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## STRUCTURE AND MORPHOLOGY OF ULTRADISPERSED ERBIUM YTTERBIUM OXIDE POWDERS OBTAINED BY DEPOSITION METHOD

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## STRUCTURE AND MORPHOLOGY OF ULTRADISPERSED ERBIUM YTTERBIUM OXIDE POWDERS OBTAINED BY DEPOSITION METHOD

### Cover Page Footnote

The research was conducted as part of Task 2.8 of the State Program for Scientific Research «Materials Science, New Materials Technologies2 for 2021–2025 in the Republic of Belarus

## STRUCTURE AND MORPHOLOGY OF ULTRADISPERSED ERBIUM YTTTERBIUM OXIDE POWDERS OBTAINED BY DEPOSITION METHOD

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The aim of research is to develop a new method for synthesis of ultra-dispersed crystalline powders of lanthanide oxides, in particular, erbium oxide doped with ytterbium oxide, intended for making transparent ceramics. The formation of nanostructured powders of the erbium oxide – ytterbium oxide system using deposition and heat treatment processes by co-deposition of salt solutions mixed with ammonium carbonate and hydrogen peroxide complex solution at temperature of 10 °C followed annealing at 100, 650, 900 and 1200 °C was studied. Studies of the morphological and structural characteristics of the powders using scanning electron microscopy and X-ray phase analysis showed that the  $Er_2O_3$ - $Yb_2O_3$  particles are great extent agglomerated, they have a laminar, plate-like morphology with the laminae thickness of about 50 nm, length of 10–15  $\mu$ m (650 °C), when sediment is annealed at 900 °C, laminar particles split into 30–60 nm diameter fibers, and during subsequent heating the fibers in agglomerates fuse together forming a mesh structure, the size of the annealed particles being 150–200 nm.

Keywords: rare earth elements oxides, erbium oxide, ytterbium oxide, ultrafine powders, deposition method

## СТРУКТУРА И МОРФОЛОГИЯ УЛЬТРАДИСПЕРСНЫХ ПОРОШКОВ ОКСИДА ЭРБИЯ – ИТТЕРБИЯ, ПОЛУЧАЕМЫХ МЕТОДОМ ОСАЖДЕНИЯ

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Цель исследования состоит в разработке нового метода синтеза ультрадисперсных кристаллических порошков оксидов лантаноидов, в частности оксида эрбия  $Er_2O_3$ , легированного оксидом иттербия  $Yb_2O_3$ , предназначенных для формирования прозрачной керамики. Изучены особенности формирования наноструктурированных порошков системы «оксид эрбия – оксид иттербия» из смешанных карбонатов лантаноидов путем осаждения карбонатом аммония с добавкой перекиси водорода из соответствующих азотнокислых солей при температуре 10 °C с последующим прокаливанием осадков при 100, 650, 900, 1200 °C. Исследования морфологических и структурных характеристик порошков с применением методов сканирующей электронной микроскопии и рентгенофазового анализа показали, что частицы  $Er_2O_3$ - $Yb_2O_3$  в значительной степени агломерированы, имеют пластинчатую морфологию с толщиной ламинатов около 50 нм и длиной 10–15 мкм (650 °C), а при прокаливании осадка при 900 °C пластины расщепляются на волокна диаметром 30–60 нм; при нагревании до 1200 °C волокна в агломератах образуют сетчатую структуру с размерами спекшихся частиц 150–200 нм.

Ключевые слова: оксиды редкоземельных элементов, оксид эрбия, оксид иттербия, ультрадисперсные порошки, метод осаждения

