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The aim of the work is to study a new method of thermochemical synthesis of luminescent powder of lutetium-aluminium garnet activated by cerium ions ($Lu_3Al_5O_{12}:Ce^{3+}$) using nitric acid salts of lutetium, aluminium and cerium, and a mixture of acetic acid and polyvinyl alcohol as a propellant. The synthesis process was carried out in a muffle furnace at an initial temperature of 350 ºC under conditions of limited presence of atmospheric air. The average size of the coherent scattering region for $Lu_3Al_5O_{12}:Ce^{3+}$ obtained from the combustion reaction is 60 nm. It was found that during the preparation of nanostructured powder in the reaction vessel the temperature sufficient for the synthesis of $Lu_3Al_5O_{12}$ develops, and the reducing environment arising from the combustion of the organic mixture favours the formation of cerium ions in the tricharged state. The emission spectrum of the obtained $LuAG:Ce^{3+}$ luminophore powders shows a broad band due to the energy transitions of the Ce^{3+} ion in the garnet structure, between 500 and 650 nm under excitation with a wavelength of 450 nm, which corresponds to the green emission colour.

Keywords: lutetium-aluminium garnet, cerium ions, nanostructure, sol-gel burning

ОСОБЕННОСТИ ПОЛУЧЕНИЯ НАНОСТРУКТУРИРОВАННЫХ ПОРОШКОВ ЛЮТЕЦИЙ-АЛЮМИНИЕВОГО ГРАНАТА, ЛЕГИРОВАННОГО ЦЕРИЕМ МЕТОДОМ ЗОЛЬ-ГЕЛЬ-ГОРЕНИЕ

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Цель работы – изучение нового способа термохимического синтеза люминесцирующего порошка лютеций-алюминиевого граната, активированного ионами церия ($Lu_3Al_5O_{12}:Ce^{3+}$) с использованием азотнокислых солей лютеция, алюминия и церия, а в качестве горючего – смеси уксусной кислоты и поливинилового спирта. Процесс синтеза осуществляли в муфельной печи при начальной температуре поджига 350 ºС в условиях ограниченного присутствия атмосферного воздуха. Средний размер области когерентного рассеяния частиц $Lu_3Al_5O_{12}:Ce^{3+}$, полученного в результате реакции горения, составляет 60 нм. Установлено, что при получении наноструктурированного порошка в реакционном сосуде развивается температура, достаточная для синтеза $Lu_3Al_5O_{12}$, а восстановительная среда, возникающая при горении органической смеси, способствует формированию ионов Ce в трехзарядном состоянии. Спектр излучения полученных порошков люминофора $LuAG:Ce^{3+}$ демонстрирует широкую полосу, обусловленную энергетическими переходами иона Ce^{3+} в структуре граната, между 500 и 650 нм при возбуждении длиной волны 450 нм, что соответствует зеленому цвету излучения.

Ключевые слова: лютеций-алюминиевый гранат, ионы церия, наноструктура, золь-гель горение

ZOL-GEL-YONISH USULIDA SERIY BILAN LEGIRLANGAN NANOTUZILISHLI LUTETSIY-ALYUMINIY GRANAT KUKUNLARINI ISHLAB CHIQARISH XUSUSIYATLARI

