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# ANALYSIS OF SPECTRAL AND KINETIC CHARACTERISTICS OF CATHODOLUMINESCENCE OF $Y_2O_3$:Eu THIN FILMS AS A RED COMPONENT OF FULL-COLOR HIGH-RESOLUTION FLUORESCENT DISPLAYS

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The spectra and kinetics of the rising and decay of cathodoluminescence (CL) of thin $Y_2O_3$:Eu films obtained by the method of RF-magnetron sputtering are studied. Based on the shape of the CL spectra at different energies and excitation current densities, the possibility of the formation of irregular solutions of yttrium and europium oxide and the features of the structure of surface and bulk layers are shown. The dependence of the CL intensity on the energy of the exciting electrons and the current density of the electron irradiation has been investigated. The CL decay time constant for the 612 nm luminescence is determined, the value of which is in the range (1.8 – 4.1) ms. It is shown that this quantity is a complex function of the type of film deposition atmosphere, the concentration of the activator, and the duration of the exciting pulses. The features of CL rising are investigated and it is proposed to analyze the structural perfection of $Y_2O_3$:Eu thin films based on the delay in CL rising.

*Key words*: yttrium oxide, cathodoluminescence, thin films.

## Introduction

Among the large number of materials for optoelectronics, luminescent materials are of particular importance, which are used to create displays, scintillators, and means for recording and visualizing information. One of the widespread crystal matrices of phosphors is cubic yttrium oxide $Y_2O_3$, activated by ions of rare-earth metals [1–4]. Among them, the most studied phosphor of micron dispersion is phosphorus $Y_2O_3$:Eu with a red emission region. The results of studying this material can be used as basic information for analyzing the characteristics and mechanism of luminescence when studying the influence of size effects and structural perfection on the emission efficiency of submicron phosphors [5]. For this purpose, we used the method of local cathodoluminescence (CL), which has a number of features in comparison with traditional optical research methods [6]. This method is characterized by high sensitivity to changes in the electronic structure of the material (impurity and structural defects), makes it possible to study the change in the luminescent properties of structures and materials in depth from 10–20 nm to several micrometers. In addition, the high excitation energy of luminescence makes it possible to study optical transitions, the excitation of which requires an energy of more than 6 eV (from the vacuum ultraviolet region). This method also allows one to study the processes of energy transfer between

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high-energy states, the electronic structure of defects, which are channels for nonradiative recombination. All these factors determine the kinetics of luminescence onset and decay, which was the reason for its study in $Y_2O_3$:Eu thin films.

Note that a number of methods are used to obtain films based on $Y_2O3$; therefore, the films differ in optical and luminescent properties due to their different perfection. In this regard, in this work, we investigate the spectral and kinetic properties of CL of thin $Y_2O_3$:Eu films obtained by radio-frequency (RF) ion-plasma sputtering, which is optimal for obtaining the most homogeneous semiconductor and dielectric films [7].

## Experimental technique

Thin $Y_2O_3$:Eu films with a thickness of 0.2 – 1.0 μm obtained by RF ion-plasma sputtering in an atmosphere of 100% oxygen or 100% argon in a system using the magnetic field of external solenoids for compression and additional ionization of the plasma column on fused silica $\upsilon$-$SiO_2$ substrates. The initial components were ИтО-И grade $Y_2O_3$ and “ос.ч”grade $Eu_2O_3$. The activator concentration was 2.5 and 5.0 mol.%. After the deposition of the films, they were heat treated in air at 950–1050 °C. X-ray diffraction studies have shown the presence of a polycrystalline structure of the films with a preferred orientation in the (222) plane. The form of the obtained diffractograms is practically analogous to the diffractograms of pure $Y_2O_3$ films presented by us in [8]. In this case, in $Y_2O_3$: Eu films deposited in an argon atmosphere, the reflection from the (440) plane has a slightly higher intensity.

