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Dispersion properties of thin films of $(La_{0.06}Ga_{0.94})_2O_3$:Eu

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Refractive index dispersion of $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films prepared by radio-frequency ion-plasma sputtering was investigated. It was found that the films have a polycrystalline structure corresponding to the monoclinic structure of β- Ga_2O_3. It is shown that the freshly deposited films are characterised by an abnormal dispersion, and after annealing in argon, a normal dispersion of the refractive index is observed. It was found that at normal dispersion, the spectral dependence of the index of refraction in the visible spectrum is mainly a function of electronic transitions from the 2p-state oxygen band, forming the upper filled level of the valence band to the lower level of conduction band, which is formed by hybridized 2p states of oxygen and 4s states of gallium. Two single-oscillator approximation models were analysed and compared for the films annealed in argon, and the approximating parameters, scattering energy, ionicity of the chemical bond, coordination number, and static refractive index were determined.

Keywords: thin film, lanthanum and gallium oxide, refractive index, dispersion.

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Introduction

Due to the possibilities of practical use in modern optoelectronics and instrumentation, researchers are currently showing great interest in metal oxide materials. The large band gap, high dielectric constant, and transparency for eigenluminescence make them popular in the development of full-colour screens, reflective coatings, gas sensors, and ultraviolet detectors [1-3]. Among these compounds, a number of interesting properties have been discovered in recent studies in β-Ga_2O_3 films obtained by various methods [4-7]. Pure or doped β-Ga_2O_3 films used as transparent conducting electrodes [8], phosphors that exhibit photoluminescence [9-11], cathodo-luminescence, or electroluminescence [12-14], depending on the method of preparation and the type of dopant. The optical and electrical properties of β-Ga_2O_3 films are determined by method of preparation, the type of deposition and the subsequent technological techniques, as well as by the introduction of impurities that can purposefully change the properties of oxide thin films. To this end, we have studied $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films where some Ga^{3+} ions were replaced by La^{3+} ions, which did not require local compensation of the electric charge. This is due to the fact that luminescent processes in β-Ga_2O_3 films are largely determined by local energy levels within the bandgap [15-17]. In addition, the introduction of the Eu^{3+} activator makes it possible to realise the red luminescence colour of these films, which can be used as the red component of a full-colour luminescent screen [18-20].

The study of the optical properties of such films, in particular the dispersion properties and their connection with the energy structure and crystal-chemical properties, seems to be quite relevant: when creating optical light filters, enlightening optical parts or creating a luminescent screen, the refractive index determines the reflective properties of the films, and the dispersion properties determine their spectral distribution. This is what led to the conduct of these studies.

I. Methodology of the experiment

$(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films with a 0.3-1.0 μm thickness were obtained by RF ion-plasma sputtering on

fused quartz υ-SiO_2 substrates. The RF sputtering was carried out in a system that uses the magnetic field of external magnets for compression and additional ionisation of the plasma column. The concentration of the activator Eu^{3+} was 1 wt. %. After the films were deposited, they were heat treated in an argon atmosphere at 1000-1100°C.

The polycrystalline structure of the obtained films has been demonstrated by X-ray diffraction studies, and the diffractograms themselves are close to the diffractograms of thin β-Ga_2O_3 films described by us in [21]. The results obtained indicate that after argon heat treatment, the structure of thin $(La_{0.06}Ga_{0.94})_2O_3$:Eu films matches to the monoclinic crystal structure of β-Ga_2O_3 with a dominant orientation in the (002) and (111) planes.

Using an OXFORD INCA Energy 350 energy dispersive spectrometer, elemental analysis of the samples was carried out at a number of points on the surface of the films. The calculations confirmed that the percentage of components and activator in the obtained films corresponded to their percentage in the compound $(La_{0.06}Ga_{0.94})_2O_3$:Eu.

