text{BO}_3)_4$ in an external magnetic field oriented along the easy axis of the crystal

The investigation of the optical absorption spectra in $\text{TbFe}_3(\text{BO}_3)_4$ at various temperatures (2–70 K) are presented in the more recent work Ref. 13. With decreasing the temperature below T_N , the splitting of three absorption lines in the transition region ${}^7F_6 \rightarrow {}^5D_4$ was found in the α -polarized spectrum ($\mathbf{k} \parallel C_3$). The maximum values of the splitting appeared to be different for the three lines (4.7, 15.7, and 20 cm^{-1}) and smaller than the splitting observed for the transition ${}^7F_6 \rightarrow {}^7F_2$,⁷ so it is naturally to associate them with the splitting of the terbium excited states in an effective magnetic field of the magnetically ordered iron subsystem.

In Fig. 1(a) (lower curve), the absorption spectrum of $\text{TbFe}_3(\text{BO}_3)_4$ in the region of the transition ${}^7F_6 \rightarrow {}^5D_4$ in the absence of an external magnetic field at temperature 2 K is shown. The lines, resulted from the splitting of three doublets, are indicated by $1a$, $1b$, $2a$, $2b$ and $3b$ (the line $3a$ is not observed at $T = 2 \text{ K}$, since it corresponds to the transition from the upper sublevel of the ground state¹³). The lines, marked by "t", reproducing the temperature behavior of the corresponding lines without the symbol "t", were interpreted as phonon wings of electron absorption lines.¹³

The absorption spectra in $\text{TbFe}_3(\text{BO}_3)_4$ were investigated in the external magnetic field directed along the easy axis of the crystal C_3 at the temperature 2 K. The relevant series of spectra is presented in Figs. 1(a) and 1(b), and the dependences of line energies on the magnetic field are shown in Fig. 2. At $H < H_c$, the linear in a magnetic field splitting of lines caused by the non-equivalence of terbium magnetic sublattices with respect to an external magnetic field is observed. In the high-field magnetic phase, the non-equivalence of the sublattices disappears. As a result, a

