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Up-conversion luminescence of $CaF_2{-}SrF_2{-}HoF_3$ solind solutions upon excitation of the $^5I_7$ level of Ho^{3+} ions

© M.K. Bubnov[1], S.A. Bukarev[1], S.V. Gushchin[1], V.A. Konyushkin[2], S.V. Kuznetsov[2,3], A.A. Lyapin[1¶], A.N. Nakladov[2], P.A. Ryabochkina[1], V.V. Voronov[2], P.P. Fedorov[2]

[1] N.P. Ogarev National Research Mordovian State University,
430005 Saransk, Russia
[2] Prokhorov Institute of General Physics, Russian Academy of Sciences, Moscow, Russia
[3] Kazan Federal University, Kazan, Tatarstan, Russia
[¶] e-mail: andrei_lyapin@mail.ru

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Up-conversion luminescence spectra of $CaF_2{-}SrF_2{-}HoF_3$ single crystals were investigated upon excitation by laser radiation with a wavelength of 1940 nm to the $^5I_7$ level of Ho^{3+} ions. The $CaF_2{-}SrF_2{-}HoF_3$ single crystals were grown by the vertical directional crystallization method. For these excitation conditions, quantitative characteristics of the up-conversion luminescence of Ho^{3+} ions were determined: energy yield, chromaticity coordinates, and correlated color temperatures. The possibility of increasing the efficiency of solar cells due to the conversion of infrared radiation in $CaF_2{-}SrF_2{-}HoF_3$ crystals was considered.

Keywords: up-conversion luminescence, rare-earth ions, fluorides, luminescence yield.

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Introduction

Solar panels are a safe, clean and cheap source of energy. They are currently used in spacecraft, portable electronics, powering buildings, electric cars, etc. However, it should be noted that modern solar panels only convert part of the radiation of the solar spectrum into photocurrent. The long-wave conversion edge for crystalline silicon, gallium arsenide and perovskite based batteries is around 1100, 870 and 820 nm respectively [1]. As a result, the losses in solar radiation conversion by the silicon batteries are about 20% [1]. In papers [2–5], options have been proposed to increase the efficiency of solar cells by up-converting infrared (IR) radiation by materials on the back side of the panel. Up-conversion materials absorb the IR radiation that has passed through the panel and transform it into visible and near-infrared radiation, which is re-converted by the solar panel into photocurrent.

At present, the issue of increasing solar cell efficiency by additional up-conversion of solar radiation in the $1.4-1.6\,\mu$m area into photocurrent using materials doped with Er^{3+} ions is widely discussed in the literature6-18. These materials exhibit a wide absorption band in the spectral area $1.4-1.6\,\mu$m (transition $^4I_{15/2}-^4I_{13/2}$ ions Er^{3+}) and intense up-conversion luminescence in the spectral range $400-1100$ nm when excited to the level of $^4I_{13/2}$, which corresponds well to the maximum spectral sensitivity of silicon solar cells. The papers [6–18] have widely presented the results of up-conversion luminescence studies of various fluoride, oxide, oxysulfide (Y_2O_2S, BaY_2F_8, CaF_2, SrF_2, Gd_2O_2S, $NaYF_4$, La_2O_2S, and others.) compounds doped with Er^{3+} ions when excited to the $^4I_{13/2}$ level by both monochromatic and broadband radiation. Real tests have been carried out on up-conversion materials when incorporated into solar panels [5,17–27].

It is worth noting that materials doped with Ho^{3+} ions also exhibit effective up-conversion luminescence when excited by IR radiation to energy levels $^5I_6$ [28,29] and $^5I_7$ [30–37]. Previously, the up-conversion luminescence of Ho^{3+} ions when excited to the $^5I_7$ level has been investigated in materials CaF_2 [30–32], SrF_2 [32,33], ZBLAN [34,35], PbF_2 [36], $ZrF_4{-}BiF_3$ [37].

