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Original article

The Study of the Luminescence of Solid Solutions Based on Yttrium Fluoride Doped with Ytterbium and Europium for Photonics

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Abstract

The majority of the global market for solar photovoltaic devices is based on silicon technology. It is very important to increase their efficiency through the use of luminescent coatings, including those converting radiation from the UV-blue region of the spectrum into the near-infrared range, where silicon absorbs radiation most efficiently (Stokes or down-conversion luminescence), or from the infrared region of the spectrum in the near-infrared range (up-conversion luminescence). The aim of this research was to synthesize and study the spectral-kinetic characteristics of single-phase solid solutions of $Y_{1-x-y}Eu_xYb_yF_3$ and to determine the quantum yield of down-conversion luminescence.

Using the method of high-temperature melting, single-phase samples of solid solutions of $Y_{1-x-y}Eu_xYb_yF_3$ with orthorhombic system were synthesized. For the series of samples with different Eu^{3+}/Yb^{3+} ratios, upon double doping with these ions, the formation of the corresponding solid solutions with a crystal lattice of the β-YF_3 phase was confirmed. Their chemical composition was determined using the energy dispersion analysis, and it was established that it corresponds to the nominal one. It was shown that both Eu^{3+} and Yb^{3+} ions become luminescent upon excitation at wavelengths of 266 nm and 296 nm. This indicates these compounds as promising sensitisers of UV radiation. In this case, upon excitation at a wavelength of 266 nm, luminescence of Eu^{2+} ions was recorded. The maximum quantum yield values (2.2 %) of the ytterbium down-conversion luminescence in the near-infrared wavelength range upon excitation at a wavelength of 266 nm were recorded for YF_3:Eu:Yb with the Eu^{3+}:Yb^{3+} ratios of 0.1:10.0 and 0.05:5.00.

Keywords: rare earth fluorides, phosphors, solar panels, down-conversion luminescence.

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1. Introduction

The majority of the global market for solar photovoltaic devices is based on silicon technology. According to a report by the Fraunhofer Institute for Solar Energy (Fraunhofer ISE) in 2017, the widespread use of silicon is based on its availability and low cost of raw materials, the perfection of the technology for producing silicon of the required purity, and its non-toxicity to humans and the environment [1]. This is due to a significant simplification of the technology for purifying cheap silicon to an acceptable level [2]. From 2008 to 2017, there was a significant decrease in the cost of solar electricity from 3 US dollars / W to 0.3 US dollars / W [1]. It should be pointed out that failed solar panels can now be recycled as waste electronic components (*e-waste*). This strongly distinguishes them from new intensively developed organohalide materials with a perovskite structure of the RPbX type (R is an organic radical, X is Br or I, or their solid solution) [3–5]. It should be noted that most of these substances are less chemically stable and decompose over several years, and the recycling of heavy elements requires specific industries and significant investments.

One of the significant disadvantages of silicon is its low efficiency (less than 25 % even for the best samples [6,7]) of converting sunlight into electricity. In reality, the 22.5 % efficiency of solar energy conversion was achieved in devices produced at one of the largest silicon solar panel production plants located in Novocheboksarsk, Russia. There are various options for increasing the efficiency of silicon solar cells with multilayer structures, structures with different surface architecture types, and luminescent coatings [8, 9]. The photosensitivity spectrum and the maximum generation of electricity by silicon do not coincide with the spectrum of the Sun [6]. The maximum photosensitivity of silicon is in the range of 900–1100 nm, which coincides with the spectral range of radiation of trivalent ytterbium ions. As a result, the efficiency of solar-silicon photovoltaic cells may be increased by using luminescent coatings.

Phosphors are suggested to be used for this purpose. They transform radiation from the UV-blue spectrum region (down-conversion luminescence) [6-11] or from the IR spectrum region (up-conversion luminescence) [12–14] to the near-infrared range due to a number of various processes, including step transitions between the states of the corresponding ions, energy transfer, or cooperative processes. In this range, silicon absorbs radiation most efficiently [6].