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Ishning maqsadi seriy ionlari ($Lu_3Al_5O_{12}:Ce^{3+}$) bilan faollashtirilgan lyutetsiy-alyuminiy granatining lyuminestsent kukunini lyutetsiy, alyuminiy va seriyning nitrat tuzlari hamda yoqilg‘i sifatida sirka kislotasi va polivinil spirti aralashmasidan foydalangan holda termokimyoviy sintez qilishning yangi usulini o‘rganishdan iborat. Sintez jarayoni atmosfera havosi cheklangan sharoitda 350 ºC boshlang'ich olov haroratida muffeL pechida amalga oshirildi. Yonish reaksiyasi natijasida olingan $Lu_3Al_5O_{12}:Ce^{3+}$ zarralarining kogerent sochilish hududining o‘rtacha kattaligi 60 nm. Reaksiya idishida nanostrukturali kukunni ishlab chiqarish jarayonida $Lu_3Al_5O_{12}$ sintezi uchun etarli harorat hosil bo'lishi va organik aralashmaning yonishi paytida paydo bo'ladigan qaytaruvchi muhit uch marta zaryadlangan holatda Ce ionlarining shakllanishiga yordam berishi aniqlandi. Olingan $LuAG: Ce^{3+}$ luminofor kukunlarining emissiya spektri yashil emissiya rangiga mos keladigan 450 nm to'lqin uzunligi bilan qo'zg'alish ostida 500 dan 650 nm gacha bo'lgan granat strukturasidagi Ce^{3+} ionining energiya o'tishlari tufayli keng diapazonni ko'rsatadi.

Kalit ***so'zlar****: lutetsiy alyuminiy granati, seriy ionlari, nanostruktura, zol-gel yonishi*

Introduction

Materials with garnet crystal structure are widely used in various optical devices – lasers, luminescent radiation converters, scintillators [1 – 4].

Lutetia are characterised by high temperature stability of luminescence and low temperature quenching effects compared to other luminophores [5]. In addition, high density and radiation stability of lutetium-aluminium garnet-based luminophores make them promising for application in detectors of various types of ionising radiation-scintillators [6–10].

Another application of garnet-based phosphors is LED-based lighting, which utilises the effect of converting blue light from light-emitting diodes (LEDs) into white light using luminophore. Most commonly garnet-based microcrystalline powders activated with cerium ions ($Y_3Al_5O_{12}:Ce^{3+}$) are used for this purpose [11–13].

Recently, a new direction in LED lighting has appeared – creation of transducers (converters) of LED and laser diode radiation into white light remote from the LED. Such converters can be made on the basis of luminescent garnet powders distributed in glass or transparent crystalline matrices [14–17].

Blue laser diodes (BLD), due to their high electro-optical conversion efficiency, high directivity and small size are promising in the field of sol-state lighting. BLDs as part of fluorescent converters are expected to replace LEDs in displays, automobile headlights and other advanced lighting applications. Of greatest interest is the use of powders based on lutetium-aluminium garnet activated by cerium, as well as sol-alloyed with transition metals and REE due to its higher luminescence efficiency, short attenuation period and temperature stability [18–20].

Introduction of some amount of transition metals, in particular iron, into the composition of lutetium-aluminium garnets can significantly change the characteristics of such luminophores, in particular, it became possible to change the emission from green to orange. The obtained luminophores had high quantum yield (55–67%), excellent thermal stability of photoluminescence (until 200 °C) and high colour rendering, which makes them promising candidates for high-power light-emitting systems [21].

Replacing part of the aluminium with gallium provides tunable light emission (from green to blue), which increases the flexibility of the luminophore $Lu_3Al_{5-x}Ga_xO_{12}:Ce^{3+}$ application [22].

Luminophore-glass composites are promising for the creation of efficient converters. For example, the composite $Lu_3Al_5O_{12}:Ce^{3+}$ in silica glass (LuAG: Ce-PiSG) made by plasma sintering at a temperature of 1060 °C had high transparency and thermal stability, and the emitters demonstrate high luminosity (298 $lm \cdot W^{-1}$) and luminous flux (1069 lm) [23].

LuAG-based luminescent radiation converter was used as a visible light amplifier for dye-sensitive solar cells [24]. LuAG particles emitting green light with a wavelength from 470 to 650 nm are capable of decomposing dye molecules and can be used in devices for catalytic decomposition of organic substances.

A luminophore converter based on lutetium-aluminium garnet powder $Lu_3Al_5O_{12}:Ce^{3+}$ distributed in a glass matrix was used to create a new lighting module [25]. By increasing the glass thickness from 0.7 to 1 mm, the maximum illuminance increases from 1306 to 1356 lux.

There are several variants of low-temperature synthesis of ultradisperse luminophores: direct synthesis in salt melts, hydroxide coprecipitation, thermochemical synthesis [26–28].

