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Features of absorption of thin films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ obtained using the method of pulsed laser deposition

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The electrical, optical, and magneto-optical properties of $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ thin films with a Curie temperature near room temperature are studied. It is shown that in the region of the phase transition there is a sharp change in the behavior of the temperature dependences of the electrical resistance and optical transparency of the films, which is associated with the insulator-metal transition. In a narrow temperature region near the transition, a change in the external magnetic field leads to the appearance of negative magnetoresistance and magnetotransmission of unpolarized light in the spectral range from 1 to 12 μm. It is shown that the effect of magnetic transmission (magnetoabsorption) in films is mainly due to the contribution of free charge carriers. The magnetic transmission spectra are sensitive to the magnetic and electronic inhomogeneities of the films.

Keywords: Thin films, manganites, magnetoabsorption, colossal magnetoresistance, IR spectroscopy, metal-insulator transition, magnetic inhomogeneities.

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Introduction

Studies of magneto-optical (MO) effects in strongly correlated compounds based on doped lanthanum manganites are usually limited by the Faraday and Kerr effects, magnetic circular or linear dichroism in the fundamental absorption region, and the temperature range near the ferromagnet/paramagnet magnetic phase transition, which, as a rule, is below room temperature [1–3]. An external magnetic field affects the processes of interaction of light with impurity states, localized and delocalized charge carriers in the infrared (IR) range, which makes the main contribution to the magnetotransport and MO properties of doped manganites. In the IR range of the spectrum in manganites in a narrow region near the Curie temperature (T_C) giant MO effects (magnetic transmission and magnetic reflection of light) are observed in unpolarized light [4].

The magnetic transmission and magnetoreflection of light in manganites exceeds by several times the intensity of traditional MO effects linear in magnetization, and therefore they are promising for practical applications, for example, for controlling the intensity (modulation) of a light flux in the IR region or non-contact detection of magnetic fields and temperature changes. From this point of view, manganites with high T_C are of the greatest interest. For example, for $La_{2/3}Ba_{1/3}MnO_3$ monocrystals it is ~ 340 K [5]. Despite the large number of studies on the optical properties of doped lanthanum manganites (for example, [6–9] and references in them), the studies on the magnetic transmission and magnetic reflection of unpolarized light in $La_{2/3}Ba_{1/3}MnO_3$ are single.

In this study the experimental data on the magnetoresistance and magnetotransmission of light in the IR range of the spectrum in $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ films of various thicknesses are obtained. The influence of the features of the film state and heat treatment on the MO effects behavior in films is studied. With the help of the magnetorefractive effect theory, it was only possible to qualitatively describe the behavior of the magnetic transmission of light in $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ films, that indicates the need to improve the theory for doped manganites. High sensitivity to an external magnetic field and large magnitudes of MO effects in $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ films can be used to create non-contact electromagnetic field sensors and IR range modulators operating at room temperature.

Samples and experimental procedure

Thin films of composition $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ with thickness $d = 80$ nm (hereinafter — film *1* /) and 110 nm (hereinafter film *2*) were obtained by irradiating a ceramic target of stoichiometric composition $La_{0.7}Ba_{0.3}MnO_3$ ($a_0 = 3.909$ Å) with a pulsed KrF laser ($\lambda = 248$ nm) using the pulsed laser deposition (PLD) technique described in [10]. The films were deposited on single-crystal $SrTiO_3$ (001) ($a_0 = 3.905$ Å) substrates at a temperature of 730°C and an oxygen pressure in the chamber of $P = 0.4$ mBar. After the deposition, the films were annealed in the growth chamber for 30 min at an oxygen pressure of 500 mBar and a temperature of 630°C. The film thickness was determined from the deposition time of the reference sample and controlled with a ZYGO optical

