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Optical spectroscopy of Ho³⁺, Pr³⁺ co-doped YScO₃ crystal

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Abstract: YScO₃ single crystal co-doped with Ho³⁺ and Pr³⁺ ions has been successfully grown by the Czochralski method. The absorption spectrum, fluorescence spectrum and fluorescence decay curve were measured at room temperature. The absorption cross section of Ho,Pr:YScO₃ at 649 nm and 1130 nm was calculated to be 0.93×10^{-20} cm² and 0.32×10^{-20} cm², respectively. Judd-Ofelt (J-O) theory was performed to evaluate the spontaneous emission probabilities and the radiative lifetimes. Ho,Pr:YScO₃ exhibited strong emission at 2040 nm and 2890 nm with a emission cross section of 0.80×10^{-21} cm² and 1.22×10^{-20} cm², respectively. The fluorescence lifetime of the Ho³⁺:⁵I₆ and ⁵I₇ levels were calculated to be 0.31 ms and 5.89 ms, respectively. All the results indicate that the Ho,Pr:YScO₃ crystal could be a potential laser material for 2.9 μm laser operation.

Keywords: Ho,Pr:YScO₃; 2.9 μm; Spectroscopic property

1. Introduction

Laser sources operating in the 3 μm spectral range have attracted much interest due to their potential applications in biomedical treatments, remote atmosphere sensing and military defense [1-3]. The reason is that this region is overlapped by strong absorption bands of both water and CO_2 [4]. Moreover, laser around 3 μm wavelength can be used as a pumping source for infrared optical parametric oscillators. Among various active ions (Er^{3+} , Dy^{3+} ions, etc.) [5,6], Ho^{3+} ion is a promising candidate for 3 μm laser owing to the ${}^5\text{I}_6 \rightarrow {}^5\text{I}_7$ transition. Unfortunately, the self-terminating transition (${}^5\text{I}_6 \rightarrow {}^5\text{I}_7$) of Ho^{3+} ions, namely, the upper energy level ${}^5\text{I}_6$ has a shorter lifetime than the lower energy level ${}^5\text{I}_7$ [7], extinguished the probability of population inversion for lasing at around the 3 μm wavelength. Co-doping with Pr^{3+} ions is an effective way to depopulate the ${}^5\text{I}_7$ multiplet of Ho^{3+} for population inversion and practical laser operation [8-13]. Up to now, laser operations have been demonstrated in Ho,Pr:LiLuF_4 [14,15], Ho,Pr:LiYF_4 [16,17] and Ho,Pr:CaF_2 [18].

Mixed sesquioxide YScO_3 crystallizes in a cubic C (bixbyite) structure, and its disordered structure comes from the random distribution of Sc^{3+} and Y^{3+} [19,20]. When doped with rare earth ions, it will lead to an inhomogeneous broadening of emission bands which is beneficial for tunable and ultrashort laser operation. Large-size YScO_3 crystals can be grown by the conventional Czochralski method using iridium crucibles due to the melting point below 2150 $^\circ\text{C}$ [21]. In 2021, YScO_3 crystals doped with Yb^{3+} and Er^{3+} have been grown by the Czochralski method and the laser operation of Yb:YScO_3 and Er:YScO_3 was demonstrated [22]. In 2022, Suzuki et al. reported a continuous-wave Tm:YScO_3 laser pumped by 780 nm laser diode, delivering as high as 0.5 W laser output at around 2100 nm with a slope efficiency of 45% [23].

In this work, Ho^{3+} , Pr^{3+} co-doped YScO_3 crystal was grown by the Czochralski method for the first time. Room temperature spectroscopic properties including absorption, emission spectra, Judd–Ofelt analysis and fluorescence lifetime were discussed in detail.

