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OPTICAL AND MAGNETO-OPTICAL PROPERTIES OF $Na_{0.375}Tb_{0.625}F_{2.25}$ CRYSTAL

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Optical and magneto-optical spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal have been studied within the ultraviolet and visible spectral range. It has been established that within the wavelength range of 535-560 nm there is an intense luminescence band caused by intra-configurational 4f→4f transitions $^5D_4 \rightarrow {}^7F_5$. In the luminescence band $^5D_4 \rightarrow {}^7F_5$ and $^5D_4 \rightarrow {}^7F_4$ the radiative 4f→4f transitions have been identified, and it has been established that the magnetic dipole transitions dominate in the secondary emission spectra. Analysis of magneto-optical research data has demonstrated that the C-term of magneto-optical activity plays a significant role in the mechanism underlying the occurrence of magneto-optical effects in luminescence bands caused by "forbidden" 4f→4f transitions. A comparative analysis of the dispersion dependence of the Verdet constant of the $Na_{0.375}Tb_{0.625}F_{2.25}$ single crystal showed that it is about 90% of the Verdet constant of the well-known $Tb_3Ga_5O_{12}$ crystal, which demonstrates a good magneto-optical properties of the new synthesized crystal.

Keywords: *Fluoride crystals; Rare-earth ions; Magneto-optical properties; Luminescence; Energy levels; Magnetic circular polarization; Verdet constant*
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INTRODUCTION

Single crystals of inorganic fluorides have long been used as optical materials with high transparency within a wide range of wavelengths from vacuum UV to mid-IR. Scintillators made from alkaline and alkaline earth fluorides are also well known. Until recently, they were simple compounds like CaF_2, LiF, BaF_2, MgF_2, LaF_3 and some others doped with activator-ions such as rare earth elements and some 3d elements [1,2]. The new field of quantum electronics required new active media with qualitatively other laser parameters differed from those of commercial CaF_2 and BaF_2 [3,4]. A worldwide search for laser crystals began among which metal fluorides became the leading materials. A main idea of obtaining qualitatively new materials was to move from single-component metal fluoride crystals to multi-component ones (in basic composition, with no activator). This approach to the problem of new fluoride materials has provided significant advances in the field of inorganic material science, which later expanded the boundaries of quantum electronics. Currently, there is a need for new scintillators for high-energy physics and related fields. New areas of application of highly transparent optical media require crystalline matrices with better mechanical, chemical, thermal properties, as well as better radiation stability and absorption of γ-rays, etc. Moreover, it has become necessary to control luminescence and other functional characteristics mainly determined by a type of activator ions and by crystalline matrices as well.

Rare-earth fluorides having the fluorite structure $Na_{0.5-x}R_{0.5+x}F_{2+2x}$ and rare earth ion activators attract great attention due to the possibility of obtaining highly concentrated (in contrast to glasses) media with high optical transparency, low melting point and low refractive indices as compared to oxide crystals [3-5]. The properties of these materials are determine by an electronic structure of the rare-earth impurity centers, which is largely determined by the crystal field acting on the rare earth ion. When doped, the rare-earth ion R^{3+} replaces a metal cation in the fluorite structure, which causes distortion of the crystal lattice near the impurity. It seems relevant to study a crystal structure of the rare-earth impurity centers, since the main contribution to the crystal field of a rare-earth ion is made by its neighboring environment.

Materials doped with rare-earth ions (RE) are key elements of modern devices for generating, transmitting and controlling optical signals. On the basis of glassy and crystalline matrices doped with rare-earth ions the effective solid-state lasers, fiber lasers and amplifiers, radiation visualizers, etc. have been created [6]. The variety of optical effects observed in such media has determined the undying interest in rare-earth ions for more than 50 years. As for magneto-optical properties, the search for paramagnetic crystals with effective Faraday rotation has been conducted for many years among various materials [7]. For oxides, the rare-earth garnets crystals containing Tb^{3+} ions (primarily, terbium gallium garnet $Tb_3Ga_5O_{12}$) were found, having wide practical application in Faraday insulators and gates for various laser devices [8,9]. A lot of Faradaic paramagnetic media contain the rare-earth elements (REE) as active ions. However, most REE ions in crystals have intense absorption bands and, as a result, small transparency ranges within the near ultraviolet (UV), visible and infrared (IR) ranges, which complicates their practical use. Taking into account the need to ensure high optical transparency of rare-earth crystals, the choice is reduced to practically a few ions (Tb^{3+}, Pr^{3+} и Ce^{3+}) with high magneto-