Investigations of CL properties were carried out in the mode of pulsed electronic excitation. The luminescence spectra were recorded on a setup based on an СФ-4А spectrophotometer using an ФЭУ-79 photomultiplier, the signal from which was fed to a resonant amplifier and recorded by an ammeter, and was also transmitted through an analog-to-digital interface converter to an IBM/PC computer for recording the luminescence spectrum. The scanning of wavelengths by the monochromator was carried out using a stepper motor, which was guided by a computer through a control unit. The luminescence spectra were investigated in the range 200–800 nm at 295 K. The kinetics of combustion and decay were recorded using an ФЭУ-79 photomultiplier. The next digitization of the signal from the photomultiplier, which was carried out with an С1-117 electronic oscilloscope, made it possible to obtain a data array containing information on the kinetics of expansion and decay in a form convenient for subsequent processing.

## Results and discussion

Typical CL spectra of the obtained $Y_2O_3$:Eu films at different energies of exciting electrons and current densities of electron irradiation are shown in Fig.1. The luminescence spectra of the films exhibit narrow luminescence bands caused by intracenter transitions between the electron shells within the $Eu^{3+}$ activator. The wavelength of the maximum radiation is $\lambda_{max}$= 612 nm, which corresponds to the red color of the glow. All observed electronic transitions $^5D_0$–$^7F_J$ for $Eu^{3+}$ ions marked in Fig.1. They include allowed magnetic dipole transitions $^5D_0$-$^7F_1$ (for $Eu^{3+}$ ions in $C_2$ and $C_3$ in $Y_2O_3$ lattice sites) and allowed electric dipole transitions $^5D_0$–$^7F_2$ (for $Eu^{3+}$ ions only at $C_2$ sites). The unit cell of cubic $Y_2O_3$ contains 32 yttrium ions, which can be replaced by other rare earth ions. Of these, 8 occupy a centrally symmetric position ($C_{3i}$ symmetry), and the remaining 24 have a lower $C_2$ symmetry [9, 10]. Thus, $Eu^{3+}$ ions can be in one of two nonequivalent positions, leading to a difference in the luminescence spectra. Taking into account that the radii of the $Y^{3+}$ and $Eu^{3+}$ ions are rather close to each other and equal, respectively, 0.90 and 0.94 Å, the activation by europium ions $Y_2O_3$ retains the cubic structure of the crystal lattice with insignificant changes in the parameters [11].

