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Spectral Characteristics of Photoluminescence Synthesized in the Field of Radiation YAGG Phosphors with Different Al/Ga Ratio

In the study the results of luminescence spectra investigating and excitation of ceramics luminescence with different Al/Ga ratios, synthesized by irradiating a mixture of initial materials with different prehistories were presented. Mixtures of oxides powders with stoichiometric composition corresponding to $Y_3Al_xGa_{5-x}O_{12}$: Ce, where x ranged from 0 to 5, were prepared for synthesis. All initial materials had a purity of no less than analytical grade. The synthesis was carried out under the influence of an electron beam with an energy of 1.4 MeV and a power density of 20 kW/cm^2. The yield of the synthesis reaction, the ratio of the sample mass to the mixture, was 90–95 %. Research has shown that decreasing the Al/Ga ratio results in a shift of the luminescence band towards the short-wavelength region of the spectrum. Additionally, the excitation spectrum changes. As the Al/Ga ratio decreases, the excitation band shifts towards the long-wavelength region around 350 nm. The characteristics of luminescence are influenced by the prehistory of the initial materials.

Keywords: Al/Ga ratio; luminescence; radiation synthesis; high-energy electron beam.

Introduction

Materials based on metal oxides have found wide application in electronics, photonics, optics, laser technology, dosimetry [1–4] due to the variety of functional properties possible for practical use. They are used in the form of crystals, ceramics, films, powders. Most often these materials are complex in composition. Materials are characterized by high resistance of their functional properties to external influences: temperature, radiation, and aggressive media. The range of such materials is very wide and continues to grow.

The main obstacle in expanding the range of possible applications is the complexity of their synthesis. The most widespread method is thermal synthesis, using which many materials with a variety of functional properties have been obtained [5]. However, the thermal synthesis methods of refractory materials are complicated, characterized by the duration of the process, as a rule of thumb for a few dozen hours. This restrains the possibility of expanding the range of materials, optimization of processes in order to increase the main functional characteristics. Less common are such synthesis methods as sol-gel [6], co-precipitation [7] and similar [8]. The main disadvantage of all these methods is the duration of the process, the need to use additional substances to facilitate synthesis, which must be discarded at the final stages of synthesis.

Combustion, flame heating methods allow to accelerate the synthesis process considerably [9, 10]. However, they require complicated procedures for purification from additionally introduced substances. The methods of pulse flame sintering [11, 12], which have been spreading in recent years, are universal, fast, but do not exclude the necessity of purification from additionally used substances. It is difficult to obtain voluminous materials with these methods.

The radiation method of synthesis by direct impact of powerful flux of high-energy electrons on the mixture of stoichiometric composition seems promising. The method was first implemented in 2017 [13, 14]. Radiation method allows to provide synthesis of ceramics in time less than 1 s without using any substances to facilitate the process. The main features and examples of synthesis are described in [15–17].

At present, in the formation period of the radiation synthesis method, it is urgent to prove the possibility of its implementation for obtaining multicomponent materials from powders of refractory monocomponent metal oxides. The confirmation of obtaining such a material can be the correspondence of its properties to the obtained materials properties by other methods. The synthesis of activated yttrium-aluminium-gallium garnets (YAGG), whose structural and luminescent properties are well studied, is of interest. The present work involves the synthesis of YAGG: Ce ceramics under different modes of radiation exposure, the study of

luminescent properties and comparison with the available information on the properties of materials of similar composition were obtained by other methods used.

Ceramic synthesis

The synthesis of ceramics was carried out by direct impact of a powerful electron beam with an energy of 1.4 MeV on the mixture in a massive copper crucible using the UNU 'Stand ELV-6' of the INP SB RAS. The synthesis of $Y_3(Al_xGa_{1-x})_5O_{12}$: Ce ceramics with different Al/Ga ratios in the composition was carried out by electron fluxes with the energy of 1.4 MeV. The electron beam had a diameter at the mixture surface of 1 cm, scanned in the transverse direction of the crucible with an amplitude of 5 cm and a frequency of 50 Hz. During synthesis, the crucible with internal dimensions of 10x5 cm^2 was stretched relative to the scanning electron beam at a speed of 1cm/s. As a result of synthesis in the crucible samples were formed in the form of plates with the crucible dimensions. The total time of electron flux impact on the mixture treated surface of crucible was 10s. The synthesis of ceramics was realized only due to the radiation energy flux, only from the mixture materials, without additives of other materials facilitating the process.

