

## Original articles

Research article

<https://doi.org/10.17308/kcmf.2022.24/10256>Up-conversion luminescence in mixed crystals  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$  doped with  $\text{Er}^{3+}$  ions excited at 1532 nmA. S. Nizamutdinov<sup>1</sup>✉, S. A. Bukarev<sup>2</sup>, S. V. Gushchin<sup>2</sup>, A. A. Shavelev<sup>1</sup>, M. A. Marisov<sup>1,3</sup>, A. A. Shakirov<sup>1</sup>, A. G. Kiiamov<sup>1</sup>, A. V. Astrakhantseva<sup>1</sup>, A. A. Lyapin<sup>2</sup><sup>1</sup>Kazan Federal University,  
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## Abstract

Increasing the efficiency of upconversion luminophores is an important problem in materials science. Here we report on approach of use of disordered crystals namely solid solutions  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$  doped with  $\text{Er}^{3+}$  ions to improve spectral characteristics and efficiency of upconversion from near IR spectral region. It is shown that investigated compound provide up to 9.4% of external energy yield of the up-conversion luminescence at an excitation power density of 6 W/cm<sup>2</sup> at 1532 nm for the 10 at.% of  $\text{Er}^{3+}$  doping. Also the investigated crystals of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  allow the control of the CCT parameter in the range of 2384–5149 K by changing the concentration and power density of the excitation. Advantages revealed in this work for crystalline compounds  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  such as wide absorption bands in the infrared spectral range, a high external energy yield, and a controllable distribution of intensity of luminescence bands makes them prospective to improve the efficiency of double-sided solar cells.

**Keywords:** Upconversion luminescence, External energy yield, Correlated color temperatures,  $\text{Er}^{3+}$ , Fluoride crystals, Solid solutions

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## 1. Introduction

Up-conversion materials doped with rare earth ions are widely used for various practical applications. These materials are used as markers to detect counterfeit products, laser radiation visualizers, solid-state lasers, biological sensors, and in solar panels to increase their efficiency. Fluoride matrices are characterized by large band gap and low energy spectrum of phonons which provides low level of nonradiative losses of excited population of levels and make them efficient in luminescent and upconversion applications [1–4].

One of the approaches to increase the efficiency of luminophores is to investigate disordered crystal lattices. First, such lattices may provide broadening of spectral lines which leads to increase of energy transfer efficiency between impurity ions and upconversion luminescence efficiency. Second, distortions of crystal lattice may lead to increase of electronic transitions probabilities which are relatively low for forbidden intraconfigurational  $f$ - $f$  transitions of rare-earth ions. Despite low probabilities the  $f$ - $f$  transitions of rare earth ions are widely used since they possess spectral lines in a variety of spectral ranges. One of the most popular luminescent ions for upconversion investigation is  $\text{Er}^{3+}$  ion [5,6]. Due to relatively large lifetime of excited states and significant manifestation of cross-relaxation processes it provide efficient upconversion with emitting photons of energy of two and higher photons of excitation radiation [7]. High efficiency of upconversion process in  $\text{Er}^{3+}$ -doped fluorides make it possible to achieve the laser oscillation at wavelengths of about  $0.55 \mu\text{m}$  (the  ${}^4\text{S}_{3/2}$ - ${}^4\text{I}_{15/2}$  transition) when pumping at  $980 \text{ nm}$  [8]. Also the record value for internal photoluminescence quantum yield for upconversion luminescence was measured for  $\text{Er}^{3+}$ - $\text{Yb}^{3+}$  ion pair [9, 10]. More interesting that  $\text{Er}^{3+}$  ions allow excitation of anti-stokes luminescence under pumping at  $1530 \text{ nm}$  where intensity of the sun light is still significant and photo effect from crystalline silicon is already absent. As it is known from literature the efficiency of  $\text{Er}^{3+}$  upconversion luminescence excited at  $1530 \text{ nm}$  can be even higher than that for  $980 \text{ nm}$  excitation most

