Er,Yb:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd) – Novel 1.5 μm Laser Crystals

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Abstract

The search for new crystalline host materials for the usage in lasers emitting in the eye-safe spectral range of 1.5–1.6 μm is an important task. The aim of this work was to study the growth technique, spectroscopic properties and laser characteristics of new active media – crystals Er^{3+},Yb^{3+}:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd).

Calcium-yttrium Er^{3+},Yb^{3+}:$Ca_3Y_2(BO_3)_4$ (CYB) and calcium-gadolinium Er^{3+},Yb^{3+}:$Ca_3Gd_2(BO_3)_4$ (CGB) oxoborate crystals co-doped with erbium and ytterbium ions were investigated. Polarized absorption and emission cross-section spectra were determined. The lifetimes of $^4I_{11/2}$ and $^4I_{13/2}$ energy levels of Er^{3+} ions were measured and ytterbium-erbium energy transfer efficiencies were estimated. The calculation of the gain cross-section spectra was performed. By using of Er^{3+},Yb^{3+}:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd) crystals the laser performance was realized, for the first time to the best of our knowledge. The laser characteristics were studied in a quasi-CW (QCW) laser operation.

The wide band with a peak at the wavelength of 976 nm is observed in the absorption spectra of both crystals. This peak coincides with the emission wavelength of the pump laser diodes for Yb-doped active media. The maximum value of absorption cross-section was 1.7×10^{-20} cm^2 for polarization $E // b$ for both crystals. The lifetimes of the upper laser level $^4I_{13/2}$ of Er^{3+} ions were 580 ± 30 μs and 550 ± 30 μs for Er,Yb:CYB and Er,Yb:CGB crystals, respectively. The energy transfer efficiencies from ytterbium to erbium ions for an Er,Yb:CYB and Er,Yb:CGB crystals were 94 % and 96 %, respectively. According to gain spectrum of the Er,Yb:CYB crystal the gain band peak is centered at the wavelength of 1530 nm. The maximum QCW output power was 0.5 W with slope efficiency of 13 % regarding to absorbed pump power for an Er,Yb: CYB crystal. The laser beam parameter M^2 did not exceed < 1.5.

Based on the obtained results, it can be concluded that these crystals are promising active media for lasers emitting in the spectral range of 1.5–1.6 μm for the usage in laser rangefinder and laser-induced breakdown spectroscopy systems, and LIDARs.

Keywords: erbium, ytterbium, borate crystals, spectroscopy, laser performance.

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Кристаллы Er,Yb:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd) – новые среды для лазеров, излучающих в спектральном диапазоне 1,5 мкм

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Поиск новых кристаллических матриц для применения в лазерах, излучающих в условно безопасном для глаз спектральном диапазоне 1,5–1,6 мкм, является актуальной задачей. Целью данной работы являлось изучение технологии роста, спектроскопических свойств и генерационных характеристик новых активных сред – кристаллов Er^{3+},Yb^{3+}:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd).

В качестве исследуемых образцов использовались кристаллы кальций-иттриевого Er^{3+},Yb^{3+}:$Ca_3Y_2(BO_3)_4$ (CYB) и кальций-гадолиниевого Er^{3+},Yb^{3+}:$Ca_3Gd_2(BO_3)_4$ (CGB) оксоборатов, соактивированных ионами эрбия и иттербия. В результате определены спектры поперечных сечений поглощения и стимулированного испускания в поляризованном свете. Определены времена жизни энергетических уровней $^4I_{11/2}$ и $^4I_{13/2}$ иона эрбия, а также проведена оценка эффективности переноса энергии от ионов иттербия к ионам эрбия. Выполнен расчет спектров поперечных сечений усиления. Впервые с использованием кристаллов реализована лазерная генерация, изучены генерационные характеристики в квазинепрерывном режиме генерации.

