Growth and Characteristics of Yb-doped $Y_3Ga_5O_{12}$ Laser Crystal

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***Abstract*— Ytterbium (Yb) doped rare-earth garnet $Y_3Ga_5O_{12}$ (YGG) single crystal was grown by the optical floating zone method for the first time, to our knowledge. Its structure and cell parameter were determined by X-ray powder diffraction. The thermal properties of Yb:YGG, including specific heat, thermal expansion coefficient, thermal diffusion coefficient, and thermal conductivity, were investigated. The optical properties of the crystal were also studied and the effective gain cross sections were calculated. With the crystal cut along the ⟨111⟩ direction, laser performance was also demonstrated by using a laser diode as the pump source. All the results show that Yb:YGG can be an excellent laser medium for applications to tunable and ultrafast pulsed lasers.**

***Index Terms*— Optical materials, solid lasers, ytterbium compounds.**

I. Introduction

SINCE the discovery of neodymium (Nd)-doped garnet lasers in the 1960s [1], active ion-doped garnet crystals have been widely investigated and identified as promising laser materials due to their large gain and excellent thermal, chemical, and mechanical properties. Nd-doped yttrium aluminum garnet (YAG) lasers have been a commercial success and their output power has reached several tens of kilowatts. With gallium replacing aluminum in the YAG crystal, another garnet material named yttrium gallium garnet (YGG) is obtained [2]–[4]. Previous studies on this material have found that this crystal has good thermal conductivity (9 W/mK) and is an excellent host for substitutionally accepting both trivalent rare-earth and transition-metal ions [4]–[10]. Constrained by a high melting temperature [11], at present all YGG crystals are grown in an atmosphere free of oxygen. This condition makes it difficult to obtain high-quality crystals because of change in the valence of Ga and the volatization of Ga_2O_3. The optical floating zone method is a suitable growth method for producing high-temperature crystals since the crystal growth process does not require a crucible. Furthermore, crystals can be grown in an oxygen atmosphere [12]. The method is ideal for the growth of high-quality YGG crystals, because the change in the valence of Ga and the volatization of Ga_2O_3 can be greatly decreased in an oxygen atmosphere. To date, however, to our knowledge, there have been no YGG crystals grown by this method.

In recent years, with the development of solid-state lasers, ytterbium (Yb) doped crystal lasers have exhibited outstanding properties. This material typically has broad absorption and emission bands and small quantum defects, which make it more suitable for certain laser applications, such as high-power laser systems, tunable lasers, and ultrafast laser pulse generation.

Considering the characteristics of active Yb ions and YGG crystals, it is proposed that Yb-doped YGG crystal will be an excellent laser crystal much like its isomorph Yb:YAG, which has been widely studied and applied in high-power and ultrafast lasers. Unfortunately, in the past, this crystal was investigated mainly as a scintillator [13] and its thermal, optical, and laser properties, which are basic and crucial for a laser material, have not been studied systemically. A Yb:YGG mode-locked laser with a width of 245 fs has been reported recently, proving that this crystal is an excellent ultrafast laser material [14]. In this paper, we report for the first time the successful growth of a Yb:YGG crystal by the optical floating zone method. We have studied its thermal and optical properties, and its continuous-wave (CW) laser performance.

II. Experiments

The Yb:YGG crystal was grown by the optical floating zone method with a four-ellipsoidal-mirror furnace (Crystal Systems Inc., FZ-T-12000-X-I-S-SU). In the furnace, there are four 3-kW Xenon lamps used as the heating source. The maximum temperature can reach as high as 3000 °C. The raw materials used are Yb_2O_3, Ga_2O_3, and Y_2O_3 powders with a purity of 99.99%. They were mixed stoichiometric proportions to the formula $Yb_{0.3}Y_{2.7}Ga_5O_{12}$, and then pressed into a rod with the dimensions of 6 mm diameter and 60 mm length. The rod was sintered at 1500 °C for 4 h in air to obtain a polycrystalline material. A Nd-doped YAG crystal rod cut along the ⟨111⟩

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direction with a diameter of 8 mm was used as the seed. The crystal was grown at a rate in the range of 5–8 mm/h in an oxygen atmosphere with purity up to 99.9%. The feed and seed rods were rotated at 30 rpm in opposite directions. In order to relieve the thermal stress inside the crystal, the as-grown crystal boule was annealed in air at a temperature of 1000 °C for 10 h.