probably due to longer lifetime of  ${}^4\text{I}_{15/2}$  state [11] and it can provide efficient energy harvesting due to stronger absorption cross-section at  $1.5 \text{ micrometers}$  spectral range [11,12]. For example, the microcrystalline powder of  $\beta$ -phase  $\text{NaYF}_4$  with 20% of  $\text{Er}^{3+}$  have shown 0.34% of external quantum efficiency at an irradiance of  $1090 \text{ W}\cdot\text{m}^{-2}$  ( $0.03 \text{ cm}^2\cdot\text{W}^{-1}$ ) at  $1522 \text{ nm}$ , polycrystalline samples of the same  $\text{NaYF}_4$ :  $\text{Er}^{3+}$  (20%) have provided 2.5% being incorporated into an optically transparent (refractive index similar to that of the phosphors) acrylic adhesive medium (weight ratio 0.4:0.6) [13] and 6.5% being incorporated into perfluorocyclobutane (PFCB) host matrix [14] external quantum efficiency when irradiated at  $1523 \text{ nm}$ .

The material under investigation is from family of  $\text{BaY}_2\text{F}_8$  fluoride matrices which are characterized by monoclinic structure and significant vibrational broadening. It is already known that it provides significant efficiency of conversion from IR spectral range, for example, the external quantum efficiency (EQE) at  $1557 \text{ nm}$  measured values of 6.5% and 4.1% have been reached under  $8.5 \text{ W cm}^{-2}$  power density illumination for the 30%  $\text{Er}^{3+}$  and 20%  $\text{Er}^{3+}$  samples, respectively [15]. We studied the upconversion luminescence of mixed crystals  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$  doped with  $\text{Er}^{3+}$  ions upon excitation by infrared laser radiation at a wavelength of  $1532 \text{ nm}$  to the  ${}^4\text{I}_{13/2}$  energy level of  $\text{Er}^{3+}$  ions.

Aim of this work is to study the efficiency of light conversion in  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$ : $\text{Er}$  crystals from the spectral range of photon energies lower than perovskite and silicon photoeffect maxima to near IR and visible spectral ranges. Namely, the spectral characteristics of up-conversion luminescence and its efficiency under laser excitation to the  ${}^4\text{I}_{13/2}$  energy level of  $\text{Er}^{3+}$  ions have been investigated.

## 2. Research methods and characterization of materials

Single crystals of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$ : $\text{Er}$  (1 at.%, 5 at.%, 10 at.%, 20 at.%, 30 at.%) were grown in Kazan Federal University by the Bridgman method, which consists in moving the crucible with the melt from the high-temperature zone to the lower-temperature. Crystal growth was implemented in graphite crucibles.

The absorption spectra of  $\text{Er}^{3+}$  ions in the studied fluoride crystals were registered in a Perkin Elmer Lambda 950 double-beam spectrophotometer with a double monochromator. The upconversion luminescence spectra of  $\text{Er}^{3+}$  ions were registered using spectroradiometer OL-770 VIS/NIR (slit spectral width 100  $\mu\text{m}$ ). To excite the upconversion luminescence of  $\text{Er}^{3+}$  ions to the  $^4\text{I}_{13/2}$  level, we used a CW fiber laser with  $\lambda_{\text{rad}} \sim 1532$  nm. The diameter of the laser beam incident on the sample was measured using a BEAMAGE-3.0 beam analyzer (GenTec-EO) and appeared to be  $420 \pm 10$   $\mu\text{m}$ .

To calculate the chromaticity coordinates and correlated color temperatures (CCT), the CIE 1931 (X, Y) standard colorimetric system was used.

To determine the internal energy yield ( $B_{\text{in}}$ ) of up-conversion luminescence, an experimental setup was elaborated based on OL IS-670-LED integrating sphere and OL-770 VIS/NIR spectroradiometer. Continuous laser radiation after a quartz achromatic depolarizer was focused onto a sample located at the center of the sphere. The spectral power of the upconversion luminescence was measured using a spectroradiometer. The integrating sphere and the spectroradiometer were connected by

an optical fiber. The experimental setup was corrected for spectral sensitivity using a Gooch & Housego reference halogen lamp with a known spectral output power. The power of the laser radiation incident on the sample was measured using a power meter based on an integrating sphere with high sensitivity.

The external energy yield ( $B_{\text{ex}}$ ) of up-conversion luminescence was determined as the ratio of the power of upconversion luminescence in a certain spectral range to the power of laser radiation incident on the sample.

All measurements were performed at room temperature.