Analysis of available literature data showed that previously the issue of increasing solar cell efficiency by up-conversion of two-micron radiation by $CaF_2{-}SrF_2{-}HoF_3$ crystals was not discussed. At the same time, these compounds have a wide absorption spectrum and low phonon energy (350 cm $^{-1}$ [38]), which allows to potentially consider them as efficient up-conversion materials. It should be noted that for practical applications of up-conversion luminescence in solar cells, polycrystalline materials doped with rare-earth ions are generally used. However, preliminary studies of up-conversion luminescence spectra should be carried out on model objects in the form of transparent single-crystals, because in comparison with powdered materials they minimize scattering processes in the study of absorption and luminescence spectra.

In accordance with this the aim of the present paper was to study the up-conversion luminescence of Ho^{3+} ions of solid solutions $CaF_2{-}SrF_2{-}HoF_3$ under their excitation by laser irradiation with wavelength 1940 nm level $^5I_7$ of these ions.

Characteristics of objects and research methods

Single-crystals of solid solutions $CaF_2-SrF_2-HoF_3$ were grown by vertical directional crystallization in graphite crucibles in vacuum. CaF_2 (GOI optical single-crystals), SrF_2 (99.99, NEVATORG), HoF_3 (99.99, LANHIT Ltd.) were used as starting reagents. The reagents except calcium fluoride were pre-melted with fluorination in flat-bottomed graphite crucibles with a lid (pressure CF_4 0.1 atm) to remove oxygen-containing impurities. The cake was heated to a temperature of 1370° C, fluorinated in an atmosphere of CF_4 for 1 h, and then the crucible was moved from the hot zone to the cold zone at a rate of 7 mm/h. After the crucible movement was finished, the crucible was cooled successively to room temperature for 4 h.

X-ray diffraction analysis of the synthesized samples was carried out on a Bruker D8 Advance diffractometer with CuK_α-radiation in the range 2θ $15°-80°$. The lattice parameters have been calculated in Powder 2.0 ($\Delta Q < 5.0$)).

To investigate the spectral luminescence properties of $CaF_2-SrF_2-HoF_3$ single-crystals, disks of 10 mm diameter and 1 mm thickness were cut out of crystal boules. The sample compositions corresponded to the saddle point areas of the three-component systems [39,40] and the growth rate was estimated from the data [41].

The chemical composition was determined by energy-dispersive analysis (EDX) on a Carl Zeiss NVision 40 microscope.

A Perkin Elmer Lambda 950 scanning double-beam spectrophotometer was used to record the absorption spectra of Ho^{3+} ions in the fluoride single-crystals under study.

Up-conversion luminescence spectra of Ho^{3+} ions in the 400−1100 and 1100−1300 nm bands were recorded with the OL− 770 VIS/NIR spectroradiometer and Horiba FHR 1000 spectrometer, respectively. A continuous fiber laser with an emission wavelength of 1940 nm was used to excite the up-conversion luminescence of Ho^{3+} ions to the $^5I_7$ level. The diameter of the laser beam incident on the sample was 1.5 mm. In the present paper laser monochromatic radiation is used to detect the presence and reveal the up-conversion luminescence patterns of solid solutions $CaF_2-SrF_2-HoF_3$. In real-world application tests of up-conversion luminescence of $CaF_2-SrF_2-HoF_3$ materials, broadband IR radiation sources shall be used to improve solar cell efficiency.

The standard CIE 1931 (X, Y) [42,43] colorimetric system was used to calculate chromaticity coordinates.

The energy yield of up-conversion luminescence of Ho^{3+} ions was determined by the formula [44–46]

$$B_{\text{en}} = P_{\text{lum}}/P_{\text{abs}}, \tag{1}$$

where P_{lum} — up-conversion luminescence power in spectral range 400−1100 nm, P_{abs} — absorbed power of laser radiation with wavelength 1940 nm.

