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Eygeniy N. PODDENEZHNY

Sukhoi State Technical University of Gomel, Gomel, Belarus, podd-evgen@yandex.ru

Andrei A. BOIKO

Sukhoi State Technical University of Gome, Gomel, Belarus, boiko@gstu.by

Ekaterina E. TRUSOVA

Belarusian State Technological University, Minsk, Belarus, trusova@belstu.by

Ekaterina R. KRAVCOVA

Belarusian State Technological University, Minsk, Belarus, katak1623@gmail.com

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SYNTHESIS OF NANOSTRUCTURED CERIUM-DOPED YTTRIUM-ALUMINUM GARNETS BY METHOD OF SOL-GEL BURNING IN A MICROWAVE OVEN

Eygeniy N. PODDENEZHNY¹ (podd-evgen@yandex.ru)

Andrei A. BOIKO¹ (boiko@gstu.by)

Ekaterina E. TRUSOVA² (trusova@belstu.by)

Ekaterina R. KRAVCOVA² (katak1623@gmail.com)

¹Sukhoi State Technical University of Gomel, Gomel, Belarus

²Belarusian State Technological University, Minsk, Belarus

The aim of research is to study the new method of thermochemical synthesis of yttrium-aluminum garnet luminescent powder, cerium doped ($Y_3Al_5O_{12}:Ce^{3+}$, YAG:Ce) using the nitric acid salts of yttrium, aluminum and cerium, with the mixture of carbamide and hexamethylenetetramine used as fuel. Synthesis was performed in a microwave oven with the power of 800 W and frequency of 2450 MHz in a porcelain crucible closed with a perforated heat-resistant cover. The average size of coherent scattering region (CSR) of YAG:Ce particles (nominal diameter D) obtained in the crucible during the microwave heating is 50.3 nm. It has been established that when YAG powder is produced by burning, the reaction cup reaches the temperature sufficient for $Y_3Al_5O_{12}$ compound synthesis, and that the reducing environment arising due to the combustion of the organic mixture contributes to formation of Ce ions in tricharged state. Further calcination of the resulting powder at the temperature of 450, 900 and 1100 °C in the open air leads to formation of practically single-phase YAG structure. The radiation spectrum of the obtained YAG:Ce luminescent powders has a wide bandwidth caused by the energy transitions of Ce^{3+} ion in the garnet structure between 490 and 750 nm at LED excitation at 450 nm. It corresponds to yellow-green radiation that with blue LED radiation creates the sensation of white light. The highest intensity of the yellow-green luminescence is reached by the YAG:Ce powders calcinated at 900 °C in the open air.

Keywords: yttrium aluminum garnet, cerium ions, nanostructure, sol-gel burning

СИНТЕЗ НАНОСТРУКТУРИРОВАННОГО ИТТРИЙ-АЛЮМИНИЕВОГО ГРАНАТА, АКТИВИРОВАННОГО ИОНАМИ ЦЕРИЯ МЕТОДОМ ЗОЛЬ-ГЕЛЬ-ГОРЕНИЕ В СВЧ-ПЕЧИ

Евгений Н. ПОДДЕНЕЖНЫЙ¹ (podd-evgen@yandex.ru)

Андрей А. БОЙКО¹ (boiko@gstu.by)

Екатерина Е. ТРУСОВА² (trusova@belstu.by)

Екатерина Р. КРАВЦОВА² (katak1623@gmail.com)