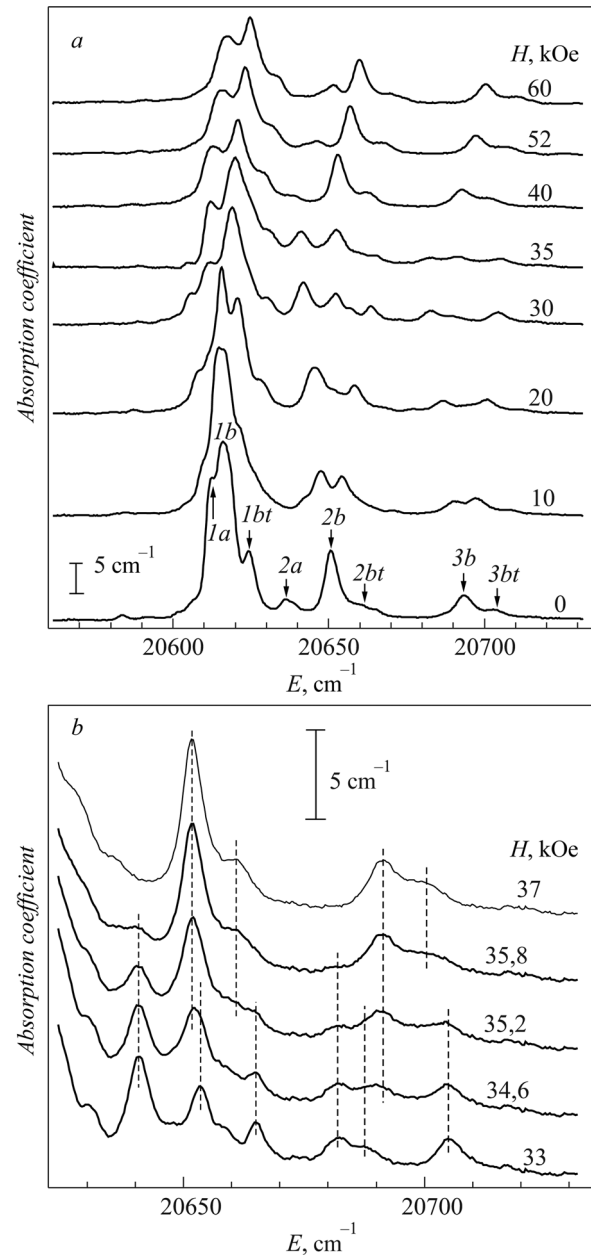


FIG. 1. The absorption spectrum in $\text{TbFe}_3(\text{BO}_3)_4$ in the transition region ${}^7F_6 \rightarrow {}^5D_4$ in Tb^{3+} (α -polarization) in an external magnetic field $\mathbf{H} \parallel C_3$, $T = 2 \text{ K}$ (a); the part of the spectrum in the field range corresponding to the spin-reorientation phase transition (b).

reconstruction of the absorption spectrum accompanied by decreasing the number of lines and by displacement of the existing lines takes place.

Let us consider the behavior of the lines directly in the region of the spin-reorientation transition in more detail. As known, an investigation of spectra in the phase-transition region can provide with an accurate enough information about the order of a transition. So, the step-wise change of a line position at the phase transition along with the simultaneous existence in the spectrum of the lines corresponding to the antiferromagnetic and high-field phases indicate the presence of an intermediate state.^{10,14}

In Fig. 1(b), the part of the absorption spectrum in $\text{TbFe}_3(\text{BO}_3)_4$, where character of the behavior of the absorption line in the region of the spin-reorientation transition (33–37 kOe) can clearly be traced, is shown. In Figs. 1(b)

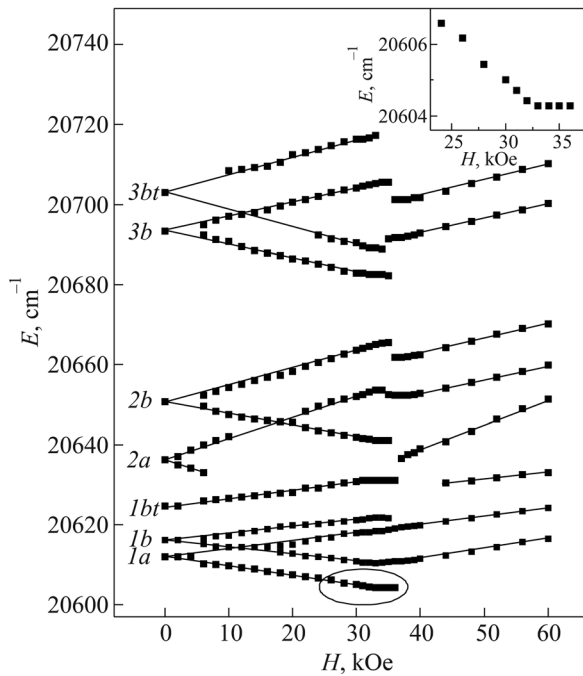


FIG. 2. The dependence of positions of absorption lines in $\text{TbFe}_3(\text{BO}_3)_4$ (the optical transition ${}^7F_6 \rightarrow {}^5D_4$ in Tb^{3+}) on an external magnetic field $\mathbf{H} \parallel \mathbf{C}_3$, $T = 2$ K. Inset: a fragment of the dependence $E(H)$ of low-frequency component of the line $1a$ in the phase-transition region.

and 2, it is seen that the line positions at the phase transition change in the step-like way. In addition, in some field range, the overlapping of the spectra from the simultaneously coexisting phases (antiferromagnetic and high-field) can be observed (Fig. 1(b)). We direct the attention to the fact that in the field range corresponding to the phase transition, the energies of the lines do not change with increasing magnetic field (see the inset in Fig. 2). This indicates that an internal field in the crystal in this region remains constant that is also typical for the first-order phase transition.^{10,15,16}

