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# Mid-infrared laser operation of $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$ sesquioxide ceramic

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## Abstract

A transparent sesquioxide ceramic  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  was fabricated by vacuum sintering at 1780 °C for 3 h with lanthana acting as a sintering additive. It was of single-phase nature (sp. gr.  $Ia\bar{3}$ ) with a close-packed microstructure (average grain size: 17  $\mu\text{m}$ , pore content: 5 ppmv) and high transparency of 80% at 1.1  $\mu\text{m}$ . The  $\text{La}^{3+}$  ions induced additional inhomogeneous broadening in the absorption and emission spectra of  $\text{Er}^{3+}$  ions. For the  ${}^4\text{I}_{11/2} \rightarrow {}^4\text{I}_{13/2}$  transition, the stimulated-emission cross-section  $\sigma_{\text{SE}}$  was  $1.06 \times 10^{-20} \text{ cm}^2$  at 2719 nm and the luminescence lifetimes of the  ${}^4\text{I}_{11/2} / {}^4\text{I}_{13/2}$  states amounted to 2.73 / 5.91 ms, respectively. A  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic laser generated 300 mW at 2840 nm with a slope efficiency of 27.7% and a laser threshold of only 17 mW.

Keywords: transparent ceramic, sesquioxides, erbium ions, mid-infrared, optical spectroscopy, laser

## 1. Introduction

Cubic yttrium sesquioxide (yttria)  $\text{Y}_2\text{O}_3$  is a well-known host material for doping with trivalent rare-earth ions [1,2]. It possesses a bixbyite or C-type structure (bixbyite is a  $(\text{Fe},\text{Mn})_2\text{O}_3$  mineral) with sp. gr.  $T_h^7 - Ia\bar{3}$ . Yttria offers two substitutional rare-earth sites with symmetries  $C_2$  (3/4 of ions) and  $C_{3i}$  (1/4 of ions) both with VI-fold oxygen coordination, however, electric dipole transitions are forbidden for ions entering the latter sites [1]. Like other cubic sesquioxides,  $\text{Y}_2\text{O}_3$  offer strong crystal fields leading to large Stark splitting of multiplets of the dopant ions in  $C_2$  sites which results in broadband emission properties. As a host matrix,  $\text{Y}_2\text{O}_3$  benefits from high thermal conductivity ( $\kappa = 12.8 \text{ Wm}^{-1}\text{K}^{-1}$  at room temperature [3]), good thermo-mechanical properties,

broadband transparency (0.24 – 9  $\mu\text{m}$ ) and relatively low phonon energy (among oxide materials). The latter is essential for reducing the rates of multiphonon non-radiative relaxation from excited-states of rare-earth ions, e.g., for emission in the mid-infrared.

Cubic  $\text{Y}_2\text{O}_3$  doped with erbium ( $\text{Er}^{3+}$ ) ions is attractive for laser operation in the mid-infrared, at 2.8  $\mu\text{m}$ , according to the  ${}^4\text{I}_{11/2} \rightarrow {}^4\text{I}_{13/2}$  electronic transition [4,5]. 2.8  $\mu\text{m}$  lasers are of practical importance for medical applications.

As the melting point of  $\text{Y}_2\text{O}_3$  is extremely high (2425 °C), the growth of single-crystals constitutes a complicated task. As an alternative, the transparent ceramic technology has emerged to offer rare-earth-doped  $\text{Y}_2\text{O}_3$  ceramic laser elements. In this way, the sintering temperature can be reduced down to about 1800 °C. Still, making yttria ceramic of sufficient quality and

size for practical applications requires the use of costly and low-productivity methods, such as a combination of hot pressing or free sintering followed by hot isostatic pressing (HIPing) [6-9].