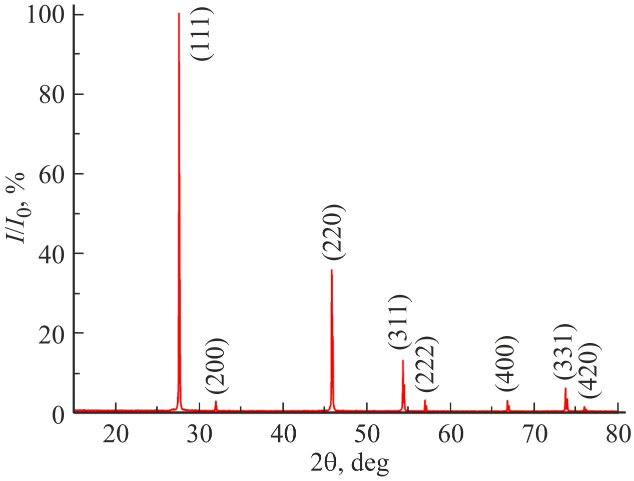

Figure 1. X-ray diffraction of a $CaF_2-SrF_2:Ho$ (3.4%).

An experimental setup based on an OLIS−670−LED integrating sphere and an OL-770 VIS/NIR spectroradiometer was assembled to determine the energy yield. Continuous laser light through a quartz achromatic depolarizer was focused on a sample located in the center of a sphere. The spectral power of up-conversion luminescence was then recorded using a spectroradiometer. The integrating sphere and spectroradiometer were connected by an optical waveguide. The correction of the experimental setup for spectral sensitivity was carried out with a reference halogen lamp OL 245-TSF-X Gooch & Housego with a known spectral irradiance. The incident laser power on the sample was measured with a S148C Thorlabs integrating sphere based power meter.

All measurements were made at room temperature.

Experimental results and discussion thereof

The compositions of the $Ca_{0.600}Sr_{0.400-x}Ho_xF_{2+x}$ single-crystals grown are shown in the Table. X-ray pictures of the samples revealed a cubic crystal structure; the results of the calculation of the lattice constants are shown in the Table. A typical X-ray diffraction pattern of a $CaF_2-SrF_2:Ho$ (3.4%) sample is shown in Fig. 1. An evaluation of the actual chemical composition by energy-dispersive analysis revealed a good match between the nominal and actual compositions.

The absorption spectrum of the $CaF_2-SrF_2:Ho$ (3.4%) single-crystal in the wavelength range 250−2250 nm is represented by bands due to transitions $^5I_8 \to {}^5G_5$, $^5I_8 \to {}^5G_6$, $^5I_8 \to {}^5F_2 + {}^3K_8$, $^5I_8 \to {}^5F_3$, $^5I_8 \to {}^5S_2({}^5F_4)$, $^5I_8 \to {}^5F_5$, $^5I_8 \to {}^5I_4$, $^5I_8 \to {}^5I_5$, $^5I_8 \to {}^5I_6$, $^5I_8 \to {}^5I_7$ of ionsHo^{3+} (Fig. 2). Crystals $CaF_2-SrF_2:Ho$ exhibit broad absorption bands in the $1.15\,\mu$m area (transition $^5I_8 \to {}^5I_6$ of Ho^{3+} ions) and $1.9\,\mu$ m (transition $^5I_8 \to {}^5I_7$ of Ho^{3+} ions), excitation of which can produce up-conversion luminescence [30].

Concentration range of crystals $Ca_{0.620}Sr_{0.380-x}Ho_xF_{2+x}$

Nominal composition, mole fractions			Composition by EDX, mole fractions			Parameter of lattice, Å	Designation
CaF_2	SrF_2	HoF_3	CaF_2	SrF_2	HoF_3		
0.620	0.375	0.005	0.624	0.370	0.006	5.5988(6)	CaF_2-SrF_2 : Ho (0.6%)
0.620	0.370	0.010	0.626	0.361	0.013	5.592(1)	CaF_2-SrF_2 : Ho (1.3%)
0.620	0.365	0.015	0.622	0.360	0.018	5.5999(1)	CaF_2-SrF_2 : Ho (1.8%)
0.620	0.350	0.030	0.613	0.353	0.034	5.5934(5)	CaF_2-SrF_2 : Ho (3.4%)