To determine the refractive index *n*, absorption coefficient α and film thickness *h* in semiconductor and dielectric films, spectrophotometric techniques are the most common. Most of them are based on the analysis of transmission spectra. Although all of these techniques are based on common theoretical assumptions, their application to real experimental data gives slightly different results for the same sample. Based on a comparative analysis of a number of such methods conducted in [22], it was found that among the spectrophotometric techniques considered, the Valeev method [23] is optimal, which was used in our study to determine the optical constants of thin films of $(La_{0.06}Ga_{0.94})_2O_3$:Eu from the interference pattern in the transmission spectrum. The transmission spectra were measured on a CM 2203 spectrofluorimeter with a Hamamatsu R928 measuring head.

II. Results and discussion

The characteristic transmission spectra $T(\lambda)$ for unannealed (I) and annealed in an argon atmosphere (II) of $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films are shown in Fig. 1. As shown in Figure 1, the interference pattern is clearly visible in the transmission spectra, which makes it possible to calculate the optical constants by the interference technique.

The dispersion dependences $n(\lambda)$ for the obtained films are shown in Fig. 2. When using the methodology of [23], the error in determining *n* due to simplifications, according to [22], does not exceed 0.1%.

It is stated in [22] that this method should be used mainly in spectral regions with a weak change in the average transmittance, which is observed in the measured transmission spectra of thin films of $(La_{0.06}Ga_{0.94})_2O_3$:Eu. In areas with a sharp change in the average transmittance *T*, the error of the method [23] increases to 3%. It should be noted that in the transparency region, films II are characterised by a higher refractive index *n* than films I and are characterised by a significant normal dispersion, especially in the UV region near the absorption edge. Films I are characterised by anomalous dispersion (Fig. 2).

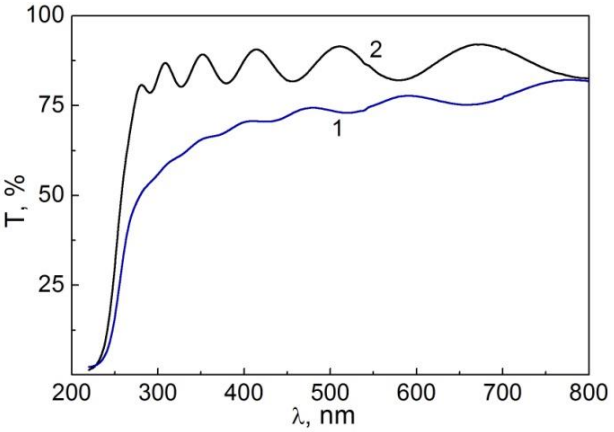

Fig. 1. Transmittance spectra of $(La_{0.06}Ga_{0.94})_2O_3$:Eu I (1) and II (2) thin films on fused quartz substrates, T = 295 K.

To describe the normal dispersion of the refractive index of II films in the studied spectral range, we used a single-oscillator three-parameter model [24], which is to some extent a slightly modified Zelmeyer approximation:

$$n^2 - A = \frac{E_0 E_d}{E_0^2 - E^2}. \qquad (1)$$

Where *A* is the approximation coefficient; E_0 is the energy of the maximum of the absorption band, which determines the spectral course of the refractive index; E_d is the dispersion energy (oscillator strength). Dispersion energy is a measure of the average strength of the optical transitions between the bands and is determined by [24]:

$$E_d = \beta N_C Z_a n_e, \qquad (2)$$

where N_C is the coordination number; Z_a is the valence of the anion; n_e is the number of valence electrons per formula unit. The parameter β depends on the degree of ionicity of the chemical bond: for ionic compounds β = 0.26 and for covalent compounds β = 0.37.

The approximation parameters in equation (1) could be determined by regression analysis for the studied films and are given in Table 1. For comparison, Table 1 also shows similar approximation parameters obtained earlier for β- Ga_2O_3 [25] and $(Y_{0.06}Ga_{0.94})_2O_3$ [26] films annealed in argon.The difference of the coefficient A from unity indicates the presence of other absorption bands besides the band with the maximum E_0, which affect the course of the dispersion curve. These bands can be in both the UV and IR regions of the spectrum. Comparison of the coefficient *A* for thin films $(La_{0.06}Ga_{0.94})_2O_3$:Eu and β-$Ga_2O_3$ or $(Y_{0.06}Ga_{0.94})_2O_3$ shows that among the analysed films, the dispersion curve in $(La_{0.06}Ga_{0.94})_2O_3$:Eu films is least determined by the absorption band with a maximum of E_0 and its course is more significantly influenced by other absorption bands. This situation looks quite natural, since such films correspond to the most disordered compound.