В спектрах поглощения для обоих кристаллов наблюдается полоса с пиком на длине волны 976 нм, что согласуется с длиной волны испускания лазерных диодов, применяемых для накачки активных элементов с ионами Yb^{3+}. Наибольшее значение поперечного сечения поглощения для обоих кристаллов составило $1{,}7 \times 10^{-20}$ см^2 для поляризации $E \,//\, b$ на длине волны 976 нм. Время жизни верхнего лазерного уровня $^4I_{13/2}$ составило 580 ± 30 мкс и 550 ± 30 мкс для кристаллов Er,Yb:CYB и Er,Yb:CGB соответственно. Эффективность переноса энергии от ионов иттербия к ионам эрбия составила 94 % для кристалла Er,Yb:CYB и 96 % для Er,Yb:CGB. Расчет спектра усиления для кристалла Er,Yb:CYB, показал что максимум полосы усиления находится на длине волны 1530 нм. Максимальное значение выходной мощности в квазинепрерывном режиме генерации составило 0,5 Вт при дифференциальной эффективности по поглощенной мощности накачки 13 % для кристалла Er,Yb:CYB, параметр распространения лазерного пучка M^2 не превышал 1,5.

На основе полученных результатов, можно сделать вывод, что данные кристаллы являются перспективными активными средами для лазеров, излучающих в спектральном диапазоне 1,5–1,6 мкм, для применения в составе систем лазерной дальнометрии, лазерно-искровой эмиссионной спектрометрии и лидаров.

Ключевые слова: эрбий, иттербий, кристаллы боратов, спектроскопия, лазерная генерация.

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Introduction

The laser radiation in the spectral range of 1.5–1.6 μm is attractive for application in rangefinders and LIBS (Laser Induced Breakdown Spectroscopy) systems [1] due to its eye-safety. This radiation is strongly absorbed in an eye's cornea and can not damage the sensitive retina. Also due to high transparency of the atmosphere 1.5–1.6 μm radiation is of great interest for application in LIDAR (Light Identification Detection and Ranging) systems [2]. Nowadays there are many laser sources emitting in this range, lasers based on Er^{3+} and Yb^{3+} co-doped materials are ones of the most widespread. The main requirements to erbium, ytterbium co-doped materials for achieving efficient laser operation in the spectral range 1.5–1.6 μm are the following [3]:

– efficient absorption of pump power by Yb^{3+} ions and further efficient energy transfer from ytterbium to erbium ions;

– fast non-radiative relaxation from $^4I_{11/2}$ energy level to $^4I_{13/2}$ energy level of Er3+ ions for minimization of losses deal with back energy transfer from erbium to ytterbium ions and upconversion transitions from the $^4I_{11/2}$ energy level to upper ones.

Nowadays $REAl_2(BO_3)_4$ oxoborate crystals are the leading Er^{3+},Yb^{3+} co-doped crystalline laser materials, because they possess necessary spectroscopic properties required for efficient laser operation at near 1.5 μm [4–7]. However crystals with sizes not more than $20 \times 10 \times 10$ mm^3 can be grown only by top seed solution growth technique (TSSG) that is characterized with a long-term (about a month) growth period [8].

In the contrast to Er,Yb:$REAl_2(BO_3)_4$ crystals, homogeneous and free of impurity phases and scattering centers Er,Yb:$Ca_3RE_2(BO_3)_4$ ones with dimensions up to Ø 20×80 mm^3 can be grown by the well managed Czochralski method. To date several articles devoted to the crystal growth and spectroscopy investigation of Er,Yb:$Ca_3RE_2(BO_3)_4$ crystals were published. Moreover, polarized absorption and emission cross-section spectra, energy transfer efficiency as well as laser performance was not reported. In this paper we demonstrate laser related spectroscopy and, for the first time to our knowledge, laser operation of Er,Yb:$Ca_3RE_2(BO_3)_4$ crystals.

