FORMATION OF LUMINESCENT COATINGS WITH THE USE OF LOW MELTING GLASS

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The aim of the present research is to develop compounds and technologies for applying the luminous vitrocrystalline compounds to smooth and ribbed glass substrates in LED lamp structure with a remote photoconverter that contains oxide low melting glass as the base and nanostructured cerium–doped yttrium–aluminum garnet powder as the luminophore. The luminescent coatings based on nanostructured cerium doped yttrium-aluminum garnet powder and low melting glass of composition BaO-Bi_2O_3-B_2O_3-K_2O-SiO_2 where prepared. YAG:Ce^{3+} powder was synthesized by the method of thermochemical reaction. Silica glass powder was introduced into the composition as diffuse scattering component, improving the lighting characteristics. Spectral-luminescent characteristics of coatings were studied.

Keywords: light converter. LED, luminophor, coatings, low melting glass, silica glass, yttrium-aluminum garnet

ФОРМИРОВАНИЕ ЛЮМИНЕСЦЕНТНЫХ ПОКРЫТИЙ С ИСПОЛЬЗОВАНИЕМ ЛЕГКОПЛАВКИХ СТЕКОЛ

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Целью настоящего исследования является разработка составов и процесса нанесения люминофорной стеклокристаллической композиции на стеклянные подложки в конструкции светодиодного светильника с удаленным фотопреобразователем, содержащей в качестве основы оксидное легкоплавкое стекло, а в качестве люминофора – наноструктурированный порошок иттрий-алюминиевого граната, активированный ионами церия. Изготовлены люминесцентные покрытия на основе наноструктурированного порошка иттрий-алюминиевого граната, допированного ионами церия и легкоплавкого стекла на основе ситстемы BaO-Bi_2O_3-B_2O_3-K_2O-SiO_2. Порошок YAG:Ce^{3+} синтезирован методом термохимической реакции. Для улучшения световых и спектрально-люминесцентных характеристик покрытий в состав вводили диффузно-рассеивающий компонент – порошок кварцевого стекла. Изучены спектрально-люминесцентные характеристики покрытий.

Ключевые слова: светопреобразователь, светодиод, люминофор, покрытия, легкоплавкое стекло, кварцевое стекло, иттрий-алюминиевый гранат

OSON ERUVCHAN SHISHALARDAN FOYDALANGAN HOLDA LYUMINESCENT QOPLAMALARNI TAYYORLASH

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Ushbu tadqiqotning maqsadi masofaviy fotoo'zgartirgichli nurdiodli chiroq konstruksiyasidagi shisha asosga lyuminofor shishakristall, ya'ni asos sifatida oson eruvchan shihsa va lyuminofor sifatida seruy ionlari bilan faollashtirilgan itriy-alyuminiy granati saqlovchi nanotuzulishli kukun kompozitsiyalarni qoplash jarayoni va tarkiblarini ishlab chiqishdan iborat. BaO-Bi_2O_3-B_2O_3-K_2O-SiO_2 tizimi asosida oson eruvchan shisha va seriy qo'shilgan itriy-alyuminiy granat nanostrukturali kukuni asosida lyuminessent qoplamalar tayyorlangan. YAG:Ce^{3+} kukuni termokimyoviy reaksiya usuli bilan sintez qilindi. Yorug'lik va spectral-lyuminessent xususiyatlarini yaxshilash uchun tarkibga diffuzion sochuvchi komponent sifatida kvarts shisha kukuni kiritildi. Qoplamalarning spektral-lyuminessent xarakteristikalari o'rganilai.

Kalit so'zlar: yorug'lik konvertori, LED, fosfor, qoplamalar, erituvchi shisha, kvarts shishasi, itriy alyuminiy granatasi

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Introduction

LED lamps are widely used due to their cost–effectiveness, energy efficiency as compared to incandescent lamps, and have a long service life [1–3]. Such lighting devices are based on white light–emitting diodes (LEDs) of various shades – from pure white (cold light) to pinkish yellow (warm light). The most common method of producing white LEDs is the use of microcrystalline luminophores that convert light emitted by blue chips into a wide spectrum corresponding to solar spectrum with the use of microcrystalline luminophores manufactured based on cerium–doped yttrium–aluminum garnet (YAG:Ce^{3+}) [4–6]. The technology for creating white LEDs involves applying a ready compound consisting of a luminophore, an organic binder and stabilizing additives to the surface of the crystal, after which the structure is heat treated. With this approach due to LED heating during its operation, conversion efficiency and brightness of LED decrease over time; spectral composition of light emission also changes over time. There are several publications that suggest that the luminophore should be put away at some distance from the chip, which would reduce the thermal degradation of the light converting layer, increase the lifespan of LED device, increase homogeneity of light emission and improve the lamp light emission pattern [10–12]. Such converter can be manufactured in form of semi-translucent composite or ceramic plate, and luminescent coating on transparent substrate [13, 14]. Besides, this approach can also offer a structural variety of forms of the remote converter – flat, hemispherical, spherical, and at the same time allows the increase of lighting device efficiency due to the structural features by 25–30% [15].