Mid-infrared laser operation of Er:Y<sub>2</sub>O<sub>3</sub> ceramics was reported [4,5,10,11]. Sanamyan *et al.* designed a diode-pumped 2 at.% Er:Y<sub>2</sub>O<sub>3</sub> ceramic laser delivering 380 mW at 2.7 μm with a slope efficiency of 15% [4]. Ding *et al.* developed a diode-pumped 7 at.% Er:Y<sub>2</sub>O<sub>3</sub> ceramic laser with a slab geometry generating 13.4 W at 2.84 μm at the expense of a low slope efficiency of 16% and low beam quality [5]. Recently, Mužík *et al.* studied heavily doped ceramic, 15 at.% Er:Y<sub>2</sub>O<sub>3</sub>, yielding 0.95 W at 2.73 μm with still a moderate slope efficiency of 17.4% [11].

Increasing the processability of fabrication of Er:Y<sub>2</sub>O<sub>3</sub> ceramics can be achieved by introducing sintering additives. Heterovalent Zr<sup>4+</sup> or Hf<sup>4+</sup> ions are among the most commonly used sintering additive for yttria [12,13]. However, the photodarkening of such media makes it almost impossible to use them in lasers [14]. Lanthana (La<sub>2</sub>O<sub>3</sub>) is another efficient sintering additive [15]. The 10 mol% La:Y<sub>2</sub>O<sub>3</sub> solid solution is consolidated into laser-quality ceramics only by free vacuum sintering (without subsequent HIPing), as demonstrated previously for (Yb<sub>x</sub>La<sub>0.10</sub>Y<sub>0.9-x</sub>)<sub>2</sub>O<sub>3</sub> ceramics [16-18].

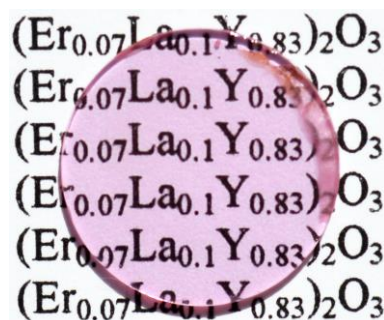
In the present work, we extend this approach to Er<sup>3+</sup>-doped Y<sub>2</sub>O<sub>3</sub> ceramics. (Er<sub>0.07</sub>La<sub>0.10</sub>Y<sub>0.83</sub>)<sub>2</sub>O<sub>3</sub> ceramics were fabricated by vacuum sintering and their structural, spectroscopic and laser properties were studied, for the first time, to the best of our knowledge.

## 2. Fabrication of ceramic

The nanopowders of (Er<sub>0.07</sub>La<sub>0.10</sub>Y<sub>0.83</sub>)<sub>2</sub>O<sub>3</sub> were obtained by the self-propagating high-temperature synthesis (SHS) with the use of metal nitrates as oxidants and metal acetates as a fuel. The starting reagents were A<sub>2</sub>O<sub>3</sub> (A = Y, La, Er) oxides (purity: 4N, Polirit, Russia), nitric acid HNO<sub>3</sub> (6N), and acetic acid CH<sub>3</sub>COOH (4N, Khimreaktiv, Russia).

At the first stage, yttrium acetate and nitrates of yttrium, erbium, and lanthanum were obtained by dissolving oxides in the respective acids. The aliquots of these solutions were mixed to obtain a SHS precursor containing the desired ratio of metals and nitrate and acetate groups in a molar ratio of 1.6:1. The precursor was dried and placed in a quartz flask in a furnace preheated to 750 °C. After combustion, the powder was calcined in air at 900 °C for 5 h. The powder deagglomeration was carried out in a planetary ball mill (ball diameter: 10 mm) with a silicon nitride grinding elements. The milling medium was absolute isopropyl alcohol, and the total time was 20 h in the mode of 10 min milling / 10 min cooling. After drying, the powder was again annealed at 900 °C for 1 h in air to remove residual organic compounds. The powder was then compacted uniaxially in a steel mold into

