

# Comparative characteristics of Yb:(YLa)<sub>2</sub>O<sub>3</sub> laser ceramic samples

I.L. Snetkov, I.B. Mukhin, O.V. Palashov

**Abstract.** Two samples of laser ceramics synthesised at Russian institutes by two different methods are studied. The optical quality of the samples is measured, and efficient (36.6%) lasing in one of the samples is obtained under pumping at a wavelength of 940 nm.

**Keywords:** laser ceramics, yttrium oxide, lanthanides, disk laser.

## 1. Introduction

For the last two decades, significant advance in the technology of synthesis of optically transparent ceramics has been achieved [1, 2]. This technology allows one to faster and cheaper synthesise large-size optical element (including active ones) of high optical quality comparable with the quality of single crystal. In addition, it makes it possible to control the spatial distribution of dopant in the volume of elements [2] and achieve a higher dopant concentration than in single crystals [3]. The flexibility of the technology allows one to synthesise ceramics from a great variety of materials with a cubic symmetry and dope them with various ions. This is especially important in the case when it is impossible (or economically inefficient) to grow a corresponding single crystal with comparable aperture and optical quality due to a high melting temperature, specific features of crystallisation, or existence of phase transitions in the material. Rare-earth sesquioxides Re<sub>2</sub>O<sub>3</sub> (Re = Y, Lu, Sc) doped with Yb<sup>3+</sup> ions are examples of such materials. Owing to the higher thermal conductivity coefficient [4–7], better thermo-optical properties responsible for the thermal lens and thermally-induced depolarisation [8, 9], and a wider gain spectrum [10, 11] than those of widely used Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub>, these materials are undoubtedly promising for fabrication of active elements of lasers with high average and peak powers.

This work is devoted to the study of yttrium ceramics Y<sub>2</sub>O<sub>3</sub>. This material has a high refractive index, wide (0.3–7 μm) transmission spectrum, good chemical stability, and high mechanical strength. An additional advantage of this ceramics over the above-mentioned sesquioxides is a considerably lower cost of initial materials. The first mention of the synthesis of translucent yttrium ceramics dates

back to 1966 [12]. A year later, the authors of [13] reported that they synthesised transparent yttrium ceramics doped with Eu<sup>3+</sup> ions. Lasing was obtained for the first time in Nd<sup>3+</sup>-doped Y<sub>2</sub>O<sub>3</sub> ceramics synthesised using ThO<sub>2</sub> as a sintering additive (Nd-doped Yttralox) [14, 15]. Lasing in Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> was demonstrated for the first time comparatively recently [16]. To date, lasing in Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> ceramics has been achieved in a cw regime with a power of 70 W and a slope efficiency  $\eta_{\text{slope}} = 70\%$  [17], as well as in a mode-locking regime with a power of 7.4 W and  $\tau_{\text{pulse}} = 547$  fs [17] or 540 mW and  $\tau_{\text{pulse}} = 68$  fs [18]. All the latest results were obtained using ceramics produced by Konoshima Chemical Co., Ltd. (Japan), which proved itself as one of the best manufacturers of laser ceramics.

In the present work, we study and compare the optical quality and lasing characteristics of two samples of Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> ceramics synthesised by different technological methods in two Russian institutes.

## 2. Ceramic elements

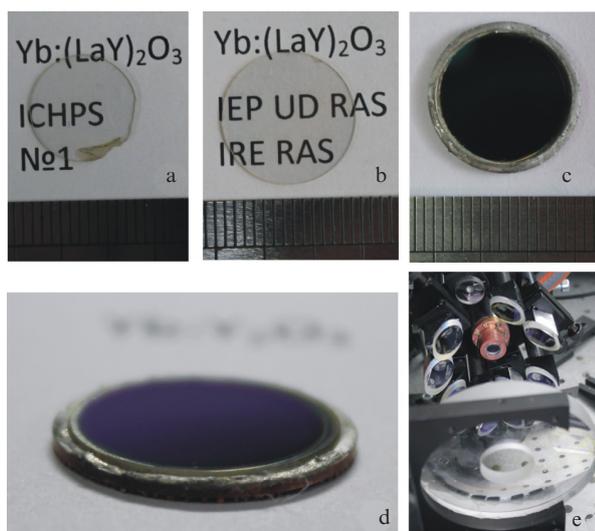
We studied two samples of Yb<sup>3+</sup>-doped Y<sub>2</sub>O<sub>3</sub> laser ceramics synthesised by different technologies. The first sample (similar to the sample studied in [19]) was synthesised at the Institute of Chemistry of High-Purity Substances, Russian Academy of Sciences (ICHPS RAS). The powders for sintering of the Yb<sub>0.1</sub>Y<sub>1.7</sub>La<sub>0.2</sub>O<sub>3</sub> composition were synthesised by the SHS method using acetate–nitrates of metals [20, 21]. As initial materials, yttrium oxide (99.99%), lanthanum oxide (99.99%), ytterbium oxide (99.99%), nitric acid (99.9999%), and acetic acid (99.9999%) were used. The powders were not additionally milled or granulated. To make compacts, the powders were uniaxially pressed with a pressure of 500 MPa into disks 15 mm in diameter and 2 mm in thickness. Sintering was performed in a vacuum furnace (SNVE 1.3.1) with tungsten heaters (residual pressure ~ 10<sup>-3</sup> Pa) and a heat shield [19].