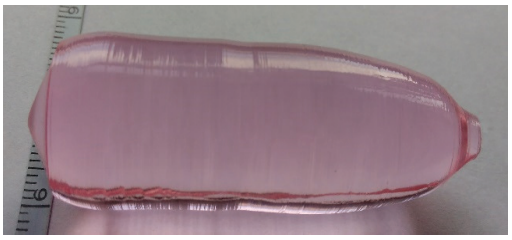
Experimental details

Crystal growth

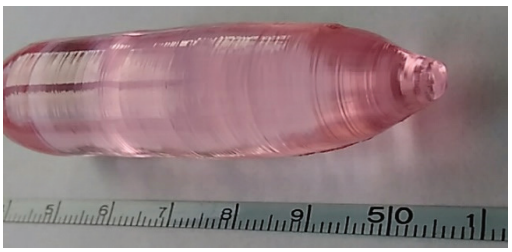
$CaCO_3$ (99.99 %), RE_2O_3(99.99 %) (RE = Y, Gd), Er_2O_3(99.99 %), Yb_2O_3(99.99 %) and B_2O_3 (99.95 %) compounds were used as reagents for solid state synthesis of the charge. The stoichiometric mixture of the initial reagents was placed into a platinum crucible. The mixture was heated at the rate of 50 °C/h to 110 °C, 230 °C, 450 °C and 750 °C and kept for 10 h at each temperature. The compound formation was carried out according to reaction:

$$3CaCO_3 + 0.88RE_2O_3 + 0.02Er_2O_3 + 0.1Yb_2O_3 + 2B_2O_3 \rightarrow Ca_3RE_{1.76}Yb_{0.2}Er_{0.04}(BO_3)_4 + 3CO_2\uparrow.$$

The obtained material was finely ground. The charge was placed in Ir crucible for crystal growth. Er,Yb co-doped $Ca_3RE_2(BO_3)_4$ crystals were grown by the Czochralski method using an automated and equipped with a weight control system «Kristall 3M» puller. The growth process was carried out in inert (argon) atmosphere. The pulling and rotation rates were 1.5 mm/h and 20 rpm, respectively. The axial temperature gradient at the crystal-melt interface was 50 °C/cm. The crystals were grown along the crystallographic axis [001]. No impurity phases and gas bubble inclusions were detected [9]. Boules of Er,Yb:$Ca_3Re_2(BO_3)_4$ (Re = Y, Gd) crystals of high optical quality were produced with 20 mm in the diameter and 80 mm in the length (Figure 1).



a



b

Figure 1 – The Er,Yb:$Ca_3Y_2(BO_3)_4$ (*a*) and Er,Yb:$Ca_3Gd_2(BO_3)_4$ (*b*) crystals

Spectroscopic technique

Two polished cubes cut from Er(2 at.%),Yb(10 at.%):$Ca_3Y_2(BO_3)_4$ and Er(2 at.%),Yb(10 at.%):$Ca_3Gd_2(BO_3)_4$ crystals with

dimensions of $5 \times 5 \times 5$ mm^3 oriented along the a, b and c crystallographic axes were used to record polarized absorption spectra. The room-temperature polarized absorption spectra were recorded with *Varian Cary* 5000 *UV-Vis-NIR* spectrophotometer in the wavelength ranges from 850 to 1100 nm and 1400–1650 nm.

Lifetime measurements were performed using optical parametric oscillator based on the β-$Ba_2B_2O_4$ crystal and pumped by the third harmonic of a Q-switched Nd:YAG laser. The luminescence radiation was detected at 1.5 μm using a monochromator, fast InGaAs photodiode and 500 MHz digital oscilloscope. To prevent reabsorption caused by significant overlap of the absorption and emission bands, measurements of Yb^{3+} luminescence kinetics were performed using with a fine powder of the crystals immersed in glycerin [11].

The energy transfer efficiency was measured by estimation of the $^2F_{5/2}$ level lifetime shortening in Er,Yb-codoped crystals and Yb-single doped crystal according to the formula (1) [12]:

$$\eta = k / \tau^{-1} = \tau(1/\tau - 1/\tau_0), \quad (1)$$

where k is the energy transfer rate; τ is the ytterbium $^2F_{5/2}$ level lifetime in Er,Yb-codoped crystal; τ_0 is the ytterbium $^2F_{5/2}$ level lifetime in Yb single-doped crystal.