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optical efficiency and transparency over a wide spectral range [9]. The previously studied anisotropic fluoride crystals ($LiTbF_4$, KTb_3F_{10}, $Ca_{1-x}Tb_xF_{2+x}$ ($0.03 < x \leq 0.32$), etc.) are inferior in magneto-optical characteristics to $Tb_3Ga_5O_{12}$ crystals, because the application of anisotropic materials is associated with precise orientation of the optical axis, since in directions that do not coincide with the optical crystal axis the Faraday effect is significantly complicated by conventional birefringence. Thus, the study of the magneto-optical properties of a new isotropic cubic-syngony crystal with the fluorite structure $Na_{0.5-x}R_{0.5+x}F_{2+2x}$ and increased concentration of terbium ions is an urgent and topical task since this makes it possible to expand the range of fluoride cubic crystals with good magneto-optical properties.

The use of $Na_{0.375}Tb_{0.625}F_{2.25}$ crystals in practical applications requires a detailed study of their optical and magneto-optical properties, which will contribute to a deep understanding of the fundamental features of the magneto-optics of RE ions in fluoride crystals. Therefore, a motivating factor of this work was a detailed analysis of the experimental data of optical (absorption, luminescence) and differential methods of magneto-optical studies, including magnetic circular dichroism - MCD and magnetic circular polarization of luminescence - MCPL, to be carried on over the visible spectral range with the samples of $Na_{0.375}Tb_{0.625}F_{2.25}$ crystals.

MATERIALS AND METHODS

The object of the study was $Na_{0.375}Tb_{0.625}F_{2.25}$ crystals of the fluorite type. The cubic phases crystallizing in the fluorite structural type are formed in binary fluoride systems MF_m–TbF_3 (M = Na, K, Ca, Sr, Ba, Cd, Pb) [10]. In the NaF–RF_3 systems, the fluorite phases $Na_{0.5-x}R_{0.5+x}F_{2+2x}$ contain >50 mol.% of RE. The $Na_{0.5-x}R_{0.5+x}F_{2+2x}$ crystals was grown by the Bridgman method at the Institute of Crystallography (Russia).

The absorption and fluorescence spectra were obtained with a high-resolution diffraction monochromator MDR-23 (LOMO, Russia). The wavelength range in the absorption experiments was within 220 nm - 700 nm with an average instrumental resolution of 0.03 nm. Since the scattering at the sample was negligible, the absorption value is represented as a dimensionless Absorbance, which is the common logarithm of the ratio of incident to transmitted radiant power through a material. The luminescence spectra were measured upon excitation by a mercury lamp with a UV filter, the average instrumental resolution was 0.05 nm. The spectra was registered with photomultiplier by using the method of stabilization of the average photocurrent when scanning along the entire line shape.

The optical spectra were recorded at 90 and 300 K. For low-temperature measurements, the crystal was attached to the cold finger of a Dewar flask filled with liquid nitrogen (78 K). In a magneto-optical experiment performed with the MDR-23 monochromator, natural (completely unpolarized) UV radiation was projected onto the sample located in a longitudinal (relative to light propagation) outer magnetic field H. The UV radiation (240-400 nm) was used to excite the luminescence of sample in the visible spectral range, and the MCPL degree spectra were measured at the emission band associated with the 4f→4f transitions. The circularity degree P of partially polarized radiation is determined by the ratio: $P=\frac{I_+-I_-}{I_++I_-}$, where $I_\pm$ are the intensities of two orthogonally polarized circular light components [11]. The values of P were measured by a method of radiation polarization modulation with a photoelastic modulator [12]. The relative error of the measured values of P and luminescence intensity in the experiments $I=\frac{1}{2}(I_++I_-)$ did not exceed ~ 5%.

RESULTS AND DISCUSSION

The optical absorption spectra of the fluoride crystal $Na_{0.375}Tb_{0.625}F_{2.25}$ were studied within the spectral range of 220-500 nm for temperature of T = 90 K and 300 K. The absorption spectra of the crystal under study within the visible and ultraviolet spectral ranges are presented in Fig. 1.