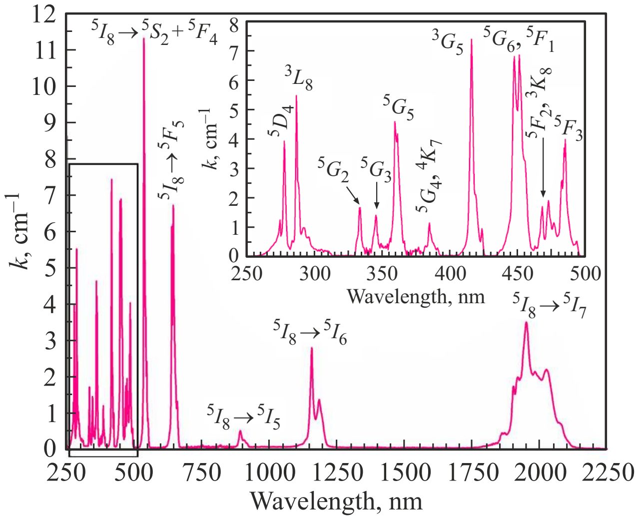

Figure 2. Absorption spectrum of CaF_2-SrF_2 : Ho (3.4%) in visible and IR spectral bands, $T = 300$ K.

The absorption spectrum of the CaF_2-SrF_2 : Ho (3.4%) for the electronic transition $^5I_8 \to {}^5I_7$ ion Ho^{3+} in the spectral range 1700 – 2200 nm, spectra of solar radiation above the atmosphere (solar spectrum AM0) and at the Earth's surface (solar spectrum AM1.5) are shown in Fig. 3. It is seen that the absorption band of the CaF_2-SrF_2 : Ho crystal (3.4%) partially overlaps with the spectrum of the Sun's radiation at the Earth's surface and agrees well with its emission above the atmosphere (Fig. 3). The dotted arrow shows the up-conversion luminescence excitation wavelength at the $^5I_7$ level of Ho^{3+} ions ($\lambda_{ex} \sim 1940$ nm) in crystals CaF_2-SrF_2 : Ho.

The analysis of up-conversion luminescence spectrum of golmium ions in CaF_2-SrF_2 : Ho crystal (3.4%) under excitation at 1940 nm wavelength revealed that the highest relative luminescence intensity in 400–1100 nm spectral area is observed for the band corresponding to $^5F_5 \to {}^5I_8$ ion transition Ho^{3+}, located in 650 nm area (Fig. 4).

The spectral sensitivity of crystalline silicon based solar cells is located in the spectral range 0.36–1.2 μm [47,48] (Fig. 4). Fig. 4 shows that the most intensive luminescence bands of Ho^{3+} ions corresponding to transitions $^5F_5 \to {}^5I_8$, $^5I_5 - {}^5I_8$ and $^5F_5 \to {}^5I_7$ in spectral area 400–1000 nm, are situated close to the area of maximum spectral sensitivity of silicon.

The long-wave conversion threshold of a silicon solar panel is about 1.2 μ m. Fig. 4 shows that the luminescence band corresponding to the electronic transition $^5I_6 \to {}^5I_8$ of ions Ho^{3+} (1100–1275 nm) is located at the edge of the spectral sensitivity of silicon and gives practically no contribution to the photocurrent. This luminescence of ions Ho^{3+} is a significant loss channel.

A diagram of the energy levels of Ho^{3+} ions showing the optical transitions between the energy levels of Ho^{3+} ions corresponding to up-conversion luminescence is shown in Fig. 5. Also Fig. 5 shows transition $^5I_8 \to {}^5I_7$ of ions Ho^{3+}, to which excitation was carried out. Mechanisms of inter-ion interaction are responsible for the up-conversion luminescence of Ho^{3+} ions in fluoride crystals with fluorite structure when excited to the $^5I_7$ level by ionsHo^{3+} [30].

The color coordinates of the up-conversion luminescence are shown in Fig. 6. CaF_2-SrF_2 : Ho single-crystals exhibit glow in the red area when the $^5I_7$ level of Ho^{3+} ions is excited by laser radiation with a wavelength of 1940 nm and a power density of 28 W/cm ^{2}. When the power density varies from 28 to 209 W/cm^2, the chromaticity coordinates change only slightly.