The stimulated emission-cross section spectra in the spectral range 1450–1650 nm were calculated by the integral reciprocity method (2) [13] using the calculated absorption cross-section spectra and radiative lifetime of $^4I_{13/2}$ energy level of Er^{3+} ions presented in [14]:

$$\sigma_{em}^{\alpha}(\lambda) = \frac{3\exp(-hc/(kT\lambda))}{8\pi n^2 \tau_{rad} c \sum_{\gamma} \int \lambda^{-4} \sigma_{abs}^{\gamma}(\lambda) \exp(-hc/(kT\lambda)) d\lambda} \sigma_{abs}^{\alpha}(\lambda), \quad (2)$$

where τ_{rad} is the radiative lifetime of an active center (Er^{3+}); c is the speed of light in vacuum; α and γ denote light polarization; h and k are the Planck's and Boltzman's constants, respectively; T is a host crystal temperature; n is the refractive index of the crystal and σ_{abs} is the ground state absorption cross-section.

The gain cross-section spectra $g(\lambda)$ were calculated for different inversion parameters β by using the following equation (3):

$$g(\lambda) = \beta\, \sigma_{em}(\lambda) - (1-\beta)\sigma_{abs}(\lambda), \quad (3)$$

where $\beta = N_{ex} / N_{tot}$ is the ratio of the population of excited Er^{3+} ions manifold to the total erbium ions concentration.

Setup for laser experiments

The laser performance of the Er,Yb:$Ca_3Re_2(BO_3)_4$ (Re=Y,Gd) crystals was investigated in Z-shaped cavity (Figure 2). The 2-mm-thick antireflection coated for both pump and lasing wavelengths *a*-cut Er(2 at.%),Yb(10 at.%):$Ca_3Re_2(BO_3)_4$ crystal was wrapped in indium foil for good thermal contact and mounted between two copper slabs with the hole in the center to permit passing of pump and laser beams. The temperature of an active element was kept at 20 °C. As a pump source a 976 nm fiber-coupled laser diode (Ø 105 μm, NA = 0.22) was used. To minimize thermal effects inside the crystal the quasi-continuous wave (QCW) mode of laser diode operation with a duty cycle of 10 % was chosen. After passing lens system the pump beam was focused into ~ 100 μm spot ($1/e^2$ intensity) inside the crystal. The cavity-mode diameter at the active element was close to the pump beam waist. Three output couplers (OC's) with different transmittances were used during laser experiments. The experimental setup is shown in Figure 2.

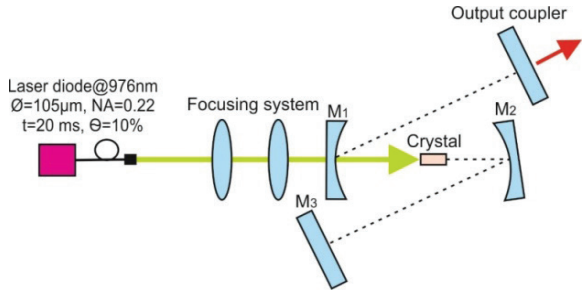


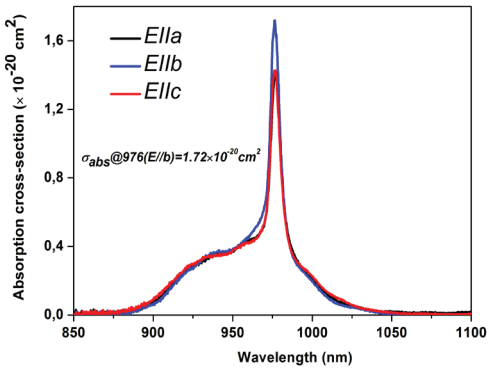
Figure 2 – Experimental setup for laser experiments

Result and discussion

Spectroscopy

The room-temperature polarized absorption cross-section spectra of the Er,Yb:$Ca_3Y_2(BO_3)_4$ (CYB) and Er,Yb:$Ca_3Gd_2(BO_3)_4$ (CGB) crystals in the spectral range of 850–1100 nm are shown in Figures 3*a* and 3*b*, respectively. The peak absorption cross-sections around 976 nm (the wavelength is close to emission wavelengths of commercial InGaAs laser diodes) corresponding to the transitions of $^2F_{7/2} \rightarrow {}^2F_{5/2}$ of Yb^{3+} ions and $^4I_{15/2} \rightarrow {}^4I_{11/2}$ of Er^{3+} ions, for both crystals is around 1.7×10^{-20} cm^2 for polarization $E // b$. Thus, the pump beam polarization corresponded to the *b* axis of the crystal will be preferable.