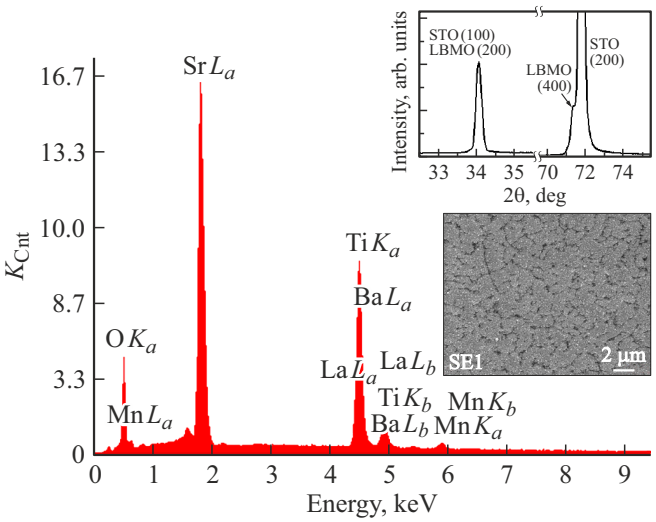

Figure 1. Energy-dispersive spectrum of a $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ ($d = 110$ nm) film on a $SrTiO_3$ substrate. In the inserts: at the top — X-ray diffraction data; at the bottom — AFM data of the film surface (the size of the scan area is $20 \times 20\,\mu$m).

profilometer. The composition of the target and the obtained films was controlled by X-ray spectral microanalysis on a JEOL JXA-733 electron microscope with an absolute error in determining the concentration of chemical elements ± 0.01. This method did not allow to estimate the oxygen content; for this reason, the value δ is not given. The film morphology was analyzed using an atomic force microscopy (AFM) probe microscope „Solver Next“ (NT-MDT). The insert in the Fig. 1 shows an image of the relief of the film surface without clearly expressed features. Such a relief is observed, as a rule, under growth regimes similar to layer-by-layer one. This is typical of the PLD technique and the used high temperatures of the substrate. Note, that the average degree of film roughness was ~ 25ṅm, which is somewhat higher than the usual values for the films obtained by magnetron sputtering [11]. Nevertheless, according to the data of X-ray diffraction studies performed using a DRON-4M diffractometer, the single-phase nature of the films is confirmed by the presence of clearly pronounced and closely spaced reflections from the film and substrate and a small number of weak additional lines. X-ray diffraction studies were performed both immediately after film growth and after additional heat treatment of the samples at $T = 700^\circ$C in an oxygen flow in a muffle furnace for 6 h. After heat treatment, only the lines belonging to the pseudocubic structure of manganite remained on the X-ray patterns (Fig. 1). According to X-ray diffraction data, the crystal lattice constant of the films in the cubic approximation was $a_0 = 3.97$ Å, which is close to the values $a_0 = 3.91$ Å for the target and $a_0 = 3.92$ Å for $La_{0.7}Ba_{0.3}MnO_3$ single-crystal films obtained by magnetron sputtering [11]. The observed difference a_0 can be explained by different degrees of roughness, mechanical stresses, and possible slight structural distortions of the lattice in the films under study [11,12]. A slight change in thickness does not affect the structural parameters of the resulting films. The temperature T_C of the samples, determined from the data of the equatorial Kerr effect, agrees with the data of measurements of the magnetization [8,13] and amounted to 301 and 310 K for the films *1* and *2* respectively.

