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Citation: Chin. Phys. B . 2018, 27(9): 097801. **doi:** 10.1088/1674-1056/27/9/097801

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Chin. Phys. B . 2018, 27(1): 017802. **doi:** 10.1088/1674-1056/27/1/017802

Crystal growth and spectral properties of Tb:Lu_2O_3*

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(Received 15 May 2018; revised manuscript received 25 June 2018; published online 28 August 2018)

The crystal growth, x-ray diffraction pattern, absorption spectrum, emission spectrum, and fluorescence lifetime of a Tb:Lu_2O_3 single crystal were studied. Excited at 483 nm, the peak absorption cross-section was calculated to be 3.5×10^{-22} cm^2, and the full width at half maximum was found to be 2.85 nm. The Judd–Ofelt (J–O) intensity parameters Ω_2, Ω_4, and Ω_6 were computed to be 3.79×10^{-20} cm^2, 1.30×10^{-20} cm^2, and 1.08×10^{-20} cm^2, with a spectroscopic quality factor Ω_4/Ω_6 being 1.20. The emission cross-sections of green emission around 543 nm and yellow emission around 584 nm were calculated to be 9.43×10^{-22} cm^2 and 1.32×10^{-22} cm^2, respectively. The fluorescence lifetime τ_{exp} of $^5D_4$ was fitted to be 1.13 ms. The data suggest that the Tb:Lu_2O_3 crystal could be a potential candidate for green and yellow laser operation.

Keywords: fluorescence spectra, laser materials, excited states

PACS: 78.55.–m, 42.70.Hj, 78.47.da **DOI:** 10.1088/1674-1056/27/9/097801

1. Introduction

Visible lasers are very attractive for their widespread applications, such as optical storage devices, display technology, medicine, materials processing, and more.[1,2] Recently, visible lasers have aroused much attention due to the invention of the InGaN laser diode,[3] because the emission wavelength of InGaN laser diode matches well with the absorption wavelength of most rare-earth ions, which is favorable for laser operation.

Pr^{3+} is a famous rare earth ion in the visible laser operation. Efficient laser operations in deep red, red, orange, green, and blue regions have been demonstrated for Pr^{3+}-doped laser materials.[4] Unfortunately, for Pr^{3+}, there is no yellow emission transition. The electronic configuration of trivalent terbium ions is [Xe]4f^8, with a large energy gap ($\sim$ 15000 cm^{-1}) between the metastable $^5D_4$ excited state and the lower ^7F_J ($J = 6, 5, \ldots, 0$) multiplets,[5], which determines the presence of multiple visible emissions in red, yellow, green, and blue spectral ranges, high luminescence quantum efficiency, and a long lifetime of the emission state (ranging from a few hundred microseconds to a few milliseconds).[6] Particularly, the yellow emission corresponding to the $^5D_4 \rightarrow {}^7F_4$ transition makes Tb^{3+} ions a good candidate for yellow laser operation, which is not included by Pr^{3+} ions. Various materials doped with Tb^{3+} ions have been researched for fluorescence imaging and green phosphors.[7–9] In 1973, the stimulated emission at 544.5 nm was first demonstrated in Tb:$LiYF_4$ crystals.[10] Recently, pumped by the InGaN laser diode, high-efficiency room temperature continuous wave lasers emitting green ($^5D_4 \rightarrow {}^7F_5$) and yellow ($^5D_4 \rightarrow {}^7F_4$) light from Tb^{3+}-doped fluoride crystals have also been achieved, with the maximum slope efficiencies being 58% around 545 nm in the green region and 20% around 585 nm in the yellow region.[6,11]

The laser performance of rare-earth ions doped fluoride crystals strongly depends on the temperature. Thus it is necessary to find a new single crystal host, which not only owns the low phonon energy similar with fluoride crystals, but also has better thermal and mechanical properties than fluoride crystals. The sesquioxide Lu_2O_3 belonging to the bixbyite structure with the cubic space group $Ia3$[12] meets these requirements well. They possess higher thermal con-

*Project supported by the National Natural Science Foundation of China (Grant No. 61621001), the National Key Research and Development Program of China (Grant Nos. 2016YFB1102202 and 2016YFB0701002), and the Fundamental Research Funds for the Central Universities, China. We acknowledge the help of MOE Key Laboratory of Advanced Micro-Structured Materials and School of Physical Science and Technology, Guangxi University.