2. Experimental

2.1. Crystal growth

Ho,Pr:YScO_3 crystal was grown by the Czochralski method. Raw materials of Ho_2O_3 , Pr_6O_{11} , Sc_2O_3 , Y_2O_3 , Al_2O_3 with purity of 99.99% were weighed according to the formula $(\text{Ho}_{0.005}\text{Pr}_{0.0005}\text{Y}_{0.4945}\text{Sc}_{0.4945})_2\text{O}_3$ and then mixed together for 6 h. The mixture was cold pressed into the form of the block under 200 MPa, and then placed into an alumina crucible, heated up to 1200 $^\circ\text{C}$ and kept at this temperature for 20 h. The charge was then loaded into an iridium crucible for crystal growth. A YScO_3 crystal was used as the seed. The pulling rate was 1 mm/h, the rotation rate was 10-30 rpm and the growth atmosphere was nitrogen. Finally, the Ho,Pr:YScO_3 crystal was obtained. Fig. 1 shows the polished samples for spectroscopic and laser experiments. The crystals were greenish and no inclusions and bubbles were observed in the samples.

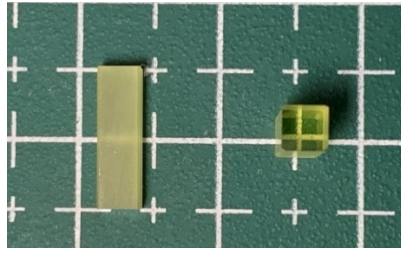


Fig. 1. Photograph of the polished Ho,Pr:YScO₃ crystal.

2.2. Spectral measurements

The Ho,Pr:YScO₃ crystal was cut and polished to the dimensions of $10 \times 10 \times 1$ mm³ for spectral measurements. The absorption spectrum was carried out on the spectrophotometer (Lambda 950, PerkinElmer UV-VIS-NIR) from 350-2200 nm with 1 nm as a footstep. The fluorescence spectrum and fluorescence decay curve were tested by Edinburgh Fluorescence Spectrophotometer (FLS1000) in the range of 1800-3200 nm under the excitation of 640 nm. All the measurements were performed at room temperature.

3. Results and discussion

3.1. Optical absorption spectrum

Fig.2 shows the room temperature absorption spectrum of Ho,Pr:YScO₃ crystal in the wavelength range of 350-2100 nm. Obviously there are six absorption bands centered at around 421 nm, 455 nm, 543 nm, 649 nm, 1130 nm and 1940 nm with the full width at half maximum (FWHM) of 9.9 nm, 3.6 nm, 7.6 nm, 16.7 nm, 16.4 nm and 13.2 nm, respectively. The absorption bands are ascribed to the transitions from the ground state ⁵I₈ to the different excited states: ⁵G₅, ⁵G₆+⁵F₁, ⁵S₂+⁵F₄, ⁵F₅, ⁵I₆, and ⁵I₇. The absorption coefficient and absorption cross section at 649 nm was calculated to be 0.63 cm⁻¹ and 0.93×10^{-20} cm², respectively. The absorption peak at 1145 nm, corresponding to the ⁵I₈ → ⁵F₅ transition of Ho³⁺ ions, matches well with the emitting wavelength of well-developed 1150 nm Raman fiber laser. The absorption coefficient and absorption cross section was 0.20 cm⁻¹ and 0.27×10^{-20} cm², with FWHM of 14.1 nm. The wide absorption band is mainly caused by the multisite characteristic of YScO₃, which ensures the steady absorption of the pumping light. Due to the low doping concentration of Pr³⁺ ions, the absorption band at around 1500 nm corresponding to the ³H₄ → ³F₃+³F₄ transition of Pr³⁺ ions, could not be observed. While, the ³H₄ → ³F₃+³F₄ transition usually has the most intense absorption in Pr³⁺-doped oxide crystals [24].