The formation of oxide luminophores based on nitric acid salts combustion processes in various organic compounds is attractive due to its simplicity, low cost, and the possibility of using both thermal and laser energy to ignite the mixture [28, 29].

Colloidal chemistry methods are also used for the synthesis of ultradisperse garnet particles [30, 31]. The paper [32] describes the study of structural and luminescence characteristics of LuAG:Ce obtained by colloidal-chemical method with additional introduction of different amounts of Lu_2O_3 (up to 30 wt.%) and finds that the introduction of Lu_2O_3 nanoparticles in the basic system leads to some suppression of luminescence intensity in LuAG:Ce composites, which is apparently caused by a decrease in the concentration of optically active cerium ions in the LuAG:Ce phase due to the diffusion processes of Ce^{3+} during heat treatment.

Thus, the development of various variants of production of oxide ultradisperse luminophores based on lutetium-aluminium garnet, including

promising for glass-crystalline and ceramic light converters, has an important scientific and practical significance.

The present paper is devoted to the study of a new method for the synthesis of luminescent nanostructured LuAG:Ce^{3+} powder by sol-gel combustion method using nitric acid salts of lutetium, aluminium and cerium, and a mixture of acetic acid and polyvinyl alcohol as a propellant.

Research methods

nm $Lu(NO_3)_3$ $6H_2O$, obtained by dissolving lutetium oxide in nitric acid; $Al(NO_3)_3 \cdot 9H_2O$; $Ce(NO_3)_3 \cdot 6H_2O$; acetic acid (CH_3COOH); polyvinyl alcohol were used as starting materials. All reactants were analytical grade. The amount of dopant – cerium relative to the molecular composition of LuAG was 5.0 at.%.

The technique of thermochemical synthesis of luminescent powder of cerium-doped lutetium-aluminium garnet of $Lu_{2,95}Al_5Ce_{0,05}$ O_{12} includes the following steps. Pour acetic acid into a chemical glass beaker, pour nitrates of lutetium, aluminium and cerium, stir with a glass rod until the sediment completely disappears, add polyvinyl alcohol (PVAL) and stir till complete dissolution, then pour the mixture into a heat-resistant non-metallic container – a porcelain evaporation bowl and put in a chamber drier at a temperature of 80–90 ° C for 6 h to form a gel-like precursor. Then the porcelain bowl with the gel is covered with aluminium foil with holes and placed in a muffle furnace heated to 350 ºC. In the furnace under the influence of rapid heating a rapid chemical reaction of oxidation-reduction takes place, the temperature spontaneously rises to 1200–1300 ºC. Then the sample is kept in the furnace at 650 °C for 2 hours. During this time the bound water is removed and organic residues are burnt out. After the furnace has cooled down, the foamed product is extracted from the porcelain bowl with a bright yellow colour and a greenish tint.

The spongy structure of the foamy product is easily broken down to individual agglomerates and subjected to grinding in a planetary mill in a zirconium dioxide beaker for 15 minutes.

Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) measurements have been performed by Q-1500D derivatograph (Hungary). The heating range was up to 1000 °C.

The crystal structure was investigated by X-ray diffraction (XRD) measurements using diffractometer DRON–7.

SEM/TEM measurements have been performed by Vega II LSH microscope, Tescan and JEM 2100 transmission microscope (JEOL, Japan).

Luminescence spectra were registered with spectrofluorimeter Fluorolog-3, HORIBA Scientific, USA (λexc. is 450 nm).