The quantum yield of up-conversion luminescence in the visible range or near-infrared range upon excitation in the range of 1.5–2.0 µm is very low [15–17], as two low-energy IR photons should be converted to one photon with higher energy in the near-infrared spectral range (NIR). The quantum efficiency of down-conversion luminescence is higher than that for up-conversion, because one ultraviolet or visible high-energy photon is converted to one or two NIR photons. The quantum efficiency of down-conversion luminescence in fluoride phosphors has been studied a lot for various matrices [18, 19], but the quantum yield, which is important for practical applications, has not been estimated.

The aim of this research was to synthesize and study the spectral-kinetic characteristics of solid solutions of $Y_{1-x-y}Eu_xYb_yF_3$ and to determine the quantum efficiency and quantum yield of down-conversion luminescence of ytterbium in the near-infrared range.

2. Experimental

Samples of yttrium fluoride-based solid solutions doped with ytterbium and europium were synthesized using the method of high-temperature melting. Yttrium fluoride, europium fluoride, and ytterbium fluoride had a purity degree of 99.99 % (*LANHIT,* Russia). The samples of $Y_{1-x-y}Eu_xYb_yF_3$ solid solutions were synthesized in a vacuum oven at a temperature of 1155 °C. The mixture was placed in the vacuum oven in a graphite crucible and was gradually heated to 940 °C, then the vacuum pumping was turned off, a mixture of gases (CF_4 and Ar) was introduced, and then it was smoothly heated to the melting temperature. The obtained melt was fluorinated and held at the process temperature for 30 minutes and then it was cooled to room temperature for 3 hours. The obtained samples were ground in an agate mortar.

All the samples were studied by X-ray diffraction analysis using a Bruker D8 Advanced diffractometer (Cu*K*a radiation), their unit cell parameters were calculated in Powder 2.0 software (DQ < 10). Their chemical composition was evaluated using a Carl

Zeiss NVision 40 scanning electron microscope with an energy dispersion spectrometer. Diffuse reflection spectra were recorded using a Lambda 950 Perkin Elmer spectrophotometer. The luminescence spectra were recorded on a Stellarnet EPP2000 spectrometer with a spectral resolution of 0.5 nm. A tunable wavelength laser system based on an Al_2O_3:Ti laser with second and third harmonic generators (LOTIS TII, 10 Hz, 10 ns) and a wavelength converter based on stimulated Raman scattering in gaseous H_2 were used as an impulse excitation source. The luminescence kinetics were recorded using MDR-23 and MDR-3 monochromators, a photomultiplier FEU-100 was used as a photodetector in the visible region of the spectrum, and a photomultiplier FEU-62 was used in the IR region of the spectrum. The time scan of the luminescence kinetics signals was carried out by a BORDO digital oscilloscope with a bandwidth of 200 MHz and a dynamic range of 10 bits. The direct measurement of the quantum yield of Stokes luminescence was carried out using a Thorlabs IS200 integrating sphere and a SOLAR S100 spectrometer, calibrated using a wide-range temperature lamp TRSh-2850 and a yellow glass optical filter ZhS-16. When measuring the quantum yield of luminescence, we used the technique from [20], which involves correcting the spectral characteristics of the luminescence recording system and calibrating the optical system using light sources with a given intensity.

3. Results

The X-ray diffraction pattern of the $Y_{0.949}Eu_{0.001}Yb_{0.05}F_3$ solution single-phase sample is provided in Fig. 1. It is typical for the whole series of samples. The results of indexing in orthorhombic system (structural type β-YF_3) are summarized in Table 1. The formation of a solid solution is confirmed by the absence of additional peaks compared to the corresponding JCPDS data and a change in the unit cell parameters of an undoped sample: YF_3 (a = 6.353 Å, b = 6.850 Å, c = 4.393 Å, JCPDS card # 74-0911).

The chemical composition was analysed based on energy dispersion analysis (Table 1), the results showed that the real composition corresponded to the nominal composition within the limits of the determination error of ± 0.5 mol%.

