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## MICROWAVE SYNTHESIS OF YTTRIUM OXIDE NANOSTRUCTURE POWDERS, DOPED WITH YTTERBIUM AND ERBIUM IONS

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## Cover Page Footnote

The work was carried out within the framework of tasks 1.2 of the State Research Program «Photonics and electronics for innovation» of the Republic of Belarus

## MICROWAVE SYNTHESIS OF YTTRIUM OXIDE NANOSTRUCTURE POWDERS, DOPED WITH YTTERBIUM AND ERBIUM IONS

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The aim of the study was to develop microwave synthesis of luminescent yttrium oxide powders doped with ytterbium and erbium ions ( $Y_2O_3:Yb^{3+}, Er^{3+}$ ) in the process of homogeneous precipitation from a reaction mixture of yttrium, ytterbium and erbium chlorides. The characteristics of the obtained powders were studied by X-ray diffraction, scanning and passing electron microscopy, differential thermal analysis, IR spectroscopy and photoluminescence under IR excitation. From luminescence spectra of  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples obtained by excitation with an IR laser at a wavelength of 975 nm, it can be concluded that when the calcination temperature of the sample's changes, the radiation color of powders changes from green to red, which can be used to produce phosphors of different colors.

**Keywords:** homogeneous precipitation, nanostructured powders, yttrium oxide, ytterbium and erbium ions, up-conversion, anti-stokes luminescence

## МИКРОВОЛНОВЫЙ СИНТЕЗ НАНОСТРУКТУРИРОВАННЫХ ПОРОШКОВ ОКСИДА ИТТРИЯ, ЛЕГИРОВАННОГО ИОНАМИ ИТТЕРБИЯ И ЭРБИЯ

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Целью исследования была разработка микроволнового синтеза люминесцентных наноструктурированных порошков оксида иттрия, легированного ионами иттербия и эрбия ( $Y_2O_3:Yb^{3+}, Er^{3+}$ ) в процессе гомогенного осаждения из реакционной смеси хлоридов иттрия, иттербия и эрбия. Характеристики полученных порошков изучали методами рентгеновской дифракции, сканирующей и проходящей электронной микроскопии, дифференциально-термического анализа, ИК-спектроскопии и фотолюминесценции при ИК возбуждении. Из спектров люминесценции образцов  $Y_2O_3:Yb^{3+}, Er^{3+}$ , полученных при возбуждении ИК-лазером на длине волны 975 нм, установлено, что при изменении температуры обжига образцов цвет излучения порошков изменяется от зеленого до красного, что может быть использовано для производства люминофоров разного цвета.

**Ключевые слова:** гомогенное осаждение, наноструктурированные порошки, оксид иттрия, ионы иттербия и эрбия, up-конверсия, антистоксова люминесценция

## YTERBIY VA ERBIY IONLARI BILAN DOPLANGAN NANOSTUZILIK ITTRIY OKSID KUKULARINING MIKROTO'LQINLI PECHDA SINTEZI

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Tadqiqotning maqsadi itriy, itterbiy va erbiyning reaksiya aralashmasidan bir hil cho'kish jarayonida ytterbiy va erbiy ionlari ( $Y_2O_3:Yb^{3+}, Er^{3+}$ ) bilan qo'shilgan lyuminescent nanostrukturali itriy oksidi kukunlarining mikroto'lqinli sintezini ishlab chiqishdan iborat edi. Olingan kukunlarning xarakteristiklari rentgen nurlari diffraksiyasi, skanerlash va uzatish elektron mikroskopiyasi, differentsial termal tahlil, IQ-spektroskopiya va IQ qo'zg'atish ostida fotolyuminesentsiya bilan o'rganildi.  $Y_2O_3:Yb^{3+}, Er^{3+}$  namunalarning 975 nm to'lqin uzunligida IQ lazer bilan qo'zg'atish yo'li bilan olingan lyuminescent spektrlaridan ma'lum bo'ldiki, na'munalarning yonish harorati o'zgaranda, kukun nurlanishining rangi yashildan qizil rangga o'zgaradi, bu usulni turli rangdagi lyuminozorlarni ishlab chiqarish uchun ishlatilish mumkin.