After annealing, the phase and structure of the Yb:YGG crystal were investigated by using X-ray power diffraction (XRPD) with a Seifert X-ray powder diffractometer at room temperature (T_0). Ground Yb:YGG crystalline powder was used as the sample. Using a Mettler Toledo DSC822^e differential scanning calorimeter, the specific heat was studied with a sample mass of about 100 mg. The density of Yb:YGG at room temperature was measured by the buoyancy method and also calculated from the structure and lattice parameter obtained by XRPD. By using a Yb:YGG sample with dimensions of 6 mm × 6 mm × 2.41 mm and a Perkin–Elmer thermal-mechanical analyzer, the average linear thermal expansion of Yb:YGG was measured over the temperature range from 28 °C to about 490 °C. A sample for measuring the thermal diffusion coefficient was cut along the ⟨111⟩ direction with dimensions of 6 mm × 6 mm × 1.2 mm and coated with graphite on the faces. This coefficient was measured with a Nanoflash model LFA447 using the laser pulse method over the temperature range from 29 °C to 290 °C. The fluorescence lifetime was measured with a laser-diode (LD) as the pump source. A crystal sample with dimensions of 3 mm × 3 mm × 3 mm was measured, and the two opposite faces along the ⟨111⟩ direction were polished. With the same sample, the absorption spectrum of Yb:YGG was measured at room temperature with a V-570 JASCO UV–vis–NIR spectrophotometer.

A CW Yb:YGG laser was constructed with a plano-concave resonator. A fiber-coupled LD with a center wavelength of about 932 nm was used as the pump source. The output beam was focused onto the laser crystal with a spot radius of about 400 μm. A numerical aperture of 0.22 was set using focusing optics. A concave mirror M1 with a curvature of 100 mm, which acts as a total reflector, was antireflection (AR) coated at 932 nm on the pump face as well as high reflection at 1020–1200 nm and high-transmission coated at 932 nm on the other face. A plane mirror was used as the output coupler (OC) with an output transmission of 3% at 1030 nm. The distance between the two mirrors M1 and OC is about 15 mm. The dimensions of the crystal are 3 mm × 3 mm × 3 mm along the ⟨111⟩ direction. The two opposite faces of the crystal were polished and AR coated at 1020–1200 nm. To remove the heat generated in the crystal in the laser experiments, it was wrapped with indium foil and held in a water-cooled copper block at a temperature of 15 °C.

III. Results and Discussion

A Yb:YGG crystal was successfully grown using the optical floating zone method. The as-grown crystal is shown in Fig. 1. From this illustration, it can be seen that the crystal has no inclusions and is colorless. No light scattering was observed using a He–Ne laser. All the observations indicate that the as-grown crystal is of excellent quality and can be used for laser experiments. The length of the as-grown crystal is about 35 mm with a diameter of about 8.5 mm. By X-ray fluorescence analysis, the segregation coefficient of Yb ions in YGG was determined to be 0.98.

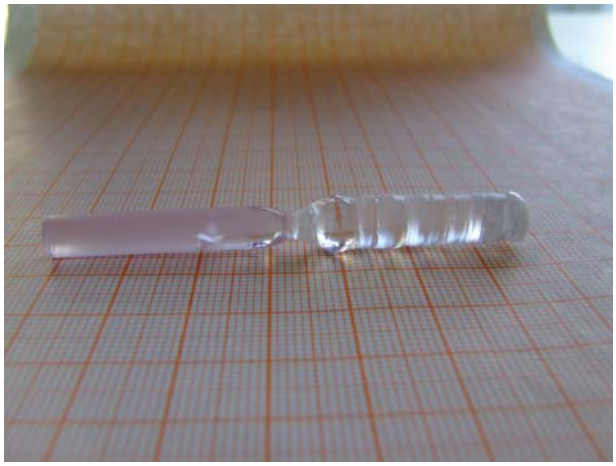

Fig. 1. As-grown crystal Yb:YGGboule.