Luminescence of Eu^{3+} ions was recorded in the YF_3 samples doped with Eu and Yb ions, both upon excitation in the 399 nm region, characteristic of europium, and upon excitation in the UV region of the spectrum (296 and 266 nm). In the
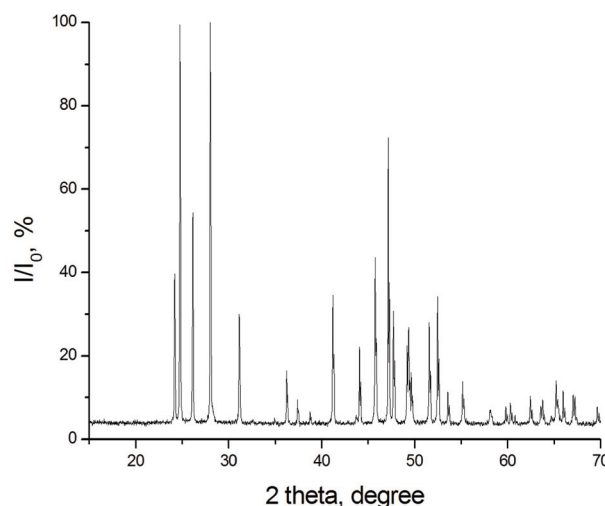

Fig. 1. The X-ray diffraction pattern of the $Y_{0.949}Eu_{0.001}Yb_{0.05}F_3$ solid solution sample

Table 1. The unit cell parameters and results of energy dispersion analysis of the $Y_{1-x-y}Eu_xYb_yF_3$ solid solutions

$Y_{1-x-y}Eu_xYb_yF_3$				
Nominal composition	Unit cell parameters, Å			Composition according to EDX
	a	b	c	
YF_3:Eu(0.05 мол.%):Yb(1.0 мол.%)	6.365(1)	6.859(2)	4.3909(7)	YF_3:Eu(0.2 мол.%):Yb(2.1 мол.%)
YF_3:Eu(0.1 мол.%):Yb(5.0 мол.%)	6.345(2)	6.839(3)	4.384(1)	YF_3:Eu(0.4 мол.%):Yb(6.4 мол.%)
YF_3:Eu(0.1 мол.%):Yb(1.0 мол.%)	6.342(2)	6.838(2)	4.3862(9)	YF_3:Eu(0.2 мол.%):Yb(1.9 мол.%)
YF_3:Eu(0.05 мол.%):Yb(10.0 мол.%)	6.339(2)	6.834(2)	4.384(1)	YF_3:Eu(*):Yb(12.9 мол.%)
YF_3:Eu(0.05 мол.%):Yb(5.0 мол.%)	6.348(1)	6.842(2)	4.3859(9)	YF_3:Eu(0.1 мол.%):Yb(7.1 мол.%)
YF_3:Eu(0.1 мол.%):Yb(10.0 мол.%)	6.3597(8)	6.851(1)	43891(6)	YF_3:Eu(0.1 мол.%):Yb(13.7 мол.%)

* - the europium concentration was not determined, since it is below the detection limit of the energy dispersion analysis.

corresponding spectra provided in Fig. 2, we can see the $^5D_0-^7F_n$ (691, 650, 615, 590 nm), $^5D_1-^7F_n$ (568, 556, 537, 526 nm), $^5D_2-^7F_n$ (511, 489, 464 nm) transitions, typical for Eu^{3+} [21]. Moreover, upon 266 nm excitation, a rather intense and wide luminescence band with a maximum in the region of 430 nm was recorded, which may correspond to the $5d-4f$ interconfigurational transitions of Eu^{2+} ions [22]. It is important that the luminescence of Yb^{3+} ions was recorded only upon excitation at 266 nm and 296 nm (Fig. 2), and in the first case, it appeared to be more intense.

The luminescence kinetics of Eu^{3+} at a wavelength of 615 nm and Yb^{3+} at a wavelength of 1020 nm upon excitation at a wavelength of 266 nm for the YF_3 matrix are provided in Fig. 3. The luminescence decay is non-exponential, for Yb^{3+} ions also with an increasing the concentration of Eu^{3+}. In our case, the luminescence decay kinetics of Eu^{3+} ions can be divided into the fast component, which shortens with an increasing concentration of Yb^{3+}, and the slow component, which becomes longer. Apparently, the fast component corresponds to the radiative lifetime of Eu^{3+} luminescence,