**Kalit so'zlar:** bir jinsli cho'kish, nanostrukturali kukunlar, itriy oksidi, itterbiy va erbiy ionlari, yuqoriga konversiya, anti-Stokes luminesans

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### Introduction

Yttrium oxide ( $Y_2O_3$ ) is the preferred matrix for introducing rare earth element (REE) ions in the production of phosphors [1, 2], laser ceramics [3], and scintillators [4]. Yttrium oxide has excellent physical and chemical properties, such as optical transparency in a wide spectral range of 0.29–8  $\mu\text{m}$ , high melting point (2450  $^{\circ}\text{C}$ ), large band gap (5.8 eV), high thermal conductivity, chemical and atmospheric durability [5]. Among

the emission properties of  $Y_2O_3$  activated by rare earth ions, up-conversion (anti-stokes luminescence) is the most attractive phenomenon, not only due to the photophysical mechanism, but also due to a wide range of practical applications, for example, as highly efficient phosphors [6, 7], solid-state lasers [8], biosensors in medical technology field [9]. It was previously established [10, 11] that  $Y_2O_3$ , activated by  $Yb^{3+}$  and  $Er^{3+}$  ions, is one of the models of a conversion system that generates radia-

tion in the visible region under infrared irradiation (IR). The sensitive  $\text{Yb}^{3+}$  ion is excited by an IR radiation source and transfers its energy to the  $\text{Er}^{3+}$  activator ion, which emits a photon in the visible region of the spectrum. Nanostructured powder materials of the  $\text{Y}_2\text{O}_3:\text{Yb}^{3+}, \text{Er}^{3+}$  system was obtained by co-precipitation [12], sol-gel [13] and combustion methods [14, 15]. The use of microwave methods for heating the reaction mixture in the process of nanoparticle synthesis is promising and is of interest due to its simplicity, easy controllability, and environmental friendliness [16]. This article describes a microwave method for the synthesis of nanostructured  $\text{Y}_2\text{O}_3$  powders activated with  $\text{Yb}^{3+}$  and  $\text{Er}^{3+}$  ions, as well as the influence of synthesis modes and subsequent heat treatments on the structural, morphological and spectral-luminescent characteristics of the resulting materials.

### Research methods

Solvothermal synthesis of nanostructured powders of the  $\text{Y}_2\text{O}_3:\text{Yb}^{3+}, \text{Er}^{3+}$  system was carried out by heating the reaction mixture using microwave radiation. The radiation power during the homogeneous deposition of precursor particles was 140 W, the radiation frequency was 2.45 GHz, and the process time was 120 minutes under conditions of rotation of the container, which provided for the return of the evaporating liquid [17]. The mixtures  $\text{YCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{YbCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{ErCl}_3 \cdot 6\text{H}_2\text{O}$  were used as the initial reactants. The chloride/urea molar ratio is taken as 1/1. The amount of doping additives – ytterbium and erbium chlorides was calculated from the point of view of their optimal concentration in the  $\text{Y}_2\text{O}_3$  structure to obtain luminescent radiation with the highest quantum yield and is 4.0 at.% for  $\text{Yb}^{3+}$  and 1.0 at.% for  $\text{Er}^{3+}$  [18].

After completion of the precipitation process, the sediment is washed 3 times with distilled water and centrifuged at a speed of 3000 rpm. The sediments are dried at a temperature of 80–90 °C for 6 hours. Firing of the precursor – hydrated oxides – is carried out in air at temperatures of 700 and 1100 °C for 2 hours.

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.

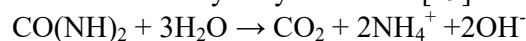
SEM/TEM measurements have been performed by Vega II LSH microscope, Tescan and JEM 2100 transmission microscope (JEOL, Japan).