tablets (Ø15 mm, thickness: 2 – 2.5 mm) at a pressure of 250 MPa. The green-body sintering was carried out in a furnace with an oil vacuum (at a pressure of no more than 10<sup>-2</sup> Pa), a graphite heat insulation, and tungsten heaters at 1780 °C for 3 h. The heating was carried out at a rate of 10 °C/min up to 900 °C, then at 4 °C/min up to the sintering temperature, and the cooling was carried out in the switched off furnace mode. The ceramic was then annealed in air at 900 °C for 5 h to reduce oxygen vacancies and was mirror-polished with diamond pastes and slurries from 50 down to 0.5 μm. The final sample thickness was 1.2 mm. The polished ceramic disk is shown in figure 1. It is transparent and rose-colored due to the Er<sup>3+</sup> doping.

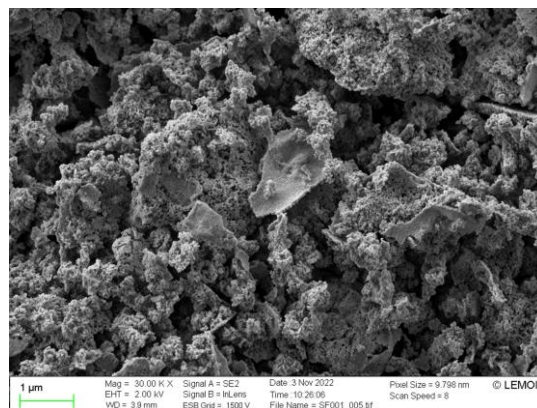


**Figure 1.** A photograph of an air-annealed and polished (Er<sub>0.07</sub>La<sub>0.10</sub>Y<sub>0.83</sub>)<sub>2</sub>O<sub>3</sub> ceramic disk.

## 3. Results and discussion

### 3.1. Structural study

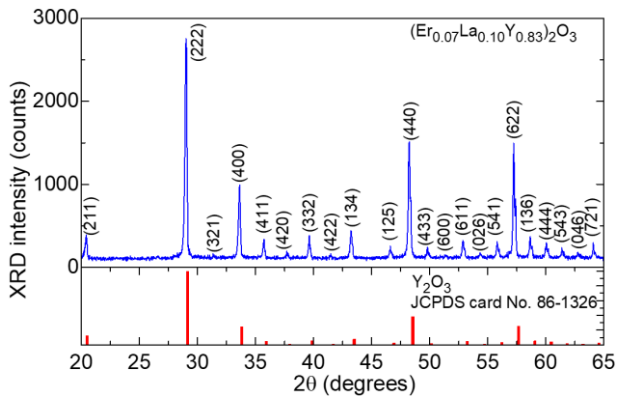
The typical morphology of sesquioxide powders obtained via SHS from acetatonitrate precursors is determined by nanosized primary particles combined into polydisperse agglomerates up to tens of μm in size [19,20]. The planetary ball milling allows to get rid of most of the agglomerates, however, some of them with a size about 1 μm and a rigid structure remain (figure 2). They are the most likely sources of pores in sesquioxide ceramics [21].



**Figure 2.** High-resolution scanning electron microscope (HRSEM) image of a planetary ball milled sesquioxide nanopowder (Er<sub>0.07</sub>La<sub>0.10</sub>Y<sub>0.83</sub>)<sub>2</sub>O<sub>3</sub>.

The specific surface area of the annealed  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  powder was  $S_{\text{BET}} = 17.9 \pm 0.1 \text{ m}^2/\text{g}$ , measured by the nitrogen adsorption according to the Brunauer–Emmett–Teller (BET) method.