The optical and MO properties of the films were studied in the IR wavelength range $0.8 \le \lambda \le 30\,\mu$m and the temperature range $200 \le T \le 360$ K using an original cryomanetooptical installation based on an IKS-21M prism spectrometer at normal passage through the sample of unpolarized (natural) light from an emitter (a globar with an operating temperature of $T = 1350^\circ$C) [14]. In the course of the measurements the sample was fixed on a copper holder of a flooded nitrogen cryostat equipped with optical windows of KRS-5. In the installation simultaneously with the optical parameters, the electro- (ρ) and magnetoresistance ($Delta\rho/\rho_0 = (\rho_H - \rho_0)/\rho_0$) of the films at direct current (where ρ_H and ρ_0 — electrical resistance of the sample in magnetic field and without field, respectively) were measured. Contacts on the samples were obtained using ultrasonic soldering with pure indium solder. Field and spectral dependences of the magnetic transmission ($\Delta t/t_0 = (t_H - t_0)/t_0$, where t_H and t_0 — the intensity of light transmitted through the sample in a magnetic field and without a field, respectively) and magnetoresistances were measured in magnetic fields $H \le 8$ kOe directed along the propagation axis of the light beam and perpendicular to the film plane. The relative error in determining $\Delta t/t_0$ was $\sim 0.5\%$. The possible influence of film anisotropy and related changes in MO properties, as well as various polarization effects, was not considered in the study due to their smallness under the given experimental conditions.

Experimental results and discussion

The optical properties of the films of doped manganites with the effect of colossal magnetoresistance (CMR) were considered, for example, in the studies [2,5,15–20]. The absorption spectra (K) of the obtained films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ at $\lambda < 2\,\mu$m are formed by the absorption edge, associated with fundamental transitions $^5E_g - ^5T_{2g}$ and transitions within the electronic Jahn-Teller cluster $[MnO_6]^{10-}_{JT}$. At $\lambda \ge 5\,\mu$m the contribution of delocalized charge carriers appears, and at $\lambda \ge 8\,\mu$m — transitions inside the hole cluster $[MnO_6]^{8-}_{JT}$ and phonon absorption of the substrate and film (Fig. 2, *a*). As the temperature decreases below T_C, the absorption sharply increases in the entire spectral range under consideration, mainly due to the strong contribution of delocalized charge carriers, which masks other features. A similar behavior of light absorption in doped manganites was observed in the studies [8,9,21–24]. The interaction of light with free charge carriers is most clearly manifested in a sharp change in the intensity of light transmitted through the film at a temperature

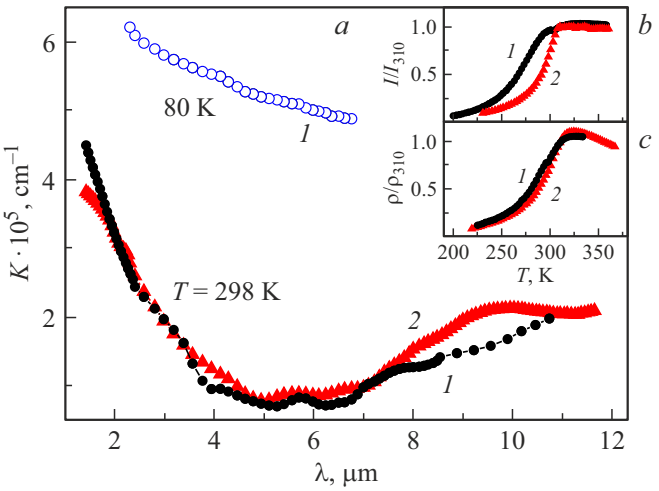

Figure 2. (a) Absorption spectra of K films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ (1 — 80 nm, 2 — 110 nm) at various temperatures; (b) temperature dependences of relative change in transparency I/I_{310} and (c) relative change in electrical resistance of films rho/ρ_{310}.

below T_C (Fig. 2, b), which is an optical analogue of the observed metal-insulator transition in the electrical resistance (Fig. 2, c) [4].