Powder mixtures of Y_2O_3, Al_2O_3, Ga_2O_3 oxides in stoichiometric ratio were prepared for synthesis, Ce_2O_3 was added for activation in the amount of 0.5 % from the total mass of the mixture. All the initial powders were obtained from Hebei Suoyi New Material Technology Co., Ltd. (K1, K6, K7, K8) and Chemreactiv (F800, ITO). The mixtures compositions are shown in Table 1. The activator was added in the amount of 0.5 % of the mixture mass of the main components. The initial powders had purity degree not lower than 99.95, close distribution of particle sizes of powders. Sample numbers in the table means their number in the accounting system adopted by the authors. All experiments of ceramics synthesis were carried out using electron fluxes with energy 1.4 MeV, power density 20kW/cm^2.

Table 1

Description of samples and initial mixture composition

№	Sample, Initial mixture composition description	Yield% Weight M_m / M_o
630	$Y_3Al_5O_{12}$: Ce- Al_2O_3(43 %) (F800), Y_2O_3(57 %)(ИТО), Ce_2O_3 (0.5 %)(K1)	90,1
631	$Y_3Al_4GaO_{12}$: Ce — Al_2O_3 (32 %) (F800), Y_2O_3 (53 %)(ИТО), Ga_2O_3 (15 %)(K8), Ce_2O_3 (0.5 %)(K1)	94,5
632	$Y_3Al_3Ga_2O_{12}$: Ce- Al_2O_3 (28.5 %) (F800), Y_2O_3 (50 %)(ИТО), Ga_2O_3 (27.5 %)(K8), Ce_2O_3 (0.5 %)(K1)	96,6
633	$Y_3Al_2Ga_3O_{12}$: Ce-Al_2O_3 (14 %) (F800), Y_2O_3 (47 %)(ИТО), Ga_2O_3 (39 %)(K8), Ce_2O_3 (0.5 %)(K1)	97,4
634	$Y_3Al_1Ga_4O_{12}$: Ce -Al_2O_3 (6.7 %) (F800), Y_2O_3 (44.3 %)(ИТО), Ga_2O_3 (49 %)(K8), Ce_2O_3 (0.5 %)(K1)	97,4
635	$Y_3Ga_5O_{12}$: Ce -Y_2O_3 (42 %)(ИТО), Ga_2O_3 (58 %)(K8), Ce_2O_3 (0.5 %)(K1)	97,7
636	$Y_3Al_5O_{12}$: Ce -Al_2O_3 (43 %) (K7), Y_2O_3 (57 %)(K6), Ce_2O_3 (0.5 %)(K1), Ce_2O_3 (0.5 %)(K1)	96,5
637	$Y_3Al_4GaO_{12}$: Ce -Al_2O_3 (32 %)(K7), Y_2O_3 (53 %)(K6), Ga_2O_3 (15 %) (K8), Ce_2O_3 (0.5 %)(K1)	96,3
638	$Y_3Al_3Ga_2O_{12}$: Ce- Al_2O_3(K7) (28.5 %) (K7), Y_2O_3 (50 %)(K6), Ga_2O_3 (27.5 %)(K8), Ce_2O_3 (0.5 %)(K1)	96,3
639	$Y_3Al_2Ga_3O_{12}$: Ce -Al_2O_3 (14 %)(K7), Y_2O_3 (47 %)(K6), Ga_2O_3 (39 %)(K8), Ce_2O_3 (0.5 %)(K1)	97,2
640	$Y_3AlGa_4O_{12}$: Ce -Al_2O_3 (6.7 %)(K7) (K7), Y_2O_3 (44.3 %)(K6), Ga_2O_3 (49 %)(K8), Ce_2O_3 (0.5 %)(K1)	96,6
641	$Y_3Ga_5O_{12}$: Ce+ Y_2O_3 (42 %)(K6), Ga_2O_3 (58 %)(K8), Ce_2O_3 (0.5 %)(K1)	94,4

As shown in the table, the synthesis was carried out using initial materials from different manufacturers to check the validity of obtained samples.