The second sample was synthesised at the Institute of Electrophysics, Ural Division, Russian Academy of Sciences (IEP UD RAS) and sintered at the V.A. Kotelnikov Institute of Radioengineering and Electronics, Russian Academy of Sciences (IRE RAS). As initial materials, La<sub>2</sub>O<sub>3</sub> (99.99% REO), Yb<sub>2</sub>O<sub>3</sub> (99.99% REO), and Y<sub>2</sub>O<sub>3</sub> (99.99% REO) commercial powders were mixed in the stoichiometric proportion to obtain a composition Yb<sub>0.12</sub>Y<sub>1.61</sub>La<sub>0.27</sub>O<sub>3</sub>. From this mixture, a nanosized powder was obtained by laser evaporation in vacuum [22] and additionally ground in a zirconium ball mill. Then, the powder was compacted by cold isostatic pressing into a disk 20 mm in diameter and 4 mm in thickness. Sintering was performed in a vacuum furnace with graphite heaters at a temperature of 1625 °C for 12 h [23].

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From the ceramic samples, we made two optical elements 0.5 mm thick (Figs 1a and 1b) and studied their optical properties. The quality was measured by the method used in [19, 21]. A probe beam with a wavelength of 1075 nm was sent through the sample under study, and the power of the passed signal was measured immediately behind the sample (at a distance smaller than 5 mm) and at a distance of 5 m from the sample. At the same positions, we measured the radiation power in the absence of the sample. From the experimental data, we estimated the transmission and scattering of the samples under study at the wavelength of the probe radiation. The transmission was 82.6% for the first element and 82.4% for the second with an error of  $\pm 0.5\%$ , which rather well coincides with the theoretical transmission for the studied material. The scattering was 3.9% and 3.2%, respectively, which agrees with scattering losses of 0.78 and 0.64  $\text{cm}^{-1}$ . Then, from these optical elements, we fabricated active laser elements by coating their faces by a dielectric layer, antireflecting from one side and highly reflecting from the other for the pump (940 nm) and lasing (1030 nm) wavelengths. For efficient cooling, the elements with dielectric coatings were soldered with indium to a copper heat sink of the laser head (Figs 1c and 1d) [24, 25]. Cooling was performed by water. A photograph of the disk laser head with an active element is presented in Fig. 1e.

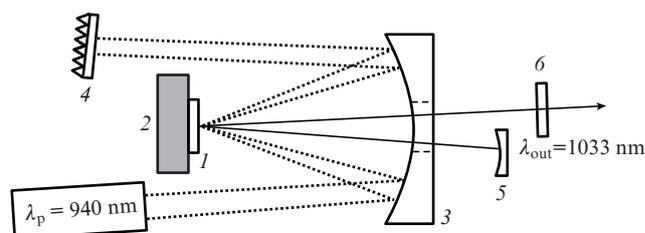


**Figure 1.** Photographs of the studied samples 0.5 mm thick after optical treatment (a, b), of the samples soldered to the copper heat sink (c, d), and of a disk laser head with an active element (e).

The quality of the active elements was additionally controlled after soldering. We measured the power of a probe beam (1075 nm) incident on the active element and passed through it, as well as the power of radiation reflected from the front antireflection-coated face of the active element. The total losses in the first and second samples were, respectively, 3.5% and 3% of the incident radiation power. The reflected radiation power was 0.17% for both samples, which testifies to a high quality of the antireflection coating. As follows from the above data, the second ceramic sample demonstrates a better optical quality than the first one both before deposition of dielectric coatings and soldering to the heat sink and after this, i.e., in the form of an active element of a disk laser.