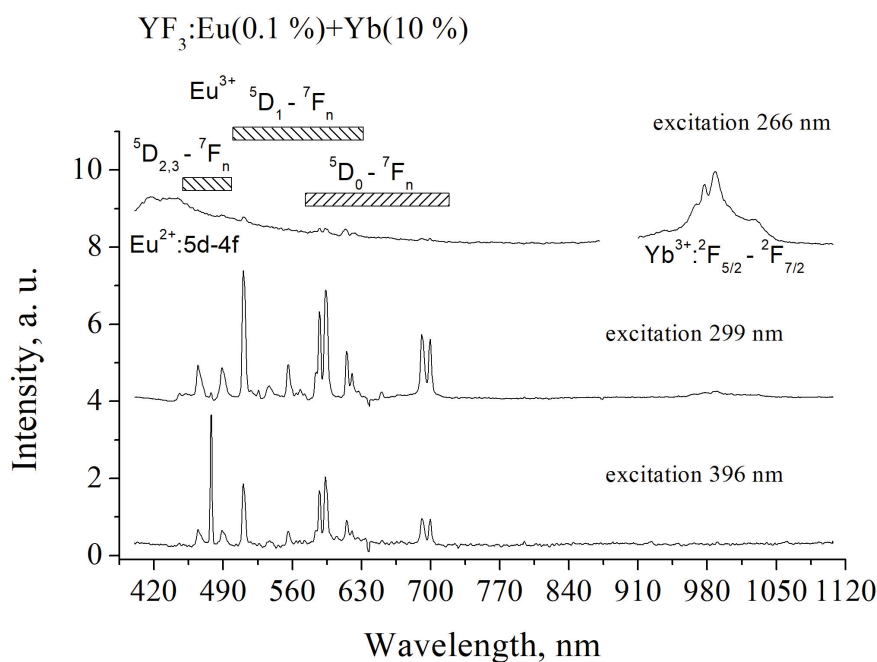**Fig. 2.** The luminescence spectra of the YF_3:Eu:Yb samples upon excitation at wavelengths of 399, 296, and 266 nm
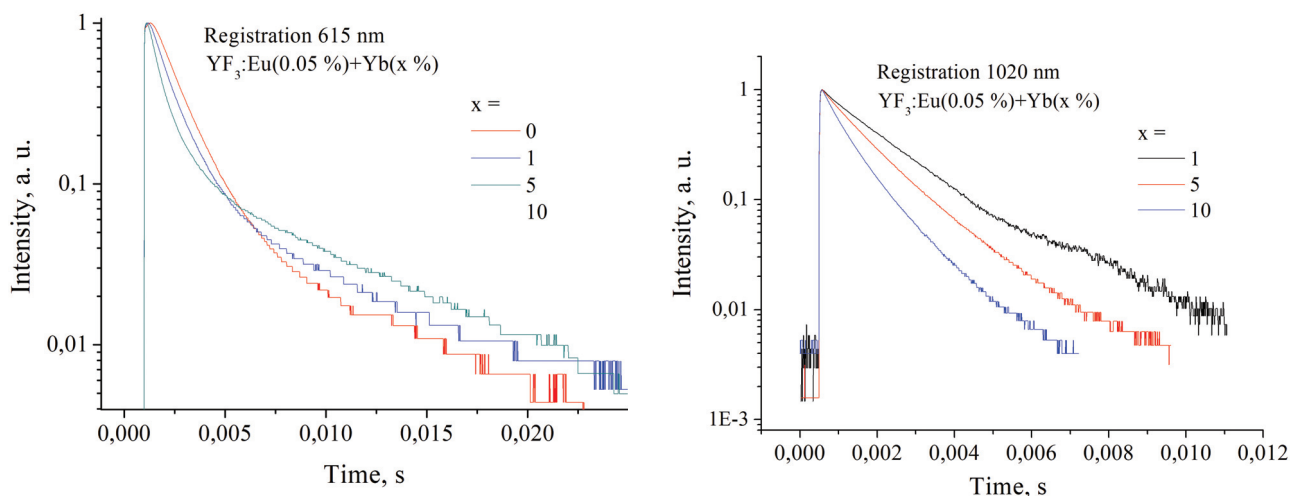

Fig. 3. The luminescence kinetics of Eu^{3+} and Yb^{3+} ions in the YF_3 samples upon excitation at a wavelength of 266 nm

which undergoes quenching due to energy transfer to Yb^{3+} ions. Meanwhile, the slow component is determined some kind of channel for refilling of the $^5D_0$ state population of Eu^{3+} ions. To evaluate the luminescence lifetime the mean lifetime was used, which was calculated using formula (1) [23]:

$$t_{avg} = \frac{\int t \cdot I(t)dt}{\int I(t)dt}, \qquad (1)$$

where $I(t)$ is the dependence of the luminescence intensity over time, t is time.