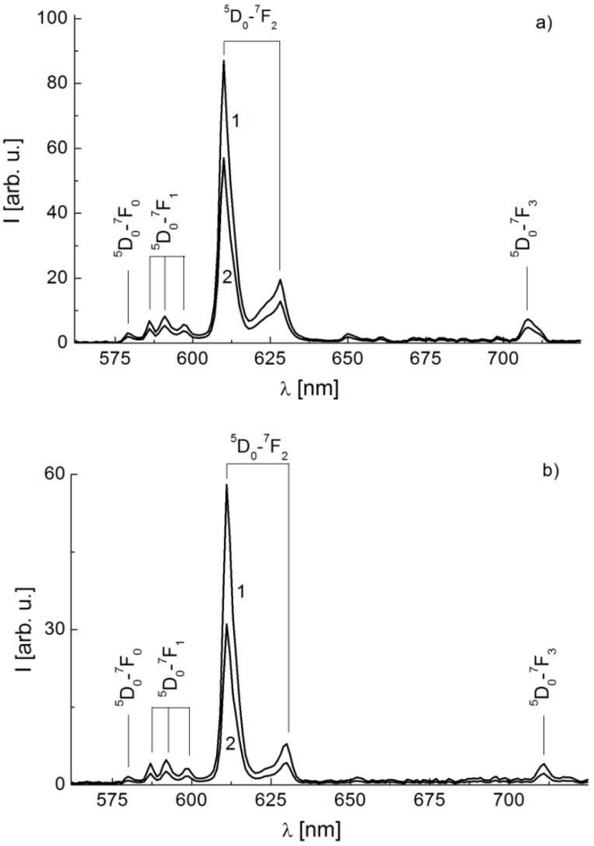

Fig.1. CL spectra of $Y_2O_3$:Eu thin films at different energies of exciting electrons (a) and current densities of electron irradiation (b). The activator concentration is 5%, the atmosphere of deposition is Ar (100%) (a) and $O_2$ (100%) (b). Parameters of electronic excitation pulses: pulse duration $5\times10^{-4}$ s, pause between pulses 0.1 s; a) the current density of the electron beam is $j= 5\times10^{-2}$ A/m$^2$, the energies of the exciting electrons are 5.0 keV (1) and 4.5 keV (2); b) the energy of exciting electrons is 6 keV, the current density of the electron beam is $12\times10^{-3}$ A/m$^2$(1) and $6.5\times10^{-3}$ A/m$^2$ (2).

When describing the luminescence spectrum of $Y_2O_3$:Eu, the so-called "asymmetric ratio" $I_{612}/I_{596}$ of the intensities of the luminescence bands at 612 and 596 nm is often used, which corresponds to the ratio of the number of $Eu^{3+}$ cations in the corresponding states of local symmetry $N_{C2}/N_{C3i}$ [5, 11–14]. Luminescence in the 612 nm band is determined by the $^5D_0$-$^7F_2$ electric dipole transition, which is very sensitive to the immediate environment of the emitting $Eu^{3+}$ ion. The band with a maximum at 596 nm is determined by the $^5D_0$–$^7F_1$ magnetic dipole transition, which is insensitive to the nearest environment [10, 13–15]. The amplitude intensity ratio $I_{612}/I_{596}$ is used to assess the ratio $N_{C2}/N_{C3i}$ [5, 13, 15, 17] and to analyze the structural perfection of $Y_2O_3$:$Eu^{3+}$.

For $Y^{3+}$ ions in the $Y_2O_3$ matrix, the equilibrium ratio $N_{C2}/N_{C3i}$ in an ideal lattice is 3:1. With a uniform substitution of $Eu^{3+}$ ions for $Y^{3+}$ ions, we should obtain a similar result. However, in thin $Y_2O_3$:Eu films, this ratio should be significantly higher, the value of which also depends on the energy of bombarding electrons. For example, at an excitation electric current density of $5\times10^{-2}$ A/m$^2$, an increase in the electronic excitation energy from 4.5 to 5.0 keV leads to an increase in this ratio from 10.4 to 14.0 (Fig.1). Note that fairly close results for the amplitude ratio $I_{612}/I_{596}$ = (8–10) were obtained in [5] when studying the photoluminescence of $Y_2O_3$:Eu powder materials of different dispersion, obtained by the sol-gel method.

Our results show that with an increase in the energy of bombarding electrons, that is, with an increase in the depth of their penetration into a thin $Y_2O_3$:Eu film, the relative contribution of the emission of $Eu^{3+}$ ions in the $C_2$ sites relative to the $C_{3i}$ sites increases in the glow. This deviation most likely indicates the formation of irregular solutions of yttrium and europium oxide in the $Y_2O_3$:Eu thin film during its deposition. An increase in the energy of exciting electrons and, accordingly, their penetration depth shows that the relative number of $Eu^{3+}$ ions in the $C_{3i}$ position prevails on the surface relative to the depth. An increase in the relative amount of $Eu^{3+}$ ions in the $C_2$ position in the depth of the film deposited in an argon atmosphere (Fig.1) is most likely due to a decrease in the local symmetry of the environment of europium ions by oxygen ions. Since the films were fired in air, the local symmetry of $Eu^{3+}$ ions near the surface first of all increased during annealing.