### 3. Experimental results

#### 3.1. Up-conversion luminescence of $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}$ crystals

In accordance with X-ray powder diffraction patterns (Fig. 1), for the synthesized  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$  crystals, the samples represented the same phase as their homologue  $\text{BaY}_2\text{F}_8$ , belonging to the monoclinic system, space group C12/m1 [16]. The differences in the position of the reflections are very small, although for the  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8$  mixed crystal they are slightly shifted to the region of small angles.

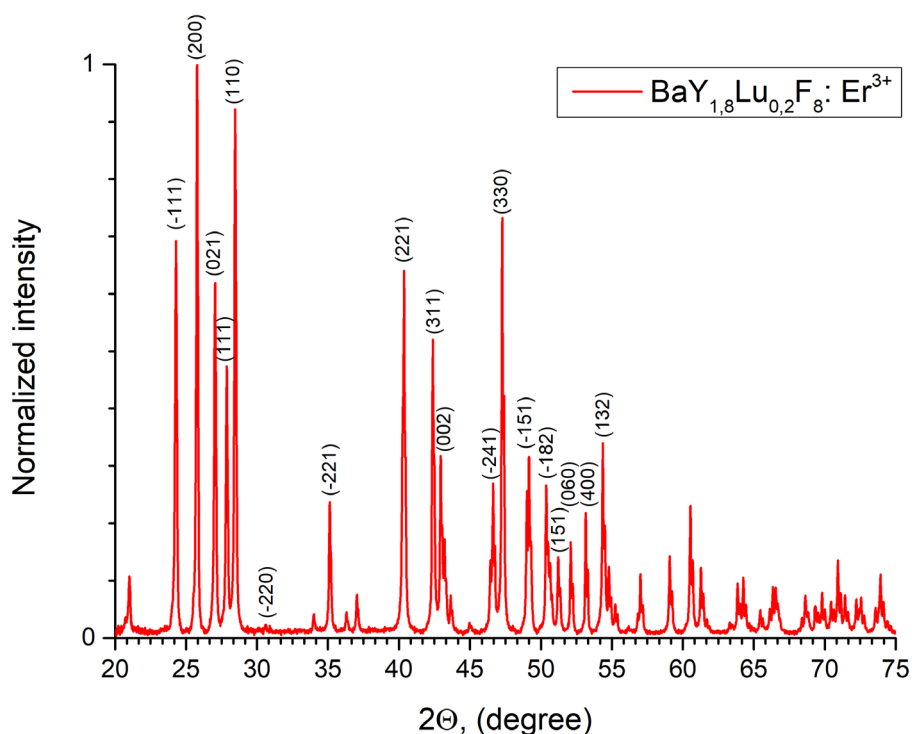
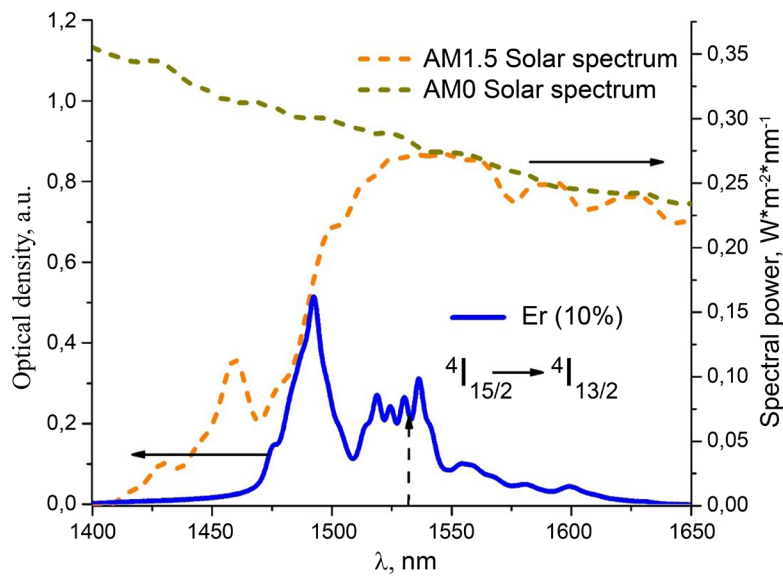


Fig. 1. X-ray pattern of the crystalline compound  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$