### 3. Study of laser properties

The optical scheme of the laser is shown in Fig. 2. As a pump source, we used a fibre-coupled diode laser (Laserline LDM 2000) with the wavelength  $\lambda_p = 940$  nm. The pump radiation was focused by a spherical mirror (3) on the sample under study (1) into a spot 2 mm in diameter and, after one V-shaped pass through the sample, was deflected to an absorber (4) (in this process, 30% of radiation power was absorbed).



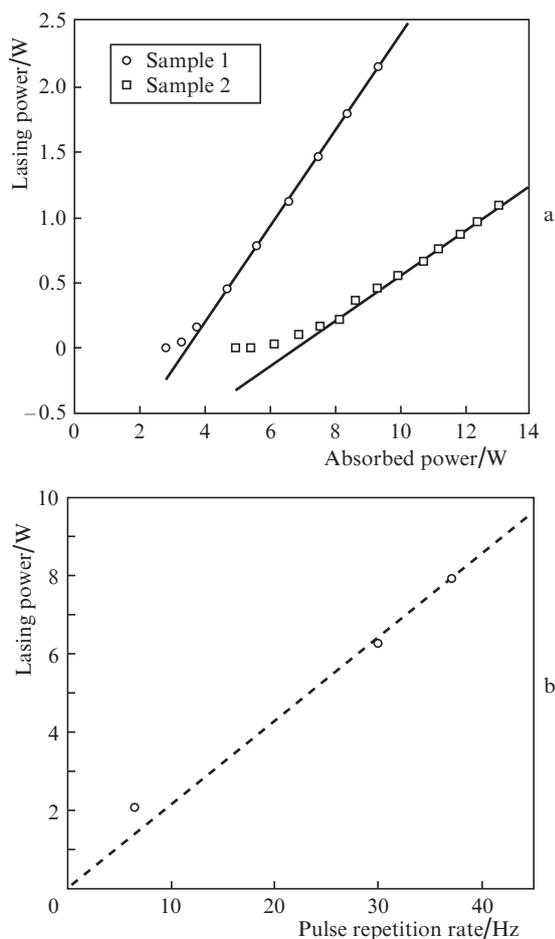
**Figure 2.** Optical scheme of the laser.

The active element mounted on a heat sink (2) was placed into a cavity formed by mirrors (5) (spherical mirror with a radius of 30 mm and a reflection of  $\sim 100\%$  at a wavelength of 1030 nm) and (6) (plane output mirror) and the rear mirror surface of the element. The used output mirrors had reflectances of 98%, 95%, and 90% at a wavelength of 1030 nm. The length of one cavity shoulder was 40 mm.

Upon quasi-cw pumping by pulses with a duration  $\tau = 3$  ms and a repetition period  $T = 150$  ms, the laser operated in the free-running mode. The laser power was measured using Ophir 3A and Ophir 10A power meters. Lasing was observed with all the three used output mirrors and differed only by the threshold and the slope efficiency. The best output mirror transmissions from the point of view of the slope efficiency were 10% for the first sample and 5% for the second. This can occur because the second sample may have higher losses independent of its optical quality. The best results on the laser power for both elements are presented in Fig. 3a. Lasing in the first sample occurred with the maximum at a wavelength of 1033 nm as the average absorbed pump power exceeded the threshold of 2.8 W, while lasing in the second sample occurred with the maximum at 1032 nm at the average absorbed pump power exceeding the threshold of 4.9 W. The lasing slope efficiencies were 36.6% and 17.5%, respectively.

The dependence of the average lasing power of the first sample on the repetition rate of 3-ms pump pulses was measured at the output mirror transmission of 10% (Fig. 3b). The average output power at a repetition rate of 37 Hz was 8 W. With a further increase in the pump pulse repetition rate, the average output power ceased to increase due to parasitic thermal effects caused by high heat release in the active elements.

As follows from Fig. 3, the second sample, despite a better optical quality, has a twofold lower lasing efficiency; at the same time, it demonstrated intense visually observed luminescence in the visible region. The visible and near-IR luminescence spectra of the first and second samples were measured using a SOLAR TII S150-2 spectrometer under identical conditions upon excitation at 940 nm with identical powers. The luminescence beam was focused by a short-focal-length quartz lens 40 mm in diameter without dielectric coatings on the receiving fibre of the spectrometer, which measured the spectrum of radiation incident on the fibre (Fig. 4, solid



**Figure 3.** Dependences of the average output laser power on the average absorbed pump power in a quasi-cw regime ( $\tau = 3$  ms,  $T = 150$  ms) for samples 1 and 2 (a), as well as dependence of the average lasing power of sample 1 on the pump pulse repetition rate (b).

curves). The spectrum of attenuated laser radiation was also recorded on the spectrometer (Fig. 4b, dashed curves). The black dashed curve in Fig. 4a shows the near-IR luminescence spectrum reduced in amplitude on the half-wavelength scale.