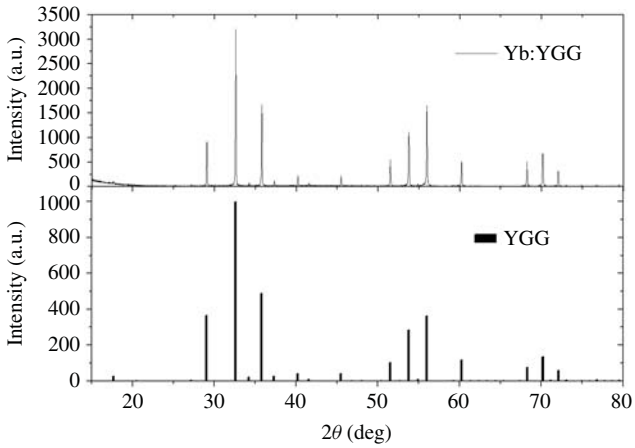

Fig. 2. XRPD patterns of Yb:YGG and YGG.

Structure is the primary characteristic of a crystal and determines most of its physical properties. By using ground Yb:YGG crystalline powder, the XRPD pattern was recorded from 15° to 80°. The results are shown in Fig. 2. The YGG pattern from the JCPDS files [15] is also shown for comparison. The figure shows that the Yb:YGG crystal has a pattern very similar to that of YGG. From the Yb:YGG pattern, we can confirm that the crystal has a garnet structure with the cubic symmetry space group $Ia3d$. The cell parameter a is calculated to be 12.29 Å, which is similar to that of pure YGG (12.27 Å) [15].

The specific heat (C_p) is one of the important factors that greatly influence the optical damage threshold in a laser crystal. Generally, the larger the specific heat of a crystal, the smaller the temperature change when it absorbs a fixed amount of pump power, resulting in a higher damage threshold. The specific heat of Yb:YGG at different temperatures is presented in Fig. 3. It can be seen that the specific heat increases with temperature and its behavior is the same as most crystalline

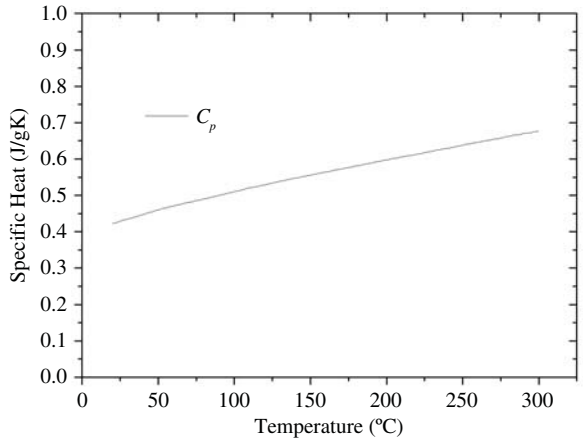

Fig. 3. Dependence of specific heat of Yb:YGG on temperature.

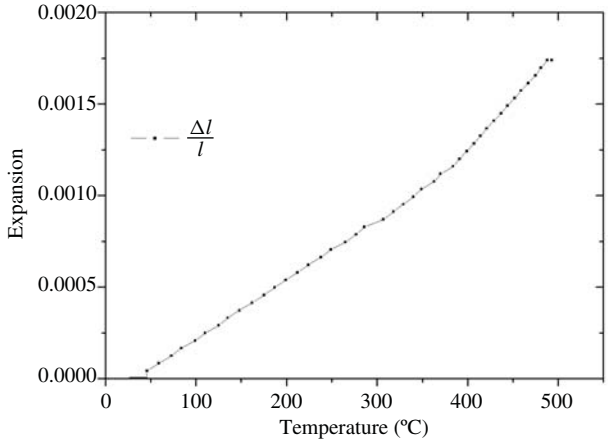

Fig. 4. Dependence of thermal expansion of Yb:YGG on temperature.

materials [16]–[19]. At room temperature, this value is measured to be 0.43 J/gK, which is slightly smaller than that of Yb:YAG with the same Yb-doping concentration [16], but larger than that of a pure GGG crystal [19], both of which have been applied to construct high-power laser systems. Therefore, it is assumed that Yb:YGG will have a high optical damage threshold as in the case of Yb:YAG and GGG.