Figure 1 shows photos of typical synthesized samples in crucibles. $Y_3Al_5O_{12}$: Ce ceramics have a characteristic bright yellow color. The color changes when Ga is introduced instead of Al.

$Y_3Al_3Ga_2O_{12}$: Ce- Al_2O_3 (28.5 %) (F800), Y_2O_3 (50 %)(ITO), Ga_2O_3 (27.5 %), Ce_2O_3 (0.5 %)(K1)

$Y_3Al_3Ga_2O_{12}$: Ce- Al_2O_3(K7) (28.5 %) (K7), Y_2O_3 (50 %)(K6), Ga_2O_3 (27.5 %), — Ce_2O_3 (0.5 %)(K1)

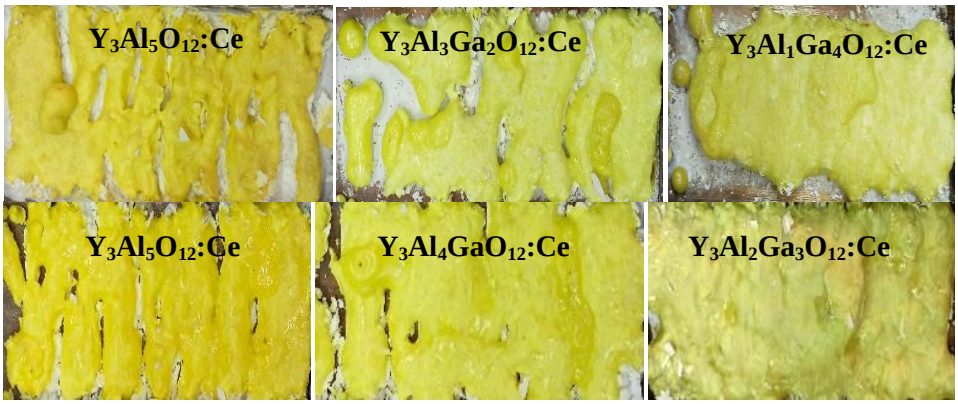

Figure 1. Photo of typical synthesized samples in crucibles

Table 1 shows the values of synthesis reaction yield. In this article we understand the synthesis reaction yield as the ratio of the synthesized sample mass to the mass of the mixture used. The mass of the obtained ceramic samples is in the range of 44–58 g. One of the reasons for the difference may be the poorly controlled process of pressing the mixture in the crucible during its leveling before synthesis and the crucibles size used. The mixture amount in the crucible was such that the electron flux did not reach the crucible bottom. The remainder of the mixture could be used in further experiments. It should be emphasized that to date the authors have succeeded in selecting initial materials that provide ceramics with a high yield of 90 to 97 %.

Spectral properties of luminescence

Excitation and luminescence spectra were measured using a Cary 5000 UV-Vis-NIR spectrophotometer. The measurements were performed as follows. Three small ceramic samples were spun off from different areas of each synthesized plate. The excitation and luminescence spectra of each were measured. In the measurements, the excitation flux was directed to the outer surface of the sample, which was facing the incoming electron flux during synthesis. Each sample was then crushed mechanically to powder. The powders were poured into cuvettes, and the excitation and luminescence spectra of each were measured. An example of excitation and luminescence spectra measurements results is shown in Figure 2. The excitation (a) and luminescence (b) spectra of ceramics (630-1, 630-2, 630-3) and their powders (630P-1, 630P-2, 630P-3) are shown. The difference in the luminescence spectra of the ceramic samples and their powders is clearly visible. The difference of luminescence spectra of samples of the same ceramics taken from one and the same plate, ceramics and complete coincidence of spectra of powders from them draws attention.

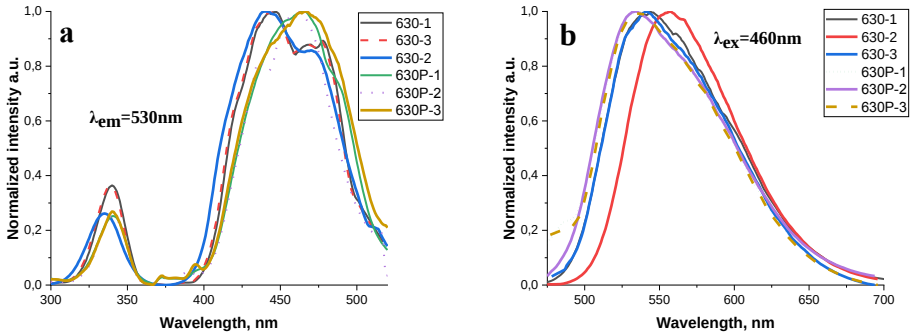

Figure 2. Excitation spectra (a) of ceramics (630-1, 630-2, 630-3)
and their powders (630P-1, 630P-2, 630P-3) and luminescence (b).