¹Гомельский государственный технический университет, Гомель, Беларусь

²Белорусский государственный технологический университет, Минск, Беларусь

Цель работы – изучение нового способа термохимического синтеза люминесцирующего порошка иттрий-алюминиевого граната, активированного ионами церия ($Y_3Al_5O_{12}:Ce^{3+}$, YAG:Ce) с использованием азотнокислых солей иттрия, алюминия и церия, а в качестве горючего – смеси карбамида и гексаметиленetetрамина. Процесс синтеза осуществляли в СВЧ-печи при мощности 800 Вт и частоте 2450 МГц в фарфоровом тигле, закрытом перфорированной термостойкой крышкой. Средний размер области когерентного рассеяния (ОКР) частиц (условный диаметр D) для YAG:Ce, полученного в тигле при СВЧ – нагреве, составляет 50,3 нм. Установлено, что при получении порошка YAG:Ce методом горения в реакционном сосуде развивается температура, достаточная для синтеза соединения $Y_3Al_5O_{12}$, а восстановительная среда, возникающая при горении органической смеси, способствует формированию ионов Ce в трехзарядном состоянии. Дальнейшее прокаливание полученного порошка при температурах 450, 900 и 1100 °C на воздухе приводит к формированию практически однофазной структуры YAG. Спектр излучения полученных порошков люминофора YAG:Ce демонстрирует широкую полосу, обусловленную энергетическими переходами иона Ce^{3+} в структуре граната, между 490 и 750 нм при возбуждении светодиодам на 450 нм. Это соответствует желто-зеленому излучению, что вместе с синим излучением светодиода создает ощущение белого света. Наибольшую интенсивность желто-зеленой люминесценции демонстрируют порошки YAG:Ce, прокаленные при 900 °C в воздушной среде.

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

MIKROTO'LQINLI PECHDA ZOL-GEL-KUYDIRISH USULIDA FAOLLASHGAN SERIY IONLARINI YORDAMIDA NANOSTRUKTURALI ITTRIY-ALYUMINIY GRANAT SINTEZI

Eygeniy N. PODDENEJNY¹ (podd-evgen@yandex.ru)

Andrey A. BOIKO¹ (boiko@gstu.by)

Ekaterina E. TRUSOVA² (trusova@belstu.by)

Ekaterina R. KRAVCOVA² (katak1623@gmail.com)

¹Suxoy davlat texnika universiteti Gomel, Gomel, Belarus

²Belarus davlat texnologiya universiteti, Minsk, Belarus

Ishdan maqsad seriy ionlari ($Y_3Al_5O_{12}:Ce^{3+}$, YAG:Ce) bilan faollashtirilgan lyuminestsent ittri-yalyuminiy granat kukunini itriy, alyuminiy va seriyning nitrat tuzlari hamda yozilg'i sifatida karbamid va geksametilentetramin aralashmasi yordamida termokimyoviy sintez qilishning yangi usulini o'rganishdan iborat. Sintez jarayoni mikroto'lqinli pechda 800 Vt quvvatda va 2450 MGts chastotada teshikli issiqlikka chidamli qopqoq bilan yopilgan chimni tigelda amalga oshirildi. Mikroto'lqinli pechda tigel yordamida qizdirish davomida olingan YAG:Ce uchun zarrachalarning koherent sochilish mintaqasining (CSR) o'rtaacha kattaligi (shartli diametr D) 50,3 nm. Qizdirish yo'li bilan YAG:Ce kukuni hosil bo'lganda, reaksiya idishida $Y_3Al_5O_{12}$ birikmasini sintez qilish uchun yetarli bo'lgan harorat paydo bo'ladi va organik aralashmaning yonishi vaqtida paydo bo'ladigan qaytaruvchi muhit Ce ionlarining hosil bo'lishiga uch marta zaryadlangan holat yordam berishi aniqlandi. Olingan kukunni havoda 450, 900 va 1100°C haroratda keyingi qizdirish deyarli bir fazali YAG tuzilishini shakllantirishga olib keladi. Natijada paydo bo'lgan YAG:Ce fosfor kukunlari emissiya spektri 450 nm LED tomonidan qo'zg'atilganda granat tarkibidagi Ce^{3+} ionining energiya o'tishlari natijasida yuzaga keladigan 490 va 750 nm keng diapazonni ko'rsatadi. Bu sariq-yashil emissiyaga mos keladi, LEDning ko'k emissiyasi bilan birga oq yorug'lik tuyulishini yaratadi. Sariq-yashil lyuminestsentning eng yuqori intensivligini 900 °C neytral muhitda qizdirilganda YAG:Ce kukunlari ko'rsatadi.