In a narrow temperature range near T_C, in doped manganites, the influence of an external magnetic field manifests itself to the maximum, which leads to the appearance of the effect of negative magnetoresistance $\Delta\rho/\rho_0$ and its optical (high-frequency) component in the form of effect of giant negative magnetic transmission of light $\Delta t/t_0$, which is caused by the suppression by magnetic field of magnetic moment fluctuations near the magnetic phase transition (Fig. 3). Note, that the temperatures of the maxima of the effects $\Delta t/t_0$ and $\Delta\rho/\rho_0$ are close to T_C of the films obtained from the Kerr effect and magnetization data. When the film temperature deviates from T_C, the effects of magnetotransmission and magnetoresistance quickly drop to zero. Such behavior $\Delta t/t_0(T)$ and $\Delta\rho/\rho_0(T)$ is typical of manganites with CMR, as was observed in [2,4,6,9,25–27]. However, in our case, in $\Delta t/t_0(T)$ and $\Delta\rho/\rho_0(T)$of the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ a prolonged low-temperature drop was observed (Fig. 3), which can be associated with the magnetic inhomogeneity of our samples, as was found for other manganite systems, for example, in [6,21].

It is known from the studies [28–32], that post-growth heat treatment of films in oxygen stimulates the improvement of the crystal structure, magnetic and electrical characteristics of manganites, bringing them closer to the parameters corresponding to those for monocrystals. A change in oxygen stoichiometry affects the ratio of concentration of Mn^{3+}/Mn^{4+} ions responsible for the appearance of ferromagnetic exchange interaction in manganites and, accordingly, also changes the magnetization of T_C films. In our case it was found, that additional heat treatment of the samples at temperatures below the synthesis temperature has little effect on the oxygen nonstoichiometry, but reduces the overall defectiveness (increases the optical transparency) of the films. This is also confirmed by the temperature dependences of the relative change in transmission $I/I_{310}(T)$ (Fig. 2, b) and $\Delta t/t_0(T)$ (Fig. 3, a) measured outside the region of impurity bands. As is known, the magnetic transmission is very sensitive to the changes in the volume of the ferromagnetic phase in manganites (review [4] and references therein). However, for the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$, additional heat treatment had practically no effect on the value and position of the maximum $\Delta t/t_0$, i.e. e., the stoichiometry and volume of the ferromagnetic phase in the films are stable. Note, that this behavior of $\Delta t/t_0$ is in good agreement with the well-known statement about the optimal level of substitution ($x \sim 1/3$) of trivalent ions La by divalent $3d$-metal ions in manganites according to the phase diagram [5]. In our case the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ have the maximum volume of the ferromagnetic phase, and the maximum in the dependence $\Delta t/t_0(T)$ well agrees with T_C for monocrystals of the corresponding composition.

As was established earlier in [7,8,21,33], the optical data (compared to transport and X-ray diffraction data) are more sensitive to nanosized electronic and magnetic inhomogeneities of various nature and can be used to certify magnetic films. In comparison with the absorption spectra, the magnetic transmission spectra of IR radiation in the obtained films had a more complex form (Fig. 4), mainly due to the competition of the positive contribution from interband transitions in the visible and near IR ranges and