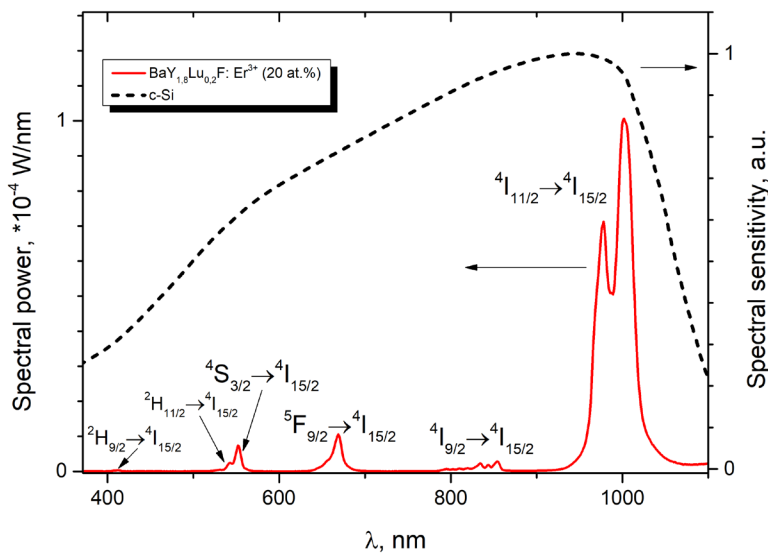
The absorption spectrum of the BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er<sup>3+</sup> crystal for the <sup>4</sup>I<sub>15/2</sub> → <sup>4</sup>I<sub>13/2</sub> electronic transition of Er<sup>3+</sup> ions in the spectral range 1400–1700 nm is shown at the Fig. 2 together with the spectrum of solar radiation above the atmosphere (the spectrum of the Sun AM0) and at the surface (the spectrum of the Sun AM1.5) of Earth. It can be seen that the absorption band of the BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er<sup>3+</sup> crystal overlaps well with the solar radiation spectrum. The dotted arrow in the spectrum also shows the wavelength of excitation of upconversion luminescence to the <sup>4</sup>I<sub>13/2</sub> level of Er<sup>3+</sup> ions in our investigation ( $\lambda_{ex} \sim 1532$  nm).

The up-conversion luminescence spectra of the BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er (20 at.%) crystal is shown at the Fig. 3, it consist of the electronic transitions <sup>2</sup>H<sub>9/2</sub> → <sup>4</sup>I<sub>15/2</sub>, <sup>2</sup>H<sub>11/2</sub> → <sup>4</sup>I<sub>15/2</sub>, <sup>4</sup>S<sub>3/2</sub> → <sup>4</sup>I<sub>15/2</sub>, <sup>4</sup>F<sub>9/2</sub> → <sup>4</sup>I<sub>15/2</sub>, <sup>4</sup>I<sub>9/2</sub> → <sup>4</sup>I<sub>15/2</sub>, <sup>4</sup>I<sub>11/2</sub> → <sup>4</sup>I<sub>15/2</sub> of Er<sup>3+</sup> ions. The highest luminescence intensity is observed for the band in the region of 1000 nm corresponding to the <sup>4</sup>I<sub>11/2</sub> → <sup>4</sup>I<sub>15/2</sub> of Er<sup>3+</sup> ions.