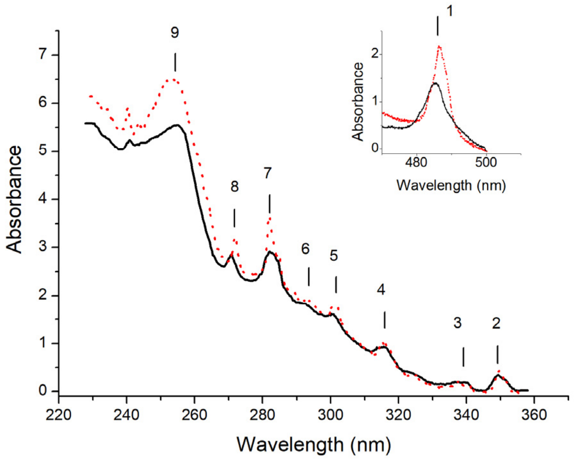

Figure 1. The optical absorption spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal at temperature of 300 K (solid line) and 90 K (dotted line).

As a result of the analysis of the obtained data, the intense absorption bands of $Na_{0.375}Tb_{0.625}F_{2.25}$ were found within the UV spectral range, as well as an absorption band at 485.5 nm (Fig. 1, inset). The presence of this absorption band is apparently associated with intra-configurational 4f→4f transitions between the sublevels of the ground multiplet $^7F_6$ and the excited multiplet $^5D_4$ of Tb^{3+} ions in $Na_{0.375}Tb_{0.625}F_{2.25}$, which is confirmed by the results of optical absorption measurements of this crystal at $T = 90$ K. It is clearly seen from Fig. 1 that when the temperature decreases to 90 K, the amplitude of absorption line 1 increases by almost 1.6 times with its simultaneous narrowing by ~2,8 nm. At the same time, the band itself shifts to the long-wave side by ~1,3 nm. Observed within the UV spectral range above 270 nm, absorption lines 2-8 can be attributed to intra-configurational transitions $^7F_6 \rightarrow {}^5D_4$ of the Tb^{3+} ions in $Na_{0.375}Tb_{0.625}F_{2.25}$. The significant value of the width of line 9 observed within the UV absorption band at a wavelength of 253.6 nm, as well as the sufficiently large value of the absorption coefficient ~32.5 cm^{-1} (at the low concentration of RE ions) measured at the maximum of this band indicate that it can be related to the allowed (by spin and by parity) electric dipole (ED) transitions occurring from the ground multiplet $^7F_6$ to the lower levels of the "mixed" excited $4f^75d$ configuration of the Tb^{3+} ion in the structure of $Na_{0.375}Tb_{0.625}F_{2.25}$.

The luminescence bands of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal caused by intra-configuration transitions $^5D_4 \rightarrow {}^7F_5$ and $^5D_4 \rightarrow {}^7F_4$ at temperature of 300 K are presented in Fig. 2 within the spectral range of wavelengths from 480 to 600 nm. From the analysis of this spectral dependence, it is seen that the most intense luminescence band is located within the wavelength range from 535 to 560 nm. For the "green" luminescence band (Fig. 3) due to intra-configuration transitions $^5D_4 \rightarrow {}^7F_5$, a decrease in the sample temperature from 300 K to 90 K is accompanied by an increase in the intensity of the luminescence line 1 (~540,8 nm) along with a simultaneous narrowing of this line and a decrease in the luminescence line 2 (~544,5 nm). The redistribution of the luminescence line intensity is due to a sharp decrease in the populations of excited Stark singlets of the $^5D_4$ multiplet when temperature decreases. Such temperature behavior of the emission line intensity near ~544 nm clearly indicates that it is associated with a radiative transition from the lowest (in energy) excited state of the $^5D_4$ multiplet.

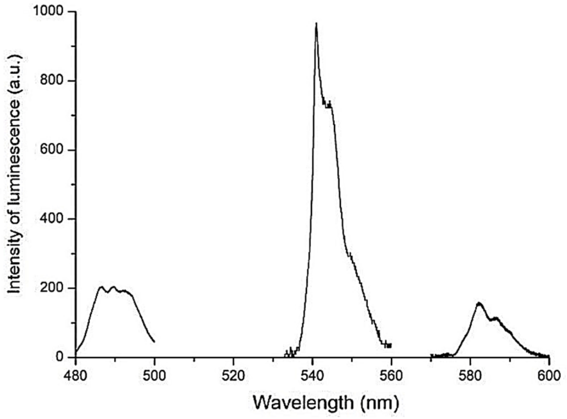

Figure 2. The luminescence spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal, recorded at the temperature of 300 K