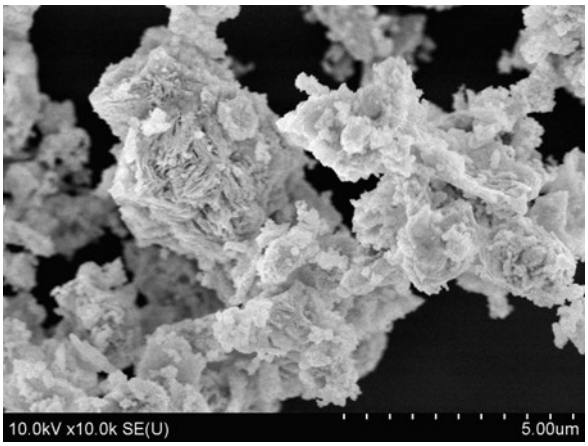
Results and Discussion

The products of interaction of nitrate lutetium, aluminium and cerium with acetic acid are chelate acetatonitrate complexes, which are formed when the mixture is heated in an evaporating porcelain cup in a muffle furnace (using the formation of lutetium oxide as an example) [33]:

$$Lu(NO_3)_3 \cdot 6H_2O + 3(1 - x)CH_3COOH = Lu(NO_3)_{3x}(CH_3COO)_{3(1-x)}aq$$

Upon heating the gel-like precursor, redox reactions between nitrate and acetate groups are initiated, which leads to the ignition of acetatonitrates. During the use of a mixture of nitrates, acetic acid and PVAL a temperature sufficient for the synthesis of the $Lu_3Al_5O_{12}$ compound and the incorporation of cerium ions into the crystal lattice of garnet develops, and combustion of the organic mixture in nitric acid salts in a limited-air environment promotes the formation of Ce ions in the tricharge state, providing bright yellow-green luminescence. Figure 1 shows the SEM-images of agglomerated powder before it is ground.

Studies of powder morphology using scanning electron microscopy showed that LuAG:Ce^{3+} powders calcined in air are agglomerated ones. The size of agglomerates is 2–5 μm. The morphology



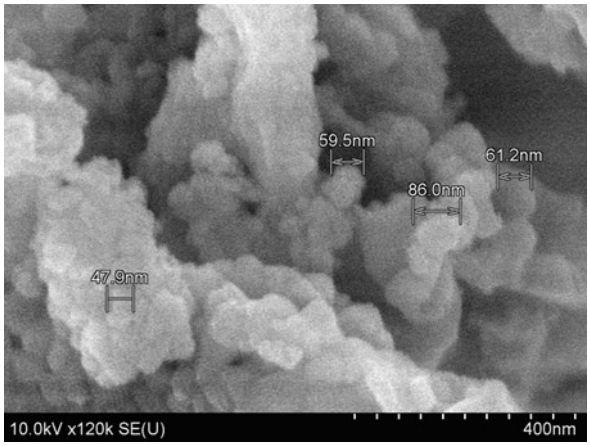


Figure 1. SEM-images of LuAG:Ce^{3+} powder (650 ºC, 1 h).

of agglomerates is characterised by the presence of a large number of pores, voids formed during combustion due to the release of gases.

The sizes of primary powder particles obtained from the combustion process followed by calcination at 650 ºC are within 47.9–86 nm.

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The sizes of primary powder particles obtained from the combustion process followed by calcination at 650 ºC are within 47.9–86 nm.

The average particle size of the powder obtained after calcination at 650 ºC, assessed by the value of broadening of the X-ray maximum for the most intense band is 60 nm, and when the powder is calcined in air at 1200 ºC, a crystalline phase with a particle size of 120 nm is formed.

Diffractograms of LuAG:Ce^3 powder samples obtained after calcination at 650 ºC and 1200 ºC for 1 h (Fig. 2) prove the formation of well-crystallised $Lu_3Al_5O_{12}$ phase (PDF#73-1368, ICDD database product). There are also reflexes belonging to the lutetium oxide Lu_2O_3 phase (PDF# 00-012-0728), apparently formed during the incomplete passage of the garnet synthesis reaction, which can affect the optical characteristics by reducing the luminescence intensity [32].

To clarify the nature of physicochemical processes taking place in the reaction vessel at increasing temperature, a differential thermal analysis (DTA) and thermogravimetric analysis (TGA) of a suspension of the initial gel obtained after evaporation of the mixture of initial components without addition of PVAL, the combustion of which can shield the processes of formation and decomposition of acetonitrate complexes was carried out.