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ductivity ($\sim$ 12.5 W/m·K) than that of fluoride materials (e.g., CaF_2 $\sim$ 7.0 W/m·K).[13,14] Compared with pure lutecia, the thermal conductivity of 3 at.% Yb^{3+}-doped lutecia only decreases slightly to 11.0 W/m·K.[15] In addition, the phonon energy of Lu_2O_3 ($\sim$ 430 cm^{-1}) is lower than that of other oxides,[15] which means that the rare earth ions doped into Lu_2O_3 own lower non-radiative transition rates between the metastable electron levels, resulting in higher radiation probability and quantum efficiency. Up to now, the optical characteristics of Tm^{3+}, Er^{3+}, and Yb^{3+} doped Lu_2O_3 crystals[16,17] have been reported. However, very few researches were focused on the Lu_2O_3 crystals doped with Tb^{3+} ions.

In this work, we study the x-ray diffraction (XRD) pattern, absorption spectrum, Judd–Ofelt (J–O) theory, emission spectrum, and fluorescence lifetime of a Tb:Lu_2O_3 crystal grown by floating zone (Fz) method in detail.

2. Experiments

2.1. Crystal growth

The Lu_2O_3 doped with trivalent terbium ions was grown by the Fz method. The Lu_2O_3 (4N purity) and Tb_4O_7 (4N purity) powders were used as raw materials and weighed precisely according to the equation $(Tb_xLu_{1-x})_2O_3$ ($x = 0.01$). After mixed evenly in an agate mortar, the raw powders were pressed into two rods and then sintered in the air at 1780 °C for 24 h. The rotation rate and growth rate were selected to be 8–10 rpm and 1–2 mm/h, respectively. High-purity argon gas was aerated into the floating zone furnace as a protective atmosphere. The grown Tb:Lu_2O_3 was slowly cooled to room temperature. There was an internal stress in the crystal due to the large temperature gradient in the growth process of the Fz method. Therefore, the grown Tb:Lu_2O_3 crystal was annealed in air at 1700 °C for 24 h to eliminate the internal stress. Then the obtained Tb:Lu_2O_3 crystal was cut into 2.5 mm×4 mm×1 mm and polished for spectral measurements.

2.2. Spectral measurements

The following measurements were performed at room temperature. The sample of Tb:Lu_2O_3 was ground into powders for XRD measurement using an Ultima IV diffractometer from Japan. As shown in Fig. 1, the diffraction peaks of the Tb:Lu_2O_3 crystal are in very good accordance with the JCPDS card of pure Lu_2O_3 crystal. The lattice parameter of the Tb:Lu_2O_3 crystal was calculated to be 1.0424 nm, which is close to that of pure Lu_2O_3 (1.0391 nm), indicating that the doping of the Tb^{3+} ions into Lu_2O_3 did not affect the phase and crystal structure obviously. The concentration of terbium in Tb:Lu_2O_3 reached 1 at.% (i.e., 2.85×10^{20} cm^{-3}), which was measured by an ICP-AES (Ultima2, Jobin–Yvon). The absorption spectrum of Tb:Lu_2O_3 from 300 nm to 2400 nm with the resolution of 1 nm was measured by a spectrometer (Lambda900). The emission spectrum together with the decay curve of Tb:Lu_2O_3 was obtained by a fluorospectrophotometer (FSP920).