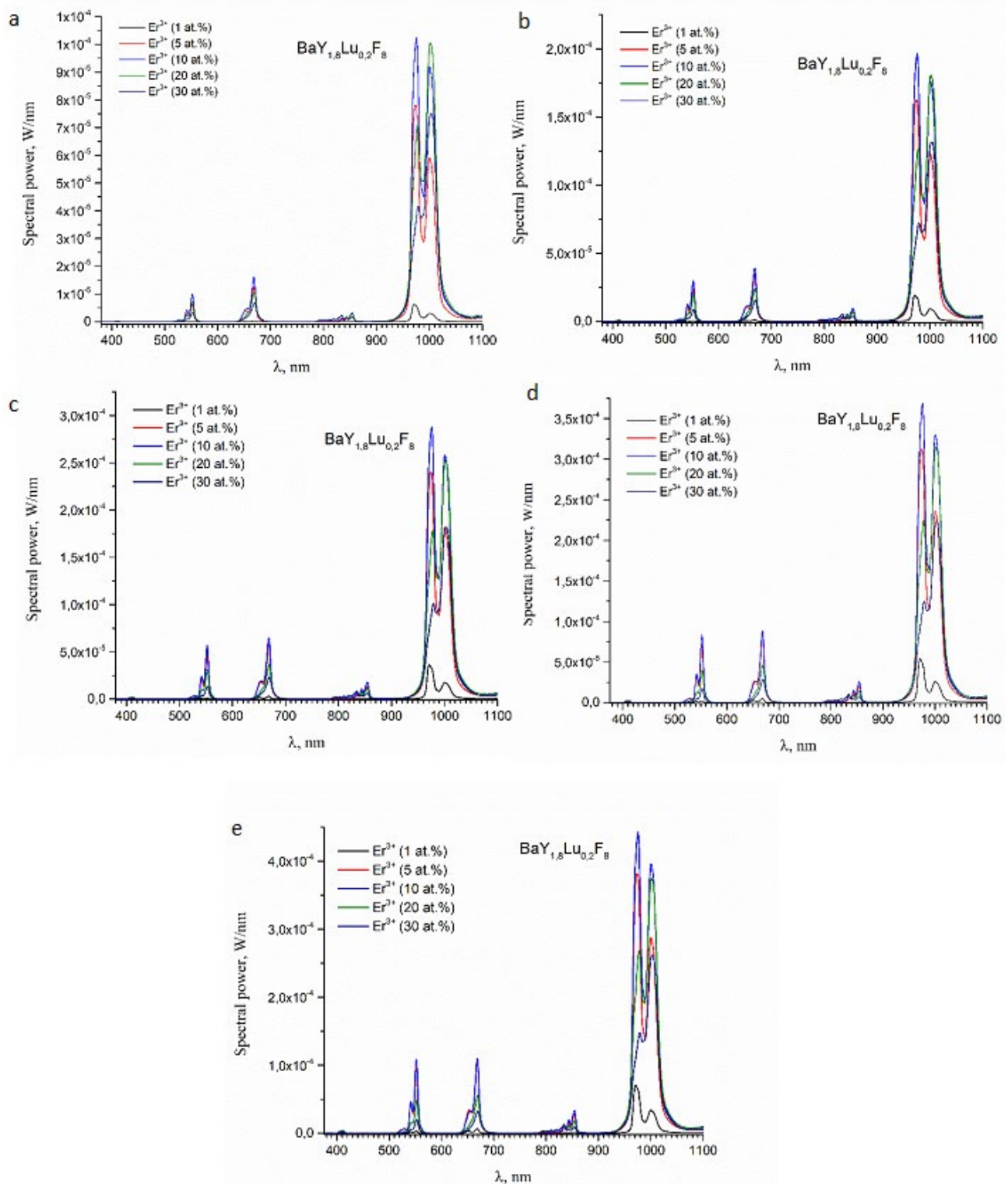
The evolution of luminescence spectra with power density of the 1532 nm laser excitation is shown at the Fig. 4. The intensity of anti-stokes luminescence rises with the excitation power and changes in the form of the spectra are observed in



**Fig. 2.** Absorption spectrum of the BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er<sup>3+</sup> (10 at.%) crystal for the <sup>4</sup>I<sub>15/2</sub> → <sup>4</sup>I<sub>13/2</sub> transition of Er<sup>3+</sup> ions and the spectrum of solar radiation above the atmosphere (AM0) and on the earth’s surface (AM1.5)



**Fig. 3.** Upconversion luminescence spectra of BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er (20 at.%) crystals and spectral sensitivity of crystalline silicon