As a rule, due to the mixing of components at the molecular level during the SHS synthesis of the precursor, solid-solution rare-earth sesquioxide powders are expected to be of single-phase nature. However, after sintering and / or subsequent annealing in air, in some cases, secondary phases enriched in lanthanum may be formed at the grain boundaries of ceramics [22]. The studied  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic was of single-phase nature (cubic, bixbyite-type structure, sp. gr.  $Ia\bar{3}$ , lattice constant:  $a = 10.6664(3) \text{ \AA}$ , density:  $\rho_{\text{calc}} = 5.4011(4) \text{ g/cm}^3$ ), as confirmed by an X-ray powder diffraction (XRD) study, see figure 3. The relative intensities of diffraction peaks for the ceramic coincide with the theoretical ones for yttria ( $\text{Y}_2\text{O}_3$ ), but their positions are shifted toward smaller angles due to the larger ionic radius of  $\text{La}^{3+}$  ( $1.032 \text{ \AA}$ ) as compared to that of  $\text{Y}^{3+}$  ( $0.90 \text{ \AA}$ ) and  $\text{Er}^{3+}$  ( $0.89 \text{ \AA}$ , for VI-fold oxygen coordination).

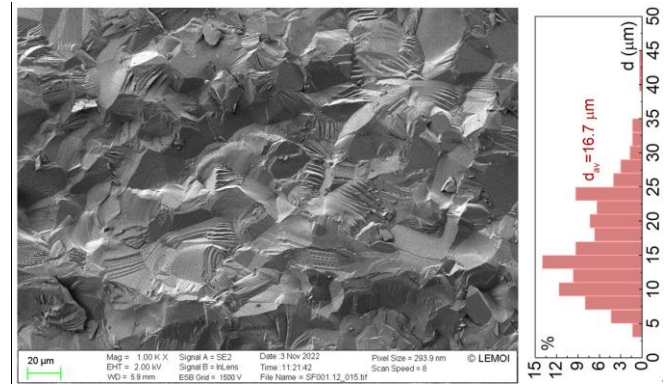


**Figure 3.** X-ray powder diffraction (XRD) pattern of the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic, *bars* – theoretical reflections for  $\text{Y}_2\text{O}_3$  (JCPDS card No. 86-1326), *numbers* – Miller's indices ( $hkl$ ).

A high-resolution scanning electron microscope (HRSEM) image of a fracture surface of the ceramic, figure 4(a), reveals a close-packed microstructure and a lack of secondary phases at the grain boundaries. The average grain size is  $\sim 17 \mu\text{m}$  and the grain size distribution is relatively broad, as shown in figure 4(b). Intense grain growth in typical for La-doped yttria [23,24]. The growth of large grains (2-3 times the average size) can also be associated with the rigid agglomerates in the SHS powder. The removal of such agglomerates, e.g., by sedimentation is a prerequisite for further improving the optical quality of the ceramic.

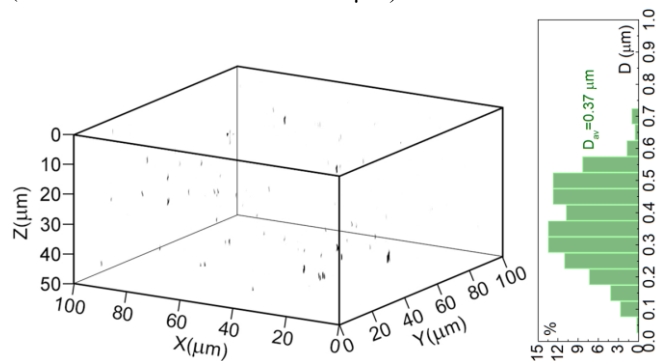
In spite of the high grain growth rate during sintering, there is no pore-grain boundary separation with the formation of intragranular porosity. The residual sub- $\mu\text{m}$  pores are located along the grain boundaries and in triple junctions and their clustering was not observed. This also leaves a room for

improvement of the ceramic quality through a longer soaking time at the sintering temperature.



**Figure 4.** (a) HRSEM image of the fracture surface of the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic; (b) the corresponding grain size distribution.

A 3D visualization of the porous structure of the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic and a pore size distribution (according to the confocal laser scanning microscopy data) are shown in figure 5. An intuitive model proposed earlier was used to estimate the residual porosity of high-density materials as the fraction occupied by pores in the studied material volume [25]. The calculated residual porosity  $P$  is as small as 0.0005 vol% and the average pore size is  $0.37 \mu\text{m}$  (with a maximum value of  $\sim 0.7 \mu\text{m}$ ).