B. The light scattering effect in the spin-reorientation phase transition region

Investigating the absorption spectra in $\text{TbFe}_3(\text{BO}_3)_4$ in the magnetic field at $\mathbf{H} \parallel \mathbf{k} \parallel \mathbf{C}_3$, it was found that an intensity of the light transmitted through the sample decreases noticeably in the field range 33–37 kOe. The dependence of transmission in the sample with the thickness of 1 mm at the frequency of 20550 cm^{-1} (outside the terbium absorption lines) on an external magnetic field is demonstrated in Fig. 3(a). It is seen that in the region of the spin-reorientation transition, a significant reduction in the direct transmitted beam of light occurs. The reason of such weakening of light is a strong scattering appearing in the sample in the phase transition region. In Fig. 3(b), a record of the scattered light ($E = 20550 \text{ cm}^{-1}$) in the direction making angle $\sim 1^\circ$ with the direction of propagation of direct light is shown. In the dependences of both transmitted and scattered light (Fig. 3), a small hysteresis (~ 0.3 kOe) is observed.

If the light exiting the sample is directed to a screen, then it is possible to trace how the picture of light scattering changes depending on an external magnetic field. The example of such a picture in the case of laser light (the wavelength is 633 nm) is presented in Fig. 4. In the absence of magnetic

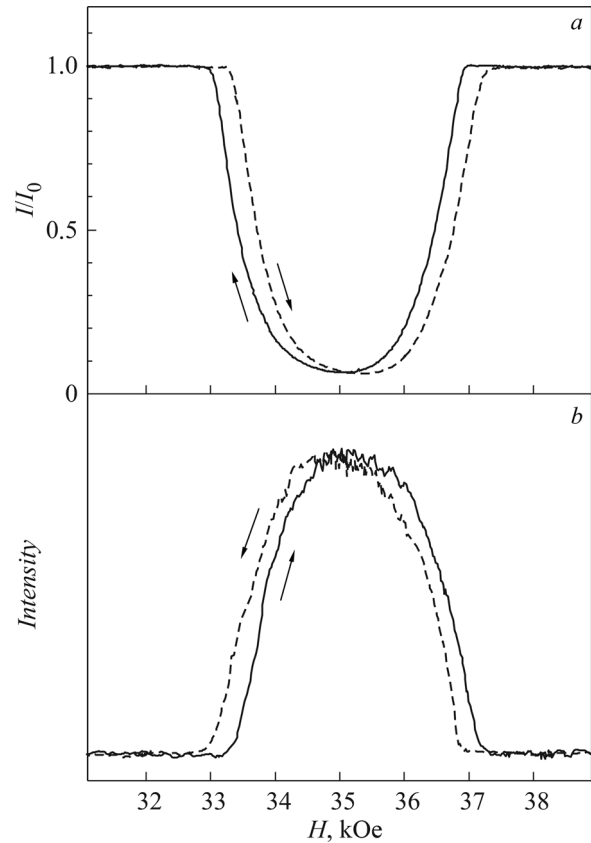


FIG. 3. The dependence of intensity of the light with $E = 20550 \text{ cm}^{-1}$ transmitted through the sample (a) and of the scattered light (b) on an external magnetic field in the vicinity of the spin-reorientation transition. The direction of the magnetic field change is shown by arrows. The sample thickness is of 1 mm. $\mathbf{H} \parallel \mathbf{C}_3$, $\mathbf{k} \parallel \mathbf{C}_3$, $T = 2$ K.

field as well as in fields not exceeding 33 kOe, there is a small spot of scattered light around the central beam (Fig. 4(a)), the shape and intensity of which do not change in the field range 0–33 kOe. In passing through the region of the phase transition (33–37 kOe), the intensity of the central beam decreases, and a wide halo of scattered light is created around it. This effect is demonstrated in the picture Fig. 4(b) (an angular radius of the spot is about 4° and is limited by a cryostat aperture). Above the phase-transition region the picture of scattering becomes similar to that below the transition (Fig. 4(c)). When the sample is illuminated by linearly-polarized as well as non-polarized light, the picture remains unchanged.