The aim of the present research is to develop compounds and technologies for applying the luminous vitrocrystalline compounds to smooth and ribbed glass substrates in LED lamp structure with a remote photoconverter that contains oxide low melting glass as the base and nanostructured cerium–doped yttrium–aluminum garnet powder as the luminophore.

Research methods

The following requirements are applied to low melting glass as the matrix for introduction of ultradispersed luminophore powder for transparent glass substrates coating:

i) glass-softening point is 450–530 °C;

ii) the temperature of complete spreading over the glass substrate during heat treatment is not higher than 600–650 °C;

iii) thermal expansion coefficient is (80–100)·10^{-7} K^{-1};

iv) chemical resistance is not lower than III class.

v) refractive index is not less than 1.6.

The low melting glass compositions offered in the scientific literature in most cases are multi-lead or boron-zinc systems that do not ensure achieving required properties in terms of thermal linear expansion coefficient values or refractive index. Therefore, accordingly, a compound is proposed as low melting glass with a melting point below 600 ◦C, wt.%: B_2O_3 15.65–19.30; ZnO 0.2–1.0; Al_2O_3 0.05–4.13; Bi_2O_3 23.18–53.38; CdO 0.1 –1.5; CuO 0.01–1,5; NiO 0.01–0.5; PbF_2 29.27–58.01[16]. The disadvantages of this glass are the use of toxic and volatile PbF_2 and relatively low refractive index (less than 1.6). The following compositions can be given as example of lead-free boron-zinc glass [17]. The latter are developed in $ZnO–B_2O_3–P_2O_5$ system, which are main components, and Al_2O_3, MgO, CaO, BaO introduced as optimizing additive. The content of the primary components is 85 wt.% and more. The limit of the primary components content (wt.%) is ZnO 45–80; B_2O_3 5–45; P_2O_5 1.0–35; optimizing: Al_2O_3 0–10; MgO 0–15; CaO 0–10; BaO 0–5 (total 15 or less). The disadvantage of this glass is the introduction of significant amounts of P_2O_5 which is a highly volatile component, and the low refractive index values. There is also bismuth-containing low melting glass [18], wt.%: Bi_2O_3 50–65; B_2O_3 25–40; Al_2O_3 1,5; ZnO 5,0; Sb_2O_3 3,5; BaO 5–15 used for pastes in microelectronics. Their disadvantages are the significant content of bismuth oxide that causes active colouring of original glass, which significantly increases absorption of the passing light.

It has been established that the introduction of BaO and CaO into the glass composition increases thermal linear expansion coefficient values of borate glass and contributes to the increase of the original glass meltability in general due to their ability to form meltable calcium-barium borates of compositions $Ca(BO_2)_2–3BaO(BO_2)_2$, $Ca(BO_2)_2–Ba(BO_2)_2$ и $2Ca(BO_2)_2–Ba(BO_2)_2$ with melting temperatures of 1056, 1088 and 1074 °C, respectively [19].

Low melting glass based on bismuth-borate system for light converting coatings that complies with specified requirements, with thermal expansion coefficient values within the range of (85–100)·10^{-7} K^{-1} and the refractive index of at least 1.7 corresponding to the refractive index of luminophore – yttrium aluminum garnet – and provides for spreading on glass substrate at temperature not exceeding 600 ◦C [20]. The glass compositions are, wt.%: Bi_2O_3 25–35; B_2O_3 30–40; ZnO 6-10; BaO 13–16; CaO 2–4; K_2O 7–10.

The raw materials (99.99 % purity) were weighted and homogenized in 50 cm^3 aluminum oxide ceramic crucible. The synthesis was performed at maximum temperature 1100°C in the electric furnace for 1 h. Obtained samples were annealed at 400 °C for 4 h in the muffle furnace. The developed glass compositions have thermal expansion coefficient values consistent with the

thermal expansion coefficient values of the glass substrate and the increased refractive index close to the refractive index of luminophore.