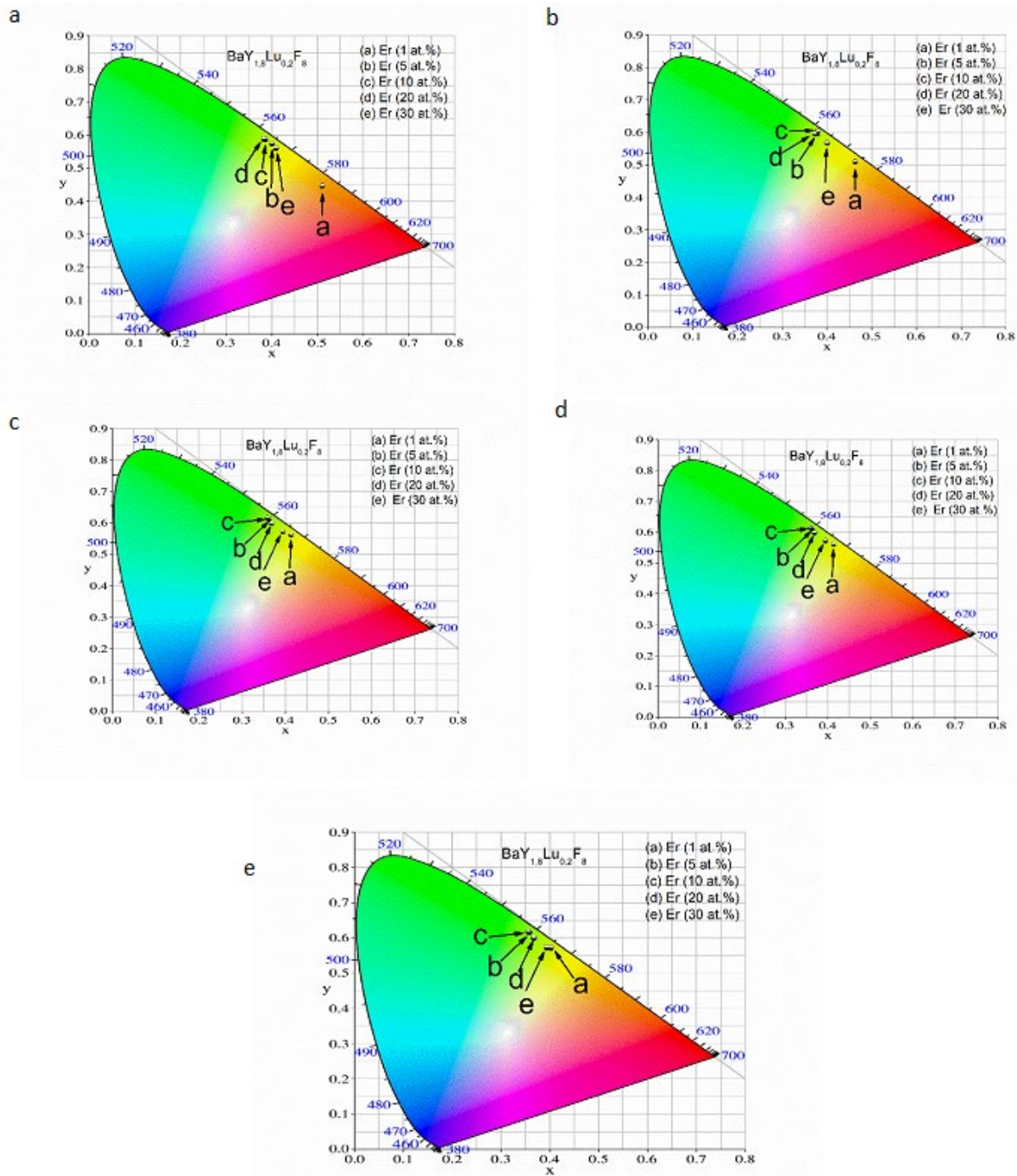
**Fig. 4.** Dependence of the intensity of the up-conversion luminescence of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  crystals on the laser excitation power density: a) 6  $\text{W}/\text{cm}^2$ , b) 12.7  $\text{W}/\text{cm}^2$ , c) 19  $\text{W}/\text{cm}^2$ , d) 25.5  $\text{W}/\text{cm}^2$ , e) 32  $\text{W}/\text{cm}^2$

the investigated range from 6 W/cm<sup>2</sup> to 32 W/cm<sup>2</sup> which are discussed further.

The highest luminescence intensity for all electronic transitions of  $Er^{3+}$  ions in the range of 300–1100 nm is observed for the sample with a concentration of 10 at.% of  $Er^{3+}$  ions. The intensity of the up-conversion luminescence of  $Er^{3+}$  ions increases

with increasing concentration up to 10 at.%, then concentration quenching is observed for samples with a concentration of 20 at.% and 30 at.%. This concentration dependence was observed for all values of the excitation power density.

