

Effect of heat-treatment on properties of ytterbium and lanthanum co-doped yttria transparent ceramics

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5 at.-% Yb³⁺ doped (La_{0.10}Y_{0.90})₂O₃ transparent ceramics are fabricated by using commercial nanopowders in H₂ atmosphere. Effect of the heat-treatment on the structure, spectral properties of (Yb_{0.05}La_{0.10}Y_{0.85})₂O₃ transparent ceramics is investigated. Laser grade ceramic samples are fabricated, and a diode-pumped laser is demonstrated, the maximum output power of 920 mW at 1 075 nm is obtained with a slope efficiency of ~ 31%, the laser pulse as short as 730 fs is also realized at the central wavelength of 1 033 nm.

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The rare-earth-ions doped transparent ceramics laser materials have attracted considerable attentions because of its numerous advantages over single-crystal. Rare earth doped YAG and Y₂O₃ are two kinds of laser transparent ceramics main on research^[1,2], Y₂O₃ is one of the most important rare earth doped laser materials due to its good optical, thermal, chemical, and mechanical properties. Compared with Nd³⁺ ions, Yb³⁺ ions have a very simple electronic structure with only two manifolds separated by about 10 000 cm⁻¹, which leads to very low quantum defects, such as excited state absorption.

Over the past fifteen years, with the development of nanocrystalline technology and a non-press vacuum sintering method^[3], the sintering temperature of Y₂O₃ transparent ceramic has been decreased to 1 700 °C, and the quality of the ceramic has been improved greatly. Consequently, ceramic lasers can be obtained with a high efficiency and ultra-short pulses that is comparable with that of single-crystal lasers^[4,5]. Laser output with the slope efficiency of 82.4 % has been obtained with the Yb³⁺:Y₂O₃ ceramics^[6], and the laser pulse as short as 68 fs has been reported with Yb³⁺ doped Y₂O₃ transparent ceramics^[7].

In our previous work, we found that the sintering temperature could decrease remarkably without influence on optical properties by doping La₂O₃ as an additive^[8]. The heat-treatment of ceramic green body is a very important factor to the microstructure, density, and transmittance, In this letter, the effect of heat-treatment on the structure and spectral properties of the (Yb_{0.05}La_{0.10}Y_{0.85})₂O₃ transparent ceramics were studied. In addition, a continuous wave (CW) laser output was reported at the wavelengths of 1 075 nm.

5-at.-% Yb:(La_{0.10}Y_{0.90})₂O₃ high purity commercial nanopowders were used to fabricate ceramic samples. The powders were mixed in absolute ethyl alcohol for 2~5 h with zirconia balls, and then calcined at 1 200 °C for 10 h in air atmosphere. Disks with 15 and 25 mm in diameter and 5 mm in thickness were isostatically pressed at 2 T/cm², some pressed plates were heat-

treated at 1 300 °C for 10~30 h, then sintered at 1 680 °C for 45 h in H₂ atmosphere without pressure. The sintered specimen were double polished (2~5 mm in thickness) for optical test, microstructure observation, spectral analysis and laser experiment.

Bulk density of the specimen was measured by the Archimedes' method. The microstructures were observed with optical microscope (BX 60F, OLYPMUS, Japan). The absorption spectrum was measured with a spectrophotometer using Xe light as pump source (Model V-570, JASCO, Japan). The fluorescence spectra excited with 940-nm Xe lamp were measured with a spectrofluorimeter (Fluorolog-3, Jobin Yvon Spex, France). All the spectroscopic analyses were made at room temperature.

Figure 1 displays the photos of (Yb_{0.05}La_{0.10}Y_{0.85})₂O₃ transparent ceramics samples before and after sintering. As shown in Fig. 1(a), sample A was heat-treated at 1 300 °C for 25 h, a significant shrinkage occurred in sample A when compared with sample B (without heat-treatment). Figure 1(b) shows that the edge of the sample A (after sintering) was black, this is because the sintering activity of ceramic green body was decreased after heat-treatment, which is not benefit for the pores elimination from grain boundaries, As a result, the edge of sample A was not compact, so its transparency is not high. Therefore, the heat-treatment temperature should be lowered.