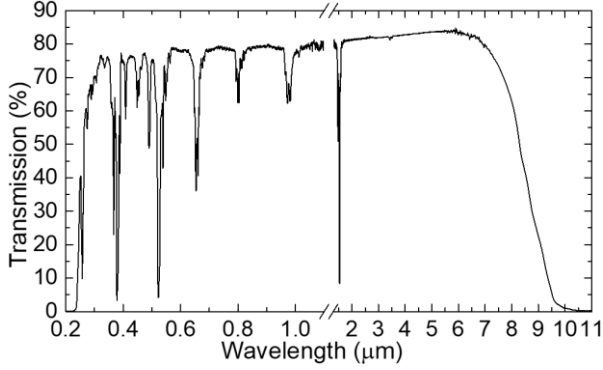


**Figure 5.** Analysis of porosity for the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic: (a) 3D confocal laser scanning microscopy observation of a volume of  $100 \times 100 \times 50 \mu\text{m}^3$ ; (b) pore size distribution.

### 3.2. Optical spectroscopy

Figure 6 shows the in-line transmission spectrum of an air-annealed and polished  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic disk with a thickness of 1.2 mm. The ceramic exhibited a very broad transparency range,  $0.24 - 9.5 \mu\text{m}$ . At  $\sim 1.1 \mu\text{m}$ , the ceramic disk had a high transmission of  $80.0 \pm 0.5\%$  being close to the theoretical limit set by Fresnel losses,  $T_0 = 2n/(n^2 + 1) = 82.4\%$  (accounting for multiple reflections, calculated based on the refractive index  $n = 1.900$  [26]). A rather sharp rise of transmission near the UV transparency edge confirms the confocal microscopy data about the low

ceramic porosity, while the gradual rise of transmission with the wavelength supports our conclusion about the sub- $\mu\text{m}$  and polydisperse structure of pores. The obtained data suggest that the pores are the main source of optical losses in the ceramic.



**Figure 6.** A transmission spectrum of an air-annealed and polished  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic disk (thickness:  $t = 1.2$  mm).

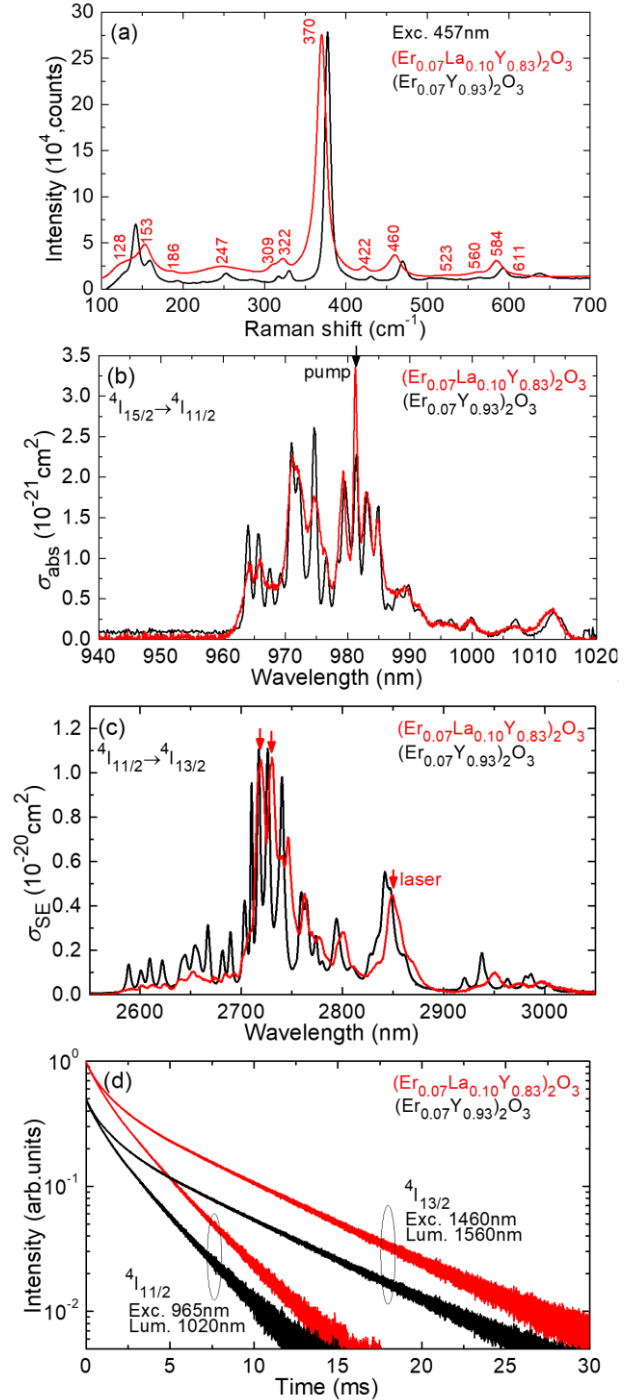
The Raman spectrum of the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic, figure 7(a), is typical for cubic bixbuite-type sesquioxides. The most intense peak appears at  $370\text{ cm}^{-1}$  (the  $A_g + F_g$  modes [27]), and it is broadened and red-shifted with respect to that for a  $(\text{Er}_{0.07}\text{Y}_{0.93})_2\text{O}_3$  ceramic ( $377\text{ cm}^{-1}$ ) indicating a formation of a substitutional solid-solution.