Clearly, the powders exhibit averaged characteristics of the excitation and luminescence spectra of the entire sample material. The excitation of ceramic samples by UV radiation occurs only in the near-surface

region. Therefore, the contribution of surface luminescence to the integral is minimal. Considering this, only the results of the spectral characteristics of the powdered materials are presented below.

In Figure 3, the excitation spectra of luminescence at the maxima of the luminescence bands and the luminescence spectra under UV excitation at 340 and 445 nm for the powders from samples 632 and 638 are presented. The ceramic samples 632 and 638 have the same composition: $Y_3Al_3Ga_2O_{12}$: Ce, but were synthesized from initial materials with different histories. Sample 632 was synthesized from powders of Al_2O_3 (F800), Y_2O_3 (ITO), Ga_2O_3 (K8), and Ce_2O_3 (0.5 %) (K1). Sample 638 was synthesized from powders of Al_2O_3 (F800), Y_2O_3 (ITO), Ga_2O_3 (K8), and Ce_2O_3 (0.5%) (K1). Sample 638 was synthesized from powders of Al_2O_3 (K7), Y_2O_3 (50%) (K6), Ga_2O_3 (K8), and Ce_2O_3 (0.5%) (K1). The results of the measurements show that the origin of the selected initial powders for synthesis does not affect the position of the luminescence spectra bands.

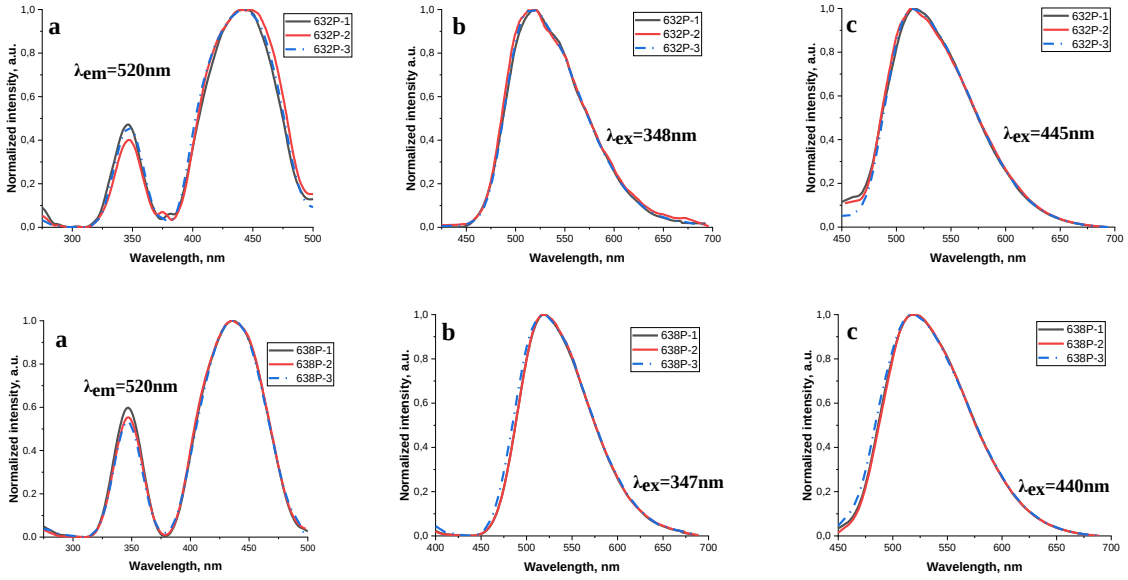

Figure 3. Excitation (a) and luminescence (b, c) spectra of powders 632 and 638

Note that the excitation and luminescence spectra of the powders do not depend on the part of the plate from which the sample was taken for measurements. This means that the luminescent properties are uniform throughout the entire plate. We emphasize that this conclusion holds well for all studies of 12 ceramic compositions.