The CCT and chromaticity coordinates of the radiation of up-conversion phosphors



**Fig. 5.** Chromatic diagram of upconversion luminescence for  $BaY_{1.8}Lu_{0.2}F_8:Er$  crystals when excited by laser radiation with a wavelength of 1532 nm and a power density of 6 W/cm<sup>2</sup> (a), 12.7 W/cm<sup>2</sup> (b), 19 W/cm<sup>2</sup> (c), 25.5 W/cm<sup>2</sup> (d), and 32 W/cm<sup>2</sup> (e)

BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er<sup>3+</sup> were studied. Fig. 5 shows location at the chromatic diagram for the up-conversion luminescence of the studied crystals upon excitation by laser radiation with a power density of 6 W/cm<sup>2</sup>, 12.7 W/cm<sup>2</sup>, 19 W/cm<sup>2</sup>, 25.5 W/cm<sup>2</sup>, and 32 W/cm<sup>2</sup>. The values of CCT and chromaticity temperatures are presented in Table 1.

The CCT and the chromaticity coordinates of the emission of fluoride phosphors depend on the excitation power density and the concentration of rare earth ions. For the BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>:Er<sup>3+</sup> (20 at.%) sample, green-yellow emission is observed with a CCT of 4767-5002 K at an excitation power density of 6–32 W/cm<sup>2</sup>. As the excitation power density increases, the CCT also increases.

**Table 1.** CCT values, chromaticity coordinates, internal and external energy yield of up-conversion luminescence in BaY<sub>1.8</sub>Lu<sub>0.2</sub>F<sub>8</sub>: Er crystals

	BaY <sub>1.8</sub> Lu <sub>0.2</sub> F <sub>8</sub> : Er (1 %)	BaY <sub>1.8</sub> Lu <sub>0.2</sub> F <sub>8</sub> : Er (5 %)	BaY <sub>1.8</sub> Lu <sub>0.2</sub> F <sub>8</sub> : Er (10 %)	BaY <sub>1.8</sub> Lu <sub>0.2</sub> F <sub>8</sub> : Er (20 %)	BaY <sub>1.8</sub> Lu <sub>0.2</sub> F <sub>8</sub> : Er (30 %)
Internal B <sub>en</sub> (6 W/cm <sup>2</sup> ), %	2.3	17.8	20.4	10.7	6.9
Internal B <sub>en</sub> (12.7 W/cm <sup>2</sup> ), %	3.2	19.4	19.0	9.9	6.2
Internal B <sub>en</sub> (19 W/cm <sup>2</sup> ), %	4.5	20.5	19.5	9.3	5.7
Internal B <sub>en</sub> (25.5 W/cm <sup>2</sup> ), %	5.1	20.2	19.0	8.9	5.4
Internal B <sub>en</sub> (32 W/cm <sup>2</sup> ), %	5.6	18.7	18.3	8.6	5.1
External B <sub>en</sub> (6 W/cm <sup>2</sup> ), %	0.4	6.4	9.4	9.0	6.5
External B <sub>en</sub> (12.7 W/cm <sup>2</sup> ), %	0.6	6.9	9.3	8.3	5.8
External B <sub>en</sub> (19 W/cm <sup>2</sup> ), %	0.7	7.1	9.3	7.8	5.4
External B <sub>en</sub> (25.5 W/cm <sup>2</sup> ), %	0.8	7.1	9.1	7.4	5.1
External B <sub>en</sub> (32 W/cm <sup>2</sup> ), %	0.9	7.0	8.9	7.1	4.8
CCT (6 W/cm <sup>2</sup> ), K	2384	4469	4767	4704	4304
CCT (12.7 W/cm <sup>2</sup> ), K	3339	4827	4943	4912	4453
CCT (19 W/cm <sup>2</sup> ), K	3922	4992	5073	4959	4511
CCT (25.5 W/cm <sup>2</sup> ), K	4249	5087	5141	4985	4553
CCT (32 W/cm <sup>2</sup> ), K	4441	5149	5181	5002	4575
Chromaticity coordinates (6 W/cm <sup>2</sup> )	X= 0.5107 Y=0.4507	X=0.4010 Y=0.5786	X= 0.3866 Y=0.5918	X=0.3824 Y=0.5928	X=0.4096 Y=0.5644
Chromaticity coordinates (12.7 W/cm <sup>2</sup> )	X= 0.4630 Y=0.5136	X= 0.3792 Y=0.6006	X= 0.3718 Y=0.6070	X=0.3734 Y=0.6003	X=0.4009 Y=0.5719
Chromaticity coordinates (19 W/ cm <sup>2</sup> )	X= 0.4316 Y=0.5454	X= 0.3689 Y=0.6110	X= 0.3635 Y=0.6151	X=0.3704 Y=0.6030	X=0.3974 Y=0.5751
Chromaticity coordinates (25.5 W/cm <sup>2</sup> )	X= 0.4137 Y=0.5660	X= 0.3627 Y=0.6167	X= 0.3590 Y=0.6191	X=0.3686 Y=0.6033	X=0.3947 Y=0.5762
Chromaticity coordinates (32 W/cm <sup>2</sup> )	X= 0.4025 Y=0.5760	X= 0.3586 Y=0.6208	X= 0.3564 Y=0.6215	X=0.3676 Y=0.6042	X=0.3934 Y=0.5774