It is necessary to note that the light scattering in the direction opposite to that of the incident light is practically not observed. The major part of light is scattered "forward" that is typical when scattering particles are equal to or greater than the wavelength.¹⁷ As such scattering particles, the magnetic domains appearing in the spin-reorientation phase transition region can be considered.

C. The visual observation of domain structure of the magnetic intermediate state

In order to be convinced in the presence of the magnetically inhomogeneous state in the region of the phase transition, the visual polarization investigations were performed on the wavelength of 546 nm. In Fig. 5, the images of domain structure of the magnetic intermediate state in

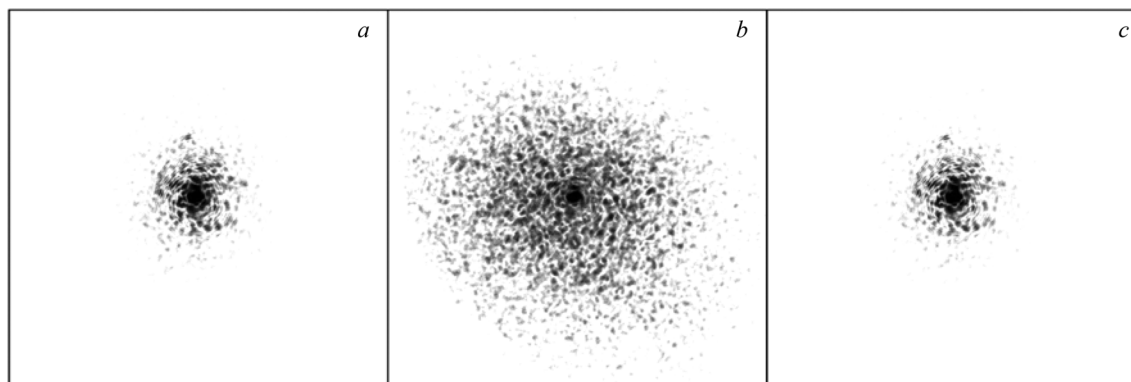


FIG. 4. Images of pattern of scattering of laser light by the sample at various magnetic fields: below the phase transition region ($H = 31$ kOe) (a); in the transition region ($H = 35$ kOe) (b); above the transition ($H = 39$ kOe) (c). The sample thickness is of 1 mm, $T = 2$ K.

$\text{TbFe}_3(\text{BO}_3)_4$ at the temperature of 8.3 K in the field range 37.6–42.6 kOe are presented. The light areas in the images correspond to the high-field magnetic phase. In the image 1, the sample below the phase transition in the magnetic field $H = 37.6$ kOe is shown. Typically, the high-field phase appeared in the form of magnetic bubbles in the magnetic field $H_1 = 38.8$ kOe (the image 2). A diameter of domains in this magnetic field is of about $8 \mu\text{m}$. With increasing magnetic field the bubbles transformed to the maze magnetic domain structure with a period about $16 \mu\text{m}$ at $H = 41.3$ kOe (the images 5–7). The further increase of magnetic field up to $H_2 = 42.6$ kOe led to the homogeneous magnetized state (the image 8).

It is naturally to assume that the magnetic domains, which appear in the phase-transition region, are those optical inhomogeneities on which light is scattered. As follows from the visual observations, the size of these optical inhomogeneities is larger than the wavelength of the light that agrees with already shown experiments on the light scattering.

The light diffraction effect of magnetic domains was first found in ferromagnetic CrBr_3 .^{18,19} The nature of this effect was associated with the presence in a crystal of the

large Faraday rotation, the sign of which depends on the direction of a magnetization vector. Thus, in a domain periodic structure the spatial modulation of magnetization leads to the spatial modulation of optical properties.

In this connection an investigation of the Faraday rotation in $\text{TbFe}_3(\text{BO}_3)_4$ and of its possible correlation with the light scattering effect is of interest.