The thermal expansion coefficient is a determining factor used to calculate the thermal conductivity at different temperatures and the absorbed pump power at the thermal fracture limit. As is well known, the thermal expansion coefficient $[\alpha_{ij}]$ of a crystal is a symmetrical second-rank tensor. From the XRPD pattern, it has been determined that Yb:YGG crystal has cubic symmetry. Based on Neumann's principle, there is only one independent principal component that can be obtained by measuring the sample cut along an arbitrary direction. The thermal expansion measured at different temperatures is shown in Fig. 4, from which it can be determined that the thermal expansion coefficient is $3.3 \times 10^{-6}\ \mathrm{K}^{-1}$ when the temperature is below 370 °C and the value became $5.3 \times 10^{-6}\ \mathrm{K}^{-1}$ in the temperature range 370–493 °C. These values are smaller than that of Nd:YAG ($7.5 \times 10^{-6}\ \mathrm{K}^{-1}$) [20] and Yb:YAG with the same Yb-doping concentration ($8.18 \times 10^{-6}\ \mathrm{K}^{-1}$) [16], which means that Yb:YGG is not especially sensitive to a rise in temperature.

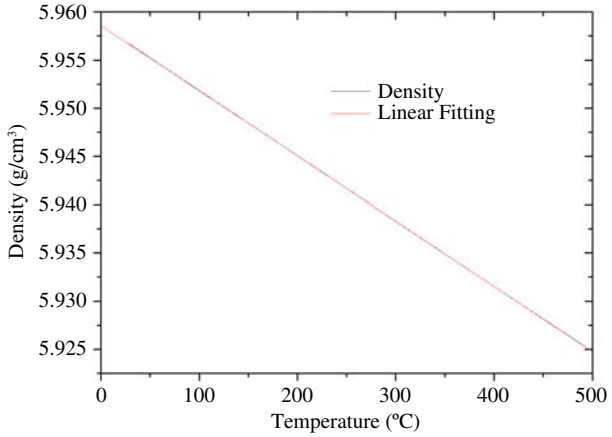

Fig. 5. Dependence of the density of Yb:YGG on temperature.

The density of the Yb:YGG was measured at room temperature by the buoyancy method with a water of density 0.9962 g cm^{-3}. The density of Yb:YGG was measured to be 5.965 g cm^{-3}. From the structure and cell parameter obtained by XRPD, the density can also be calculated using the following equation:

$$\rho_{theory} = \frac{mz}{VN_A} \tag{1}$$

where m is the atomic weight, $Z = 8$ [15] is the number of molecules in a unit cell, N_A is Avogadro's constant, and $V = a^3$ is the cell volume as obtained from the cell parameter. The calculated density is 5.957 g cm^{-3}, which agrees well with the experimental value. Due to thermal expansion, the density decreases with increasing temperature. The density (ρ_T) at temperature T is given by

$$\begin{aligned}\rho_T &= \frac{mz}{VN_A} \\ &= \frac{mz}{(a+\Delta a)^3 N_A} \\ &= \frac{mz}{(a + a \times \alpha \times \Delta T)^3 N_A} \\ &= \frac{\rho a^3}{(a + a \times \alpha \times \Delta T)^3}\end{aligned} \tag{2}$$

with $\Delta T = T - T_0$. The change in ρ_T with temperature is shown in Fig. 5. It can be seen that the density decreases with increasing temperature according to the following equation:

$$\rho_T = 5.9586 - 6.77 \times 10^{-5} \times T. \tag{3}$$

The thermal diffusivity determines the heat-transmission time in a material and is also one of the determining factors used to calculate thermal conductivity. As with the thermal expansion coefficient, the thermal diffusion coefficient $[d_{ij}]$ of a crystal is also a symmetric second-rank tensor, and there is only one independent principal component. By using the laser pulse method on a Yb:YGG sample cut along the $\langle 111 \rangle$ direction, the thermal diffusion coefficient was measured at different temperatures and is presented in Fig. 6. It can be seen that the coefficient decreases with increasing temperature. At room temperature, $d = 1.33\ \mathrm{mm}^2\ \mathrm{s}^{-1}$, which is slightly

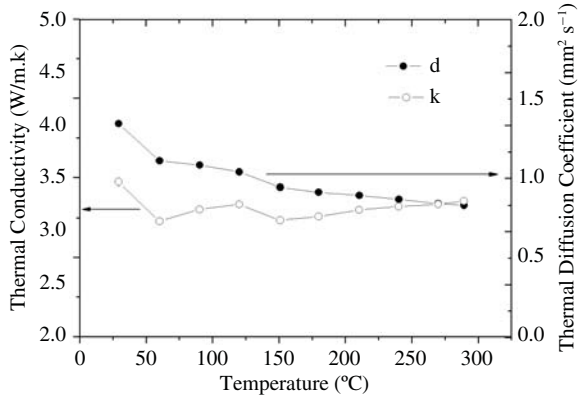

Fig. 6. Dependence of thermal diffusion coefficient and conductivity of Yb:YGG on temperature.