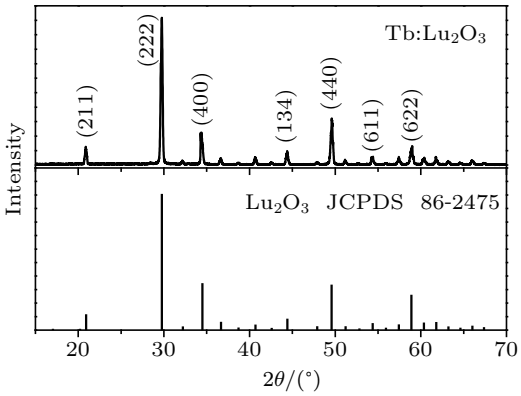


Fig. 1. Powder XRD pattern of Tb:Lu_2O_3 crystal.

3. Results and discussion

3.1. Absorption spectrum

The absorption spectra of the Tb:Lu_2O_3 crystal in the ranges of 320–495 nm and 1600–2400 nm are presented in Fig. 2. The absorption peaks centered around 328 nm, 331 nm, 339 nm, 342 nm, 344 nm, 349 nm, 353 nm, 355 nm, and 483 nm, corresponding to the transitions from $^7F_6$ to $^5H_6$,

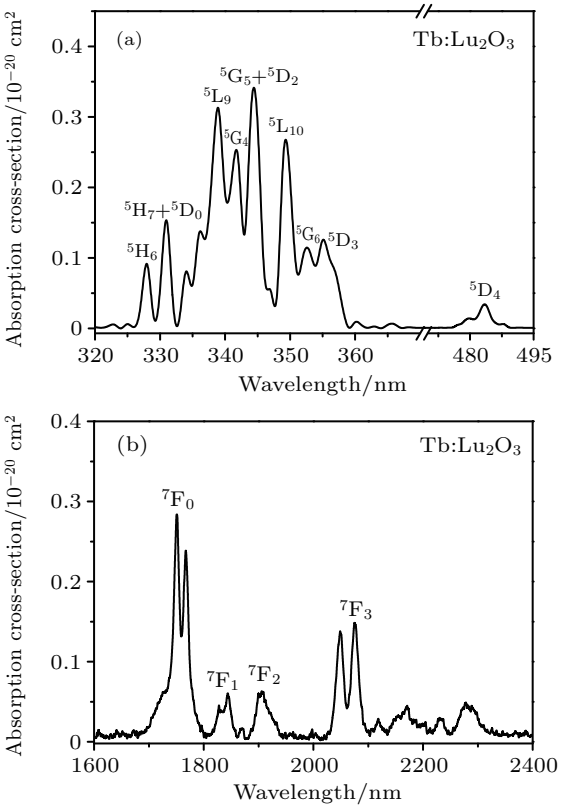


Fig. 2. Room temperature absorption spectrum of Tb:Lu_2O_3 crystal: (a) 320–495 nm, (b) 1600–2400 nm.

$^5H_7$+$^5D_0$, $^5L_9$, $^5G_4$, $^5G_5$+$^5D_2$, $^5L_{10}$, $^5G_6$, $^5D_3$, and $^5D_4$, respectively, are assigned and marked in Fig. 2(a). Figure 2(b) shows four absorption bands at 1752 nm, 1843 nm, 1906 nm, and 2075 nm, corresponding to the transitions from $^7F_6$ to $^7F_0$, $^7F_1$, $^7F_2$, and $^7F_3$, respectively. Some absorption bands overlap with each other due to the interaction of the crystal field. The absorption band around 483 nm corresponding to the $^7F_6 \rightarrow ^5D_4$ transition fits well with the emission wavelength of the InGaN laser diode.[18] At the peak wavelength of 483 nm, its absorption cross-section σ_{abs} and FWHM are 3.5×10^{-22} cm^2 and 2.85 nm, respectively. For the Tb:Lu_2O_3 crystal, its σ_{abs} is larger than that of Tb:$LiLuF_4$ crystal (2.0×10^{-22} cm^2 at 488.8 nm for π-polarization)[11] and the FWHM is larger than that of Tb:KYbW (only 1.0 nm for $E \parallel N_m$).[19]