D. The Faraday effect and its relation to the light scattering effect

As mentioned previously, above the spin-reorientation phase transition the terbium magnetic moments are oriented along the external magnetic field, whereas magnetic moments of iron — almost perpendicular to the field. A contribution to rotation of the polarization plane in the magnetic field can be made by both the terbium subsystem and the iron one. However, an exchange interaction in the subsystem of iron ions is rather strong ($H_E = 705$ kOe (Ref. 5)), so, over the investigated field range, the magnetization of the iron subsystem along the field (and along the direction of light propagation) is negligible, and hence a prevalent contribution to the Faraday rotation should be made by the terbium subsystem.

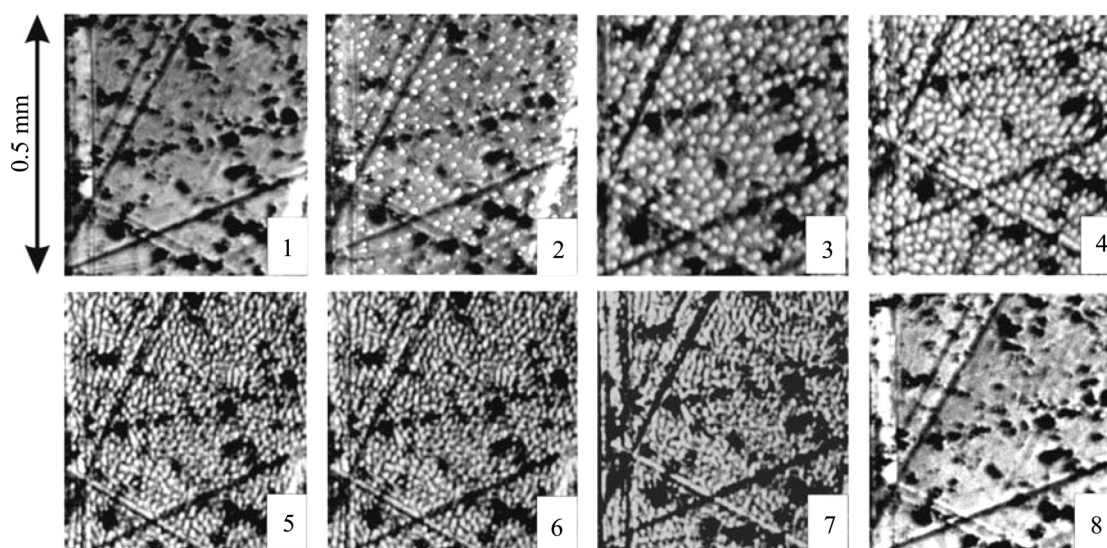


FIG. 5. Domain structure of the magnetic intermediate state in $\text{TbFe}_3(\text{BO}_3)_4$ at $T = 8.3$ K (dark areas correspond to the antiferromagnetic phase, light areas — to the high-field magnetic phase) in various magnetic fields $H \parallel C_3$: 37.6 (1), 38.8 (2), 39 (3), 39.5 (4), 40.1 (5), 41.3 (6), 42.2 (7), 42.6 (8) kOe. The sample thickness is of 0.11 mm.

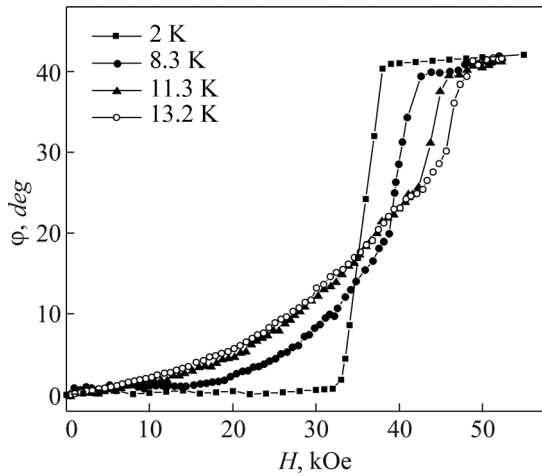


FIG. 6. Field dependences of rotation of light-polarization plane ($\lambda = 633$ nm) at various temperatures in the magnetic field $\mathbf{H}||\mathbf{k}||\mathbf{C}_3$. The sample thickness is of 0.11 mm.