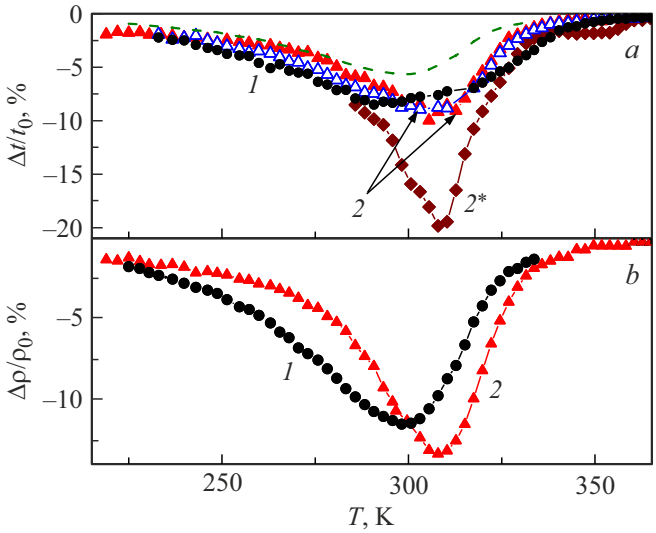

Figure 3. (a) Temperature dependences of magnetic transmission $\Delta t/t_0$ of films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ (1 — 80 nm, 2 — 110 nm) at a wavelength $\lambda = 6\,\mu$m ($\lambda = 2\,\mu$m for a film 110 nm — 2^*) in a magnetic field $H = 7.5$ kOe. Open symbols — magnetic transmission $\Delta t/t_0$ of the film 2 prior heat treatment. Dashed line — the result of calculation of $\Delta t/t_0$ by expression (2) for the film 1. (b) Temperature dependences of the magnetoresistance $\Delta\rho/\rho_0$ of 1 and 2 films in the field $H = 7.5$ kOe.

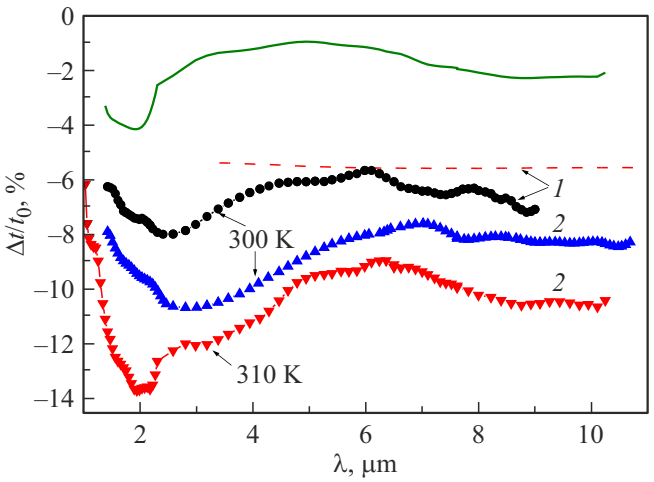

Figure 4. Spectra of magnetic transmission $\Delta t/t_0$ of the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ (*1* — 80 nm and *2* — 110 nm) at various temperatures in the field $H = 7.5$ kOe. Dashed line — calculation for film *1* by expression (2). Solid line — spectrum obtained after subtracting the magnetic transmission spectrum $\Delta t/t_0$ (for $T = 310$ K) from the spectrum $\Delta t/t_0$ (for $T = 300$ K) for film *2*.

the negative contribution to the IR region of the spectrum associated with the interaction of light with delocalized carriers.

In addition, in spectra $\Delta t/t_0$ the features in the form of peaks near $\lambda = 3\,\mu$m (~ 0.4 eV) and $9\,\mu$m (~ 0.14 eV) appeared, corresponding to the position of the impurity bands in the absorption spectrum and correspondingly associated with the transitions in electronic $[MnO_6]^{10-}_{JT}$ and hole $[MnO_6]^{8-}_{JT}$ clusters in manganites. Similar features in the magnetic transmission of manganites were also observed in the studies [2,34] and in nanostructured films of similar composition $La_{0.67}Ba_{0.33}MnO_3$ [35,36].