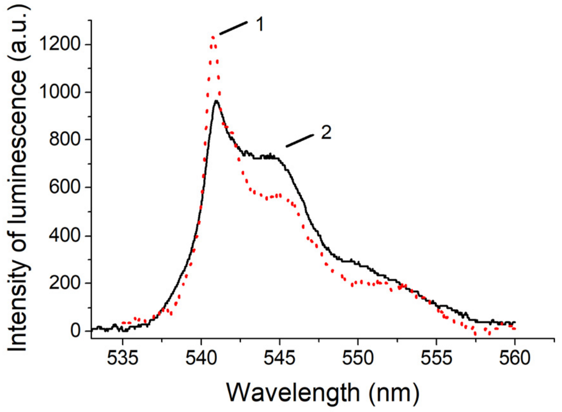

Figure 3. The spectra of the "green" luminescence band of the $Na_{0.375}Tb_{0.625}F_{2.25}$crystal, recorded at the temperature of 90 K (dotted line) and 300 K (solid line)

A comparison of the energies of the experimentally observed emission lines 1,2,3 and 4 with the theoretical scheme of the energy levels of the ground $4f^{(8)}$ configuration of the Tb^{3+} RE ions in the crystal field (CF) of O_h symmetry [13] makes it possible to identify the radiative 4f→4f transitions in the luminescence band $^5D_4 \rightarrow {}^7F_5$ and $^5D_4 \rightarrow {}^7F_4$ (Fig. 4).

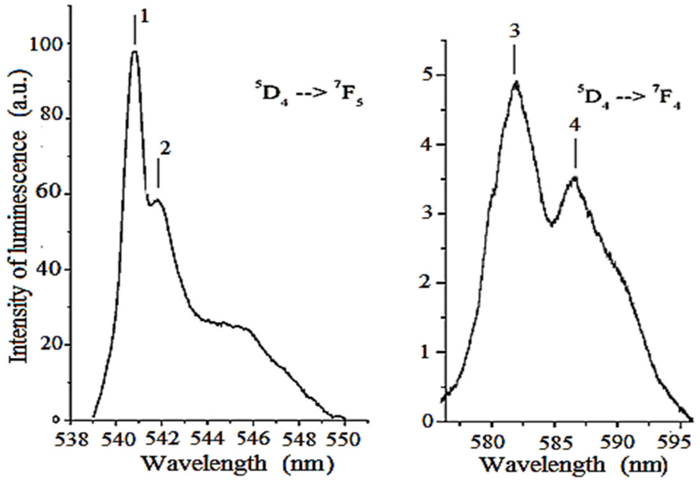

Figure 4. The luminescence spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal, recorded at the temperature of 78 K on transitions $^5D_4 \rightarrow {}^7F_5$ and $^5D_4 \rightarrow {}^7F_4$

From the scheme of radiative transitions shown in Fig. 5 it follows that the lines 1,2 and 4 are associated with magnetic dipole optical 4f→4f transitions occurring from the sublevels A_1g, T_2g (for lines 1 and 2) and Eg (for line 4) of the excited multiplet $^5D_4$ (the designations of the levels are given in accordance with [14]) to the sublevel T_1g (for lines 1 and 2) of the $^7F_5$ multiplet and the sublevel Eg (for line 4) of the $^7F_4$ multiplet. Line 3 corresponds to the electric dipole transition from the sublevel T_2g of the $^5D_4$ multiplet to the sublevel A_1g of the $^7F_4$ multiplet. The spectra of the MCPL degree recorded in an outer magnetic field $H = 10$ kOe at $T = 300$ K for the radiative 4f→4f transitions on the fluorescence band $^5D_4 \rightarrow {}^7F_4$ and $^5D_4 \rightarrow {}^7F_5$ are shown in Fig. 6 and Fig. 7, respectively.