Kalit so'zlar: itriy alyuminiy granatasi, seriy ionlari, nanostrukтура, sol-gel yonishi

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Introduction

Ultra-dispersed luminophores based on yttrium-containing oxide systems are widely used in display devices: computer screens, scoreboards, and TV screens ($\text{Y}_2\text{O}_3\text{:Eu}$, $\text{Y}_2\text{O}_3\text{:Tb}$), in the production of transparent ceramics for lasers and scintillators ($\text{Y}_2\text{O}_3\text{:Er}$, Nd) [1-4]. The anti-stokes luminescence of yttrium-based materials is actively used in the field of intellectual property protection ($\text{YVO}_4\text{:Eu}$) [5], in the solar cells (Y_2O_3 , YOF) [6], in biosensors ($\text{Y}_2\text{O}_3\text{:Yb}$) [7] and thermometry ($\text{Y}_2\text{O}_3\text{:Eu}$, Tb , Tm) [8].

Many years of worldwide research (including the 2014 Nobel Prize in Physics for the invention of blue light-emitting diodes and bright white light sources) led to development of a new class of lighting devices based on the nitride semiconductor structure covered with yellow luminophore, emission of which is perceived by the eye as white light. The cerium-doped microcrystalline yttrium-aluminum garnet powders $\text{Y}_3\text{Al}_5\text{O}_{12}\text{:Ce}^{3+}$ (YAG:Ce) [9, 10] are most commonly used as yellow luminophore.

The advantages of ultradisperse powders in high-power LEDs and light transducers remote from the emitting crystal comprise lower light dissipation on the small luminophore particles, reduced consumption of luminophore and homogeneous distribution of compound [11, 12]. Therefore, the objectives are to produce nanosized luminophores and, at the same time, to reduce the cost of the products by developing energy-saving methods for their production. Industrial methods for luminescent garnet powders synthesis are based on the energy-consuming direct synthesis reactions from relevant oxides that require high sintering temperatures (1500–1600 °C) under conditions of gas reducing environment.

There are numerous options of low-temperature synthesis of YAG:Ce-based ultra-disperse luminophores: sol-gel process [13, 14], coprecipitation of hydroxides [15–17], thermochemical synthesis [18]. Synthesis of oxide luminophores based on nitric acid salts being burnt in various organic compounds is attractive due to its simplicity, low cost and possibility of use for mixture ignition with both hear and microwave energy [19, 20]. In the view of the above-mentioned, development of new options for production of YAG:Ce-based ultra-disperse luminophores, comprising the ones that are promising for vitrocrys-

talline [21, 22] and ceramic light transducers [23, 24], is of great scientific and practical importance.

The research focuses on development of new method of YAG:Ce luminescent powder synthesis by method of sol-gel burning in microwave oven with the use of nitric acid salts of yttrium, aluminum and cerium, and the mixture of carbamide and hexamethylenetetramine as a fuel.

Research methods

Thermochemical synthesis of cerium-doped yttrium-aluminum garnet to produce luminescent powder with chemical composition $\text{Y}_{2.94}\text{Ce}_{0.06}\text{Al}_5\text{O}_{12}$, which is considered optimal for obtaining efficient luminescence in the yellow-green spectrum when excited by blue LED with wavelength of 450 nm [25], it has the following stages: weighing components 23.0 g $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 38.0 g. $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 1.0 g. $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 21.6 g. $\text{CH}_4\text{N}_2\text{O}$, dissolution of reagents in 30.0 mL of distilled water, adding 2.4 g of hexamethylenetetramine into the mixture. Hexamethylenetetramine ($\text{CH}_2)_6\text{N}_4$) added to the mixture acts as initiator of the combustion reaction. Next, components are mixed till mixture becomes translucent, and the resulting sol is placed in a heat-resistant glass into the temperature cabinet with the temperature of 80 °C. When being stirred, within 1.5–2 hours the sol increases its concentration till it reaches the viscous gel texture.

Gel is then placed in a porcelain crucible closed with a perforated ceramic lid and put into a domestic microwave oven SAMSUNG MS23F with the radiation frequency of 2450 MHz, without rotation. After 3–5 minutes at the power of 800 W, a turbulent thermochemical reaction starts in the crucible, with gases and water vapour being released. The spongy structure of the foam-like product easily breaks down into separate agglomerates and is ground in a planetary ball mill Pulverizette (Germany) in a zirconium dioxide glass for 15 min.