If we analyse the calculation of the electronic structure of the initial compound β-Ga_2O_3 [27, 28], the top

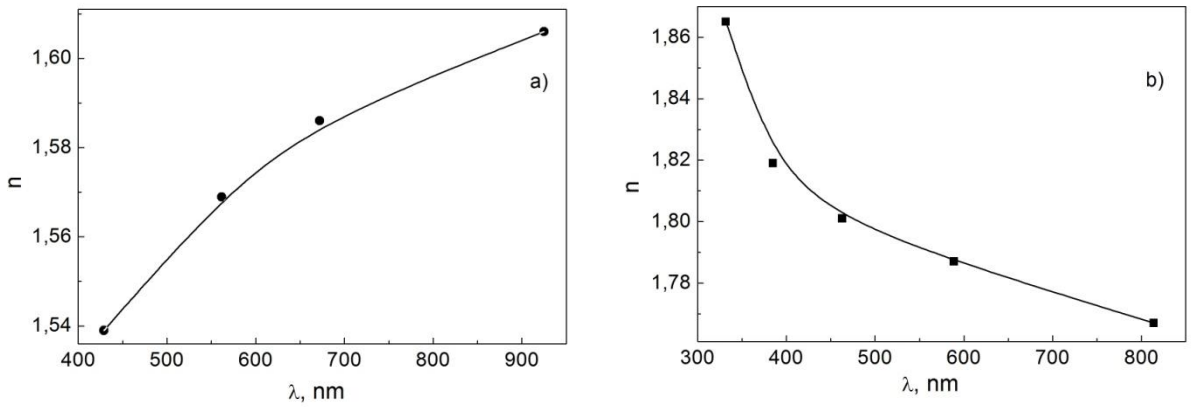

Fig. 2. Dispersion of the refractive index of thin $(La_{0.06}Ga_{0.94})_2O_3$:Eu films I (a) and II (b): points - calculation by the method [23], line - approximation by formula (1) (b).

Table 1.

Crystallochemical and energy parameters of the dispersion curve of thin films annealed in argon in the relations (1) and (4)

Thin film	A	E_0, eV	E_d, eV	f_i	N_C	n_0	E_{01}, eV
$(La_{0.06}Ga_{0.94})_2O_3$:Eu	1.98	7.18	7.78	0.96	2.40	1.665	5.91
β-Ga_2O_3 [25]	1.48	6.98	10.01	0.83	3.21	1.718	7.28
$(Y_{0.06}Ga_{0.94})_2O_3$ [26]	1.63	6.28	8.47	0.86	2.82	1.727	6.17

of the valence band in this compound is formed by 2p-orbitals of oxygen. The bottom of the conduction band is formed by hybridised 2p states of oxygen and 4s4p states of gallium. The main contribution to the bottom of the conduction band is made by 4s states of Ga (≈60%) [27-29]. In [30], a strong hybridisation of 2p (O) and 4s (Ga) states was found. According to the calculations of the electronic energy structure of β- Ga_2O_3 [29], the electronic transitions from the upper valence band to the lower conduction band energy is between 5 and 8 eV. At the same time, the maximum electron density falls at ~ 6.5 eV. The values obtained for the $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films fall within the region of the indicated densities of interband transitions. At the same time, the obtained value of E_0 is slightly higher than for the compared of films β-Ga_2O_3 and $(Y_{0.06}Ga_{0.94})_2O_3$. This situation may be due to a difference in the hybridisation of 2p states of O and 4s states of Ga, which form the bottom of the conduction band, due to the admixture of electronic states of La^{3+} and Eu^{3+} ions, which are characteristic of the $(La_{0.06}Ga_{0.94})_2O_3$:Eu compound. Based on the obtained E_0 values for the films shown in Table 1 It can be said that the spectral dependence of the visible refractive index in $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films is mainly determined by transitions from the zone of 2p-states of oxygen, forming the upper filled level of the valence band, to the lower conduction band formed by hybridized 2p states of O and 4s states of Ga with an admixture of electronic levels of La^{3+} and Eu^{3+} ions