Figure 4 shows the luminescence spectra of powders from ceramics 630–634 and 636–540 upon excitation by UV radiation in the region of 450, 350 and 220 nm. The luminescence spectra of ceramics 635 and 641 are not shown. Their luminescence is weak and could not be measured correctly by the spectrophotometer used. The luminescence of the powders clearly depends on the Al/Ga ratio in the composition. With a change in this ratio, a shift in the luminescence band and its shape occurs.

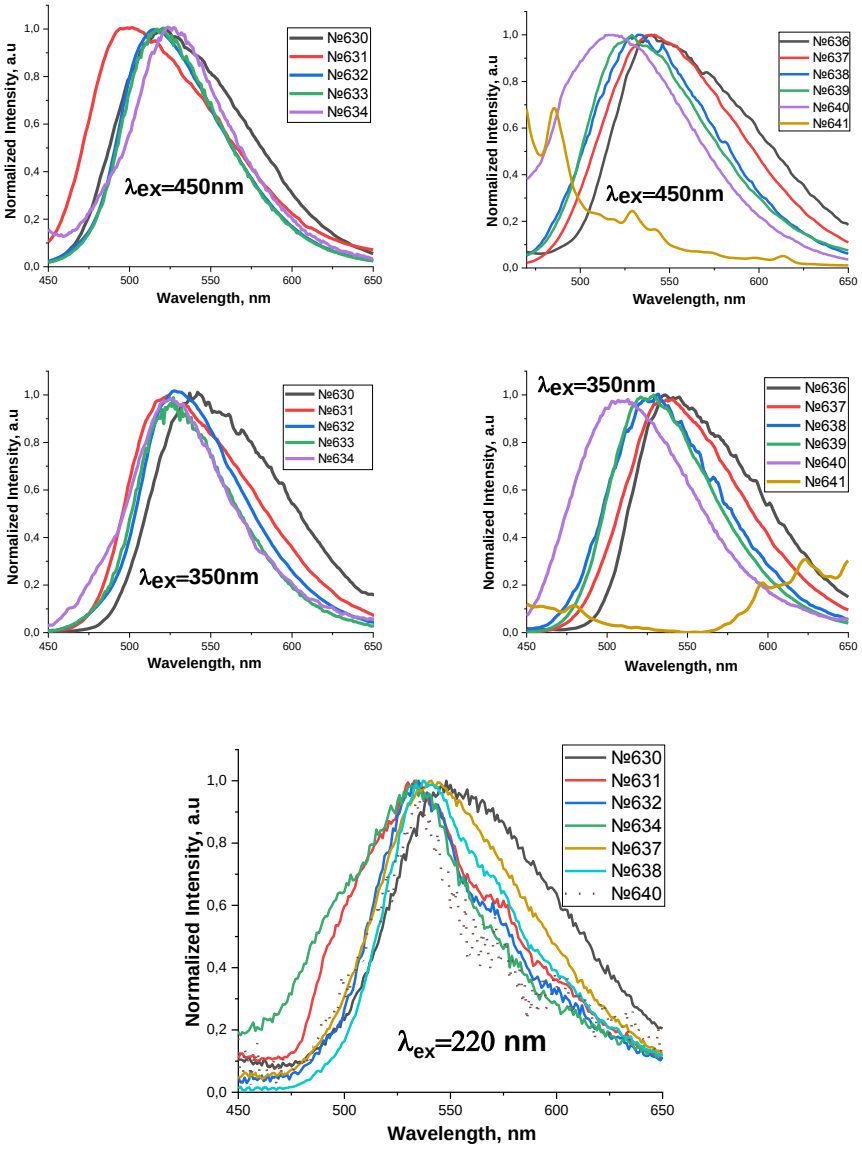

Figure 4. Luminescence spectra of powders from ceramics 630–634 and 636–540 upon excitation by UV radiation in the region of 450, 350 and 220 nm

Quantitative comparison of spectral characteristics: band positions and their half-widths are summarized in Table 2. We have intentionally provided this extensive information in table form, but not in the compact form of graphs. We believe that quantitative information in table form is more important.