The differential thermal analysis (DTA) 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).

IR spectroscopy was performed on Specord-IR-75 spectrophotometer. Measurements range was $500\text{--}4000\text{ cm}^{-1}$.

Luminescence spectra were registered with SDL-2 spectrometer, adjusted so as to take into account the spectral sensitivity of the registration system and distribution of the stimulating radiation spectral density. The luminescence spectra of obtained powders were examined during luminescence excitation with a mercury lamp light with a filter at a room temperature.

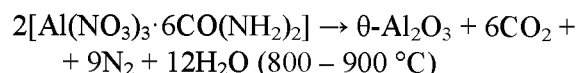
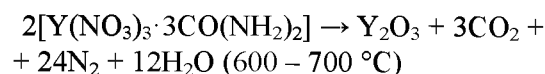
Results and Discussion

When being burnt, the gel-like mixture of nitrate salts of aluminum, yttrium, cerium with reducing agents produces a bright yellow foam-like product that is carried as fibres with the flow of reaction gases to the surface of the ceramic lid of the crucible (Fig. 1, a).

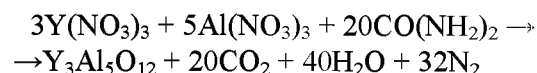
The Figure 1, b shows the SEM-images of agglomerated powder before it is ground. The size of agglomerates is $2\text{--}5\text{ }\mu\text{m}$. The agglomerates have a large number of pores, voids formed during the combustion process due to the emission of gases.

Chemical reactions during the synthesis of yttrium-aluminum garnet with carbamide used

as fuel are as follows at the first stage of the synthesis, complex compounds are formed of yttrium and aluminum nitric acid salts with carbamide, which soon decompose producing yttrium and aluminum oxides, with gas and water vapour being thereby released [26]:



At the temperature of about $900\text{ }^\circ\text{C}$, reaction between yttrium and aluminum oxides starts, with a number of intermediate steps. The overall reaction of the garnet synthesis for anhydrous salts and carbamide as fuel can be given as follows [27]:

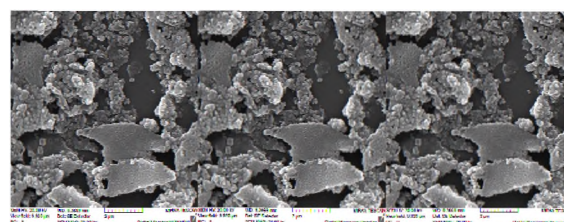


At incomplete reaction of garnet phase formation at rapid combustion impurities of perovskite YAlO_3 and yttrium aluminum monoclinic $\text{Y}_4\text{Al}_2\text{O}_9$ [28] can be formed in synthesized powder.

Carbamide with cerium nitrate also forms hydrates coordination compounds $[\text{Ce}(\text{H}_2\text{O})_2(\text{CO})$



a



b

Figure 1. Synthesized YAG:Ce sample: a – fibers on crucible lid; b – SEM-images of agglomerated powder.