According to the results of Tubbs [31], the degree of ionicity of the chemical bond is defined as $f_i = \sqrt{E_0/E_d}$. Its values for the $(La_{0.06}Ga_{0.94})_2O_3$:Eu films are also given in Table 1. As can be seen, the films obtained by us are characterised by a rather high value of fi, which exceeds this value in thin films of β-Ga_2O_3 and $(Y_{0.06}Ga_{0.94})_2O_3$ [25, 26].

Based on equation (2) and taking into account that the valence of the anion is $Z_a = 2$, we determine the coordination number of the first coordination sphere of the cation. In this case, without taking into account the insignificant percentage of impurity (1 wt. %), to determine n_e, we will proceed from the fact that $(La_{0.06}Ga_{0.94})_2O_3$=$(La_{0.06}Ga_{0.94}O_{1.5})(La_{0.06}Ga_{0.94}O_{1.5})$ and in one formula unit of $La_{0.06}Ga_{0.94}O_{1.5}$, the value of n_e = 6.

Taking into account the mixed ionic-covalent nature of the chemical bond in $(La_{0.06}Ga_{0.94})_2O_3$, the value of β is determined from the following relation

$$\beta = 0.26 f_i + 0.37\,(1 - f_i). \qquad (3)$$

As a result, for the thin $(La_{0.06}Ga_{0.94})_2O_3$:Eu films, when determining the coordination number, we obtain N_C =2.40.

When considering the crystal structure of films of β-Ga_2O_3, in which our thin $(La_{0.06}Ga_{0.94})_2O_3$:Eu films are formed, it is necessary to take into account that Ga^{3+} ions occupy two non-equivalent crystallographic positions - one with a tetrahedral and the other with an octahedral oxygen environment of Ga^{3+} ions [32]. In addition, in the structure of β-Ga_2O_3, six complexes with a tetrahedral surround of gallium atoms (GaO_4) surround each paired double cell with an octahedral oxygen surround of gallium atoms (GaO_6) [28]. Based on this, on average, there are 36 O atoms per 8 Ga atoms in an ideal β-Ga_2O_3 lattice, or each Ga atom has 4.5 O atoms on average. The N_C value obtained by us is somewhat lower than the specified value and this downward deviation is most likely due to anionic vacancies as an integral element of the structure of real oxide crystal lattices [33]. A similar situation is observed in films of β-Ga_2O_3 and $(Y_{0.06}Ga_{0.94})_2O_3$ [25, 26]. In this case, annealing of these films in an oxygen atmosphere leads to a slight increase in the coordination number of the cation.

Most semiconductor and dielectric materials exhibit

characteristic absorption in the UV region of the spectrum. The characteristic wavelengths of UV absorption or the average interband wavelength of the oscillator λ_0 can be determined from the formula for describing the dispersion in the single-oscillator Zelmeyer model [34],

$$\frac{(n_0^2-1)}{(n^2-A)} = 1 - \left(\frac{\lambda_0}{\lambda}\right)^2 \quad (4)$$