3.2. Judd–Ofelt analysis

The Judd–Ofelt (J–O) theory is a useful tool to calculate the $4f^N$ radiative transition intensities of lanthanide ions in various host materials. The detailed calculation process is the same with the literature.[19–21] The refractive index formula of Lu_2O_3 and the transition matrix elements of Tb^{3+} ions required for the calculation are taken from Refs. [5], [19], and [22]. In our J–O theory analysis, six bands corresponding to the $^7F_6 \rightarrow ^5L_{10}$, $^5G_6$+$^5D_3$, $^5D_4$, $^7F_0$+$^7F_1$, $^7F_2$, and $^7F_3$ transitions are chosen to confirm the J–O intensity parameters. The parameters of the average wavelength $\bar{\lambda}$, the refractive index n, the calculated line strengths S_{cal}, and the experimental line strengths S_{exp} are shown in Table 1. The small value of rms ΔS (0.011×10^{-20} cm^2) indicates that the fitting results between $S_{exp}(J,J')$ and $S_{cal}(J,J')$ could be considered as reasonable. The three J–O intensity parameters of Tb:Lu_2O_3 and Tb^{3+}-doped other host materials are presented in Table 2. The parameter Ω_2 depends on the asymmetry and covalence of the lanthanide ions coordination field. The high value of Ω_2 means the increase of the covalent bonding and a higher asymmetry of the lanthanide ion site in host materials.[23] As shown in Table 2, Ω_2 of Tb^{3+}-doped Lu_2O_3 is 3.79, which is larger than that of $NaPO_3$–BaF_2–(GdF_3/TbF_3) and $KYb(WO_4)_2$, but smaller than that of CdF_2, $TbAl_3(BO_3)_4$, $LiYF_4$, and GAGSe, indicating that the covalent bonding and asymmetry of Tb^{3+} in Lu_2O_3 are higher than those of $NaPO_3$–BaF_2–(GdF_3/TbF_3) and $KYb(WO_4)_2$, but lower than those of CdF_2, $TbAl_3(BO_3)_4$, $LiYF_4$, and GAGSe. Furthermore, Ω_2 is relatively sensitive because of the admixing of the configuration, while Ω_4 and Ω_6 are less sensitive to the environment of the crystal field.[23] As a spectroscopic quality factor, Ω_4/Ω_6 is generally used to predict the stimulated emission in laser hosts.[21,24] For Tb:Lu_2O_3, Ω_4/Ω_6 is calculated to be 1.20, which is larger than that of most Tb^{3+}-doped host materials listed in Table 2. This suggests that Tb:Lu_2O_3 is a very promising medium for the visible laser operation.

Table 1. The average wavelength ($\bar{\lambda}$), refractive index (n), experimental line strength $S_{exp}(J,J')$, and calculated line strength $S_{cal}(J,J')$ of Tb:Lu_2O_3 crystal.

Transition (from $^7F_6$)	$\bar{\lambda}$/nm	n	$S_{exp}/10^{-20}$ cm^2	$S_{cal}/10^{-20}$ cm^2
$^5L_{10}$	349	2.010	0.065	0.064
$^5G_6$+$^5D_3$	353	2.007	0.027	0.027
$^5D_4$	483	1.953	0.008	0.006
$^7F_0$+$^7F_1$	1764	1.899	0.573	0.562
$^7F_2$	1908	1.897	0.555	0.570
$^7F_3$	2137	1.895	0.752	0.748
rms $\Delta S/10^{-20}$ cm^2	0.011			

Table 2. Comparison of the J–O intensity parameters and the values of Ω_4/Ω_6 for Tb:Lu_2O_3 crystal and other Tb^{3+}-doped materials.