The 2–3 mm-thick -transparent glass plates, both smooth and ribbed, were used as the substrates for coating.

As the luminescent powder forming part of the light converter coating, nanostructured cerium doped yttrium-aluminum garnet powder was used that had been obtained by the thermochemical reaction of oxidation of relevant nitric acid salts in reducing agents (sucrose, citric acid and acetic acid) [21–23]. Method of synthesis of luminescent ultradispersed powder with $Y_{2,95}Ce_{0,05}Al_5O_{12}$ chemical composition, used as active filling agent, is described in the research papers [24–26].

In order to improve light and luminescent spectroscopic parameters of the coatings, a diffuse -dispersing component – silica glass powder with various granulometric composition – was introduced into the composition of the slurry (average particle size from 12 to 400 microns). Silica glass is introduced into the coating as a component that is transparent for blue light emission of the LED chip, and white light is formed by blue passing light being combined with light emission generated by cerium-doped yttrium-aluminum garnet particles. Thick film coating was also applied to the ribbed glass. In this case there is no need to introduce a light diffusion component, because emission of blue LED partially passes through the areas of ribbed surface of the substrates that are not covered by the slurry (Fig. 1, b).

The thickness and the composition of the luminescent compound layer are selected in such a way that blue light emission of LED ($\lambda_{emission}$ = 455 nm) excites broadband radiation in the phosphor with a maximum at a wavelength of 570 nm (yellow glow). Total emission of blue LED and yellow luminophore is perceived by the eye as white light or "warm" white light depending on ratio of amount to size of the silica glass that is transparent to light emission and luminophore composition.

Design of the converter that is made on the ribbed substrate with a layer of luminophore compound applied to the recesses, requires lower consumption of expensive luminescent powder and maintains uniformity of light emission. Uniform distribution of the compound containing luminophore particles based on yttrium-aluminum garnet in recesses of the glass plate ensures stability and uniformity of the luminous flux with its transformation into the broadband light emission with a longer wavelength. Light flux of blue colour uniformly passes through transparent areas of the substrate. Total emission of the blue LED and the yellow luminophore is perceived by the eye as the white or "warm" white light depending on the ratio of transparent areas to recesses filled with luminophore.

Introduction of the luminophore compound only into the recesses of the light transformer significantly reduces the consumption of luminophore while maintaining the uniformity of the luminous flux of the lamp and the uniformity of light emission.

Ground silica glass powder used as diffuse reflection filling agent, was pre-washed and etched in ultrasonic bath UZU-025 at a frequency of 50 Hz, ultrasonic power– 250 W, in 10% NH_4HF_2 aqueous solution for 30 minutes. After the powder is washed, its transparency increases, dust and defects are removed from the grain surface, and the refractive and reflective capacities increases.

Luminophore composition was prepared and applied to the glass substrate. In order to obtain light converting coating, ground frit of low melting glass containing 25 wt.% Bi_2O_3 was mixed in isopropyl alcohol medium with nanostructured cerium-doped yttrium-aluminum garnet powder and the silica glass powder with a ratio of 44,5: 11.0: 44.5 wt.%.

The resulting suspension was sprinkled on the glass substrate and dried in the air at 50◦C in the dryer for 20 minutes. The coated substrate was then placed in a muffle furnace on a ceramic stand and gradually heated to a temperature of 650 °C with a temperature rise rate of 10 °C/min. The substrate was held at 650◦C for 30 minutes, then slowly cooled down with the furnace to the room temperature [24].

Scanning electron microscope TESCAN (Czech Republic) was used to study morphology of the coating.

Luminescence of the YAG:Ce^{3+} based coating with the introduction of silica glass powders was excited in Ce^{3+} ion absorption band area within the blue area, by LED–003W–07C–020–030LM–EL–P with a wavelength in 440–460 nm spectrum, which corresponds to transitions from