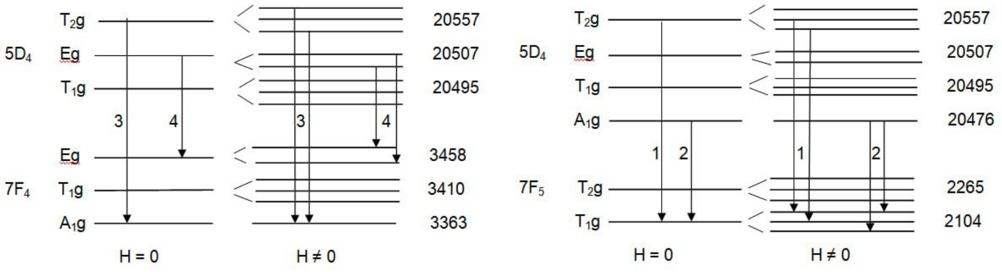

Figure 5. The scheme of the radiative transitions 4f →4f on the fluorescence band $^5D_4 \rightarrow {}^7F_4$ and $^5D_4 \rightarrow {}^7F_5$.

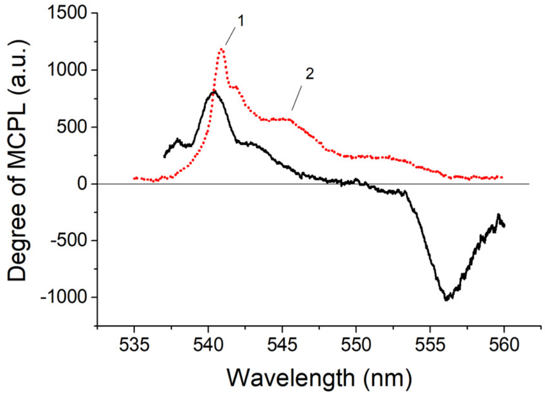

Figure 6. The MCPL degree spectrum (solid line) and the luminescence spectrum (dotted line) of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal for the $^5D_4 \rightarrow {}^7F_5$ transition at $T = 300$ K and $H = 10$ kOe

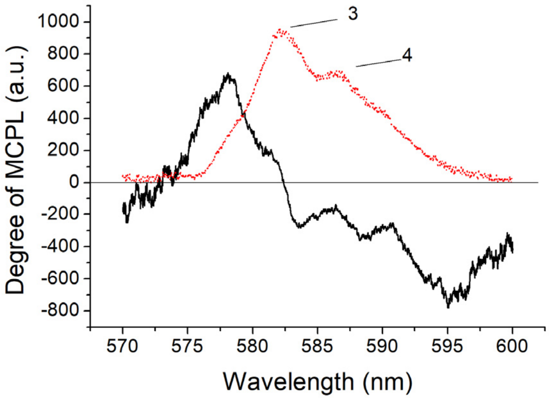

Figure 7. The MCPL degree spectrum (solid line) and the luminescence spectrum (dotted line) of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal for the $^5D_4 \rightarrow {}^7F_4$ transition at $T = 300$ K and $H = 10$ kOe

A characteristic structure of the MCPL degree spectrum for closely spaced secondary emission lines 1 and 2, 3 and 4 can be formed by superimposing the linear dependences $P(n)$ with the corresponding luminescence lines in the areas when they pass through zero both at the maxima of the luminescence lines (A-term) and at the point shifted from the maximum of the emission band [15]. This behavior of the spectral dependences of the MCPL degree is characteristic of non-Kramers (with the even number of electrons of the unfilled 4f shell) and Kramers (with the odd number of electrons) ions in RE crystals and can be described by the following expression [16,17]:

$$P = \frac{I_+ - I_-}{I_+ + I_-} = \frac{1}{2}\mu_B H \left[\frac{(\nu - \nu_0)}{\Gamma^2} \cdot \frac{A}{D_2} + \frac{1}{kT} \cdot \frac{C}{D_1}\right] \tag{1}$$

where ν is the wave number (in cm^{-1}), Γ is the width of the luminescence line, D_1 and D_2 are the "dipole transition forces" expressed through the matrix elements of the corresponding emission transitions [17]:

$$D_1 = \frac{1}{4}[|<a|\hat{D}_x|j>|^2 + |<a|\hat{D}_y|j>|^2 + |<b|\hat{D}_x|j>|^2 + |<b|\hat{D}_y|j>|^2] \tag{2}$$

$$D_2 = \frac{1}{2}[|<a|\hat{D}_x|j>|^2 + |<a|\hat{D}_y|j>|^2 + |<a|\hat{D}_x|k>|^2 + |<a|\hat{D}_y|k>|^2] \tag{3}$$