The near-IR spectra correspond to the typical luminescence spectrum of Yb<sup>3+</sup> in Y<sub>2</sub>O<sub>3</sub> [19, 21]. In the visible spectral region, one observes four groups of lines with maxima at 409.6, 490, 550, and 662 nm. Emission in these wavelength regions is usually associated with cooperative processes of the Yb<sup>3+</sup> ion interaction with other rare-earth ions [26, 27]. In particular, cooperative processes between Yb<sup>3+</sup> and Er<sup>3+</sup> ions

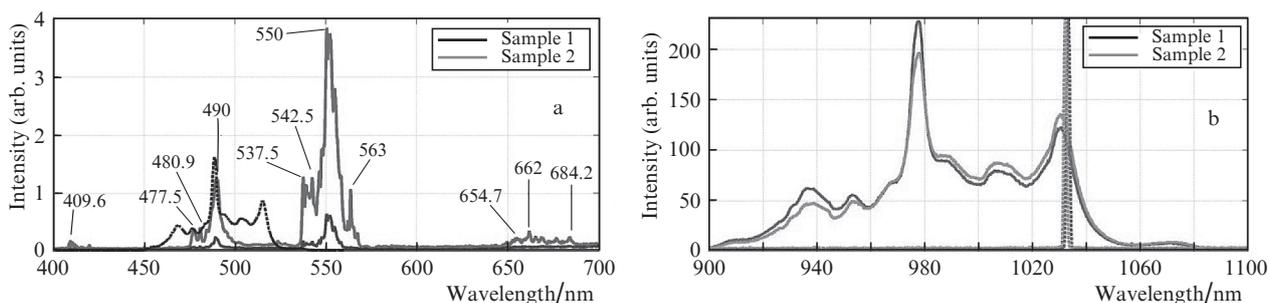
are believed to be responsible for emission in the regions of 409 [28] and 550 [29, 30] nm, as well as in the range 650–680 nm [29, 31]. The emission in the region of 490 nm is related to the cooperative luminescence of Yb<sup>3+</sup>–Yb<sup>3+</sup> clusters at the doubled Yb<sup>3+</sup> luminescence frequency [32, 33] or to cooperative processes between Yb<sup>3+</sup> and Tm<sup>3+</sup> ions [34, 35]. Similar spectra were obtained in Yb<sup>3+</sup>:Y<sub>2</sub>O<sub>3</sub> ceramics in [36]. The main difference between the spectra consists in the different intensity ratios of peaks at different wavelengths, which can point to different concentrations of rare-earth dopants in our and other samples.

In the experiment, the luminescence intensity in the regions of 490 and 550 nm observed in sample 2 exceeded the corresponding intensities in sample 1 by more than five times at the same pump power. The luminescence in the regions of 409 nm and 650–680 nm was not observed in the first sample within the experimental accuracy. The weak luminescence in the visible spectral region can testify to the chemical purity and a higher optical quality of this ceramic element and, hence, to the possibility of more efficient lasing. Thus, comparing the visible luminescence intensity of materials (before making active elements) with the luminescence intensity of a reference sample, one can not only estimate the optical quality but also characterise the laser properties of ceramic elements.

#### 4. Conclusions

We studied the optical quality and the spectral and laser properties of two samples of laser ceramics synthesised by different methods from differently prepared powders. Active elements for disk lasers are made and quasi-cw lasing is obtained. Upon pumping at a wavelength of 940 nm, the lasing slope efficiency for the ceramic element synthesised in the ICHPS RAS reached 36.6%, while the corresponding efficiency for the sample of the IEP UD RAS was 17.5%. The intensity of the visible luminescence related to the cooperative interaction of impurity ions can characterise the chemical purity of a ceramic material and serve as a criterion of its applicability for the production of active laser elements. It is the cooperative processes that are responsible for the considerable difference in the lasing efficiency of the studied ceramic samples with comparable optical quality.

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**Figure 4.** Luminescence spectra of samples 1 and 2 in the visible (a) and near-IR (b) regions.

and executed at the Institute of Applied Physics, Russian Academy of Sciences. The study of the optical quality and the spectral and lasing characteristics of the ceramic element synthesised at the Institute of Electrophysics, Ural Division, Russian Academy of Sciences, was supported by the State Order for the Institute of Applied Physics, Russian Academy of Sciences (Project No. 0035-2014-0107).

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