According to Fig. 2a, with an increase in the excitation energy, the CL intensity increases. This behavior of this dependence for $Y_2O_3$:Eu films is observed up to a certain value, which is determined by the film thickness. In particular, for the $Y_2O_3$:Eu film with a thickness of 0.20 μm, as can be seen from Fig.3a, this energy is in the region of 5.0 keV. Thereafter, the CL intensity slightly decreases, and when the excitation energy reaches 5.5 keV, it starts to increase again. This behavior of this dependence is apparently due to the fact that when the bombarding electrons reach an energy of 5.0 keV, the exciting beam begins to pass through the film, reaching the substrate. As a result, there is a redistribution of electrons between the film and the substrate, which leads to a decrease in the CL intensity in the range of 5.0–5.5 keV. It is clear that the value 5.5 keV is not decisive for the $Y_2O_3$:Eu material, but is determined by the thickness of the deposited film [18]. With a decrease (increase) in the film thickness, the electron energy at which the maximum radiation intensity is reached will decrease (increase).

It should be noted that the CL intensity is a complex function of the excitation energy and radiation dose. Since the measurements (Fig.2a) were carried out at a fixed number of bombarding electrons, it is clear that a further increase in the CL intensity at an excitation energy above 5.5 keV can be explained only by an increase in the energy of the bombarding electrons. When the duration of the exciting pulse was $4\times10^{-4}$ s; at the minimum radiation dose in this study, the above-described minimum CL intensity was not observed.

This approach can also explain the rather sharp increase in the CL intensity with an increase in the current density of the exciting beam (Fig. 2b). Analyzing the mechanism of CL excitation of the $Y_2O_3$:Eu phosphor, one should proceed from the fact that with an increase in the concentration of bombarding particles, the CL intensity should increase in direct proportion. As can be seen from Fig. 2b, the dependence we obtained in the investigated range of excitation current densities is practically linear, which is in good agreement with the proposed approach.

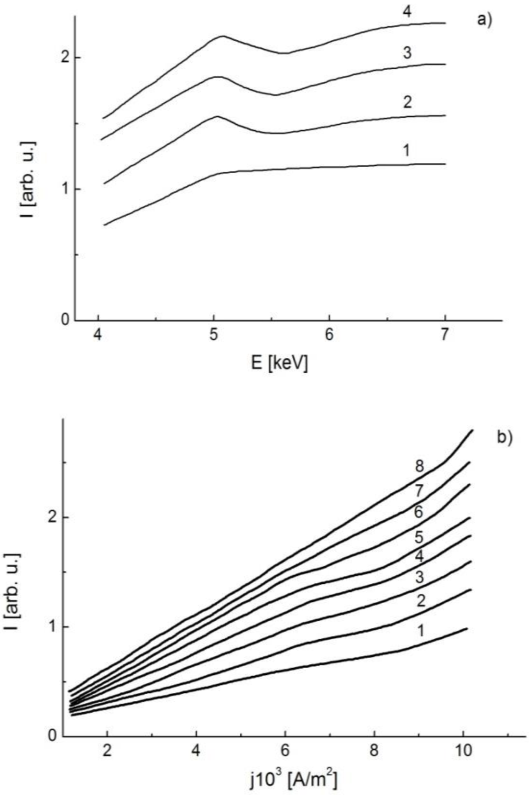

Fig. 2. Dependence of the CL intensity of $Y_2O_3$:Eu thin films on the energy of exciting electrons (a) and the current density of electronic excitation (b). Concentration of the activator 2.5% (a) and 5% (b), atmosphere of Ar sputtering (100%). Parameters of electronic excitation pulses: a) the current density of the electron beam is $10^{-2}$ A/m$^2$, the duration of the excitation pulses is $4\times10^{-4}$ s (1), $6\times10^{-4}$ s (2), $8\times10^{-4}$ s (3) , $10^{-3}$ s (4); b) the energy of exciting electrons is 2.5 keV, the pulse duration is $3\times10^{-4}$ s (1), $4\times10^{-4}$ s (2), $5\times10^{-4}$ s (3), $6\times10^{-4}$ s (4), $7\times10^{-4}$ s (5), $8\times10^{-4}$ s (6), $9\times10^{-4}$ s (7), $10^{-3}$ s (8). Pause between pulses 0.1 s.