The estimated mean luminescence lifetime values show that concentration quenching is mostly manifested for Yb^{3+} ions (Table 2). In this case, the decrease in the luminescence lifetime in the IR region with an increase in the number of Eu^{3+} ions is insignificant, which indicates an insignificant effect of energy transfer back to the Eu^{3+} ions for the YF_3 matrix. This mechanism was described in [24].

Using the integrating sphere, we determined the quantum yield of luminescence of Yb^{3+} ions (QY) upon excitation of the samples in the region of 266 nm (Table 3). The maximum values were recorded for the Eu and Yb ratios of 0.1:10.0 and 0.05:5.00 for the YF_3 (QY = 2.2 %).

4. Conclusions

Using the method of high-temperature melting, single-phase samples of solid solutions of $Y_{1-x-y}Eu_xYb_yF_3$ with different amounts of Eu^{3+} and Yb^{3+} cations were synthesized. The powder samples of these crystals were described using of X-ray diffraction, energy dispersion analysis, and optical luminescent spectroscopy. Compliance with the β-YF_3 structural type was confirmed for all samples. A monotonic change in the unit cell parameters indicates the formation of solid solutions of the same phase, while the deviation of the real composition from the nominal did not exceed ± 0.5 mol%. The study of luminescence of the samples doped with both Eu^{3+} and Yb^{3+} ions showed the luminescence spectra of Eu^{3+} and Yb^{3+} ions, which are characteristic of these compounds. Both ions became luminescent upon excitation at wavelengths of 266 and 296 nm. It was also shown that, upon excitation at a wavelength of 266 nm, a wide luminescence band with a centre of approximately 430 nm appears. It may be due to the $5d$–$4f$ transition of Eu^{2+} ions. This leads to the fact that the kinetics of the luminescence decay of Eu^{3+} ions significantly differs from the exponential. In the luminescence kinetics, a short component can be distinguished, with the lifetime decreasing when the number of Yb^{3+} ions increases, and a long component, whose lifetime increases. Most likely, there is an energy transfer from Eu^{3+} ions to Yb^{3+} ions. Using the integrating sphere, we measured the quantum yield of luminescence of Yb^{3+} ions upon excitation at a wavelength of 266 nm. The quantum yield values of down-conversion luminescence were recorded for the Eu and Yb ratios of 0.1:10.0 and 0.05:5.00 for the YF_3 (QY = 2.2 %).

Conflict of interests

The authors declare that they have no known competing financial interests or personal

Table 2. The mean luminescence lifetime, recorded at wavelengths of 615 nm and 1020 nm in the YF_3: Eu:Yb samples upon excitation at a wavelength of 266 nm

Recording wavelength	615 nm	615 nm	1020 nm	615 nm	1020 nm	615 nm	1020 nm
Composition	Yb 0%	Yb 1.0 mol%		Yb 5.0 mol%		Yb 10.0 mol%	
Eu 0.05 mol%	2.6±0,1 ms	2.3±0.1 ms	1.7±0.1 ms	3.0±0.1 ms	1.3±0.1 ms	3.7±0,1 ms	1.0±0.1 ms
Eu 0.1 mol%	2.8±0.1 ms	2,1±0.1 ms	1.6±0.1 ms	2,7±0.1 ms	1.3±0.1 ms	3.5±0.1 ms	1.1±0.1 ms

Table 3. The quantum yield of the Yb^{3+} luminescence in the YF_3:Eu:Yb samples upon 266 nm excitation

Composition	Yb 1.0 mol%	Yb 5.0 mol%	Yb 10.0 mol%
Eu 0.05 mol%	1.0 %	2.2 %	1.6 %
Eu 0.10 mol%	1.0 %	1.1 %	2.2 %

relationships that could have influenced the work reported in this paper.

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