The absorption cross-sections,  $\sigma_{\text{abs}}$ , for the  $^4I_{15/2} \rightarrow ^4I_{11/2}$  transition are shown in figure 7(b). They were calculated for an  $\text{Er}^{3+}$  concentration  $N_{\text{Er}}$  of  $17.22 \times 10^{20}$  at/ $\text{cm}^3$ . The peak  $\sigma_{\text{abs}}$  is  $0.34 \times 10^{-20}\text{ cm}^2$  at  $981.3\text{ nm}$  and the corresponding absorption bandwidth (FWHM) is less than  $1\text{ nm}$  and another broader peak is found at  $971.1\text{ nm}$  ( $\sigma_{\text{abs}} = 0.22 \times 10^{-20}\text{ cm}^2$ ). The absorption spectrum is less structured as compared to that for the  $(\text{Er}_{0.07}\text{Y}_{0.93})_2\text{O}_3$  ceramic due to the  $\text{La}^{3+}$  codoping inducing additional inhomogeneous spectral broadening similarly to other “mixed” sesquioxide ceramics [28].

The stimulated-emission (SE) cross-sections,  $\sigma_{\text{SE}}$ , the  $^4I_{11/2} \rightarrow ^4I_{13/2}$  transition falling in to the mid-infrared spectral range were calculated using the Füchtbauer-Ladenburg formula [29]. The radiative lifetime of the emitting state ( $\tau_{\text{rad}} = 5.39\text{ ms}$ ) and the luminescence branching ratio ( $B_{\text{Jr}} = 18.3\%$ ) for  $\text{Er}^{3+}$  ions in yttria were calculated using the standard Judd-Ofelt theory. The mid-infrared emission spectra of  $\text{Er}^{3+}$  ions are very broad spanning from  $2.6$  to  $3\text{ }\mu\text{m}$  and the maximum  $\sigma_{\text{SE}}$  is  $1.06 \times 10^{-20}\text{ cm}^2$  at  $2719\text{ nm}$ , see figure 7(c). Due to the reabsorption from the metastable terminal laser level ( $^4I_{13/2}$ ) caused by a resonant excited-state absorption,  $^4I_{13/2} \rightarrow ^4I_{13/2}$ , the laser operation in  $\text{Er}^{3+}$  ions frequently occurs at longer wavelengths. For the studied ceramic, it is expected around  $2849\text{ nm}$  ( $\sigma_{\text{SE}}$  amounts to  $0.45 \times 10^{-20}\text{ cm}^2$ ).