To study the mechanism of energy transfer of rare-earth activators in films of the $Y_2O_3$ matrix, we analyzed a number of kinetics of the rising and decay of CL in $Y_2O_3$: Eu films for the luminescence $\lambda_{max}$ = 612 nm. The typical form of kinetics for various films depending on the excitation energy is shown in Fig. 3 and Fig. 4. It was found that the kinetics of luminescence decay in the studied $Y_2O_3$:Eu films at different energies and irradiation doses are well approximated by monoexponential functions. Our results indicate that the decay time constant in the studied films changes depending on the atmosphere of the deposition of the films, the concentration of the activator, and the duration of the exciting bombarding pulses. In particular, it was found that $Y_2O_3$:Eu films deposited in an atmosphere of both argon and oxygen with an activator concentration of 5 mol.% luminescence brighter than films with an activator concentration of 2.5 mol.%. The decay time constant for the first films is larger than for the second ones (Table 1). In addition, for all types of films, the decay time at short excitation duration (4 ms) is longer than the decay time at a longer excitation duration (6–9 ms). This can be explained by the fact that the near-surface layers of the film are structurally inhomogeneous than the bulk ones. Therefore, most likely, after the termination of the exciting electron pulse, these inhomogeneities contribute to the luminescence kinetics, increasing the decay time due to a larger relative contribution at low radiation doses.

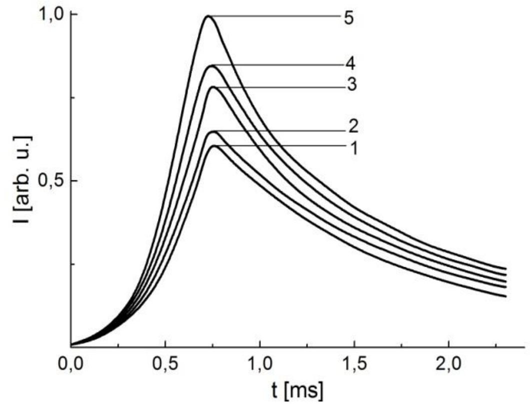

Fig.3. Dependence of the kinetics of CL rising and decay on the excitation energy and technological conditions for the production of $Y_2O_3$:Eu thin films. Parameters of electronic excitation pulses: pulse duration $5\times10^{-4}$ s; pause between pulses 0.1 s; electron beam current density $j=1\times10^{-2}$ A/m$^2$. Activator concentration 2.5%, Ar deposition atmosphere (100%), electron bombardment energy 5.5 keV (1), 6.0 keV (2). Activator concentration 5%, $O_2$ deposition atmosphere (100%), electron bombardment energy 4.0 keV (3), 5.5 keV (4), 7.0 keV (5).

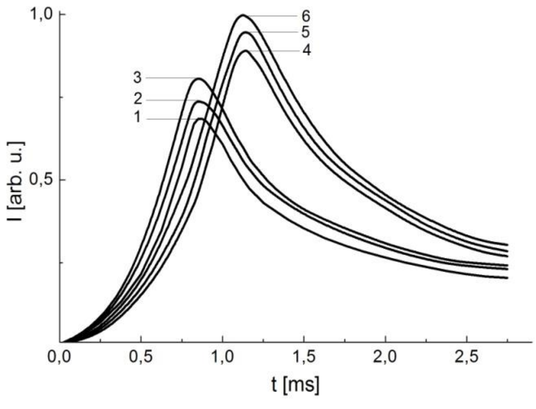

Fig. 4. Dependence of the kinetics of CL rising and decay on the excitation energy and technological conditions for the production of $Y_2O_3$:Eu thin films. Parameters of electronic excitation pulses: pulse duration $9\times10^{-4}$ s; pause between pulses 0.1 s; electron beam current density $j=1\times10^{-2}$ A/m$^2$. Activator concentration 2.5%, Ar deposition atmosphere (100%), electron bombardment energy 5.0 keV (1) 6.0 keV (2), 6.5 keV (3). Activator concentration 5%, $O_2$ deposition atmosphere (100%), electron bombardment energy 5.0 keV (4), 6.0 keV (5), 6.5 keV (6).