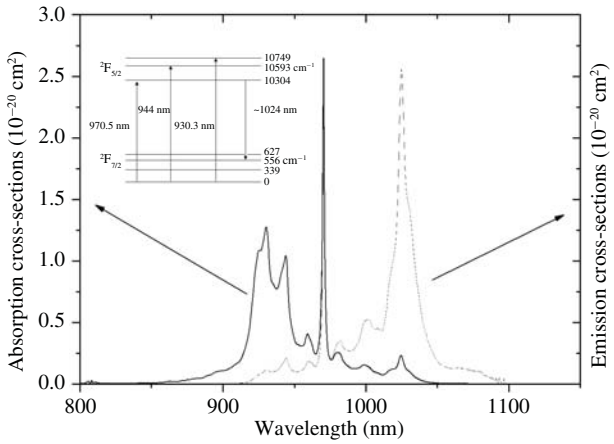

Fig. 7. Absorption and emission cross-sections of Yb:YGG. (Inset) The energy level diagram of Yb:YGG.

smaller than that of Yb:YAG (1.62 $mm^2\ s^{-1}$) with the same Yb doping concentration [16].

Based on the measured specific heat, density, and thermal diffusion coefficient, the thermal conductivity [k_{ij}] of Yb:YGG at different temperatures can be calculated using the following equation:

$$k = \rho_T c_p d. \tag{4}$$

The calculated thermal conductivity at room temperature is 3.47 W m^{-1} K^{-1}, which is smaller than that of Yb:YAG with the same Yb doping concentration (over 4 W m^{-1} K^{-1}) [16]. Compared with Yb:YAG, which has been used in high-power laser systems [21], it can be assumed that Yb:YGG will also be an excellent crystal for application at moderate and even high power levels. The change in thermal conductivity with increase in temperature is also shown in Fig. 6. It is found that, due to the small thermal expansion coefficient, the small decrease in the thermal diffusivity, and the large increase in the specific heat with increasing temperature, the variation in the thermal conductivity is only 10%, and the thermal conductivity slightly increases when temperature is raised above 150 ºC. The behavior of the thermal conductivity for Yb:YGG is different from that of other garnet crystals, such as Yb:YAG, Nd:YAG, and GGG. In the latter cases, the thermal conductivity decreases with increasing temperature. Based on the analysis of glasses [22], whose thermal conductivity increases with increasing temperature, it is proposed that, due to the random substitution of Yb, Y, and Ga ions on the octahedral sites of the lattice [4], Yb:YGG processes a disordered structure and produces the atypical behavior of thermal conductivity.

The fluorescence lifetime of the crystal was determined to be 1.78 ms, which is larger than that of Yb:YAG (1.01 ms) and Yb:GGG (0.8 ms) [23]. The absorption spectrum of a laser medium is determined by the eigenvalue multiplets of the active ions and is partly influenced by the host material. It is important for finalizing the design of laser systems. The room-temperature non-polarized absorption spectrum of Yb:YGG is shown in Fig. 7. The strongest absorption occurs at 970 nm, which is different from that of Yb:YAG and Yb:GGG [23]–[26]. The absorption cross section σ_{abs} at 970 nm is 2.7×10^{-20} cm^2, which is over three and four times than that for Yb:YAG and Yb:GGG [23], respectively. Based on the analysis in [27] and the measured absorption spectrum, we present the energy level diagram of Yb:YGG in the inset of Fig. 7.