The dependence of up-conversion luminescence intensity of crystals CaF_2-SrF_2 : Ho on the concentration of ions Ho^{3+} was investigated using the integrating sphere

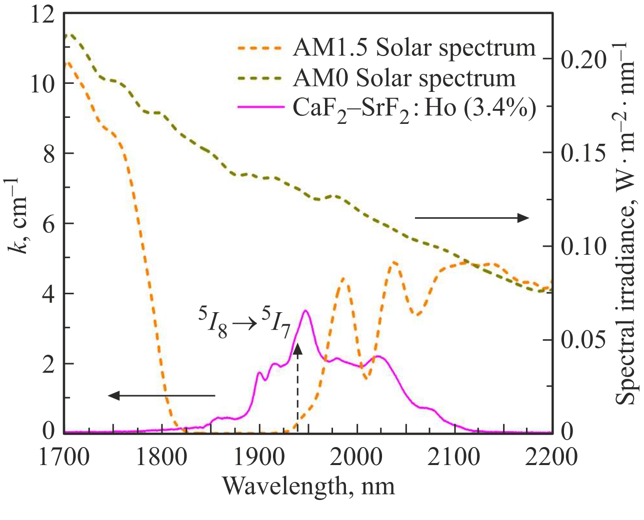

Figure 3. Absorption spectrum of the CaF_2-SrF_2 : Ho (3.4%) for the $^5I_8 \to {}^5I_7$ ion transition Ho^{3+} and spectra of solar radiation above the atmosphere (AM0) and at the Earth surface (AM1.5).

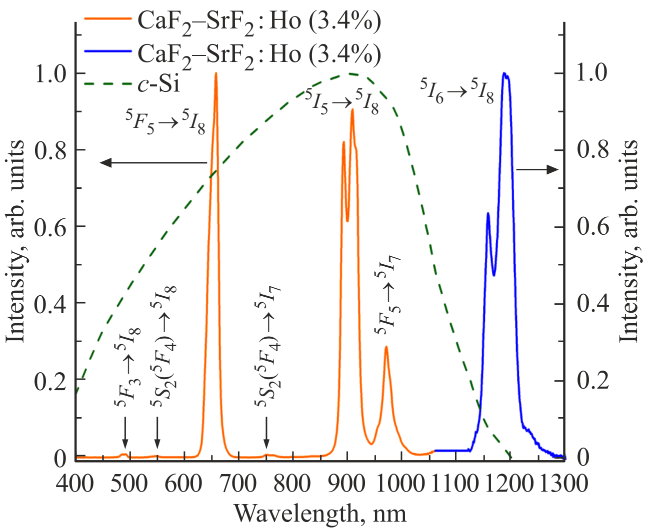

Figure 4. Up-conversion luminescence spectra of Ho^{3+} ions for $CaF_2-SrF_2:Ho$ (3.4%) crystal at $T = 300\,K$ and spectral sensitivity of single crystal silicon [47,48]. Excitation power density 28 W/cm ^{2}.

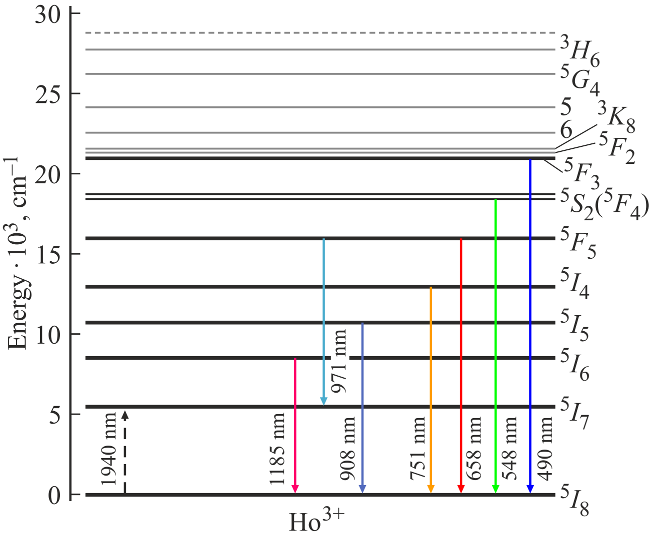

Figure 5. Energy level diagram of Ho^{3+} ions showing the transitions corresponding to the up-conversion luminescence of these ions and the $^5I_8 \rightarrow {}^5I_7$ transition of Ho^{3+} ions, to which the luminescence was excited.