As you can see from the Table 1, the decay time constant of CL in $Y_2O_3$:Eu thin films for the emission of 612 nm is in the range (1.8–4.1) ms, depending on the structural perfection of the films and the concentration of the activator.

Table 1

Dependence of the decay time constant of CL in $Y_2O_3$:Eu thin films for a 612 nm glow on the excitation time and of preliminary irradiation*

| Film spraying atmosphere, irradiation | Activator concentration, mol. % | Excitation time $t_{ex}\times10^4$, s | $\tau\times10^3$, s |
|---|---|---|---|
| Argon 100 %, | 2.5 | 4 | 3.7 |
| | 2.5 | 6 | 2.2 |
| | 2.5 | 9 | 2.2 |
| Argon 100 %, | 5.0 | 4 | 4.1 |
| | 5.0 | 6 | 3.4 |
| | 5.0 | 9 | 3.3 |
| Oxygen 100 %, | 5.0 | 4 | 2.0 |
| | 5.0 | 6 | 1.9 |
| | 5.0 | 9 | 1.8 |

* Excitation current density $10^{-2}$ A/m$^2$, voltage 4.5 keV.

Theoretical calculations of electric dipole, magnetic dipole and electric quadrupole transitions in Lanthanides show that these quantities have the following values: $\tau_{el} = 10^{-4}$ s; $\tau_{mag} = 0.5\times10^{-2}$ s; $\tau_q = 2$ s [19]. Comparing our values with these results based on the spectral composition of the radiation (Fig.1), it can be seen that we have a luminescence caused by electric dipole transitions and that the $Eu^{3+}$ ions emitting in the 612 [nm] region are located at the sites of the $Y_2O_3$ crystal lattice with the $C_2$ point symmetry.

Note also that the values of the decay time constant $\tau$ obtained by us are in good agreement with the known studies of the decay of luminescence in various $Y_2O_3$:Eu samples [20–24]. Thus, in [20], when determining the decay time of the $^5D_0$–$^7F_2$ transition in $Y_2O_3$:Eu ceramics with crystallite sizes of 10 nm, a value of 1.3 ms was obtained. When studying the damping constant in $Y_2O_3$:Eu nanocrystals, it was found in [21] that the damping time constant is in the range from 2.0 to 3.7 ms. Studies [22] show that the lifetime of 5–7 nm $Y_2O_3$:Eu nanophosphorites for an activator concentration of 2.4 mol% is 3.08 ms, and for an activator concentration of 5.2 mol%, respectively, 2.40 ms. For microcrystals, the decay time constant for the 612 nm luminescence is shorter and for an activator concentration of 3.5 mol% $\tau = 1.07$ ms, and for 5.2 mol% $\tau = 1.02$ ms. According to [23], in $Y_2O_3$:Eu single crystals, the decay time constant is $\tau\approx 4$ ms, while in [24] for $Y_2O_3$:Eu single crystals, the decay time constant is determined as 1.90 and 1.72 ms for activator concentrations of 3 and 8%.

Our studies show that such differences in the value of the decay time constant during the glow of the phosphor $Y_2O_3$:Eu in the 612 nm region, caused by the $^5D_0$–$^7F_2$ electric dipole transition, are due to the fact that this value of $\tau$ is a complex function, which is determined by the method and conditions of production, the method and dose of excitation, the perfection of the structure and the concentration of the activator.

When analyzing the kinetics of CL rising and decay, it was found that, in a number of cases, there is a delay in CL rising relative to the decay of the electronic excitation pulse (Fig. 4). This situation is more pronounced in $Y_2O_3$:Eu films deposited in an oxygen atmosphere, and this shift, in addition, depends on the energy and radiation dose. In particular, with an increase in the dose of electron irradiation (due to an increase in the pulse duration), a shift with time of the maximum of CL burnup relative to the decay of the electronic excitation pulse is observed. This effect can be explained by structural inhomogeneities, which are clearly manifested in films deposited in an oxygen atmosphere. Due to such inhomogeneities, the activator ions $Eu^{3+}$ can be located in the interstitial defect positions of the $Y_2O_3$:Eu crystal lattice, or carry out transitions due to diffusion between $C_2$ and $C_3$ and the sites of the $Y_2O_3$ matrix. Thus, the structural perfection of $Y_2O_3$:Eu thin films can be analyzed on the basis of the dependence of the CL flare-up on the energy and radiation dose.