The broad and smooth absorption band corresponding to transition of $^4I_{13/2} \rightarrow {}^4I_{15/2}$ of Er^{3+} ions is observed in the spectral range of 1400–1650 nm. The maximum absorption cross-section for Er,Yb:CYB crystal in the spectral region of 1400–1650 nm does not exceed 0.6×10^{-20} cm^2 at the wavelength of 1530 nm (Figure 4).



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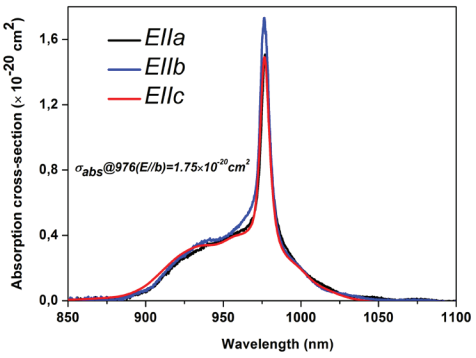


b

Figure 3 – Polarized absorption cross-section spectra of Er,Yb:CYB (*a*) and Er,Yb:CGB (*b*) crystals at room temperature

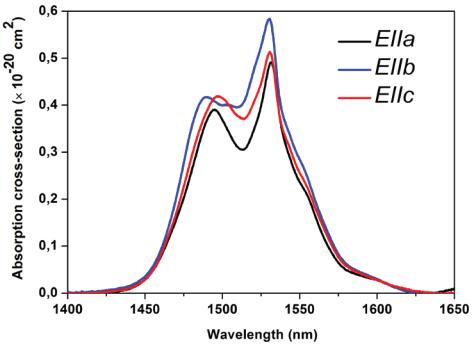
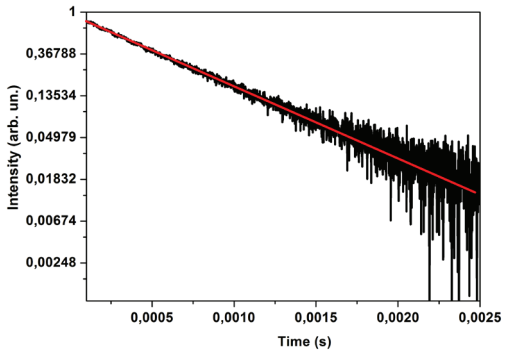


Figure 4 – Polarized absorption cross-section spectra of Er,Yb:$Ca_3Y_2(BO_3)_4$ crystal in the spectral region of 1400–1650 nm

The decay curves of 1.5 μm emission ($^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+} ions) were single exponential (Figure 5) for both crystals, and the luminescence decay times of the $^4I_{13/2}$ energy level of Er^{3+} were measured to be about 580 ± 30 μs and 550 ± 30 μs for Er,Yb:CYB and Er,Yb:CGB crystals, respectively. Taking into account, that radiative lifetimes calculated from the Judd–Offelt analysis is 2.41 ms for Er,Yb:CYB crystal and 1.98 ms for Er,Yb:CGB crystal [13] the luminescence quantum yields of the $^4I_{13/2}$ energy level was estimated to be 24 % and 28 % for Er,Yb:CYB and Er,Yb:CGB crystals, respectively. Comparatively low luminescence quantum yields are explained by the high phonon energy of the oxoborate crystals, which result in a high non-radiative $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition probability. However, it should be mentioned, that obtained values is near three times higher to those obtained for Er,Yb:$REAl_2(BO_3)_4$ crystals (10 %) [5].