(Fig. 7). It was found that in the concentration range of 0.6–3.4% Ho^{3+} ions, the intensity of up-conversion luminescence increases with increasing concentration of these ions. The most intense luminescence is characteristic of the $CaF_2-SrF_2:Ho$ (3.4%). This result may be due to an increase in the up-conversion probability of energy transfer as the concentration of Ho^{3+} ions increases. Fluoride crystals with a fluorite structure have a pronounced tendency to associate rare-earth ions into clusters. When Ho^{3+} ions form clusters in $CaF_2-SrF_2:Ho$ crystals, the distance between ions Ho^{3+} is reduced. Reducing the distance between Ho^{3+} ions in turn provides an increase in the efficiency of the inter-ion interaction of ions Ho^{3+} [49,50].

An important characteristic for evaluating the application of up-conversion IR conversion to improve solar cell efficiency is the energy yield. Fig. 8 shows the dependences of the energy yield of up-conversion luminescence of crystals $CaF_2-SrF_2:Ho$ in the spectral bands 380–780 nm (Fig. 8, *a*/) and 380–1100 nm (Fig. 8, *b*) on the concentration of Ho^{3+} ions and the excitation power density. The maximum energy yield of up-conversion luminescence B_{en} for spectral wavelength range 380–1100 nm was 0.09% at excitation power density 170 W/cm ^{2} for crystal $CaF_2-SrF_2:Ho$ (3.4%). For the spectral wavelength range 380–780 nm the maximum energy yield was 0.04%. When excited by radiation with a power density greater than 170 W/cm^2, saturation of the energy yield is observed.

Fig. 8 also shows the energy yield of up-conversion luminescence for the crystal $CaF_2:Ho$. Analysis of the literature data [31] and the results shown in Fig. 8 shows that mixed fluoride crystals $CaF_2-SrF_2:Ho$ have a higher energy yield compared to crystals $CaF_2:Ho$. This may be due to lower phonon energy of crystals $CaF_2-SrF_2:Ho$, which leads to lower up-conversion luminescence losses due to emission-free transitions. In order to confirm this hypothesis, a wider concentration range of studies is needed.

The resulting B_{en} value for radiation conversion in the two micron wavelength spectral range is one order of magnitude smaller than that for B_{en} for fluoride materials doped with Er^{3+} ions, which are used to convert radiation in the 1.5 μm range and are actively used in laboratory-scale solar cell testing. However, the B_{en} value for two-micron spectral wavelength range conversion in crystals $CaF_2-SrF_2:Ho$

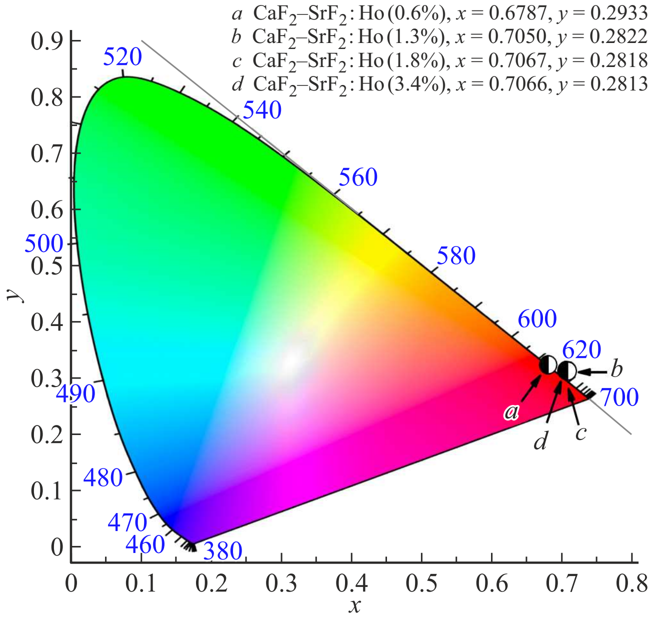

Figure 6. Chromatic up-conversion luminescence diagram for $CaF_2-SrF_2:Ho$ single-crystals when excited by laser radiation with wavelength 1940 nm.