Thus, these bands in the $\Delta t/t_0$ spectra are associated with charge and magnetic inhomogeneity centers and phase separation of the „ferromagnetic metal−paramagnetic insulator“ type in films. Note, that the intensity and position of these bands in the spectra do not depend on temperature and doping level, but may vary depending on the content of vacancies in the oxygen sublattice and the degree of defectiveness of the films [34]. As can be seen from Fig. 4, in the spectrum $\Delta t/t_0$ of the 110 nm film at $T = T_C$, in addition to the band in the $3\,\mu$m region, more intense band with a maximum at $\lambda = 2\,\mu$m was observed. Below T_C the spectrum was substantially rebuilt: the peak at $2\,\mu$m disappeared, but the band at $3\,\mu$m remained. In addition, there was a slight shift of the entire spectrum towards longer wavelengths. In order to identify the contribution of charge carriers and impurity bands for the film *2*, a difference spectrum of the magnetic transmission effects measured at $T = 300$ K and $T = T_C = 310$ K. was constructed. The resulting spectrum clearly demonstrates the presence of maxima at wavelengths 2 and $9\,\mu$m (Fig. 4). The band at $2\,\mu$m was observed by us earlier in the spectra $\Delta t/t_0$ of other manganite films, but its nature was not discussed in detail. It can be assumed, that in this case there is a superposition of two bands: a band associated with transitions in electron clusters, and a band associated with the excitation of surface or bulk plasmons, as in [37], or various geometric resonances (resonances Mi) at the boundary of inhomogeneities in the phase separation region, as in [38]. It is important to note, that the temperature dependences $\Delta t/t_0$ at wavelengths 2 and $6\,\mu$m coincide (Fig. 3, *a*). This indicates, that the band at $2\,\mu$m is also associated with the formation of a ferromagnetic phase in the film.

The calculation of light magnetoabsorption spectra $(\Delta K/K_0 = (K_H - K_0)/K_0)$ in films taking into account the data on light reflection and magnetoreflection taken from the study [13], found a broad maximum in the region $4-8\,\mu$m between the impurity bands and only a small peak in the region $2\,\mu$m (Fig. 5). Thus, we can conclude, that the main role in the absorption of IR radiation in a magnetic field is played by the contribution associated with the appearance of free charge carriers in doped manganites near and below T_C.

Let us consider the influence of the film thickness on the effect of magnetic transmission. According to the phase diagram [5], in the studied films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ the volume of the ferromagnetic phase is almost maximum. Therefore, different values of $\Delta t/t_0$ for films (Figs 3 and 4) can be related both to the thickness and to the magnetic reflection $\Delta R/R_0$ of the film according to the expression

$$\frac{\Delta t}{t} = A\exp[(K_0 - K_H)d] - 1. \qquad (1)$$

where $A = [(1-R_H)/(1-R_0)]^2$, K_0 and K_H, R_0 and R_H — light absorption and reflection coefficients in the absence and presence of a magnetic field, respectively.

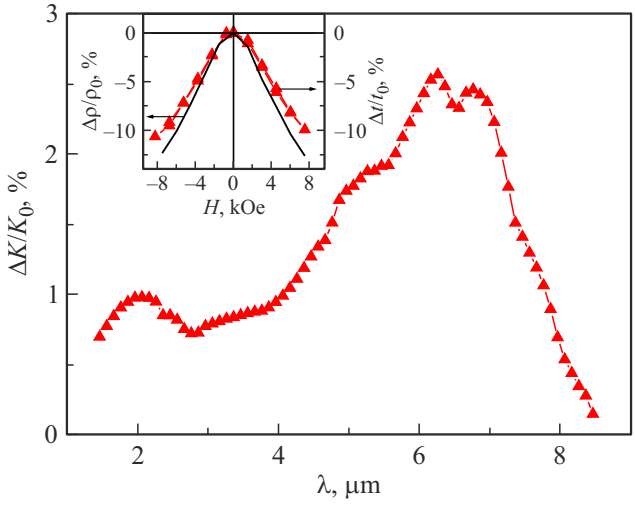

Figure 5. Magnetic absorption spectrum $\Delta K/K_0$ of the 110 nm film $La_{0.69}Ba_{0.33}MnO_{3-\delta}$ at $T = 310$ K. In the insert: field dependences of magnetoresistance $\Delta\rho/\rho_0$ and magnetic transmission $\Delta t/t_0$ of the film at wavelength $\lambda = 6\,\mu$m at $T = 310$ K.

According to the theory of the magnetorefractive effect in optimally doped manganites [4], the magnetic transmission is directly proportional to the magnetoresistance:

$$\frac{\Delta t}{t} = 0.5\,\frac{\Delta\rho}{\rho}\,t_0 k^2\,\frac{2n^2+n}{n^2+k^2}, \qquad (2)$$

where n, k — refraction indices.