## ULTRADISPERSLANGAN ERBIY-YTTERBIY OKSIDI KUKUNLARINING TUZILISHI VA MORFOLOGIYASI

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Tadqiqotning maqsadi shaffof keramikaga shakl berishga mo'ljallangan lantanoidlar oksidlari, xususan, ytterbiy oksidi  $Yb_2O_3$  bilan legirlangan erbiy oksidi  $Er_2O_3$  ning ultradispers kristall kukunlarini sintez qilishning yangi usulini ishlab chiqarishdan iborat. Lantanoidlarning aralash karbonatlaridan 10 °C haroratda tegishli nitrat kislova tuzlaridan vodorod peroksid qo'shib ammoniy karbonat bilan cho'ktirish va cho'kmalarni 100, 650, 900, 1200 °C da qizdirish orqali "erbiy oksidi - ytterbiy oksidi" tizimining nanotuzilishli kukunlarini shakllantirish xususiyatlari o'rganildi. Kukunlarning morfologik va strukturaviy xususiyatlarini skanerlovchi elektron mikroskopiyasi va rentgen fazali tahlil usullari yordamida o'rganish shuni ko'rsatdiki,  $Er_2O_3$ - $Yb_2O_3$  zarralari sezilarli darajada aglomeratsiyalangan, laminalarning qalinligi taxminan 50 nm va uzunligi 10–15 mkm (650 °C) bo'lgan plastinka morfologiyasiga ega, cho'lana 900 °C da qizdirilganda plastinkalar diametri 30–60 nm bo'lgan tolalarga bo'linadi; 1200 °C gacha qizdirilganda, aglomeratlardagi tolalar 150–200 nm o'lchamdagi yopishgan zarrachalar bilan to'rtli tuzilishni hosil qiladi.

Kalit so'zlar: noyob yer oksidlari, erbiy oksidi, ytterbiy oksidi, ultra mayda kukunlar, qo'shma cho'ktirish usuli

## Introduction

Lanthanide oxide-based optically transparent ceramics doped with rare earth element ions is promising for use as active media for solid-state lasers, luminescent converters, scintillators thanks to high optical and thermomechanical characteristics [1–5].

Erbium oxide is a promising matrix for creating lasers and fiber optic amplifiers by doping active lanthanide ions due to excellent physical properties, such as optical transparency over a wide spectrum, high melting temperature (2380 °C), wide band gap, high thermal conductivity [6–11].

To activate the sintering of rare earth oxide particles in the production of ceramics with high optical transparency, sintering additives in the form of yttrium, lanthanum, and zirconium oxides are used, introduced into the system during the formation of nanostructured powders [12–15]. In this way, transparent ceramics from erbium oxide  $\text{Er}_2\text{O}_3$  with the addition of  $\text{La}_2\text{O}_3$  were successfully produced [13].  $\text{Er}_2\text{O}_3$  ceramics with the addition of zirconium dioxide (4 at.%  $\text{ZrO}_2$ ), sintered at a temperature of 1850°C for 8 hours in a vacuum [14], demonstrated an optical transmittance of 77% in the visible range and 78% at a wavelength of 1100 nm.

Similarly, highly transparent ytterbium oxide ( $\text{Yb}_2\text{O}_3$ ) ceramics were obtained by vacuum sintering using  $\text{ZrO}_2$  as a sintering additive [15]. Transparent ceramics doped with 3%  $\text{ZrO}_2$  were found to have the best optical properties, with a transmittance of 75% at 1100 nm and about 81% in the mid-infrared region.

Recently, a new, rapidly developing field has emerged: the study and application of transparent, high-entropy optical media based on rare-earth oxides. Such materials are classified as ceramics, consisting of five or more components in equiatomic proportions [16, 17].

A new highly transparent ceramic based on rare earth oxides with the composition  $(\text{Lu}_{0.2}\text{Y}_{0.2}\text{Gd}_{0.2}\text{Yb}_{0.2}\text{Er}_{0.2})_2\text{O}_3$  was successfully fabricated by vacuum sintering with the addition of 3 at.%  $\text{ZrO}_2$  and 10 at.%  $\text{La}_2\text{O}_3$  as sintering additives [18]. A single-phase cubic structure of the ceramics from a solid solution with a relative density of 99.95% and an average grain size of  $6.91 \pm 3.28 \mu\text{m}$  was obtained. The grain boundary of the ceramics had a thickness of 1.3 nm. The linear transmittance of such ceramics reached 80% at a wavelength of 1100 nm, which was 98.7% of the theoretical value for an  $\text{Er}_2\text{O}_3$  single crystal. In addition, fluorescence

emission was observed in the ultraviolet (311 nm), visible (563, 622 nm), and near-infrared (1032, 1535 nm) spectral regions. The new high-entropy ceramics may have broad potential for use in optical applications, such as scintillators, high-conversion luminescent materials, and infrared lasers.

Nanocrystalline luminescent powders of rare earth oxides can be obtained by thermal decomposition of the corresponding salts [19, 20].  $\text{Er}_2\text{O}_3$  powders with crystal sizes of 9.7–27.2 nm were obtained in this way [19].

Nanosized  $\text{Er}^{3+}:\text{Y}_2\text{O}_3$  powders with varying erbium impurity concentrations were synthesized by decomposing a mixture of erbium and yttrium nitrate salts in the presence of a surfactant that ensures particle deagglomeration, followed by calcination at 800 °C [20]. The calculated particle sizes according to the Scherrer equation range from  $20 \pm 3$  to  $32 \pm 3$  nm.