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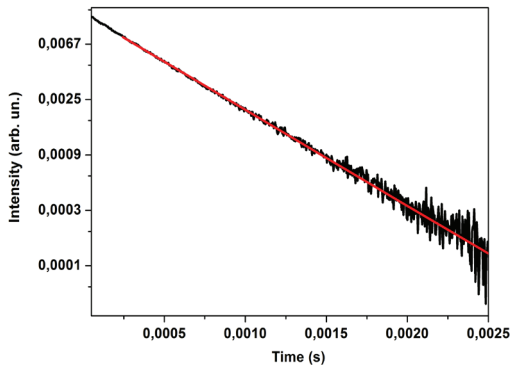


b

Figure 5 – Kinetics of luminescence decay of Er,Yb:CYB (*a*) and Er,Yb:CGB (*b*) crystals

The lifetime of the $^4I_{11/2}$ level of Er^{3+} ions was estimated in CYB and CGB crystals doped only Er^{3+} ions, because in Er-Yb co-doped crystals the process of back energy transfer from erbium to ytterbium distorts the measured lifetime of the level $^4I_{11/2}$. In erbium media, nonradiative relaxation to the $^4I_{13/2}$ level is the dominant process of emptying the $^4I_{11/2}$ level. Therefore, the time of emptying the $^4I_{11/2}$ level

corresponds to the time of filling the $^4I_{13/2}$ level. Consequently, the task of estimating the lifetime of the $^4I_{11/2}$ level of the Er^{3+} ion in Er:CYB and Er:CGB crystals was to determine the $^4I_{13/2}$ energy level rising time. The lifetime of the $^4I_{11/2}$ energy level in Er:CYB and Er:CGB crystals did not exceed 120 ns (Figure 6) (for comparison, in phosphate glass with Er^{3+} ions it is 1–3 μs [15, 16]). The short lifetime of $^4I_{11/2}$ energy level enables fast non-radiative relaxation from $^4I_{11/2}$ energy level to $^4I_{13/2}$ energy level of Er^{3+} that leads to minimization of losses dealt with back energy transfer from erbium to ytterbium ions and up-conversion transitions from to $^4I_{11/2}$ energy level to upper ones.

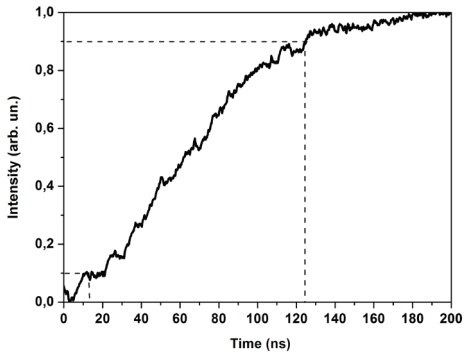


Figure 6 – The rising part of luminescence kinetics curve from $^4I_{13/2}$ energy level of Er^{3+} in the region of about 1.5 μm

The dependences of obtained lifetimes of $^2F_{5/2}$ energy level on different weight content of Yb(1 at.%):CYB and Yb(1 at.%):CGB crystalline powders in glycerin suspension are presented in Figure 7. With the decrease in weight content of crystalline powder in suspension the measured lifetimes also decreased. After considerable dilution of the powders and thus elimination of selftrapping effect the ytterbium lifetimes in Yb:CYB and Yb:CGB crystals were determined to be 680 ± 30 and 700 ± 30 μs, respectively. The $^2F_{5/2}$ energy level lifetimes were measured to be 40 ± 2 μs in Er(2 at.%),Yb(10 at.%):CYB crystal and 30 ± 2 μs for Er(2 at.%),Yb(10 at.%):CGB crystal.

By using formula (1) the energy transfer efficiency in the Er:Yb:CYB crystal doped with 2 at.% Er^{3+} and 10 at.% Yb^{3+} was calculated to be about 94 %. Very close efficiency of 96 % was obtained for Er:Yb:CGB crystal. These results suggest that the almost all of absorbed energy can be efficiently transferred from $^2F_{5/2}$ energy level of Yb^{3+} ions to the $^4I_{11/2}$ energy level of Er^{3+} ions by nonradiative resonant energy-transfer process. It also should be mentioned that energy transfer efficiencies in $Er,Yb:Ca_3RE_2(BO_3)_4$ crystals are similar to those in $REAl_2(BO_3)_4$ oxoborate crystals [4, 5] and Er and Yb co-doped glass and more efficient than in vanadates and tungstates [17, 18].