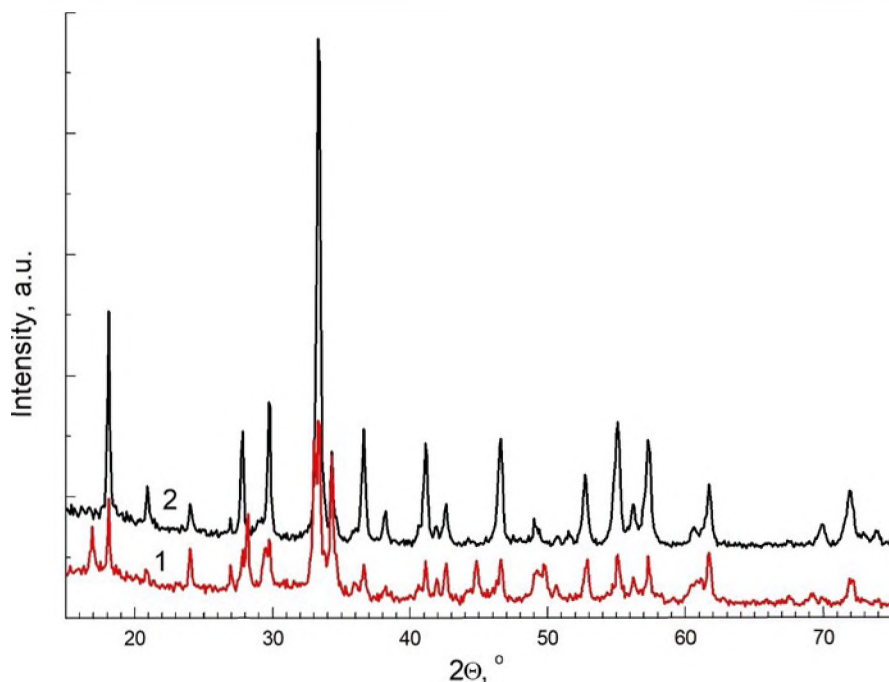


Figure 2. Diffractogram of the YAG:Ce powder:
1 – precursor without calcination; 2 – powder after calcination at 1100 °C (1 hour).

$(\text{NH}_2)_2(\text{NO}_3)_3$ that, if heated above 900 °C, decompose producing trivalent cerium oxide Ce_2O_3 [29]. Cerium ions replace part of yttrium ions in the lattice nodes, thus contributing to the formation of yttrium-aluminum garnet luminescent powder.

Diffractogram of the YAG:Ce powder sample produced by combustion in the nitrate-carbamide mixture in microwave oven confirm

presence of impurities, therefore, additional calcination at temperatures of 650, 900 and 1100 °C was performed in the open air in order to study temperature effect on the powders composition and their luminescent properties.

During the first stage, free and sorbed water is removed at the temperature range of 20–120 °C (endothermic process), at the second and the third stages (180–280 °C) the carbamide-oxide com-

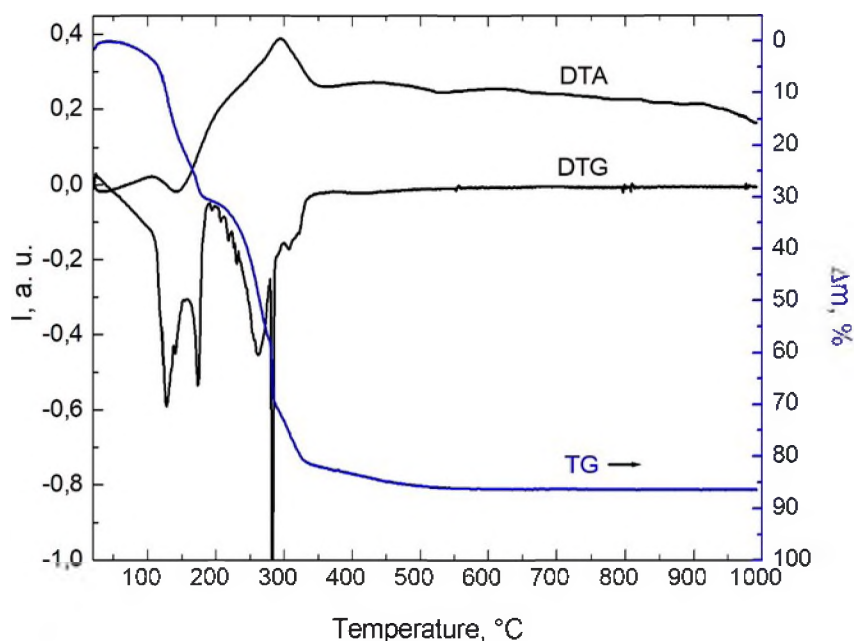


Figure 3. DTA of the heating process of a gel mixture of nitrate salts, urea and HMTA in the range of 20–1000 °C.