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

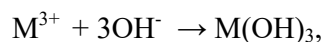
The luminescence measurements were performed by using RF-6000 spectrofluorimeter (Shimadzu, Japan)

### Results and Discussion

A common method of chemical precipitation is the interaction of liquid solutions: pouring one of the solutions (precipitant) into a certain volume of another containing the precipitated ions. The main disadvantages of this method are the presence of an excess of one of the reagents in the reaction mixture and the occurrence of local supersaturations. All this leads to the formation of agglomerated sediments. One of the options for solving this problem is the precipitation of hydrated oxides due to the slow hydrolysis of urea [19]:

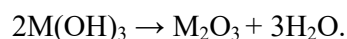


The slow release of  $\text{OH}^-$  groups leads to the precipitation of aggregated hydroxide powders:



where  $\text{M} = \text{Y}, \text{Yb}, \text{Er}$ .

During further heat treatments, free and bound water is removed and yttrium, erbium and ytterbium oxides are formed:



When nano-sized oxide particles in close contact are calcined, active impurities diffuse into the yttrium oxide lattice with partial replacement of yttrium ions by ytterbium and erbium ions. Scanning and transmission electron microscopy data indicate strong agglomeration of nanoparticles (Fig. 1), and the agglomerates of the resulting powders are easily destroyed by mechanical action.

XRD of  $\text{Y}_2\text{O}_3:\text{Yb}^{3+}, \text{Er}^{3+}$  [20] samples synthesized during the deposition process confirms that after calcination of the precursor at a temperature of 700 °C, nano-sized powders with reflections corresponding to the reflections of the cubic crystal structure of the chemical composition of  $\text{Y}_2\text{O}_3$  are already formed (card No. 41-1105 of the JCPDS catalog). The lattice parameter, according to the tabular data, is equal to 1.641 nm, and the

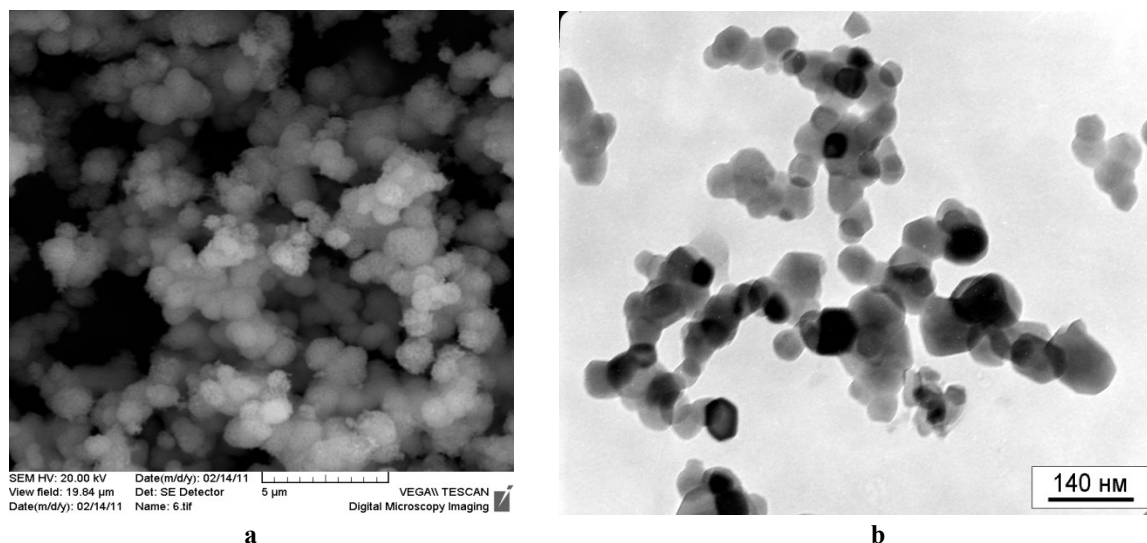


Figure 1. SEM image (a) and TEM image (b) of  $Y_2O_3:Yb^{3+}, Er^{3+}$  powder, dried and calcined at 700 °C.

coherent scattering region (conditional particle size), calculated using the Scherrer formula [2] is about 50 – 70 nm at a calcination temperature of 700 °C and 600 – 800 nm at 1200 °C. The main peak in the diffraction pattern corresponds to the [222] crystal plane and is located at  $2\theta = 28.94$ . The deviation towards larger angular values of the samples obtained upon calcination at 1200 °C ( $2\theta = 29.19$ ) indicates a broadening of the matrix lattice due to the inclusion of  $Yb^{3+}$  and  $Er^{3+}$  ions (Fig. 2).