The luminescence decay curves from the upper ( $^4I_{11/2}$ ) and lower ( $^4I_{13/2}$ ) laser levels of  $\text{Er}^{3+}$  in the  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic were measured under resonant excitation, as shown in figure 7(d). The mean luminescence lifetimes  $\langle \tau_{\text{lum}} \rangle$  are

$2.73\text{ ms}$  and  $5.91\text{ ms}$ , respectively, representing a favorable ratio for mid-infrared laser operation. For comparison, we also determined the corresponding luminescence lifetimes for an  $(\text{Er}_{0.07}\text{Y}_{0.93})_2\text{O}_3$  ceramic,  $\langle \tau_{\text{lum}} \rangle = 2.74\text{ ms}$  and  $5.88\text{ ms}$ , respectively, indicating a minor effect of  $\text{La}^{3+}$ .

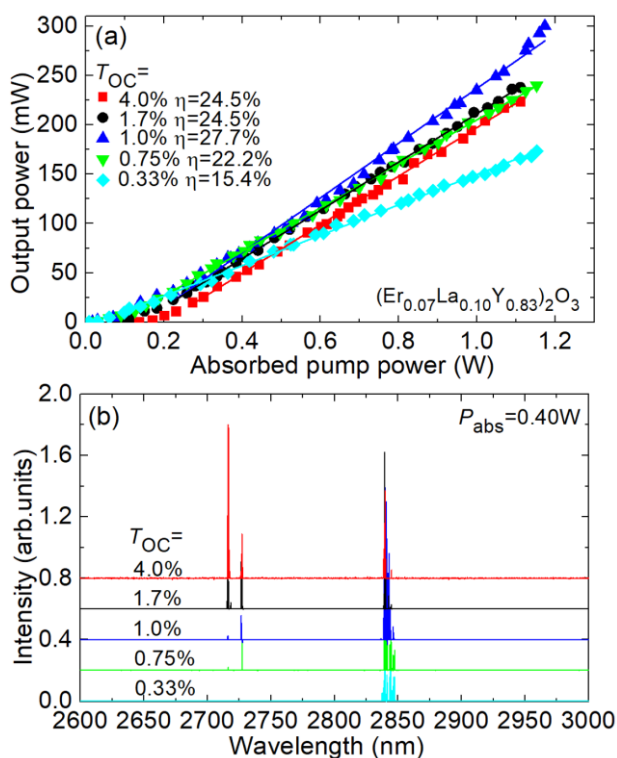


**Figure 7.** Spectroscopy of  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic: (a) Raman spectrum,  $\lambda_{\text{exc}} = 457\text{ nm}$ ; (b) absorption cross-sections,  $\sigma_{\text{abs}}$ , the  $^4I_{15/2} \rightarrow ^4I_{11/2}$  transition; (c) stimulated-emission (SE) cross-sections,  $\sigma_{\text{SE}}$ , the  $^4I_{11/2} \rightarrow ^4I_{13/2}$  transition; (d) luminescence decay curves from the  $^4I_{11/2}$  and  $^4I_{13/2}$   $\text{Er}^{3+}$  states under resonant excitation. The data for a  $(\text{Er}_{0.07}\text{Y}_{0.93})_2\text{O}_3$  ceramic are added for comparison.

### 3.3. Mid-infrared laser operation

The  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic ( $\text{Ø}12$  mm,  $t = 0.93$  mm) was polished from both sides to laser-grade quality and left uncoated. It was mounted on a passively cooled Cu-holder using a silver paste for better heat removal. A simple linear hemispherical laser cavity was used. It was formed by a flat pump mirror (PM) coated for high transmission (HT) at  $0.92 - 1.06$   $\mu\text{m}$  and high reflection (HR) at  $2.6 - 3.0$   $\mu\text{m}$ , and a set of concave output couplers (OCs) having a radius of curvature (RoC) of  $-100$  mm and a transmission at the laser wavelength  $T_{\text{OC}}$  in the range of  $0.33\% - 4.0\%$ . The ceramic element was placed close to the PM with an airgap less than  $1$  mm. The total cavity length was about  $100$  mm.