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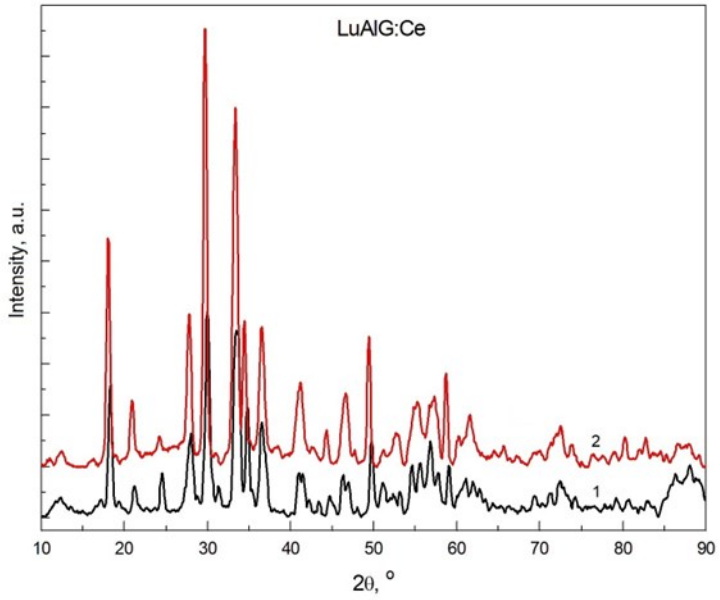


Figure 2. Diffractograms of LuAG:Ce^3 powder samples obtained after calcination at 650 ºC and 1200 ºC.

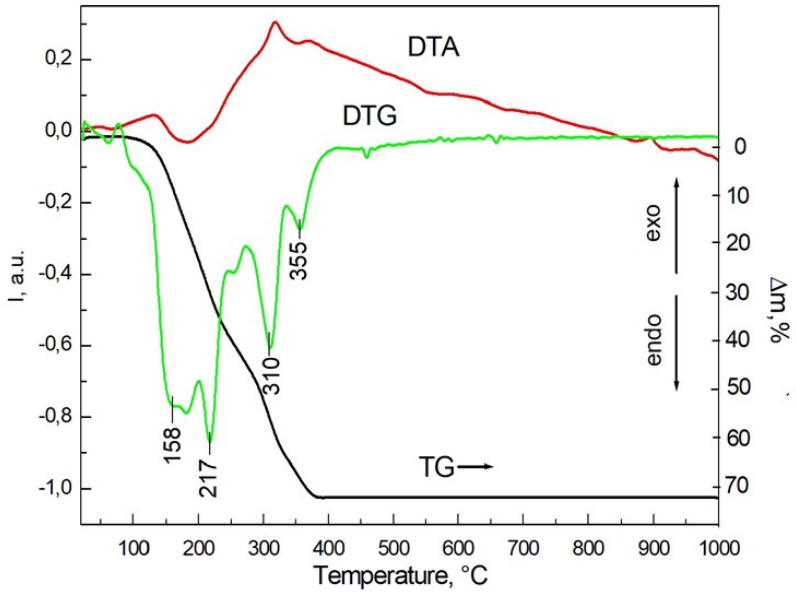


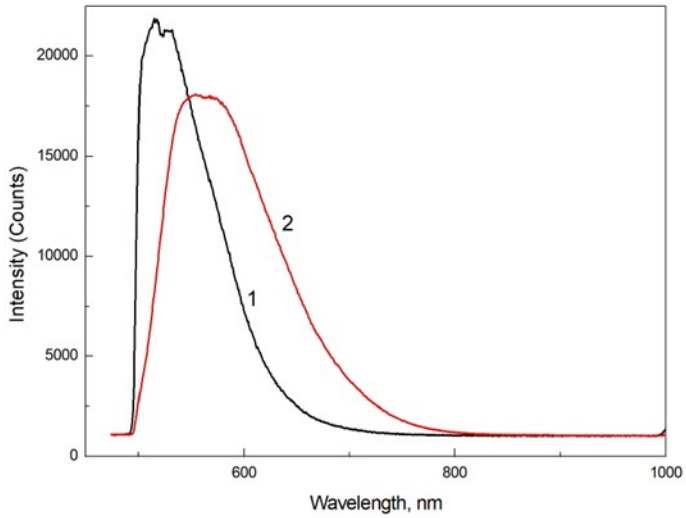
Figure 3. DTA of the heating process of a gel mixture of nitrate salts and acetic acid.

decomposition of acetonitrate complexes was carried out.