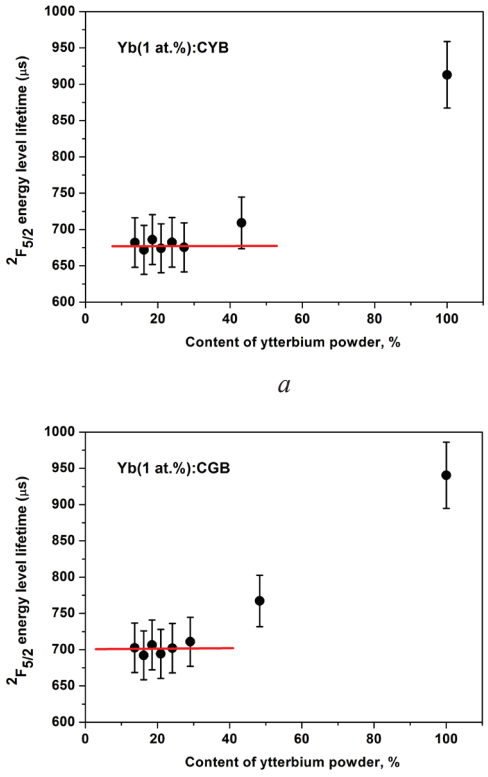
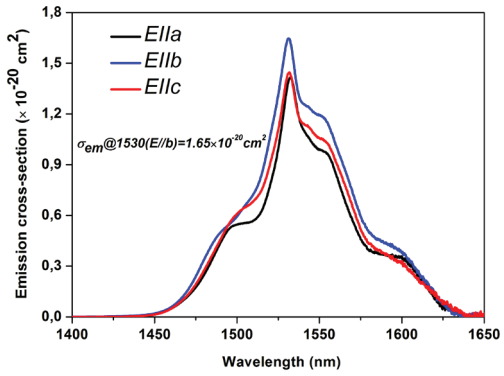


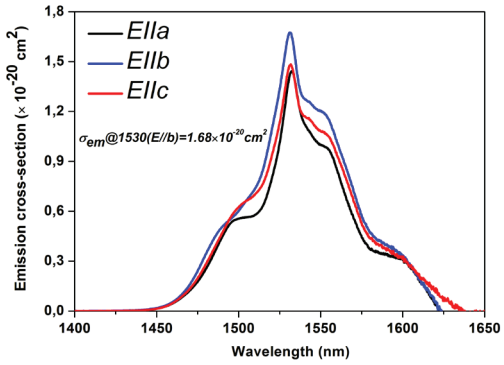
Figure 7 – The $^2F_{5/2}$ energy level lifetimes of Yb(1 at.%):CYB (*a*) and Yb(1 at.%):CGB (*b*) crystalline powder in glycerin

The stimulated emission cross-section spectra of $Er,Yb:Ca_3RE_2(BO_3)_4$ crystals in the spectral range of 1400–1650 nm are shown in Figure 8. The highest stimulated emission cross-sections of both crystals was found to be about 1.65×10^{-20} cm^2 at 1530 nm for polarization $E // b$. One can also note the low anisotropy of stimulated emission for $Er,Yb:Ca_3RE_2(BO_3)_4$ crystals.

Taking into account the similarity of absorption and emission spectra of both crystals the gain cross-section spectra $g(\lambda)$ were calculated only for Er,Yb:CYB crystal for $E // b$ polarization. The gain cross-section spectra of Er,Yb:CYB crystal for different inversion parameters β from 0.1 to 1 in the spectral range of 1400–1650 nm are shown in Figure 9. In accordance with presented spectra the maximum of gain band is centered at 1530 nm under $\beta > 0.3$, which causes the spectral position of laser emission.



a



b

Figure 8 – The stimulated emission cross-section spectra of Er,Yb:CYB (*a*) and Er,Yb:CGB (*b*) crystals

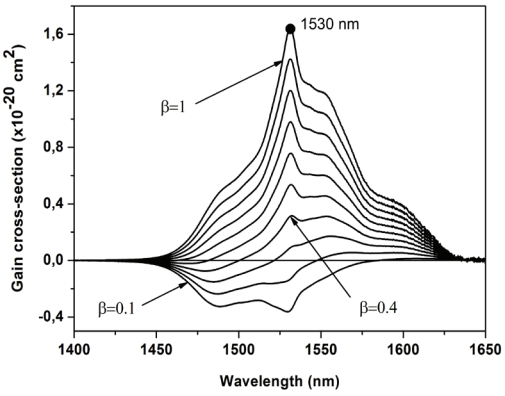


Figure 9 – The $E // b$ gain cross-section spectra of the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+} ions in Er,Yb:CYB crystals in the spectral range of 1400–1650 nm