As a pump source, we employed a continuous-wave Ti:sapphire laser (3900S, Spectra Physics) delivering up to  $3.2$  W at  $981$  nm with a diffraction-limited beam quality ( $M^2 \approx 1$ ). The pumping was into the  ${}^4\text{I}_{15/2} \rightarrow {}^4\text{I}_{11/2}$  absorption band of  $\text{Er}^{3+}$  ions. The pump beam was focused into the ceramic element through the PM using an AR-coated achromatic lens (focal length:  $f = 75$  mm) resulting in a pump spot radius  $w_p$  of  $33 \pm 5$   $\mu\text{m}$  (at the  $1/e^2$  level). The measured single-pass pump absorption was  $36.7\%$ . The residual pump power was filtered out using a long-pass filter (LP1400, Spectrogon). The laser spectra were measured using an optical spectrum analyzer (AQ6376, Yokogawa).



**Figure 8.** Mid-infrared  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic laser: (a) input-output dependences,  $\eta$  – slope efficiency; (b) typical spectra of laser emission.

The mid-infrared  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic laser generated a maximum output power of  $300$  mW at  $\sim 2840$  nm

with a slope efficiency  $\eta$  of  $27.7\%$  (with respect to the absorbed pump power) and a laser threshold of only  $17$  mW (for  $T_{\text{OC}} = 1.0\%$ ), figure 8(a). The laser threshold gradually increased with output coupling, from  $9$  mW ( $T_{\text{OC}} = 0.33\%$ ) to  $135$  mW ( $T_{\text{OC}} = 4\%$ ). The input-output dependences were linear well above the laser threshold. Further power scaling was limited by the relatively low pump absorption in a thin ceramic sample and the available pump power. It is expected by applying commercially available InGaAs laser diodes emitting at  $\sim 980$  nm as pump sources.

The typical spectra of laser emission are shown in figure 8(b). For small output coupling ( $T_{\text{OC}} \leq 1\%$ ), the laser operated at  $\sim 2840$  nm and for higher  $T_{\text{OC}}$ , the laser emission was observed both at  $\sim 2716$  and  $2840$  nm. The ceramic laser operated on the fundamental transverse mode ( $\text{TEM}_{00}$ ).

## 4. Conclusions

To conclude, vacuum sintering at  $1780$   $^{\circ}\text{C}$  using sesquioxide nanopowders of complex composition  $(\text{Er},\text{La},\text{Y})_2\text{O}_3$  obtained via self-propagating high-temperature synthesis with  $\text{La}_2\text{O}_3$  acting as a sintering additive is a viable and technologically simple way of fabricating highly transparent laser ceramics with a close-packed microstructure and a reasonably low content of pores. An  $(\text{Er}_{0.07}\text{La}_{0.10}\text{Y}_{0.83})_2\text{O}_3$  ceramic fabricated in this way exhibits broadband mid-infrared emission owing to the  ${}^4\text{I}_{11/2} \rightarrow {}^4\text{I}_{13/2}$  transition, an appealing ratio of the upper laser level ( ${}^4\text{I}_{11/2}$ ) to lower laser level ( ${}^4\text{I}_{13/2}$ ) luminescence lifetimes and enables efficient laser operation at  $2.8$   $\mu\text{m}$ . The  $\text{La}^{3+}$  ions induce additional inhomogeneous broadening to the absorption / emission spectra of  $\text{Er}^{3+}$  ions. Further power scaling is expected by applying  $\sim 980$  nm InGaAs laser diodes as pump sources.

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