where $<a|$, $<b|$ and $|j>$, $|k>$ are the wave functions of the Stark sublevels of the initial and final states of the optical transition, respectively. The first term in equation (1) determines the temperature-independent "diamagnetic" contribution to the MCPL degree of the RE ion (the A-term of MCPL [16,17]) due to the Zeeman splitting of the sublevels of the final state of the transition under the action of the outer magnetic field. The second term describes the contribution of the "paramagnetic" C-term of the MCPL degree, which depends on temperature [16,17] and on difference in the sublevel population of the initial state of the transition in the outer field. The inclined linear spectral dependence of the MCPL degree described by equation (1) is shifted from the line center by a value of the "paramagnetic" shift that determines the amplitude of the C-term of the MCPL degree [16,18].

A detailed analysis of the MCPL degree spectrum in Fig. 6 showed that in the magnetic field at the luminescence line 1 there is a magneto-optical transition due to a transition between the sublevels of the T_2g triplet of the $^5D_4$ multiplet to those of the T_1g triplet of the $^7F_5$ (*C*-term) multiplet (Fig. 5). The inclined linear dependence within the luminescence line 2 (Fig. 6) in the magnetic field is formed by means of a transition from the singlet state A_1g of the $^5D_4$ multiplet to the sublevels of the T_1g triplet of the $^7F_5$ multiplet (*A*-term) (Fig. 5). The inclined linear dependences of the MCPL degree on luminescence lines 3 and 4 (Fig. 7) also indicate the manifestation of the *C*-term of magneto-optical activity, which corresponds to transitions from the sublevels of the T_2g triplet and the Eg doublet of the $^5D_4$multiplet (Fig. 5) to the split sublevels of the $^7F_4$ multiplet in the magnetic field. Analysis of the dispersion dependences of the MCPL degree for temperatures of 90 K and 300 K showed a significant increase in the effect with a decrease in temperature (Fig. 8), which indicates a significant contribution of the temperature-dependent paramagnetic *C*-term to this magneto-optical effect.

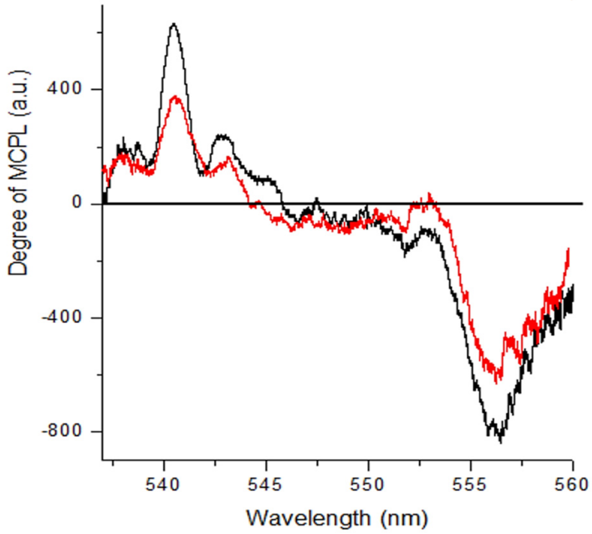

Figure 8. The MCPL degree spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal for the $^5D_4 \rightarrow {}^7F_4$ transition at *T* = 300 K (red line) and *T* = 90 K (black line) and *H* = 7 kOe.

The Faraday effect of the Tb^{3+} ion in the $Na_{0.375}Tb_{0.625}F_{2.25}$ structure was studied within the wavelength range from 400 to 700 nm at a temperature of 95 K. Fig. 9 shows this measured dispersion dependence of the Verdet constant. For comparison, the Verdet constant of the well-known terbium gallium garnet $Tb_3Ga_5O_{12}$ is also presented. A comparative analysis shows that for short wavelengths, the Verdet constant of the $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal is about 85% of that of the $Tb_3Ga_5O_{12}$ crystal, and at long wavelengths, it is more than 90% [10].