There are four endothermic peaks on the curves of mass loss during thermal treatment of the gel mixture in the range of 20–1000 °C (fig. 3). The first peak up to 180 °C is associated with partial dehydration of the gel, the next peak at 217 °C is probably caused by melting of the acetate-nitrate complex. The mass loss at temperatures above 180 °C is due to both the continued dehydration of the sample and the beginning of decomposition of nitrate groups to nitrogen oxide and oxygen. The exothermic peak at 320 °C reflects the beginning of interaction of the released oxidant with acetate groups, but the combustion reaction is superimposed on the decomposition reactions of acetate groups.

The mass of the samples decreases sharply, and after 380 °C remains practically unchanged, which proves the absence of thermal effects up to the temperature of 1000 °C. The mass loss of the gel mixture is more than 90%. At the same time, no phase transformations were detected, which indicates the complete transformation of the complex into lutetium-yttrium garnet with a cubic crystal lattice. Thus, derivatographic studies confirm the formation of crystalline phases at 320–380 ºC, the main of which is LuAG:Ce^3.

For comparison, the spectral curves of LuAG:Ce^{3+} and YAG:Ce^{3+} powders obtained with an equal amount of doping ion (5.0 at.%) are shown in Fig.4.



(1 – LuAG, 2 –YAG)
Figure 4. Luminescence spectra of powdered LuAG:Ce and YAG:Ce samples, λ_{ex} = 450 nm.

It is known that the Ce^{3+} ion is located in the Lu_2O_3 or Y_2O_3 sublattice, partially replacing the matrix ions in the 3- and 4-charge state. However, the Ce^{4+} ion is optically inactive and does not form the corresponding levels in the energy pattern. In the emission spectrum of the LuAG:Ce + Lu_2O_3 luminophore, the broad band due to energy transitions of the Ce^{3+} ion in the garnet structure is located between 500 and 650 nm and corresponds to green emission, and the luminescence intensity for LuAG:Ce^{3+} + Lu_2O_3 is higher than for YAG:Ce^{3+} obtained by thermochemical synthesis [35].

Conclusion

Novel method has been developed for the thermochemical synthesis of cerium-doped lutetium -aluminium garnet ($Lu_3Al_5O_{12}$:Ce^{3+}) luminescent powder with the use of nitrates of lutetium, aluminum and cerium, and a mixture of acetic acid and polyvinyl alcohol as fuel. The synthesis process was performed in a in a muffle furnace, in a porcelain crucible covered with perforated aluminum foil.

Pursuant to X-ray diffraction it was determined that the average size of the coherent scattering region of particles for LuAG:Ce obtained in and calcined at 650 °C is 60 nm, and 120 nm when the powder is calcified in the air at 1200 ºC. The reaction cup reaches a temperature sufficient for synthesis of $Lu_3Al_5O_{12}$ compound and penetration of cerium ions into the structure of the garnet in a three-charge state.

The calcination of the powder at 650 and 1200 ºC in the air leads to formation of two-phase structure. Small amount of the impurity phase Lu_2O_3 does not lead to a significant change in the spectral-luminescent characteristics. LuAG:Ce + Lu_2O_3 luminophore emission spectrum, when excited by a 450 nm LED, gives a wide band between 500 and 650 nm and corresponds to green emission. The luminescence intensity for LuAG:Ce + Lu_2O_3 is higher than for YAG:Ce^{3+} obtained by thermochemical synthesis.

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