## Conclusion

Based on the analysis of the shape of the CL spectra at various energies and current densities of electronic excitation, the possibility of the formation of irregular solutions of yttrium and europium oxide in thin $Y_2O_3$:Eu films and an increase in the number of $Eu^{3+}$ ions in the state of reduced $C_2$ symmetry in the depth of the film relative to the surface are shown. It was found that an improvement in energy transfer from electrons absorbed by the film to activator centers is observed with an increase in the irradiation dose, i.e. the duration of the exciting CL pulses from 3 $\times 10^{-4}$ s to $10^{-3}$ s. The obtained values of the CL decay time constant indicate that the emission of $Y_2O_3$:Eu in the 612 nm region is caused by an electric dipole transition between the $^5D_0$–$^7F_2$ energy levels. From the analysis of the dependence of the CL decay time constant on the activator concentration, preparation conditions, and irradiation time, it is shown that the near-surface layers are structurally inhomogeneous than the bulk ones. It has been established that the structural perfection of $Y_2O_3$:Eu thin films can be analyzed on the basis of the dependence of the CL flare-up and the CL decay time constant on the energy and radiation dose.

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# АНАЛІЗ СПЕКТРАЛЬНИХ ТА КІНЕТИЧНИХ ХАРАКТЕРИСТИК КАТОДОЛЮМІНЕСЦЕНЦІЇ ТОНКИХ ПЛІВОК $Y_2O_3$:Eu, ЯК ЧЕРВОНОГО КОМПОНЕНТУ ПОВНОКОЛІРНИХ ЛЮМІНЕСЦЕНТНИХ ДИСПЛЕЇВ ВИСОКОЇ РОЗДІЛЬНОЇ ЗДАТНОСТІ

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Досліджено спектри та кінетику розгоряння і загасання катодолюмінесценції (КЛ) тонких плівок $Y_2O_3$:Eu, отриманих методом ВЧ-магнетронного напилення в атмосфері кисню та аргону. Після нанесення проводилась термообробка отриманих тонкоплівкових матеріалів. На основі форми спектрів КЛ при різних енергіях та густинах струму збудження показано можливість утворення нерегулярних розчинів оксиду ітрію і європію та особливості структури поверхневих і об’ємних шарів. Проведено дослідження залежності інтенсивності КЛ від енергії збуджуючих електронів і густини струму електронного опромінення. Визначено постійну часу загасання КЛ для свічення 612 нм, величина якої перебуває в межах (1.8 – 4.1) мс. Показано, що дана величина є складною функцією від виду атмосфери напилення плівок, концентрації активатора та тривалості збуджуючих імпульсів. Встановлено, що кінетики загасання люмінесценції в досліджуваних плівках оксиду ітрію при різних енергіях та дозах опромінення добре апроксимуються моноекспоненціальними функціями. При аналізі кінетик розгоряння і загасання КЛ було встановлено, що в ряді випадків спостерігається затримка розгоряння КЛ відносно спаду імпульсу електронного збудження. Така ситуація яскравіше виражена у плівках $Y_2O_3$:Eu, напилених в атмосфері кисню і дане зміщення крім того залежить від енергії і дози опромінення. Зокрема, при збільшенні дози електронного опромінення, спостерігається зміщення з часом максимуму розгоряння КЛ відносно спаду імпульсу електронного збудження. Даний ефект можна пояснити структурними неоднорідностями, які чіткіше проявляються у плівках, напилених в атмосфері кисню. Досліджено особливості розгоряння КЛ і запропоновано на основі затримки розгоряння катодолюмінесценції аналізувати структурну досконалість тонких плівок $Y_2O_3$:Eu.

*Ключові слова*: оксид ітрію, катодолюмінесценція, тонкі плівки.

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