Laser performance of Er,Yb:$Ca_3RE_2(BO_3)_4$ *crystals*

Input-output characteristics and laser output spectrum of QCW Er,Yb:CYB diode-pumped lasers are plotted in Figure 10 and Figure 11 respectively. The maximal peak output power of 0.5 W at 1530 nm with slope efficiency of 13 % was obtained for OC with transmittance of 1.3 % when absorbed pump power was 6.8 W. The laser threshold was about 2.5 W of absorbed pump power. The laser radiation was linearly polarized ($E // b$).

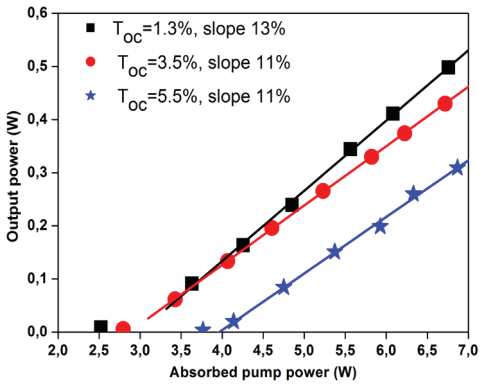


Figure 10 – Input-output characteristics of Er,Yb:CYB laser

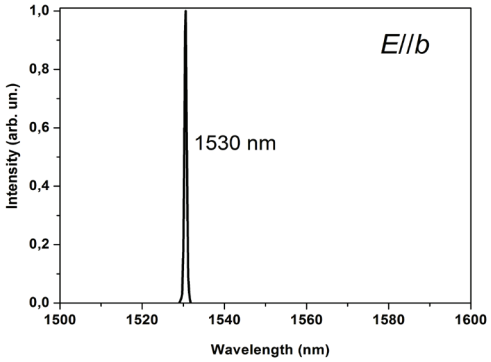


Figure 11 – Laser output spectrum

Input-output characteristics of QCW Er,Yb:CGB laser are shown in Figure 12. The similar slope efficiencies with slightly lower output powers and higher thresholds were obtained in this case. The weak dependence of the slope efficiency from the OC's transmittance evidences low passive losses of the laser resonator.

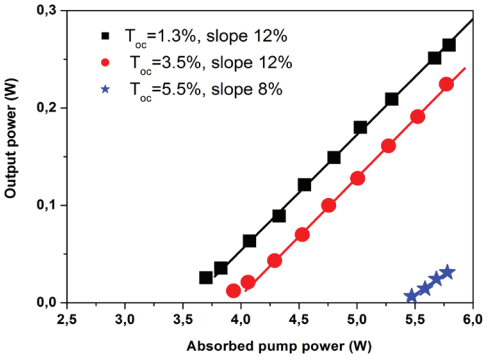


Figure 12 – Input-output characteristics of Er,Yb:CGB laser

The maximal peak output power of 0.4 W was obtained at 1530 nm, slope efficiency was estimated to be 12 % for 1.3 % OC. It should be mentioned that the spatial profile of the output beam was TEM00 mode with $M^2 < 1.5$ during all laser experiments.

Conclusions

$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd) crystals co-doped with Er and Yb ions were grown by Czochralski technique. The detailed investigation of the spectral-luminescent properties of the Er,Yb:$Ca_3RE_2(BO_3)_4$ (RE=Y, Gd) crystals were performed. Diode-pumped QCW laser operation of the Er:Yb:CYB and Er,Yb:CGB crystals was demonstrated, for the first time to our best knowledge. Maximal peak output power of 0.5 W with slope efficiency of 13 % was achieved at 1530 nm by using of Er,Yb:CYB crystal. The obtained characteristics indicate the promise of Er, Yb:CYB and Er, Yb:CGB crystals usage as active media of pulsed-pumped lasers emitting in the spectral range 1.5–1.6 μm for application in laser rangefinder, LIBS and LIDAR systems.

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