There are also methods for obtaining REE oxide powders through thermochemical reactions (combustion method) [21, 22], however, the particles of such oxides are often highly agglomerated and contaminated with combustion reaction products.

Most of the known methods for producing nanosized powders co-doped REE are based on the processes of precipitation of carbonate or oxalate precursors from aqueous solutions of salts, separation of the precipitate from the mother liquor, washing, drying in air, and calcination at high temperatures [23, 24]. For the precipitation of hydrated REE compounds, including erbium oxide, salts, most often nitrates, are used as sources, and solutions of ammonia, oxalic acid, ammonium carbonate or ammonium bicarbonate are used as precipitants. Despite the obvious disadvantages of precipitation methods - labor intensity, large volumes of solvents used, this synthesis option ensures the production of low-agglomerated powders with a narrow size distribution of nanoparticles, as well as a high degree of purity [25].

The aim of the present research is to develop a new method for synthesis of ultra-dispersed crystalline powders of lanthanide oxides, in particular, Erbium oxide  $\text{Er}_2\text{O}_3$  doped with Ytterbium oxide  $\text{Yb}_2\text{O}_3$  - additive intended for making transparent ceramics with upconversion effect.

## Research methods

Microcrystalline oxide powders  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  a purity of 99.5% and 99.92% respectively; ammonium carbonate  $(\text{NH}_4)_2\text{CO}_3$  and hydrogen peroxide (35%  $\text{H}_2\text{O}_2$ ) were used as starting components.

Heated up to 90 °C and mixed with a magnetic stirrer, the initial microcrystalline  $\text{Er}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  powders are dissolved in the concentrated nitric acid till complete dissolution. Then the sediment is dried in a loss-on-drying oven at 90 °C to remove free water and nitric acid. The initial nitrate salts are weighed out:  $\text{Er}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  – 8,0 g.,  $\text{Yb}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  – 0,8 g. and  $(\text{NH}_4)_2\text{CO}_3$  – 5,0 g.

Hydrated carbonate sediments are produced as follows.

Nitric acid salts are dissolved in the distilled water, then ammonium carbonate is dissolved in the distilled water, both solutions are simultaneously poured,  $\text{H}_2\text{O}_2$  hydrogen peroxide 35 % is added till the pH of 10–12, the mixture being stirred with a magnetic mixer. In the glass sediment is formed, it is placed in the centrifuge, deposited at a speed of about 3000 r/min, then sediment is removed, washed 3 times with distilled water and re-centrifuged, then placed in a 150 mL 107 mm-diameter porcelain evaporation bowl No.4 and evaporated in the loss-on-drying oven at the temperature of 100 °C for 7 hours.

The kinetics of the deposition reactions and crystallization conditions of mixed lanthanide carbonates in  $\text{Er}_2\text{O}_3$ – $\text{Yb}_2\text{O}_3$  dual system was examined by such methods as XRF, DTA, IR spectroscopy, SEM and energy dispersive X-ray analysis during the sedimentation and subsequent annealing at the temperature of 100, 650, 900, 1200°C.

Differential Thermal Analysis have been performed by NETZSCH STA 449F3 derivatograph (Germany). The heating range was up to 800°C.

SEM measurements have been performed by S-4800 «Hitachi» (Japan).

IR spectra were recorded using IR spectrometer Nexus Nicolet 5700 «Thermo Electron Corporation» (USA).

## Results and Discussion

Based on the analysis of derivatograms of the dried sediment (Fig. 1) we conclude that the decomposition of hydrated sediment of Erbium and Ytterbium hydroxides mixture follows a probable pattern  $\text{Er-Yb}(\text{OH}) \cdot y\text{H}_2\text{O} \rightarrow \text{Er-Yb}(\text{OH})_3 \rightarrow (\text{Er-Yb})_2\text{O}_3$ . At 60 °C the hydrated oxides mixture recrystallizes, at 90 °C free water is removed, then hydroxyl groups are removed, and at about 600 °C mixed oxides are formed (exothermic effect), it being followed by slow removal of residual hydroxyl groups to the constant mass (loss of mass of 45%).