Reflections corresponding to the formation of crystalline phases  $Yb_2O_3$  and  $Er_2O_3$  are not recorded in the diffraction patterns due to the low concentration of impurity phases.

Figure 3 shows differential thermal gravimetry (DTG) and differential gravimetry (DG) curves for a hydrated precursor sediment that has

undergone heat treatment in air (90 °C for 6 hours). The DTG curve has a peak at 134 °C and 138 °C (endo-effects), indicating the removal of free and bound water [21]. At temperatures of 350 °C and 417 °C, hydroxides apparently decompose with the release of water molecules, and at 700 – 740 °C a peak appears on the DTG curve, corresponding to the exothermic reaction of the process of formation of the crystalline phase. At the same time, the mass of the forming crystalline structure continues to fall. At a temperature of 930 °C, residual hydroxyl groups are removed with an endothermic effect [22]. Examination of the DTG and TG diagrams clearly confirms the assumption that the crystalline phase in the form of nanocrystals is completely formed at 700 – 740 °C, and with a further increase in the calcination temperature, its compaction occurs.

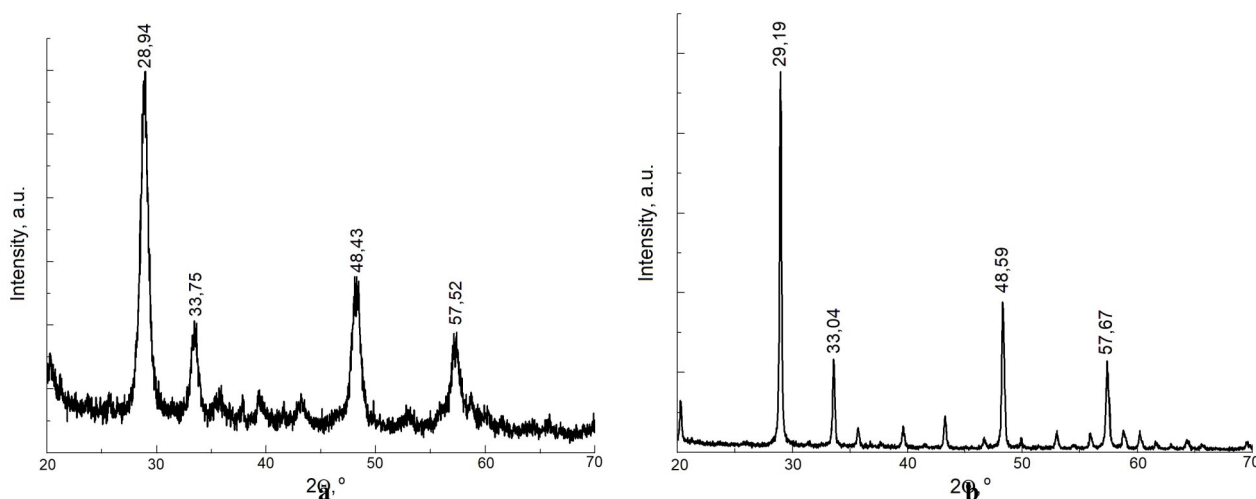


Figure 2. XRD patterns for  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples recorded after calcination of the precursor in air: a – at a temperature of 700 °C and b – at 1200 °C.