Hosts	$\Omega_2/10^{-20}$ cm^2	$\Omega_4/10^{-20}$ cm^2	$\Omega_6/10^{-20}$ cm^2	$\chi = \Omega_4/\Omega_6$	Ref.
$KYb(WO_4)_2$	1.91	2.41	4.91	0.49	[19]
CdF_2	7.90	2.99	3.09	0.96	[21]
$TbAl_3(BO_3)_4$	8.15	0.29	2.44	0.12	[26]
$LiYF_4$	28.30	1.65	2.15	0.77	[28]
GAGSe	7.50	4.40	2.40	1.83	[29]
$NaPO_3$–BaF_2–(GdF_3/TbF_3)	2.76	3.21	3.36	0.95	[30]
Lu_2O_3	3.79	1.30	1.08	1.20	this work

For the Lu_2O_3 crystal, the radiative lifetime (τ_{rad}), fluorescence branching ratio (β), and spontaneous radiation transition probability (A) of $^5D_4$ multiplet of Tb^{3+} ions are calculated and shown in Table 3. Here, we mainly pay attention to the $^5D_4 \rightarrow ^7F_5$ transition emitting green light and the $^5D_4 \rightarrow ^7F_4$ transition emitting yellow light. The fluorescence branching ratio of the $^5D_4 \rightarrow ^7F_5$ transition is 65.89%, which is the largest one among the listed transitions in Table 3, indicating the high possibility of a green emission around 543 nm. For the $^5D_4 \rightarrow ^7F_4$ transition at 584 nm, the fluo-

rescence branching ratio is 4.50%. In addition, the radiative lifetime τ_{rad} is 3.02 ms, larger than that of Tb:KYb$(WO_4)_2$ (2.08 ms)[19] and Tb:CdF_2 (2.77 ms),[21] suggesting the higher energy storage capability of the Tb:Lu_2O_3 crystal.

Table 3. The spontaneous radiation transition probability (A), fluorescence branching ratio (β), and radiative lifetime (τ_{rad}) of Tb:Lu_2O_3 crystal.

Transition (from $^5D_4$)	A/s^{-1}	$\beta/\%$
$^7F_6$	53.71	16.22
$^7F_5$	218.20	65.89
$^7F_4$	14.89	4.50
$^7F_3$	23.10	6.98
$^7F_2$	10.35	3.13
$^7F_1$	6.65	2.01
$^7F_0$	4.24	1.28
Radiative lifetime/ms	$\tau_{rad} = 3.02$	

3.3. Fluorescence spectrum

Excited by 483 nm, the room-temperature fluorescence spectrum of the Tb^{3+}-doped Lu_2O_3 crystal is shown in Fig. 3, in which three observed emissions corresponding to the $^5D_4 \rightarrow {}^7F_5$, $^7F_4$, and $^7F_3$ transitions are assigned and marked. The intensities of the three emission bands are in good agreement with the results of J–O analysis presented in Table 3. It can be seen that the $^5D_4 \rightarrow {}^7F_5$ transition at 543 nm corresponding to green emission owns the largest emission cross-section (9.43×10^{-22} cm^2) and the obtained FWHM is 4.15 nm. The emission cross-section is higher than that of Tb:KY_3F_{10} (8.0×10^{-22} cm^2 at 545 nm)[6] and Tb:LaF_3 (7.5×10^{-22} cm^2 at 543 nm).[6] Furthermore, the emission cross-section of the yellow emission band at 584 nm corresponding to the $^5D_4 \rightarrow {}^7F_4$ transition is 1.32×10^{-22} cm^2. The value is higher than that of Tb:PZABP (0.7×10^{-22} cm^2 at 582 nm),[25] Tb:LBTAF (0.58×10^{-22} cm^2 at 585 nm),[23] and Tb:TPP (1.0×10^{-22} cm^2 at 587 nm).[26] The above data indicate that the Tb:Lu_2O_3 crystal is a promising candidate for a visible laser operation.