Estimates of the value $\Delta t/t_0$ for the films using formula (2) give approximately 2–4 times lower values (dashed line in Fig. 3, *a*) compared with the experimental data. Note, that the calculation of the magnetic transmission spectra of the films using formula (2) at a fixed temperature in the approximation of linear dispersion n and k also had only qualitative agreement with the experimental curve (for example, a film *1* with a thickness of 80 nm in Fig. 4). It is logical to assume, that the difference between the theoretical and experimental curves is due both to the allowed approximations in the dispersion n and k, and the unaccounted for contribution associated with the reflection of light at the film-substrate interface for thin films with a thickness close to the depth of the skin layer [6]. The contribution of the light reflected from the substrate can increase the effective film thickness and enhance the observed effect $\Delta t/t_0$ according to the expression (1). Besides, the theory of the magnetorefractive effect does not take into account the contribution of impurity transitions and various resonance states in manganites, which, as can be seen from Figs 4 and 5, also determine the behavior and spectral dependence of $\Delta t/t_0$ in the IR region.

In contrast to the magnetization and the Kerr effect, the field dependences $\Delta\rho/\rho_0(H)$ and $\Delta t/t_0(H)$ of $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ in the region T_C had a linear behavior without saturation in magnetic fields up to 8 kOe (insert in Fig. 5).

The sign of the magnetic transmission effect in the films did not depend on the direction of the external magnetic field, i.e., the effect is even. This indicates, that the effect $\Delta t/t_0$ in unpolarized light is mainly associated with a change in the diagonal components of the complex dielectric permittivity under the action of a magnetic field, as well as the absence of a noticeable contribution of linear MO phenomena under experimental conditions, in contrast to what was observed, for example, in spinels in [39]. A change in the geometry of the magnetic field application (the field in the film plane and perpendicular to the path of the light beam) had almost no effect on the general form of the field and spectral dependences of the magnetic transmission (data not shown). This behavior, we believe, is due to the finding the maximum effect near T_C of the film. In this temperature range the fluctuations of the magnetic moments are maximum, and the system becomes insensitive to the external magnetic field orientation. It is only possible to change the absolute value of the effects of magnetic transmission and magnetoresistance in the films as a result of the influence of the demagnetizing factor.

Conclusion

Using the pulsed laser deposition method the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ with the thickness of 80 and 110 nm were obtained on the $SrTiO_3$ substrates. It is shown, that near the room temperature, in addition to the colossal magnetoresistance the films exhibit a giant effect of negative magnetic transmission (magnet absorption) of unpolarized light, which reaches 20% in a field of 7.5 kOe in a wide spectral region from 1 to 12 μ m. The complex shape of the absorption and magnetic transmission spectra with singularities near the impurity bands reflects the magnetic and electronic inhomogeneity of the obtained films. It is shown, that the main role in the magnetic absorption of light in films in the spectrum IR region is played by the influence of an external magnetic field on the fluctuations of magnetic moments near the temperature T_C and the ratio of localized and delocalized charge carriers. Additional heat treatment of the films, due to recrystallization processes, reduces the overall defectiveness and increases their optical transparency, but does not affect the magnetic transmission due to the fixed volume of the ferromagnetic conducting phase in optimally doped films. To describe the magnetic transmission in thin films of manganites in the theory of the magnetorefractive effect, it is necessary to take into account additional mechanisms of light absorption in manganites, as well as the contribution of light reflected from the substrate. The high sensitivity of electrical resistance and light transmission in the films $La_{0.69}Ba_{0.31}MnO_{3-\delta}$ to an external magnetic field can be used to create non-contact electromagnetic field sensors and MO light modulators, working at room temperature.

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Conflict of interest

The authors declare that they have no conflict of interest.

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