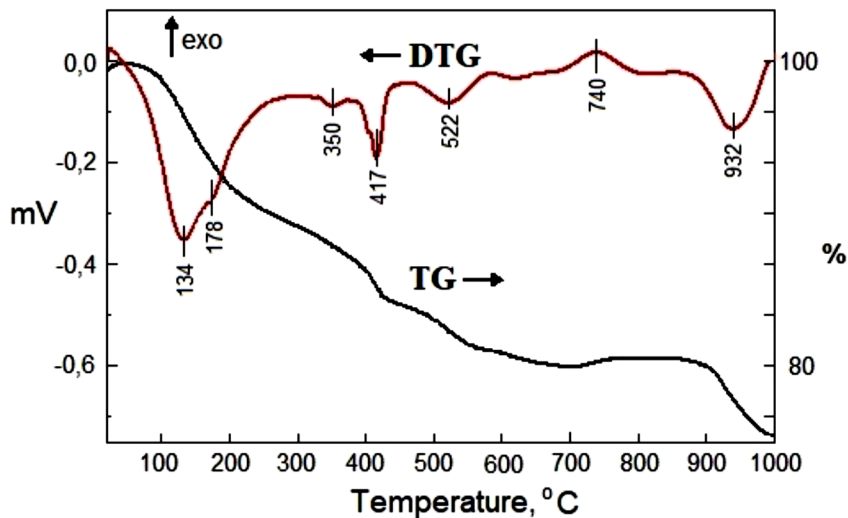


Figure 3. DTG and DG for the precursor obtained after precipitation and drying (90 °C) of a mixture of hydroxides.

Consideration of the IR spectra of  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples obtained by deposition under microwave irradiation and calcined at different temperatures: 1 – 90 °C; 2 – 700 °C; 3 – 1200 °C shows (Figure 4) that the bands at 2860–3600  $cm^{-1}$ , caused by stretching vibrations of O–H groups and vibrations of bound water molecules H–O–H, absorbed by porous particles [23], are present in all cases of processing, however, at a calcination temperature of 1200 °C, their intensity decreases approximately four times. Stretching vibrations of CO appear in the form of broad bands at 1580 (asymmetrical), 1405 and 1093  $cm^{-1}$  (symmetrical), non-planar bending vibrations of CO at 848  $cm^{-1}$  [24]. The band at 563  $cm^{-1}$  is attributed to the vibrations of the Y–O bond. The reduced concentration of hydroxyl groups, adsorbed gas impurities and residual carbon-containing impurities during additional calcination confirms the promise of high-temperature treatment in the formation of dehydrated  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples.

The luminescence spectra of  $Y_2O_3:Yb^{3+}, Er^{3+}$  measured at various concentrations of ytterbium and erbium ions were studied in detail in [25–28]. It has been shown that when the ternary system  $Y_2O_3:Yb^{3+}, Er^{3+}$  is irradiated with a diode laser with a wavelength of 978 nm, green (563 nm) or red (660 nm) radiation appears, which corresponds to the energy transitions  $^4S_{3/2} \rightarrow ^4I_{15/2}$  and  $^4F_{9/2} \rightarrow ^4I_{15/2}$  in  $Yb^{3+}$  and  $Er^{3+}$  ions depending on the excitation conditions and the concentration of doping ions.

The luminescence spectra of the powders we obtained, calcined at temperatures of 700 and

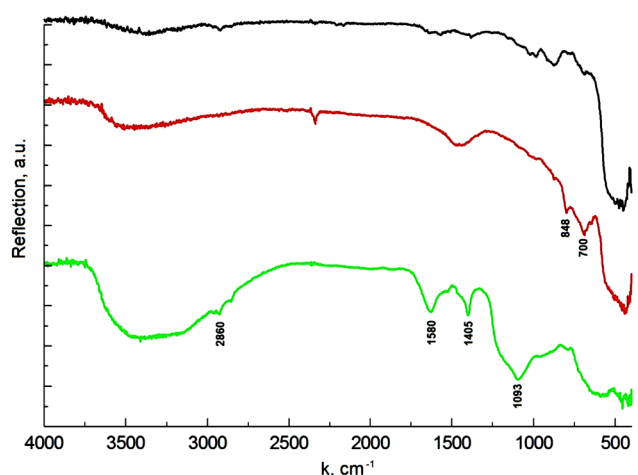


Figure 4. IR spectra of  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples obtained by deposition under microwave irradiation at different heat treatment temperatures: 1 – 90 °C (green); 2 – 700 °C (red); 3 – 1200 °C (black).