Figure 2 is the in-line optical transmittance spectra of (Yb_{0.05}La_{0.10}Y_{0.85})₂O₃ transparent ceramics. Sample B has a high transmittance, and its highest in-line

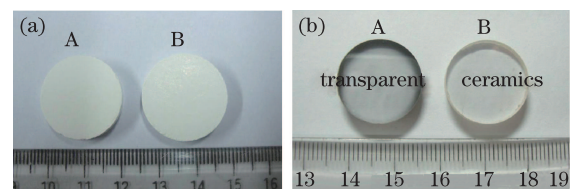


Fig. 1. Photos of (Yb_{0.05}La_{0.10}Y_{0.85})₂O₃ transparent ceramics samples (a) before and (b) after sintering.

transmittance reaches 80%, which is close to its theoretical value; this indicates that the laser grade ceramic samples have been fabricated. The highest transmittance of sample A was 60%, which is much lower than that of sample B, so there are relatively more light scattering factors in sample A, and the main scattering factor is the pores.

Figure 3 shows the optical microstructure photographs of $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ transparent ceramics. It is obvious that there are many pores (black points) existed in grains and grain boundaries of sample A, this result is accordance with that of the transmittance. Figure 3(b) inhibits that there were no second phase and almost no pores in grains or grain boundaries in sample B, which indicates that sample B has a very compact structure, and the average grain size was about $30\sim 50\ \mu\text{m}$.

The measured and relative density values of sample A and B are listed in Table 1. It is clear that both the measured and relative densities of sample A are lower than that of sample B, which indicates that sample A was not compact enough, so the suitable heat-treatment temperature still need to be further investigated.

Uncoated $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ ceramic was used for lasing experiment under laser diodes (LD) end-pumping; the ceramic sample was cut as $3\times 5\times 3$ (mm). The laser diode bar with 970 nm and a maximum power of 7 W was used as the pump source, and was focused into the ceramic by two coupling lenses, the focused pump beam had a diameter of $200\ \mu\text{m}$.

For optimizing the experiment result, two coupling-out mirror transmittances ($T = 2.5\%$ and 7%) was used, and the result of measured output power versus the incident power was shown in Fig. 4. When $T = 7\%$, with a pump

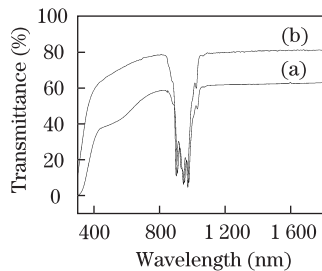


Fig. 2. In-line optical transmittance of $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ transparent ceramics samples.

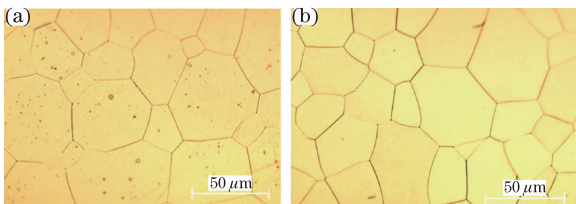


Fig. 3. Optical microstructure of $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ transparent ceramics (a) with and (b) without heat-treatment.

Table 1. Densities of $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ Transparent Ceramics

Sample	A	B
Measured Density (g/cm^3)	5.283	5.329
Relative Density (%)	98.4	99.3

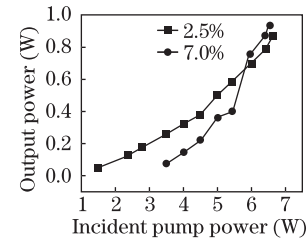


Fig. 4. Curve of laser input and output power.

power of 6.5 W, a maximum output power of 920 mW with a slope efficiency of 31 % was obtained at the wavelength 1075 nm. It is believed that more high-power and efficient lasers will be demonstrated by using a bigger transmittance of coupling-out mirror. In addition, the mode-locked pulse laser as short as 174 ps and 730 fs was also firstly realized in $(\text{Yb}_{0.05}\text{La}_{0.10}\text{Y}_{0.85})_2\text{O}_3$ ceramic^[9,10], so if by choosing a proper thickness of the gain medium and Yb^{3+} doping concentration, further optimizing the laser cavity, the even shorter pulse may be promising.

In conclusion, high optical quality 5 at.-% $\text{Yb}:(\text{La}_{0.10}\text{Y}_{0.90})_2\text{O}_3$ transparent ceramics are successfully fabricated by conventional ceramics processing and sintered without pressure in H_2 atmosphere. The transmittance and relative densities of ceramics are all decreased after the high temperature heat-treatment, which indicates that the sintering activity of ceramic body is decreased due to too high temperature heat-treatment. Therefore, the optimum temperature still needs to be investigated. When $T = 7\%$, the CW laser is obtained at 1075 nm, and the maximum output power is 920 mW with a slope efficiency of $\sim 31\%$.

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