Based on the analysis of XRD data on the obtained powders [26] (Fig. 2) it can be concluded that the samples obtained at 650 °C show main diffraction peaks broadening effect, from which it follows that at this temperature the crystalline phase of cubic Erbium oxide has already been formed, the precursor particles are  $18 \pm 2$  nm in size, at annealing temperature of 900 °C the particle size increases to

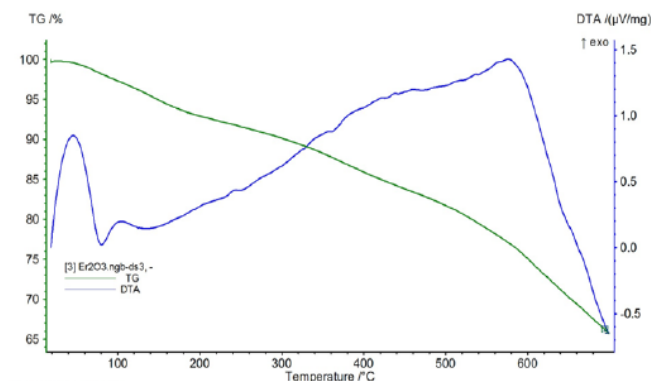


Figure 1. Derivatograms of the dried sediment.

$27 \pm 2$  nm, and at 1200 °C the particle size is within range of  $30 \pm 2$  nm. In all the cases, at the temperatures ranging from 650 to 1200 °C the powders have a cubic crystalline structure of  $\text{Er}_2\text{O}_3$  chemical composition (JCPDF 77-0777).

$\text{Yb}_2\text{O}_3$  peaks in  $\text{Er}_2\text{O}_3$  mixture in radiographs are almost indiscernible because these oxides have the same single-phase cubic structure, the difference in the peaks being only about 0.08 degrees (they almost coincide, the images must have very high resolution), Ia-3, Z=16 space group is also the same. Grid parameter  $a = 1,0435$  ( $\text{Yb}_2\text{O}_3$ );  $a = 1,0563$  ( $\text{Er}_2\text{O}_3$ ). The mixtures form solid solution, so elemental composition can be determined by EDX analysis.

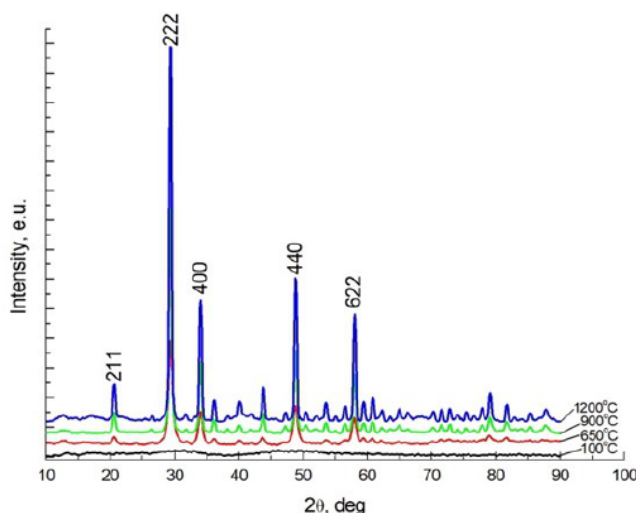


Figure 2. XRD data on the obtained powders  $\text{Er}_2\text{O}_3$ :Yb.

Table 1

Elemental composition of the obtained powders at 650 °C

Element	Atomic number	wt. %	wt. % (standardized)	Atomic %
Oxygen, O	8	14,13	15,88	55,77
Erbium, Er	68	65,91	74,09	24,89
Ytterbium, Yb	70	5,63	6,33	2,06
Carbon, C	6	3,29	3,70	17,29
Total content, %		88,96	100	100

Examination of the elemental composition of the obtained powders by energy dispersion microanalysis (EDX) that is used to determine local chemical composition of phases, showed presence of erbium, ytterbium and oxygen atoms in composition in the following ratio (Table 1).

Presence of carbon impurities in the powder annealed at 650 °C is related to residual compounds formed during decomposition of Er/Yb (OH)CO<sub>3</sub>·yH<sub>2</sub>O hydrated Erbium Ytterbium oxide compound, which is confirmed by IR spectroscopy and IR analysis; it significantly decreases during subsequent annealing: 3.29 (650 °C);

2.52 (900 °C); 2.16 (1200 °C) wt. %.

Based on analysis of SEM data for the obtained samples, it can be concluded that the powders produced after the heat treatment at 650 °C are to a great extent agglomerated, have a laminar, plate-like morphology with the laminate thickness of about 50 nm and its length of 10–15 μm (Fig 3). When the sediment is annealed at 900 °C, laminar particles split into fibres of 30–60 nm in diameter, and when fibres making up agglomerates are further annealed, they fuse forming a mesh structure, the size of the annealed particles being 150–200 nm (Fig. 4 and 5).