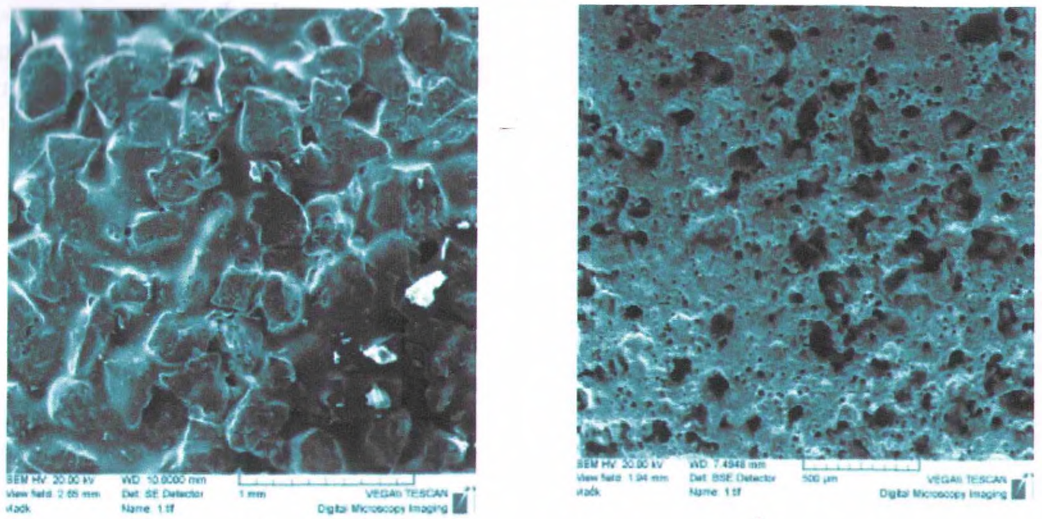


Figure 1. SEM image of phosphor coating containing quartz powder with an average particle size of 400 (a) and 12 microns (b).

$4f^2F_{5/2}$ to 5d split levels.

Luminescence spectra were recalculated using the reference cold-white LED spectrum.

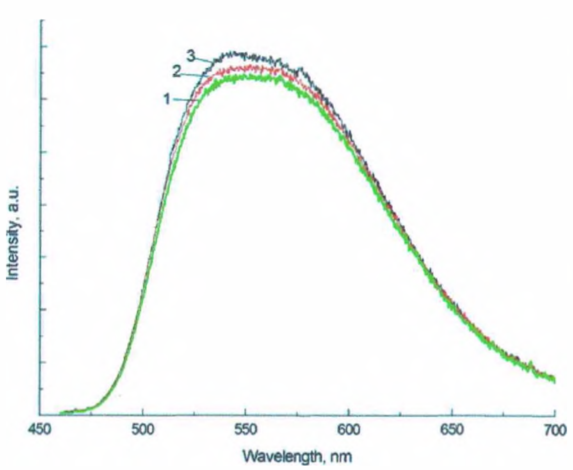
Results and Discussion

SEM image of phosphor coating filled with silica glass powder (average particle size from 12 (a) to 400 microns (b)) in a low melting glass matrix with submicron YAG:Ce powder is shown in Fig.1. Silica glass is introduced into the coating as a component that is transparent to the blue light emission of the LED chip, and white light is generated by mixing blue emission and yellow radiation generated by activated yttrium aluminum garnet particles.

Analysis of the luminescence (Fig. 2) of coatings containing transparent silica particles has proved that highest luminescence intensity has been shown by the luminophore coatings produced with the use of cerium-doped yttrium-aluminum garnet produced by being burnt in citric acid, calcified in the argon atmosphere at a temperature of 1100 °C silica glass powder with an average particle size of 150–400 μm being added as diffusion component.

The measured luminophore emission spectra had a wide band within 500–700 nm, which corresponds to transitions of Ce^{3+} ion from 5d to 4f-shell levels, with the maximum being approximately 550 nm [27]. Luminescence spectra of the samples depending on the particle size of diffuse-dispersing component of silica powder № 1 – 150 μm, № 2 – 252 μm, № 3 – 400 μm is present in Fig. 2. As the size of diffuse-dispersing powder increases, the luminescence intensity increases, too, because total amount of blue LED quanta passing through the composite and excited in yellow luminophore particles increases.

In order to create remote light transducers emitting light in a wide range of colour temperatures (from cold-white to pink), they were formed by combination of two types of luminophores emitting, respectively, yellow-green and red parts of the spectrum. For example, introduction into glass matrices of the red luminophores Y_2O_3: Eu^{3+}, which were obtained by the thermochemical reactions, together with YAG:Ce^{3+} based yellow luminophore, allows us to move light emission band to red, thus obtaining "warm" white color light from the device. In order to achieve this, a combination of two luminophores was prepared with a ratio of 80/20 wt. % (yellow / red). When such a composite is irradiated by LEDs with λ = 455 nm, a combined light emission spectrum with a wide band of 400–700 nm and



(1 – 0.15 mm, 2 – 0.25 mm, 3 – 0.4 mm)
Figure 2. Luminescence spectra of coatings depending on the size of silica glass particles.

an intensive narrow band of 612 nm is obtained, which with the use of the blue light allows to adjust the"warm" white light emission (Fig. 3).