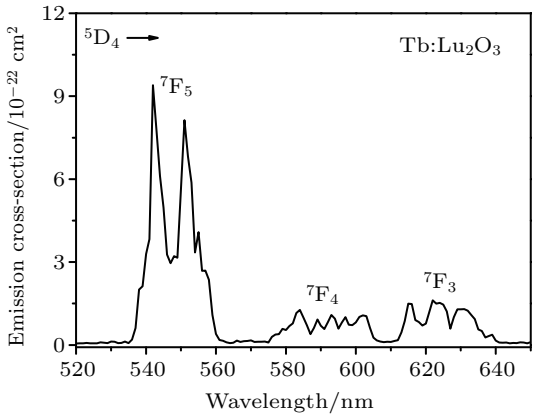


Fig. 3. Fluorescence spectrum of Tb:Lu_2O_3 crystal excited at 483 nm.

Excited at 483 nm, the fluorescence decay curve of $^5D_4$ level measured at room temperature is shown in Fig. 4. Obviously, it is single-exponential. The fluorescence lifetime τ_{exp} of $^5D_4$ level in the Tb:Lu_2O_3 crystal is fitted to be 1.13 ms. With the obtained radiative lifetime τ_{rad} (3.02 ms), the fluorescence quantum efficiency η (τ_{exp}/τ_{rad}) of the Tb:Lu_2O_3 crystal is calculated to be 37.4%, which is comparable to that of Tb:KY_3F_{10}[27] (38%) and higher than that of Tb:KYb$(WO_4)_2$[19] (19%). Furthermore, with the concentration of Tb^{3+} ions decreasing from 2.0 mol% to 0.1 mol%, the quantum efficiency was reported to change from 46% to 93% in Tb:LBTAF.[23] Hence, the quantum efficiency of the Tb:Lu_2O_3 crystal could also be optimized by changing the doped concentration of the Tb^{3+} ions.

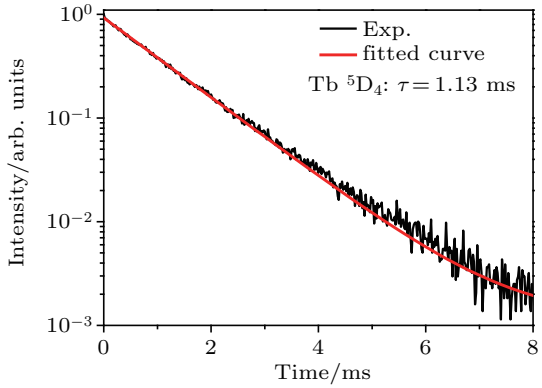


Fig. 4. (color online) Fluorescence decay curve of $^5D_4$ level of Tb:Lu_2O_3 crystal.

4. Conclusion

Tb^{3+}-doped Lu_2O_3 was successfully grown through the Fz method. The J–O theory was adopted to analyze the $4f^N$ radiative transitions of Tb^{3+} ions doped in Lu_2O_3. Around 483 nm, the absorption cross-section was obtained to be 3.5×10^{-22} cm^2 with an FWHM of 2.85 nm. As shown in the fluorescence spectrum, the $^5D_4 \rightarrow {}^7F_5$ transition around 543 nm, corresponding to the green emission, has the largest emission cross-section of 9.43×10^{-22} cm^2 with the FWHM of 4.15 nm and the emission cross-section of the $^5D_4 \rightarrow {}^7F_4$ transition around 584 nm, corresponding to the yellow emission, is 1.32×10^{-22} cm^2. With the fluorescence lifetime τ_{exp} of $^5D_4$ level fitted to be 1.13 ms and the radiative lifetime τ_{rad} of 3.02 ms, the fluorescence quantum efficiency was calculated to be 37.4%. These results reveal that the Tb:Lu_2O_3 crystal would be promising for green and yellow laser operations.

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