Table 2

Spectral characteristics of luminescence

Initial materials for synthesis		ITO, F800, K1					K6, K7, K1				
No. Sample		630	631	632	633	634	636	637	638	639	640
Ratio Al/Ga		5/0	4/1	3/2	2/3	1/4	5/0	4/1	3/2	2/3	1/4
λ_{ex}=450 nm	λ_{em} nm	521	501	516	521	525	540	540	534	529	517
	ΔE, eV	0,416	0,444	0,331	0,313	0,321	0,375	0,368	0,356	0,35	0,412
λ_{ex}=340 nm	λ_{em} nm	543	525	530	529	525	537	540	532	530	510
	ΔE, eV	0,382	0,389	0,306	0,296	0,314	0,363	0,356	0,466	0,328	0,404
λ_{ex}=220 nm	λ_{em} nm	549	535	534	-	535	-	541	537	-	535
	ΔE, eV	0,381	0,375	0,29		0,389		0,354	0,288		0,245

It follows from the results presented in Figure 4 and Table 2 that with a change in the Al/Ga ratio in the ceramic composition, there is a shift in the band position and in their half-width. It is significant that the bandshapes and their position generally do not depend on the wavelength of UV excitation. We emphasize that the studies results of the luminescence spectra dependence on the Al/Ga ratio in the ceramic composition are in good agreement with the regularities obtained in [18] in the crystals grown study by the thermal method. This means that the ceramic samples obtained by us by the radiation express method from Y_2O_3, Al_2O_3, Ga_2O_3, Ce_2O_3 powders have the same crystalline structure as those obtained by the thermal method.

For luminescent materials, not only qualitative and spectral properties are important. Information on quantitative properties is no less necessary: the value of the luminescence flux. The most correct way to assess this value are measurements of energetic (or light) brightness. Brightness is the value of the radiation flux from a unit of the emitter surface in a single solid angle.

The luminescence brightness of the phosphor depends on the excitation conditions and the direction of measurement. However, with an unchanged mutual arrangement of all excitation sources, sample, brightness meter and unchanged excitation modes, it is possible to measure the relative brightness of a series of samples. Moreover, it can be shown that when these conditions are met, the brightness of the surfaces when changing the spectra in the range of 520–545 nm, the measurement error does not exceed 5–7 %. When the specified conditions are met, the brightness of the powder surfaces is proportional to the luminous fluxes of the samples.

During photoexcitation, the brightness value reflects the probability of energy transfer to the luminescence center. In the study of luminescence dependence YAGG: Ce ceramics on the Al/Ga ratio in its ceramic composition, relative brightness measurements are of interest.

To measure relative brightness values, a stand was created based on the CS-200 brightness meter with a constant mutual placement of all stand elements. Samples in cuvettes were placed strictly in the same place. Measurements of relative brightness values of samples were performed relative to the brightness of the SDL 4000 phosphor. Standard LED modules with radiation in the range of 450 and 340 nm were used as excitation sources.

Measurement results of relative brightness values $L_{отн} = L_{cer}/L_{SDL}$, where L_{cer}, L_{SDL} — brightness of the ceramics and the standard are shown in Figure 5 in the form of histograms.

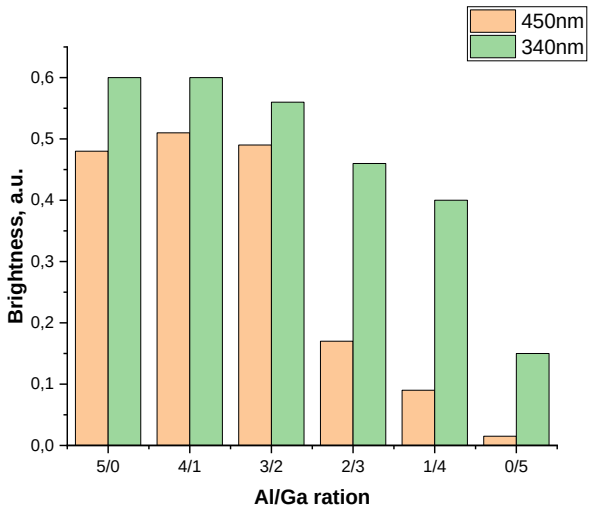

Figure 5. Dependence of relative brightness of ceramic samples on Al/Ga ratio

The dependence of the relative brightness of ceramic samples on the Al/Ga ratio under excitation in the 450 nm region qualitatively corresponds to the regularity given in [18]. With a decrease in the Al/Ga ratio, the width of the forbidden band decreases 5d the level of the Ce ion appears near the bottom of the conduction zone, passes into the conduction zone. The probability of radiative transition decreases sharply [19].