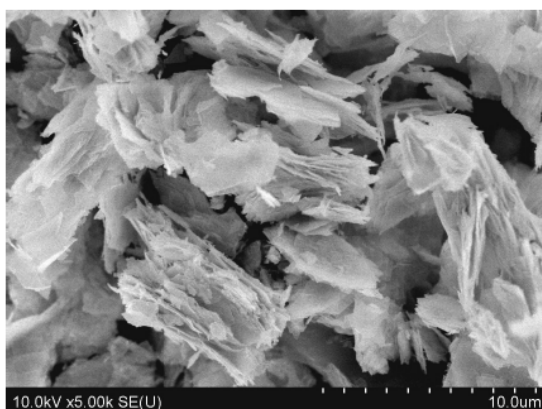


Figure 3. Morphology of Er<sub>2</sub>O<sub>3</sub>-Yb<sub>2</sub>O<sub>3</sub> powders after the heat treatment at 650 °C.

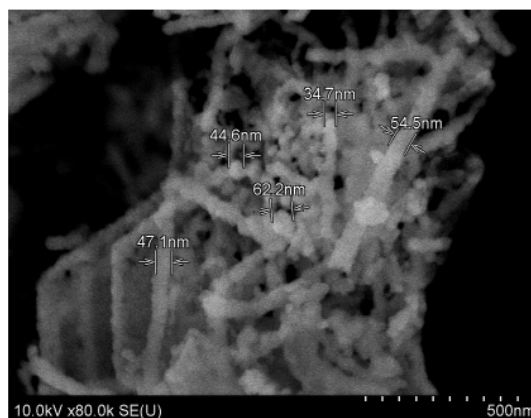
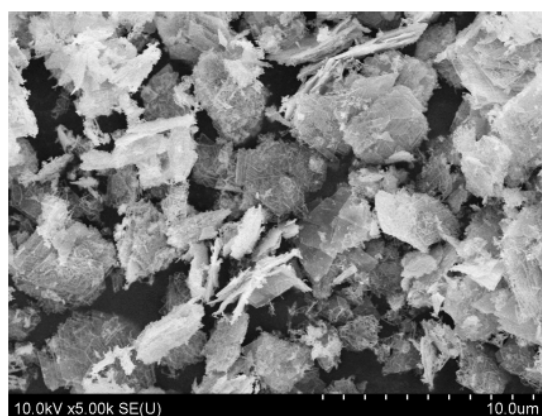


Figure 4. Morphology of Er<sub>2</sub>O<sub>3</sub>-Yb<sub>2</sub>O<sub>3</sub> powders after the heat treatment at 900 °C.

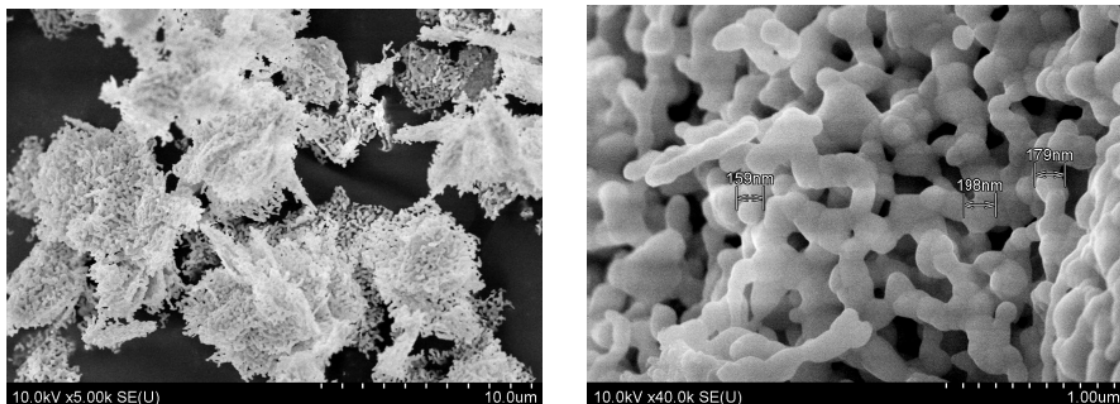


Figure 5. Morphology of  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  powders after the heat treatment at  $1200\text{ }^\circ\text{C}$ .