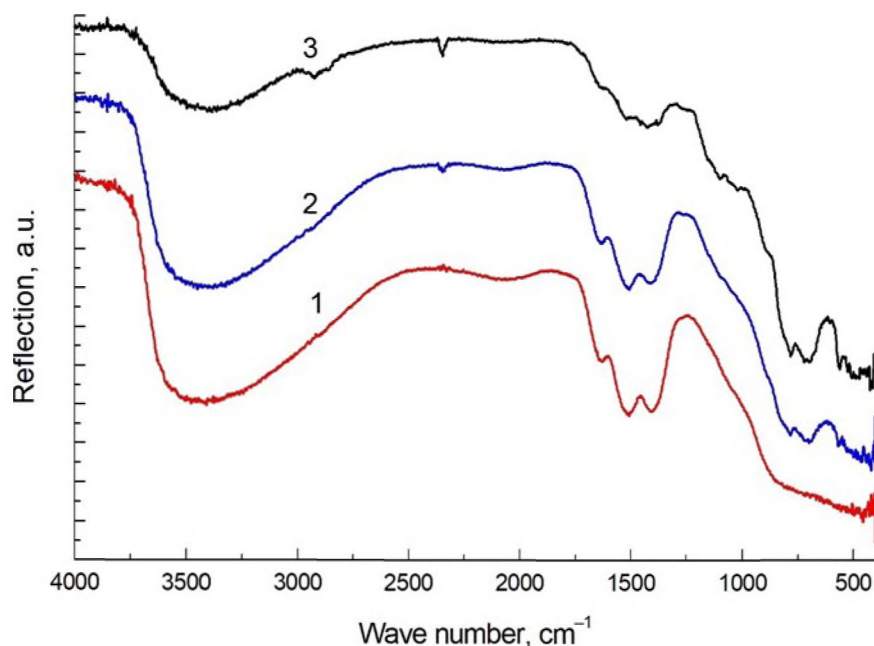


Figure 4. Infrared spectra of powders, obtained by combustion in a microwave oven and calcined for 1 hour at the following temperatures: 1– 650, 2 – 900, 3 – 1100 °C.

plexes of yttrium, aluminum and cerium are formed, and later, when the mixture is ignited (280–300 °C), the intensive combustion reaction starts, with a sudden rise in the temperature and a decrease in the mass. The crystalline phases of the yttrium-aluminium oxides appear to be formed thereby, the main one being the $Y_3Al_5O_{12}$ phase. Then follow the reactions of the nitric acid residues removal and the slow burnout of the organic impurities (330–450 °C). At the same time, the mass of the samples decreases dramatically, and after 450 °C remains almost unchanged, which proves the absence of thermal effects till temperature of 1000 °C. The mass loss of the gel mixture exceeds 90%. Thus, derivatographic examination confirms formation of the crystalline phases at the temperature of 280–300 °C, the main phase being the garnet phase.

Analysis of the infrared spectra of powders received by gel-like mixture being burned in the microwave oven and calcified in the open air at the temperature of 650, 900 and 1100 °C shows that the wide band at 3200–3600 cm^{-1} corresponds to vibrations of groups (O–H) adsorbed on porous particles of the crystalline phase, 1500 cm^{-1} band is related to the asymmetric oscillations of $-CH_2$ groups, 1600 and 1380 cm^{-1} band is associated with asymmetric and symmetric oscillations of NO_3^- groups [31], respectively (Fig. 4).

The intensity of these bands decreases as calcination temperature increases, confirming ad-

sorbed gas removal process. The absorption peak at 2300 cm^{-1} is due to fluctuations in C–O bond formed at intense burnout of organic components and penetration of the carbon impurities in the crystalline structure [27]. Furthermore, the peaks at 567, 706 and 783 cm^{-1} are characterized by fluctuations in metal-oxygen bonds (Al–O), (Y–O) [32], the intensity of which increases with increase in the calcination temperature, which proves continuation of crystalline structure formation.

Figure 5 shows the luminescence curves of powders depending on the calcination temperature. It is known that Ce ion can be located in Y_2O_3 sublattice, partially replacing yttrium ions in 3 and 4 charge state [33]. However, C^{4+} ion is optically inactive and does not form corresponding levels in the energy pattern. In YAG:Ce luminescence spectrum, a wide band caused by the energy transitions of Ce^{3+} ion in the structure of the garnet is between 490 and 750 nm and corresponds to yellow-green radiation, which creates sensation of white light with LED blue radiation.