The difference of this pattern from that measured under excitation by radiation in the 340 nm region seems unexpected. Among the most probable reasons, in our opinion, is the following. The two excitation

bands at λmac=460 and 340 nm are due to transitions $4F_{5/2}\rightarrow 5D_0$, $5D_1$, the broad luminescence band at λmac=520 and 580 nm is due to transitions $5D_0\rightarrow 4F_{5/2}$, $4F_{7/2}$ in Ce ions [20]. It is known that modification (so doping) affects the electronic structure of the luminescence centers [21, 22]. The position of 5d levels relative to 4f and cleavage changes with the change in the structure of the environment [23]. This manifests itself in a change in the appearance of the excitation and luminescence spectra and possibly leads to a change in the probability of radiative transitions.

Conclusion

We emphasize that the study results of the luminescence spectra dependence on the Al/Ga ratio in the composition of ceramics correspond well to the regularities obtained in [Zorenko, 2012 or 2014] when studying crystals grown by the thermal method. This indicates that the ceramic samples obtained by the radiation express method have the same crystal structure as the crystals grown by traditional thermal approaches. These results confirm the versatility and reliability of the radiation method of synthesis, opening new horizons for further research in the field of luminescence and creation of high-quality materials.

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Әр түрлі Al/Ga қатынасы бар YAGG сәулелену өрісінде синтезделген люминофордың фотолюминесценциясының спектрлік сипаттамалары

Мақалада әртүрлі бастапқы материалдардың қоспасын сәулелендіру арқылы синтезделген әртүрлі Al/Ga қатынасы бар керамиканың люминесценция және қозу спектрлерін зерттеу нәтижелері келтірілген. Синтез үшін $Y_3Al_xGa_{5-x}O_{12}$: Ce стехиометриялық құрамы бар оксид ұнтақтарының қоспалары дайындалды, мұндағы x 0-ден 5-ке дейін өзгереді. Барлық бастапқы материалдар аналитикалық материалдардан төмен емес тазалыққа ие болды. Синтез энергиясы 1,4 МэВ және қуат тығыздығы 20 кВт/см^2 болатын электронды сәуленің әсерінен жүргізілді. Синтез реакциясының шығымы — үлгінің массасының қоспаның массасына қатынасы — 90–95% құрады. Зерттеулер көрсеткендей, Al/Ga қатынасының төмендеуі люминесценция жолағының спектрдің қысқа толқынды аймағына ауысуына әкеледі. Сонымен қатар, қозу спектрі де өзгереді. Al/Ga қатынасы төмендеген кезде қозу жолағы шамамен 350 нм ұзын толқынды аймаққа ауысады. Люминесценцияның сипаттамаларына бастапқы материалдардың тарихы әсер етеді.

Кілт сөздер: Al/Ga қатынасы, люминесценция, радиациялық синтез, жоғары энергиялы электронды сәуле.

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Спектральные характеристики фотолюминесценции синтезированных в поле радиации YAGG люминофоров с различным соотношением Al/Ga

В статье представлены результаты исследования спектров люминесценции и возбуждения люминесценции керамики с различным соотношением Al/Ga, синтезированной путем облучения смеси исходных материалов с различной предысторией. Для синтеза были подготовлены смеси порошков оксидов со стехиометрическим составом, соответствующим $Y_3Al_xGa_{5-x}O_{12}$: Ce, где *x* варьируется от 0 до 5. Все исходные материалы имели чистоту не ниже аналитической. Синтез проводился под воздействием электронного пучка с энергией 1,4 МэВ и плотностью мощности 20 кВт/см^2. Выход реакции синтеза — отношение массы образца к массе смеси — составил 90–95 %. Исследования показали, что уменьшение соотношения Al/Ga приводит к смещению полосы люминесценции в коротковолновую область спектра. Кроме того, изменяется спектр возбуждения. При уменьшении соотношения Al/Ga полоса возбуждения смещается в длинноволновую область около 350 нм. На характеристики люминесценции влияет предыстория исходных материалов.

Ключевые слова: соотношение Al/Ga, люминесценция, радиационный синтез, высокоэнергетический электронный пучок.

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