which is a slightly modified model compared to equation (1). In relation (4), n_0 is a static refractive index that gives some indication of the structure and density of the material. The values of n_0 and λ_0 are determined by the relationship $(n^2–A)^{-1}$ to λ^{-2}. The obtained values of n_0 and the oscillator energy E_{01}, which was determined through λ_0, are given in Table 1. As shown in the table, the values of n_0 in thin $(La_{0.06}Ga_{0.94})_2O_3$:Eu films are slightly lower than in films of β-Ga_2O_3 and $(Y_{0.06}Ga_{0.94})_2O_3$. This situation is probably due to a greater disturbance of the β-Ga_2O_3 lattice structure when part of the Ga^{3+} ions with an ionic radius of 0.62 Å is replaced by La^{3+} ions with a larger ionic radius of 1.04 Å and Eu^{3+} ions with an ionic radius of 1.09 Å [35]. The replacement of a part of Ga^{3+} ions in the β-Ga_2O_3 lattice by Y^{3+} ions with an ionic radius of 0.93 Å causes smaller deformations of the crystal lattice, which leads to closer static refractive indices in films of β-Ga_2O_3 and $(Y_{0.06}Ga_{0.94})_2O_3$. It is also likely that a larger difference in the ionic radii of the cations and, accordingly, a larger lattice strain causes a larger discrepancy between the values of E_0 and E_d for the films of $(La_{0.06}Ga_{0.94})_2O_3$:Eu compared to the other two films shown in Table 1.

According to [36], the average value of the oscillator energy E_{01} is related to the static refractive index n_0 by a ratio:

$$n_0^2 = 1 + C\frac{(h\nu_p)^2}{E_{01}^2} \quad (5)$$

where $h\nu_p$ is the energy of plasma oscillations for valence electrons and C is a parameter depending on the matrix elements. As can be seen from expression (5), there is an inversely proportional relationship between the static refractive index and the average oscillator energy and a directly proportional relationship between the static refractive index and the matrix elements and the plasma oscillation energy. Considering that in $(La_{0.06}Ga_{0.94})_2O_3$:Eu films, compared to the other two films in Table 1, we observe a simultaneous decrease in n_0 and E_{01}, we can conclude that in films of $(La_{0.06}Ga_{0.94})_2O_3$:Eu, the shape of the dispersion curve is more influenced by the value of the matrix element C and the plasma oscillation energy of the valence electron ensemble compared to the average oscillator energy E_{01}.

Conclusions

The studies show that $(La_{0.06}Ga_{0.94})_2O_3$:Eu thin films obtained by radio-frequency ion-plasma sputtering are formed in a monoclinic crystal structure corresponding to the structure of β-Ga_2O_3. It has been found that freshly deposited films are characterized by an abnormal dispersion, and after annealing in an argon atmosphere, the films exhibit a normal dispersion of the refractive index, which is mainly determined by transitions from the 2p-state oxygen band, forming the upper filled level of the valence band to the lower level of conduction band, which is formed by hybridized 2p states of oxygen and 4s states of gallium. The dispersion of the refractive index of such films is determined by the peculiarities of their energy structure, crystal-chemical properties, and the energy of plasma oscillations of the valence electron ensemble.

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Дисперсійні властивості тонких плівок $(La_{0.06}Ga_{0.94})_2O_3$:Eu

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Досліджено дисперсію показника заломлення тонких плівок $(La_{0.06}Ga_{0.94})_2O_3$:Eu, одержаних методом високочастотного іонно-плазмового розпилення. Встановлено, що плівки мають полікристалічну структуру, яка відповідає моноклінній структурі β-Ga_2O_3. Показано, що для свіжонанесених плівок характерна аномальна дисперсія, а після відпалу в аргоні спостерігається нормальна дисперсія показника заломлення. Встановлено, що при нормальній дисперсії спектральна залежність показника заломлення у видимій області спектру в основному визначається електронними переходами із зони 2p-станів кисню, які формують верхній заповнений рівень валентної зони у дно зони провідності, утворене гібридизованими 2p-станами кисню і 4s-станами галію. Для плівок, відпалених у аргоні проведено аналіз та порівняння двох одноосциляторних апроксимаційних моделей, визначено параметри апроксимації, дисперсійну енергію, ступінь іонності хімічного зв'язку, координаційне число, статичний показник заломлення.

Ключові слова: тонка плівка, оксид лантану і галію, показник заломлення, дисперсія.