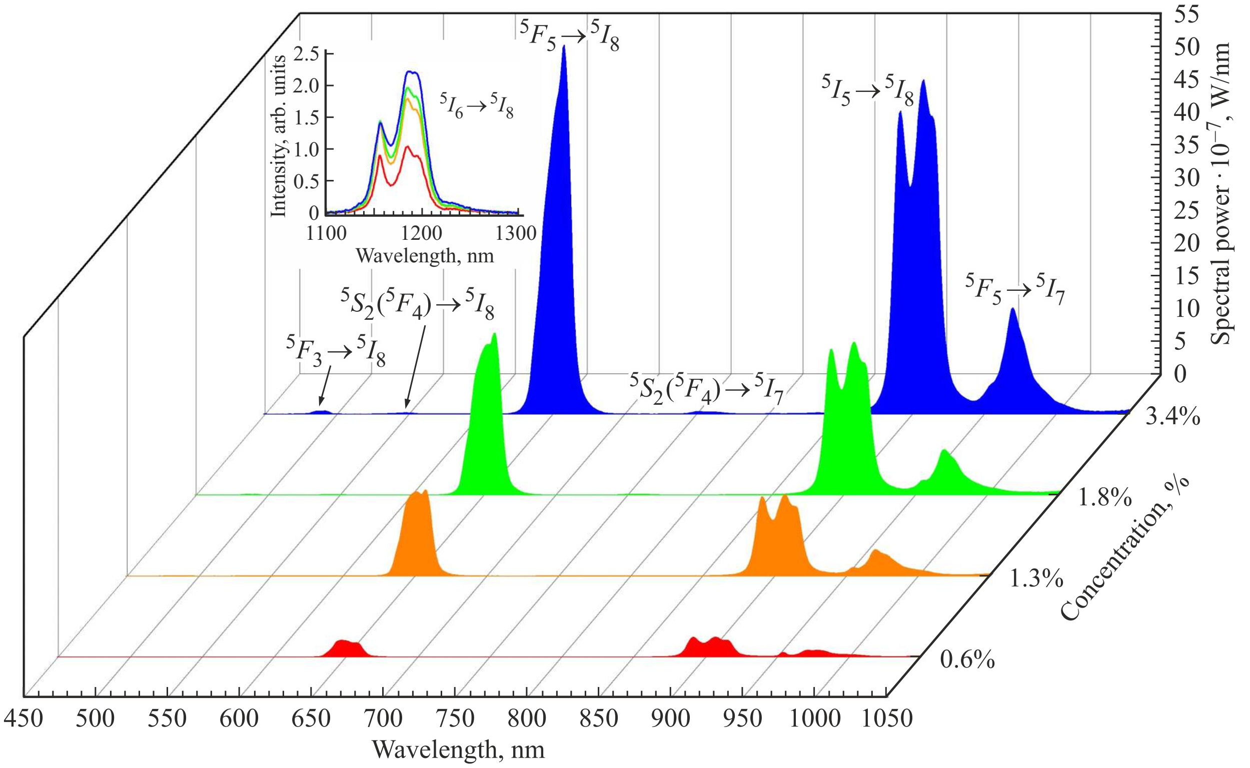

Figure 7. Dependence of up-conversion luminescence intensity of crystals $CaF_2-SrF_2:Ho$ on ion concentration Ho^{3+}.