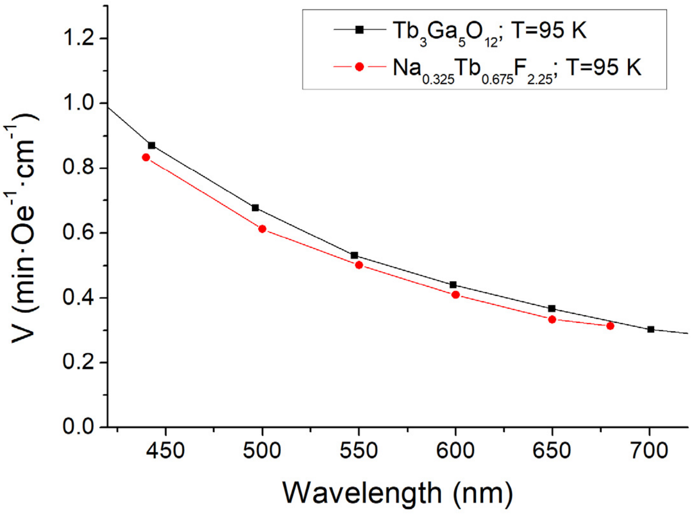

Figure 9. The dispersion dependence of the Faraday effect for the $Na_{0.375}Tb_{0.625}F_{2.25}$ and $Tb_3Ga_5O_{12}$ crystals at temperature 95 K in the magnetic field of 10 kOe.

Thus, it can be concluded that an increase in the Tb^{3+} ions concentration to $1.456 \cdot 10^{22}$ cm^{-3} in $Na_{0.375}Tb_{0.625}F_{2.25}$ crystal affects to the growth of the magneto-optical Faraday effect in a new synthesized crystal with a fluorite structure, which expands the possibilities of using this crystal under study as an analogue of terbium gallium garnet.

CONCLUSIONS

Based on the analysis of the optical spectra of the $Na_{0.375}Tb_{0.625}F_{2.25}$ single crystal it has been established that within the wavelength range of 535-560 nm there is a more intensive luminescence band caused by intra-configuration 4f→4f transitions $^5D_4 \rightarrow {}^7F_5$. Analysis of the absorption and luminescence spectra allowed us to identify the radiative 4f→4f transitions in the luminescence bands $^5D_4 \rightarrow {}^7F_5$ and $^5D_4 \rightarrow {}^7F_4$ and to establish a dominance of the magnetic dipole transitions in the secondary emission spectra. The experimentally found values of the energy levels is in agreement with the theoretical calculations of the energy levels found for the rare earth Tb^{3+} ion in a crystal environment of O_h symmetry.

Analysis of the magneto-optical research data shows that the *C*-term of the magneto-optical activity plays a significant role in a mechanism of occurrence of magneto-optical effects on luminescence bands caused by “forbidden” 4f→4f transitions.

The results of the Faraday effect study showed that the Verdet constant of the $Na_{0.375}Tb_{0.625}F_{2.25}$ single crystal is about 0.9 of the Verdet constant of $Tb_3Ga_5O_{12}$ crystal, which demonstrates the good magneto-optical properties of the new synthesized crystal and expands the range of fluoride cubic crystals with good magneto-optical features.

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ОПТИЧНІ ТА МАГНІТООПТИЧНІ ВЛАСТИВОСТІ КРИСТАЛА $Na_{0.375}Tb_{0.625}F_{2.25}$

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Оптичні та магнітооптичні спектри кристала $Na_{0,375}Tb_{0,625}F_{2,25}$ були досліджені в ультрафіолетовому та видимому спектральному діапазонах. Встановлено, що в діапазоні довжин хвиль 535-560 нм спостерігається інтенсивна смуга люмінесценції, спричинена внутрішніми конфігураційними переходами 4f→4f $^5D_4 \rightarrow {}^7F_5$. У смузі люмінесценції $^5D_4 \rightarrow {}^7F_5$ та $^5D_4 \rightarrow {}^7F_4$ ідентифіковані радіаційні переходи 4f→4f та встановлено, що у спектрах вторинного випромінювання домінують магнітні дипольні переходи. Аналіз даних магнітооптичних досліджень показав, що C-член магнітооптичної активності відіграє значну роль у механізмі виникнення магнітооптичних ефектів на смугах люмінесценції, спричинених «забороненими» переходами 4f→4f. Порівняльний аналіз дисперсійної залежності константи Верде монокристала $Na_{0,375}Tb_{0,625}F_{2,25}$ показав, що вона становить близько 90% від константи Верде добре відомого кристала $Tb_3Ga_5O_{12}$, що демонструє хороші магнітооптичні властивості нового синтезованого кристала.

Ключові слова: *кристали фториду; іони рідкісноземельних елементів; магнітооптичні властивості; люмінесценція; енергетичні рівні; магнітна кругова поляризація; стала Верде*