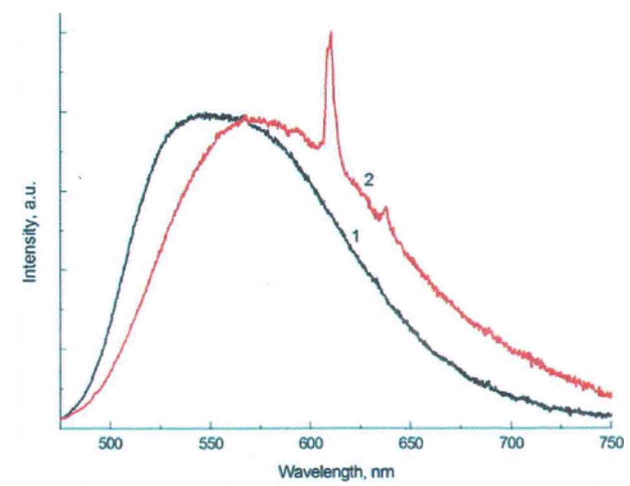


Figure 3. Emission spectrum of a phosphor coatings when irradiated with LED with λ = 455 nm: 1 with YAG:Ce^{3+} luminophore and 2 – combination of two luminophores YAG:Ce^{3+} / Y_2O_3: Eu^{3+} with a ratio of 80/20 wt. % (yellow /red).

The obtained samples of the luminescent coatings consisting of low melting glass with 25 wt.% of Bi_2O_3, nanostructured cerium-doped yttrium-aluminum garnet powder, and silica glass powder taken in ratio of 44.5 : 11.0 : 44.5 wt. % enabled to achieve white light from blue and UV-LEDs, the total light emission spectrum of which is shown in Fig. 4 with corresponding chromaticity coordinates X=0.29–0.36 and Y=0.25–0.38 (Fig. 5).

Conclusion

The composition of luminophore coating on the glass substrates for convertor that transforms light emission of blue LEDs or chips into white light has been developed based on application and firing of composite slurry using low melting bismuth-containing glass and obtained by burning cerium-doped $Y_3Al_5O_{12}$ yttrium-aluminum garnet ultradispersed powder; with additional introduction of light-diffusing silica powder. Highest luminescence intensity at excitation at wavelength λ=460 nm has been achieved by the luminophore coatings obtained with the use of diffuse-dispersing component of silica glass powder with the medium particle size of 400 μm.

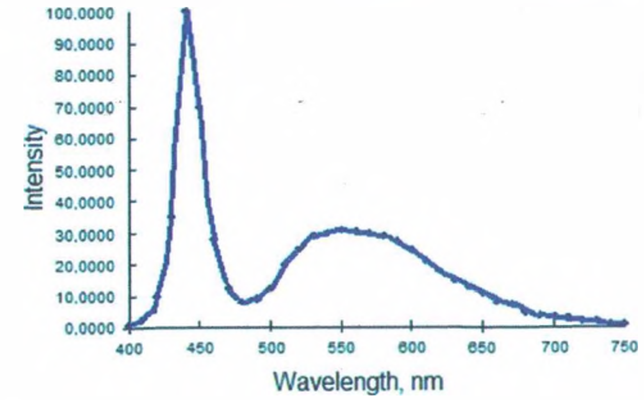


Figure 4. Total spectrum of a blue LED and a composite thick-film light converter.

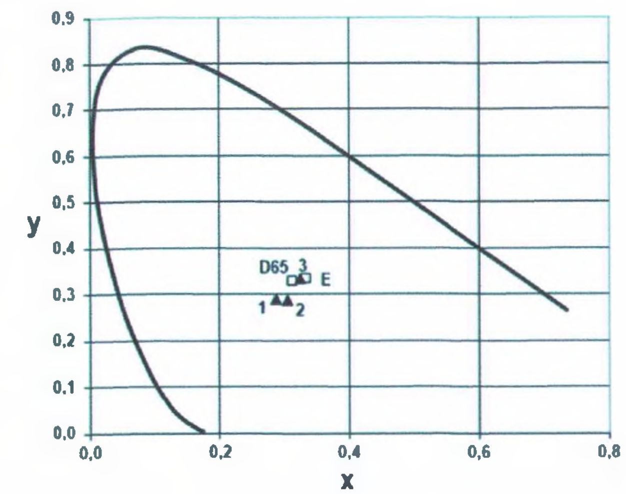


Figure 5. Color diagram for a light converter when irradiated with light from a blue LED with λ = 455 nm.

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