Using the reciprocity method, the emission cross section $\sigma_{em}(\lambda)$ can be calculated from the absorption cross section at different wavelengths λ by applying the following formula [28]:

$$\sigma_{em}(\lambda) = \sigma_{abs}(\lambda)\frac{Z_l}{Z_u}\exp\left(\frac{h\frac{c}{\lambda_{zl}} - h\frac{c}{\lambda}}{kT}\right) \tag{5}$$

where $\frac{Z_l}{Z_u} = 0.8$ [28] is the ratio of the partition functions for the lower (Z_l) and upper levels (Z_u), h is Planck constant, c is the velocity of light, $\lambda_{Zl} = 970$ nm is the zero line, and k is the Boltzmann constant. The calculated emission spectrum is also presented in Fig. 7. It is observed that the largest emission cross section is located at 1024 nm with $\sigma_{em} = 2.56 \times 10^{-20}$ cm^2, which is larger than that of Yb:YAG or Yb:GGG [23]. The full-width at half-maximum (FWHM) was calculated to be 11 nm, which is wider than that of Yb:YAG or Yb:GGG (about 10 nm) [26], [28], [29]. The large absorption and emission cross sections as well as the wide emission spectrum indicate that the Yb:YGG laser should be more efficient than lasers constructed with Yb:YAG and Yb:GGG.

Due to the presence of ground state resonance absorption at the laser wavelength for Yb lasers, the effective gain cross sections $\sigma_g(\lambda)$ is given by [30]

$$\sigma_g(\lambda) = [\beta\sigma_{em}(\lambda) - (1-\beta)\sigma_{abs}(\lambda)] \tag{6}$$

where β is the fraction of Yb ions that are excited to the upper level eigenvalue manifold. When $\sigma_g(\lambda) = 0$, we can obtain the minimum value of $\beta(\beta_{min})$, which denotes the minimum fraction of Yb ions that must be excited such that the ground-state absorption and the gain exactly balance each other. For the wavelength at 1024 nm, $\beta_{min,1024\,\mathrm{nm}}$ can be expressed by [28]

$$\beta_{\min,1024\,\mathrm{nm}} = \frac{\sigma_{abs}(1024\,\mathrm{nm})}{\sigma_{abs}(1024\,\mathrm{nm}) + \sigma_{em}(1024\,\mathrm{nm})}. \tag{7}$$

From (7) and Fig. 5, $\beta_{min,1024\ \mathrm{nm}} = 0.083$, which is seen to be larger than the value for Yb:YAG (0.055) [28], which

TABLE I
COMPARISONS OF Yb:YGG, Yb:YAG, AND Yb:GGG (OR GGG)

Crystals	Yb:YGG (9.8 at%)	Yb:YAG (10 at%)	Yb:GGG
Symmetry	Cubic	Cubic	Cubic
Thermal expansion coefficient (10^{-6} K^{-1})	3.8 or 5.3	8.18 [a]	~7 (Pure) [b]
Specific Heat (J gK^{-1})	0.43	~0.63 [a]	~0.37 (Pure) [b]
Thermal diffusion coefficient (mm^2 s^{-1})	1.33	1.62 [a]	~3 (Pure) [b]
Thermal Conductivity (W m^{-1} K^{-1})	3.47	~4.8 [a]	7 (8 at.%) [c]
Absorption Cross-sections (10^{-20} cm^2)	2.7(970 nm)	0.77 (968 nm) [c]	0.66 (971 nm) [c]
Emission cross sections (10^{-20} cm^2)	2.56 (1024 nm)	2.03 (1031 nm) [c]	2 (1025 nm) [c]
Fluorescence lifetime (ms)	1.78	1.01 [c, d]	0.8 [c]
FWHM at emission peak (nm)	11	10 [d]	~10 [e]
Positive band at effective gain cross-sections ($\beta = 0.5$)	120 nm	~100 nm [f]	~80 (nm) [e]

[a] From [16].
[b] From [19].
[c] From [23].
[d] From [28], [29].
[e] From [26].
[f] From [31].

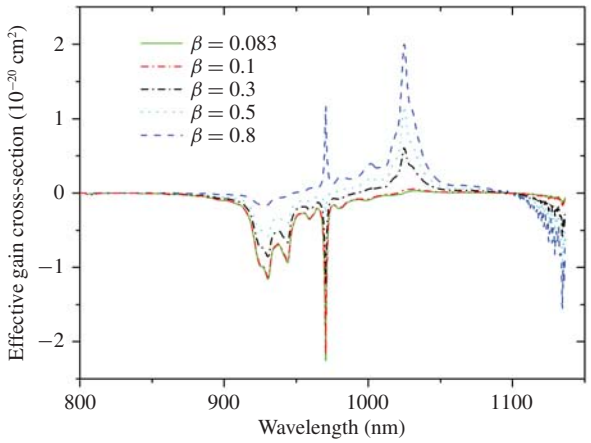

Fig. 8. Effective gain cross sections of Yb:YGG.