In Fig. 6, field dependences of rotation of the polarization plane $\varphi(H)$ measured at the temperatures $T = 2, 8.3, 11.3,$ and 13.2 K and $\mathbf{H}||\mathbf{k}||\mathbf{C}_3$ are shown. In the spin-reorientation phase transition region, a jump on the $\varphi(H)$ curves is observed. As seen in Fig. 6, with increasing the temperature the magnitude of the jump decreases, and it shifts towards high fields.

Thus, the measurements indicate that in the high-field phase, the $\text{TbFe}_3(\text{BO}_3)_4$ crystal has rather high Faraday rotation (about 40° for the 0.11-mm thick sample). At the same time, in the antiferromagnetic phase in the vicinity of the transition at the temperature 2 K, the rotation is practically absent. Therefore an appearance of the intermediate state with a spatial alternation of two phases can create in the crystal significant optical inhomogeneities causing light scattering. From values of the Faraday rotation the difference in the refractive index of the antiferromagnetic and high-field phases in the case of circularly polarized modes can be estimated: $\Delta n \approx 1.2 \cdot 10^{-3}$ at $T = 2$ K.

The magnetic linear birefringence appearing at the spin-flop transition in the iron subsystem at $\mathbf{k}||\mathbf{H}||\mathbf{C}_3$ is among other possible causes of light scattering. It is absent in the antiferromagnetic phase and comes into play when magnetic moments of iron are perpendicular to the field as well as to the direction of light propagation. In this case, three types of domains of the high-field phase can appear in the crystal. In addition, at the spin-reorientation transition the structural distortions related to the magnetostriction can appear.^{13,20,21} They also can contribute to the linear birefringence. Indeed, the visual investigations above the phase transition using crossed polarizers demonstrated that besides circular, there is also a linear magnetic birefringence. No appreciable optical inhomogeneity was found in the sample. It probably indicates that only one of three domain types of the high field phase is realized in the sample. The value of the linear birefringence in the magnetic field $H = 43$ kOe at $T = 6$ K did not exceed $2 \cdot 10^{-4}$ that is significantly smaller than the circular birefringence.

The spectral dependences of an angle of the polarization-plane rotation and of light scattering efficiency were also measured. These dependences are shown in Fig. 7. The scattering efficiency was determined as relative weaken-

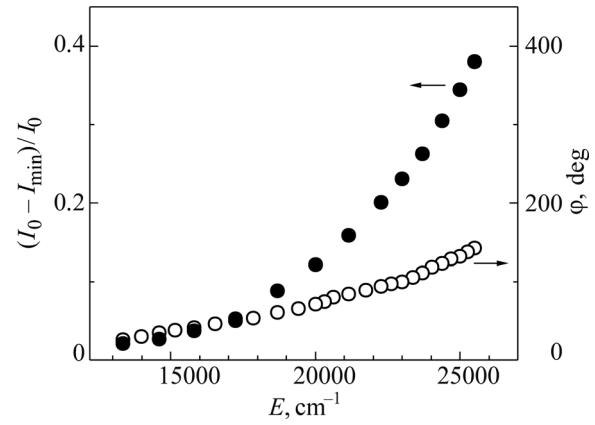


FIG. 7. Spectral dependences of light-scattering efficiency in the phase-transition region ($H = 35$ kOe) and of angle of rotation of the polarization plane in the high-field phase ($H = 40$ kOe). The sample thickness is of 0.11 mm, $T = 2$ K.

ing of a central beam of light: $(I_0 - I_{\min})/I_0$, where I_0 is the intensity of a central beam outside the phase transition region, I_{\min} is the same but during the transition at the point of the highest scattering. The spectral dependences of the Faraday effect and of the scattering reveal a correlation of these two effects (Fig. 7). A more rapid increase of the scattering with decreasing the wavelength is likely to be due to decrease of the ratio between the wavelength and size of inhomogeneities.