From analysis of fig.5, it is obvious that increase in the temperature of YAG:Ce powder treatment within the range of 650–1100 °C in air environment leads to increase in intensity of the luminescence up to temperature of 900 °C. At the same time, structure ordering, crystals growth, crystals specific surface reduction, volatilization processes occur. However, when the calcination temperature rises to 1100 °C, luminescence inten-

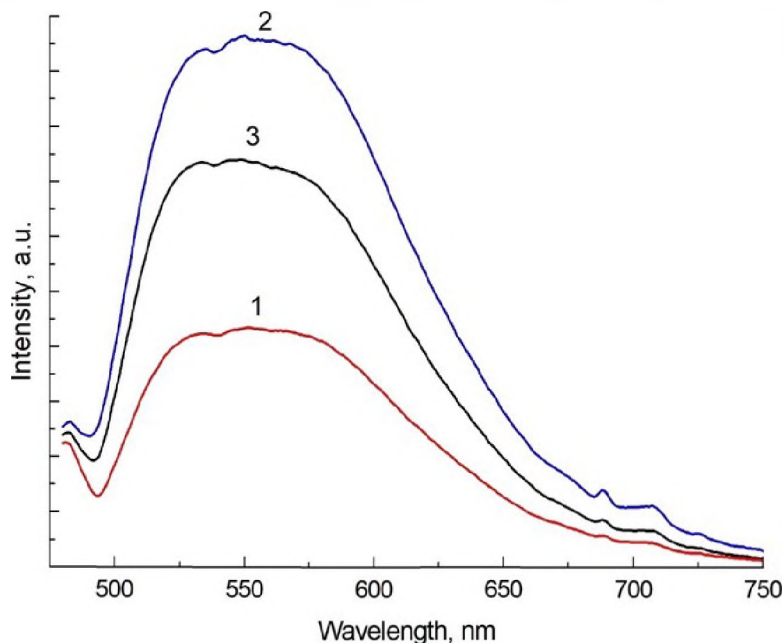


Figure 5. Luminescence spectra of powdered YAG:Ce samples depending on the calcination temperature, $\lambda_{\text{ex}} = 450$ nm.

sity decreases. This is likely due to increase of the four-charge cerium ions share in the yttrium oxide sublattice in oxidizing atmosphere and, consequently, due to decrease of the share of Ce^{3+} ions that emit yellow-green luminescence in the 530–590 nm wavelength range (curve 3).

Conclusion

Novel method has been developed for the thermochemical synthesis of cerium-doped yttrium-aluminum garnet luminescent powder with the use of nitric acid salts of yttrium, aluminium and cerium and mixture of carbamide and hexamethylenetetramine as fuel. The synthesis process was performed in a microwave oven with the power of 800 W and the frequency of 2450 MHz in a porcelain crucible closed by a perforated heat-resistant lid.

Pursuant to X-ray fluorescence analysis it was determined that the average size of a cohesive particle dispersion area for YAG:Ce produced in

crucible during microwave heating is 50.3 nm, and 0.8 μm when the powder is calcified in the air at 1100 °C. The reaction cup reaches a temperature sufficient for synthesis of $\text{Y}_3\text{Al}_5\text{O}_{12}$ compound and penetration of cerium ions into the structure of the garnet in a three-charge state. The calcination of the powder at 450, 900 and 1100 °C in the air leads to formation of a practically single-phase YAG structure, and a small amount of the perovskite impurity phase does not lead to significant change in the spectral luminescent properties. YAG:Ce luminophore emission spectrum, when excited by a 450 nm LED, gives a wide band between 490 and 750 nm that corresponds to the yellow-green radiation that with blue LED radiation creates the sensation of white light. The highest intensity of the yellow-green luminescence is shown by YAG:Ce powders calcinated at 900 °C in the air. This appears to be due to increase in the share of four-charge cerium ions in yttrium oxide sublattice during the treatment in the oxidizing atmosphere.

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