IR spectra curves for  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  powders at the temperatures of 100, 650, 900 and  $1200\text{ }^\circ\text{C}$  are shown in the Fig. 6. Based on examination of IR reflection curves received from the dried sediment samples annealed in the air atmosphere at 100, 650, 900 and  $1200\text{ }^\circ\text{C}$ , it can be concluded that  $\text{CO}_3^{2-}$  stretching vibrations appear as broad bands at 1580 (asymmetrical), 1390 and  $\text{CO}_3^{2-}$  out-of-plane deformation vibrations – at  $848\text{ cm}^{-1}$  (for the annealing temperatures of 100, 650 and  $900\text{ }^\circ\text{C}$ ) [27], their intensity decreasing as the heating temperature increases. Band at  $563\text{ cm}^{-1}$  refers to Er–O bond vibrations. Band at  $3600\text{ cm}^{-1}$  is most likely related to O–H groups vibrations, absorbed by  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3 \cdot x(\text{OH})$  porous particles [28], however, it is significantly reduced at the annealing temperature of  $1200\text{ }^\circ\text{C}$ .

Based on the analysis of the IR spectra of the dried sediment we conclude that erbium-ytterbium carbohydrate decomposes according to the scheme  $\text{Er/Yb}(\text{OH})\text{CO}_3 \cdot y\text{H}_2\text{O} \rightarrow \text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3 \cdot x(\text{OH}) \rightarrow \text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$ , two intermediate compounds being formed [29, 30].

The reduced concentration of hydroxyl groups, adsorbed gaseous impurities and the residual carbon-containing impurities resulting from annealing at  $T=1200\text{ }^\circ\text{C}$  confirms the viability of

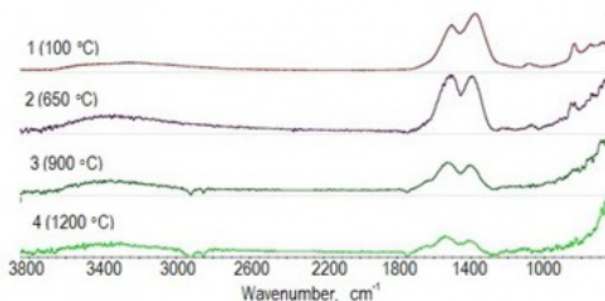


Figure 6. IR spectra curves for  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  powders at the temperatures of 100, 650, 900 and  $1200\text{ }^\circ\text{C}$ .

the high temperature treatment at formation of dehydrated  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  samples.

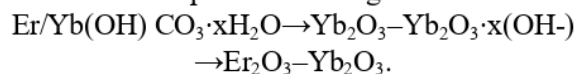
### Conclusion

A new method has been developed to produce the ultra-dispersed powders of  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  system using deposition and heat treatment processes by co-deposition of salt solutions mixed with ammonium carbonate and hydrogen peroxide complex solution at low temperature.

It has been established that during drying and annealing at 100, 650, 900 and  $1200\text{ }^\circ\text{C}$  in the air atmosphere, water is removed from sediment, further at the temperature of  $650\text{ }^\circ\text{C}$  volatile organic pollutants that may have been contained in  $(\text{NH}_4)_2\text{CO}_3$  are removed from the carbonates, and at the temperature of  $900\text{ }^\circ\text{C}$  the carbonates decompose to hydrated erbium ytterbium oxide compound.

It is shown that the reduction of concentration of hydroxyl groups, adsorbed gaseous impurities and residual carbon-containing impurities as a result of annealing at  $T=1200\text{ }^\circ\text{C}$  confirms viability of high temperature treatment for forming dehydrated  $\text{Er}_2\text{O}_3\text{-Yb}_2\text{O}_3$  samples.

The kinetics of the deposition reactions and crystallization conditions of mixed lanthanide carbonates in  $\text{Er/Yb}(\text{OH})\text{CO}_3 \cdot x\text{H}_2\text{O}$  was examined by the methods of X-ray diffraction, DTA, IR spectroscopy, SEM and energy dispersive X-ray analysis. It has been established that Erbium-Ytterbium carbonate decomposes according to scheme



The powders obtained after the heat treatment at  $650\text{ }^\circ\text{C}$  have been found to be to a great extent agglomerated, they have a laminar, plate-like morphology with the laminate thickness of about 50 nm, length of 10–15  $\mu\text{m}$ , when sediment

is annealed at 900 °C, laminar particles split into 30–60 nm diameter fibers, and during subsequent heating the fibers in agglomerates fuse together forming a mesh structure, the size of the annealed particles being 150–200 nm.

### Acknowledgments

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