E. H - T phase diagram of $\text{TbFe}_3(\text{BO}_3)_4$ and energy of interphase wall

In order to determine regions of existence of the intermediate state, the fields H_1 and H_2 were measured by means of a visual observation as well as from field dependences of rotation of light-polarization plane at various temperatures (Fig. 6). At $T = 2$ K the data obtained from field dependences of light scattering were also used.

The H - T phase diagram for the antiferromagnet $\text{TbFe}_3(\text{BO}_3)_4$ is shown in Fig. 8. Note that the data obtained from magneto-optical measurements and those from scattering investigations are in a good agreement with each other that confirms the relationship of the scattering effect with an appearance of domain structure of the intermediate state.

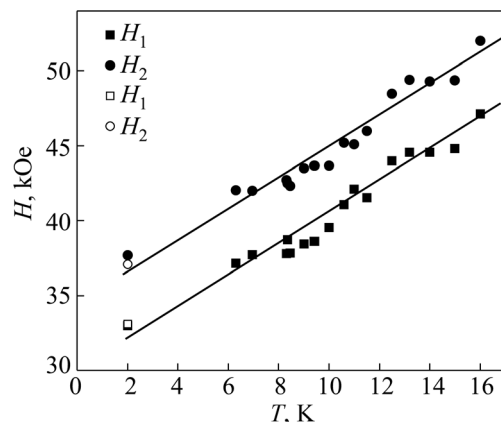


FIG. 8. The H - T phase diagram of the terbium ferroborate. Symbols (■) and (●) indicate fields H_1 and H_2 obtained from magneto-optical investigations, (□) and (○)—obtained from experiments on light scattering. The solid lines correspond to a linear fit.

Taking into account a period of maze domain structure, the energy of the wall between the antiferromagnetic and high-field magnetic phases can be calculated. This energy is determined by the following expression:¹⁵

$$\sigma = \frac{D^2 \Delta M^2 \pi F(\rho)}{t}.$$

Here t is the plate thickness, D is the period of the domain structure, ΔM is the magnetization jump at the first-order phase transition, ρ is the concentration of the magnetic phase, $F(\rho)$ is the function of ρ :²²

$$F(\rho) = \frac{1}{(2\pi)^2} - \frac{1}{8}(1 - 2\rho)^2 \ln 2 \quad \text{and} \quad 1 - 2\rho \ll 1.$$

Substituting the value $\Delta M \approx 330$ G, obtained from measurements of field dependences of the magnetization, and also $D \approx 16 \mu\text{m}$, $t = 110 \mu\text{m}$, $F(0.5) \approx 0.025$, we obtain $\sigma \approx 2 \text{ erg/cm}^2$.

IV. CONCLUSION

Spectroscopic and magneto-optical investigations of the $\text{TbFe}_3(\text{BO}_3)_4$ single crystal in the external magnetic field $\mathbf{H} \parallel \mathbf{C}_3$ have shown the following.

In the absorption spectra in the region of the spin-reorientation phase transition (33–37 kOe at $T = 2$ K), there are simultaneously lines related to two magnetic phases that indicates the appearance of an intermediate magnetic state. In the same magnetic field range, a strong scattering of light by the crystal is observed.

Visual polarization-optical investigation in the phase-transition region demonstrated that a high-field magnetic phase arises in the form of magnetic bubbles, and then transforms to a maze structure. The formation of the double-phase domain structure apparently causes the strong scattering of light by the crystal.

The performed measurements of rotation of a polarization plane and of magnetic linear birefringence at $\mathbf{H} \parallel \mathbf{k} \parallel \mathbf{C}_3$ established that in the high-field magnetic phase the circular birefringence in $\text{TbFe}_3(\text{BO}_3)_4$ is an order of magnitude higher than the linear birefringence values. This suggests that the main mechanism of light scattering at the spin-reorientation transition is the circular birefringence. This assumption is also confirmed by a correlation between spectral dependences of light-scattering efficiency and of the Faraday rotation.

The obtained magneto-optical data allowed to determine an existence domain of the magnetic intermediate state in

H - T -coordinates, and also the energy of the wall between the antiferromagnetic and high-field magnetic phases — 2 erg/cm^2 .

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