indicates that the maximum extractable fraction of the stored energy from Yb:YGG is smaller than that of Yb:YAG. For $\beta = 0.083$, 0.1, 0.3, 0.5, and 0.8, the effective gain cross section $\sigma_g(\lambda)$ was calculated and is shown in Fig. 8. The figure shows that the positive range is comparable with that of Yb:CaGB, Yb:BOYS, and Yb:CYB, all of which have been proven to be excellent crystals for constructing tunable and ultrafast lasers [31], [32]. The minimum pump intensity required to achieve transparency at the extraction wavelength can be calculated by [28]

$$I_{\min} = \beta_{\min,1024} I_{sat} \tag{8}$$

where I_{sat} is the pump saturation intensity parameter. The value is 0.38 kW cm^{-2}, which is much smaller than that of Yb:YAG (1.53 kW cm^{-2}) and comparable with those of fluorides [28].

Using a 932-nm LD as the pump source, CW laser operation was achieved in Yb:YGG with the configuration shown in Fig. 9. The change in output power with the absorbed pump power is shown in Fig. 10. The output power scales linearly with the increase in absorbed pump power, and the threshold is measured to be 5.7 W. The maximum output power is 1.37 W under a pump power of 14.79 W. The optical conversion and

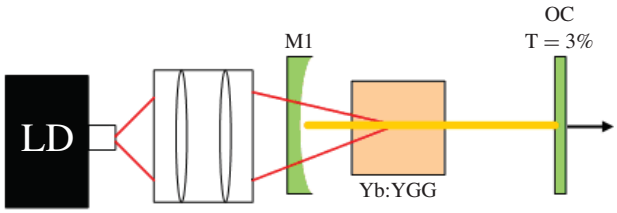

Fig. 9. Configuration of the Yb:YGG laser.

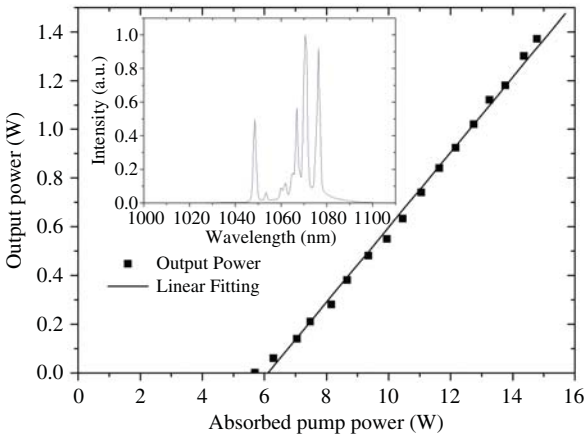

Fig. 10. Output power of Yb:YGG vs. absorbed pump power. (Inset) Laser spectrum of Yb:YGG.

slope efficiencies are 9.3 and 15.4%, respectively. The laser spectrum is also presented in the inset of Fig. 10. Based on the analysis of the cavity, it can be assumed that, with an optimized configuration, such as mirrors with an optimized curvature combination to realize mode-matching and a suitable OC with optimized transmission at the laser wavelength, a much better Yb:YGG laser with a much lower threshold, higher output power, and higher efficiency can be constructed.

IV. CONCLUSION

In conclusion, we have reported the growth of a Yb:YGG crystal using the optical floating zone method with a Yb doping concentration of 10 at.%. The structural, thermal,

optical, and laser characteristics were investigated. It has been found that the crystal possesses cubic symmetry with a cell parameter of 12.29 Å. It has comparable and even better thermal properties than Yb-doped YAG and GGG, a small change in thermal conductivity with changing temperature, large absorption and emission cross sections, and a wide emission spectrum. Detailed comparisons of Yb:YGG, Yb:YAG, and Yb:GGG (or GGG) are shown in Table I. All the measured and calculated parameters indicate that this crystal will have promising application at moderate and even high power levels, and in tunable and ultrafast, especially femtosecond, pulsed laser systems. With an LD as the pump source, a CW output power of 1.37 W was achieved. We feel that with an optimized configuration, the laser performance can be improved further.

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