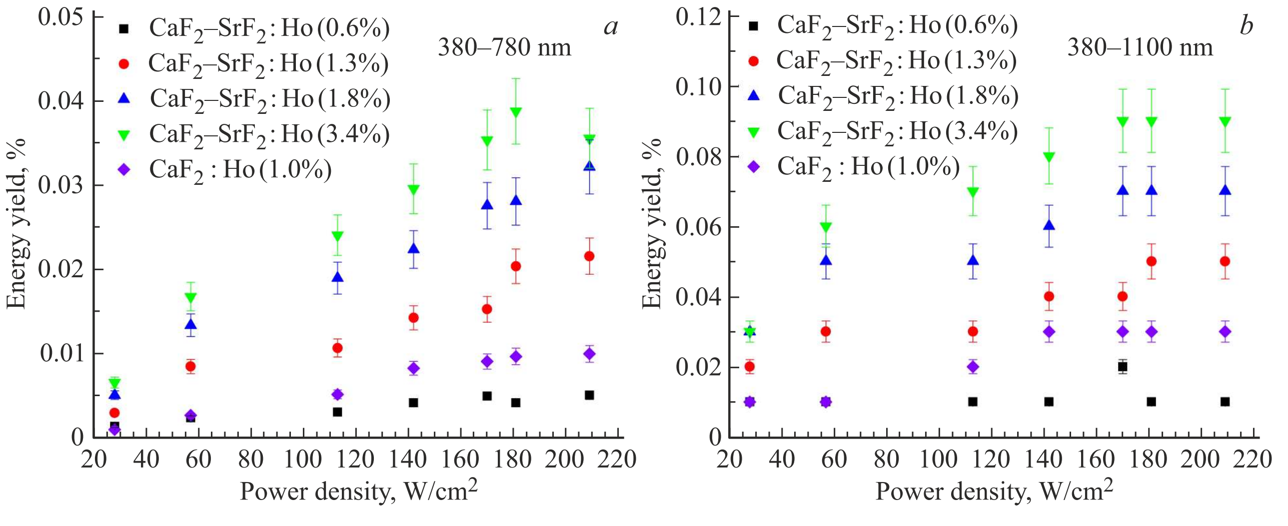

Figure 8. Energy yield dependences of up-conversion luminescence of $CaF_2-SrF_2:Ho$ and $CaF_2:Ho$ for spectral bands 380−780 (*a*) and 380−1100 nm (*b*) on excitation laser radiation power density.

may be higher after optimizing the concentration of Ho^{3+} ions as well as when they are codoped with other rare-earth ions.

Conclusion

Thus, in the present paper up-conversion luminescence characteristics of a concentration series of crystals $CaF_2-SrF_2:Ho$ grown by vertical directional crystallization method are investigated. When the $^5I_7$ level of Ho^{3+} ions was excited by laser radiation at a wavelength of 1940 nm up-conversion luminescence spectra corresponding to the electronic transitions of these ions $^5F_3 \to {}^5I_8$, $^5S_2(^5F_4) \to {}^5I_8$, $^5F_5 \to {}^5I_8$, $^5S_2(^5F_4) \to {}^5I_7$, $^5I_4 \to {}^5I_8$, $^5F_3 \to {}^5I_6$, $^5I_5 \to {}^5I_8$, $^5F_5 \to {}^5I_7$ and $^5I_6 \to {}^5I_8$ were recorded.

Crystals CaF_2- SrF $_2$:Ho exhibit stable luminescence in the red area of the spectrum when excited by laser radiation in the two-micron area.

The maximum energy yield of up-conversion luminescence Ben for the CaF_2-SrF_2:Ho (3.4%) was 0.09 and 0.04% for the 380−1100 and 380−780 nm spectral wavelength bands, respectively. It is possible to increase the B_{en} value for two-micron spectral wavelength range conversion in crystals CaF_2-SrF_2:Ho after optimizing the concentration of Ho^{3+} ions as well as when they are codoped with other rare-earth ions.

Mixed fluoride crystals CaF_2-SrF_2:Ho have a higher energy yield compared to crystals CaF_2:Ho.

The materials CaF_2-SrF_2:Ho can potentially be used in solar cells to increase the spectral range of radiation conversion.

Conflict of interest

The authors declare that they have no conflict of interest.

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