With an increase in the concentration of  $\text{Er}^{3+}$  ions from 1 at.% to 30 at.%, the CCT to a concentration of 10 at.% increases in the range of 2384–4767 K, and then decreases again to 4304 K at an excitation power density of 6 W/cm<sup>2</sup>. The observed character of the increase in CCT with an increase in the concentration of  $\text{Er}^{3+}$  ions manifests itself for all studied excitation power densities. The dependence of CCT on the concentration of rare earth ions and the excitation power density is presented in Table 1.

### 3.2. Energy yield of upconversion luminescence of $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$

Internal and external energy yield are important characteristics when evaluating the application of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}$  crystalline compounds in photovoltaics to increase the efficiency of solar cells and protect securities. The internal energy yield of up-conversion luminescence in the range of 400–1100 nm was determined as the ratio of the radiation power to the absorbed excitation power for crystals with an  $\text{Er}^{3+}$  ion concentration of 1 at.%, 5 at.%, 10 at.%, 20 at.% and 30 at.% respectively. It can be seen that the maximum yield value corresponds to the sample with a concentration of 10 at.%  $\text{Er}^{3+}$  ions. The value of the internal energy output when excited by other power densities is presented in Table 1.

The maximum value of the external energy yield of the up-conversion luminescence was 9.4% at an excitation power density of 6 W/cm<sup>2</sup> for the  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  (10 at%) crystal which appears to be higher than that for  $\text{BaY}_2\text{F}_8:\text{Er}$  result from literature [15] and at the level of  $\text{Yb}^{3+}$  codoped luminophore  $\text{BaF}_2:\text{Er},\text{Yb}$  [10].

## 4. Conclusions

In this work, we studied the upconversion luminescence characteristics of the concentration series of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  crystals grown by the Bridgman method. The studied crystals showed intense up-conversion luminescence upon excitation of  $\text{Er}^{3+}$  ions to the  $^4\text{I}_{13/2}$  level by laser radiation with a wavelength of 1532 nm. The luminescence spectra consisted of electronic transitions  $^2\text{H}_{9/2} \rightarrow ^4\text{I}_{15/2}$ ,  $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ ,  $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ ,  $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ ,  $^4\text{I}_{9/2} \rightarrow ^4\text{I}_{15/2}$ ,  $^4\text{I}_{11/2} \rightarrow ^4\text{I}_{15/2}$   $\text{Er}^{3+}$  ions.

The maximum value of the external energy yield of the up-conversion luminescence was 9.4% at an excitation power density of 6 W/cm<sup>2</sup> for the  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  (10 at.%) crystal which appeared to be of optimal chemical composition. The maximum value of the internal energy yield of the up-conversion luminescence was 20.4% at an excitation power density of 6 W/cm<sup>2</sup> for the  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  (10 at.%) crystal.

Also the investigated crystals of  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  allow the control of the up-conversion luminescence spectra shape. By changing the concentration and power density of the excitation, it was possible to obtain up-conversion radiation with CCT in the range of 2384–5149 K.

Advantages revealed in this work for crystalline compounds  $\text{BaY}_{1.8}\text{Lu}_{0.2}\text{F}_8:\text{Er}^{3+}$  such as wide absorption bands in the infrared spectral range, a high external energy yield, and a controllable distribution of intensity of luminescence bands makes them prospective to improve the efficiency of double-sided solar cells.

## Contribution of the authors

The authors contributed equally to this article

## Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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