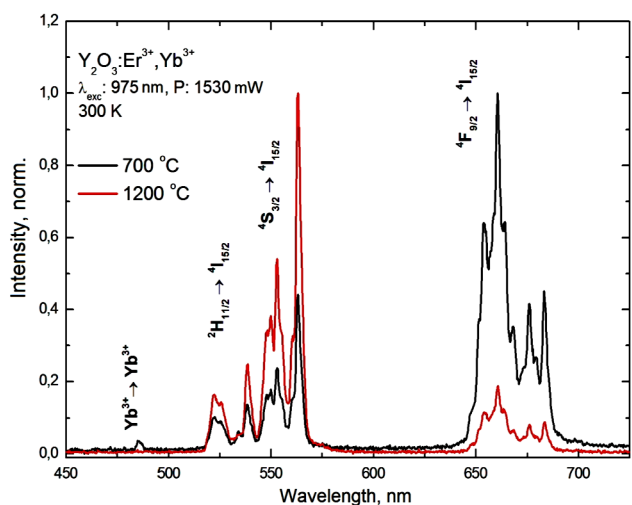


Figure 5. Luminescence spectra of powders calcined at temperatures of 700 and 1200 °C.

1200 °C, excited by an IR laser at a wavelength of 975 nm, are shown in Figure 5.

From an examination of the emission spectra of samples thermally treated in an air atmosphere, we can conclude that when calcined at a lower temperature (700 °C), radiation in the red region of the spectrum prevails, while at a higher processing temperature, the anti-stokes luminescence efficiency increases in the green region of the spectrum. It can be assumed that firing  $Y_2O_3:Yb^{3+}, Er^{3+}$  powders at 1200 °C leads to matrix compaction and a reduction in the Er – Er interionic distance, which increases the efficiency of cross-relaxation interactions of  $Er^{3+}$  ions. As a result of the transfer of excitations from  $Yb^{3+}$  ions to  $Er^{3+}$  ions, the latter are excited into the  $^2H_{11/2}$  state, from which  $^4S_{3/2}$  is populated and cross-relaxation occurs according to the scheme  $^2H_{11/2}, ^4S_{3/2} \rightarrow ^4I^{9/2} : ^4I_{11/2} \rightarrow ^4F_{9/2}$ , i.e., the population of the  $^2H_{11/2}$  and  $^4S_{3/2}$  levels decreases, and the  $^4F_{9/2}$  level increases [29, 30].

### Conclusion

Using the microwave synthesis method, nanostructured yttrium oxide powders activated with ytterbium and erbium ions of a quasi-spherical shape with primary particle sizes of 50 – 70 nm, formed at a calcination temperature of 700 °C and 0.6 – 0.8  $\mu m$  at 1200 °C, were obtained. It has been established that as a result of deposition of aggregated hydroxide powders and further heat treatments (drying and calcination in air), free and bound water is removed and oxides of yttrium, erbium and ytterbium are formed, and upon calcina-

tion, active impurities diffuse into the yttrium oxide lattice with partial replacement of ions yttrium into ytterbium and erbium ions. Thermogravimetric analysis data confirm the assumption that the crystalline phase is completely formed at 700 – 740 °C, and with a further increase in the calcination temperature, its compaction occurs. IR spectroscopy states that with additional calcination of  $Y_2O_3:Yb^{3+}, Er^{3+}$  powders at 1200 °C, hydroxyl groups and adsorbed gas impurities are almost completely removed. From an examination of the luminescence spectra of  $Y_2O_3:Yb^{3+}, Er^{3+}$  samples obtained upon excitation with an IR laser at a wavelength of 975 nm, we can conclude that upon calcination at a temperature of 700 °C, radiation in the red region of the spectrum prevails, whereas at a higher processing temperature (1200 °C) anti-stokes luminescence efficiency increases in the green region of the spectrum. It can be assumed that firing  $Y_2O_3:Yb^{3+}, Er^{3+}$  powders at 1200 °C leads to compaction of the matrix and a reduction in the Er – Er interionic distance, which increases the efficiency of cross-relaxation interactions of  $Er^{3+}$  ions. The effect of changing the emission of  $Y_2O_3:Yb^{3+}, Er^{3+}$  powders processed at different temperatures can be used to obtain green or red phosphors.

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