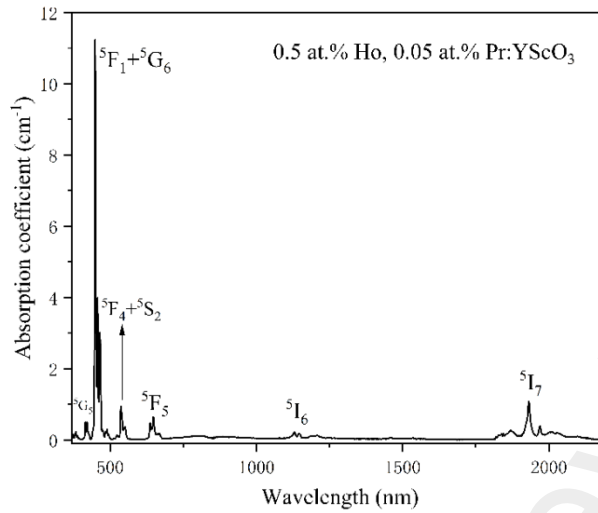


Fig. 2. The absorption spectrum of Ho,Pr:YScO₃ crystal

3.2. J-O analysis

The Judd-Ofelt (JO) theory [25,26] was used to analysis the absorption spectrum of Ho,Pr:YScO₃ crystal, and then the fluorescence branching ratios and radiative lifetimes of the excited multiplets can be evaluated. The reduced matrix elements used for absorption and emission transitions can be consulted in [27] and [28], respectively. The average wavelength of different transition, experimental and calculated line strength, and RMS deviation of Ho³⁺ ion in YScO₃ crystal are listed in Table 1. The value of rmsΔS is 1.638×10⁻²⁰ cm², which indicates a good agreement between the experimental and the calculated line strength.

Table 1. The average wavelength of different transition, experimental and calculated line strength, and RMS deviation of Ho³⁺ in YScO₃ crystal.

| Excited state (from ⁵ I ₈) | $\bar{\lambda}$ (nm) | S _{mea} (10 ⁻²⁰ cm ²) | S _{cal} (10 ⁻²⁰ cm ²) |
|---|----------------------|---|---|
| (⁵ G, ³ G) ₅ | 419 | 1.89 | 3.06 |
| ⁵ G ₆ + ⁵ F ₁ | 456 | 27.87 | 29.01 |
| ⁵ F ₄ + ⁵ S ₂ | 540 | 3.35 | 4.20 |
| ⁵ F ₅ | 649 | 2.90 | 4.16 |
| ⁵ I ₆ | 1107 | 1.22 | 2.43 |
| ⁵ I ₇ | 1956 | 3.55 | 5.76 |
| rmsΔS (10 ⁻²⁰ cm ²) | | 1.638 | |

The values of $\Omega_{2,4,6}$ were calculated to be 15.63×10⁻²⁰ cm², 5.74×10⁻²⁰ cm², 3.02×10⁻²⁰ cm², respectively. Table 2 shows the J-O parameters of different Ho³⁺,Pr³⁺ co-doped crystals. Ω_2 is related to covalency in rare earth doped material. The value Ω_2 of Ho,Pr:YScO₃ is higher than that of Ho,Pr:LiLuF₄, Ho,Pr:LiYF₄, Ho,Pr:Sc₂O₃, Ho,Pr:Lu₂O₃ and Ho,Pr:CaGdAlO₄, indicating that the covalency of Ho,Pr:YScO₃ is larger than that of Ho,Pr:LiLuF₄, Ho,Pr:LiYF₄, Ho,Pr:Sc₂O₃, Ho,Pr:Lu₂O₃ and Ho,Pr:CaGdAlO₄. Ω_4 and Ω_6 are dependent on the host environment, and the ratio of Ω_4/Ω_6 is used to judge the spectroscopic quality. The value Ω_4/Ω_6 of Ho,Pr:YScO₃ is 1.90, suggesting that Ho,Pr:YScO₃ crystal is a promising medium for efficient laser operation around 2.9 μm.

Table 2. The J-O intensity parameters of different Ho³⁺, Pr³⁺ co-doped crystals.

| Crystals | Ω_2 (10 ⁻²⁰ cm ²) | Ω_4 (10 ⁻²⁰ cm ²) | Ω_6 (10 ⁻²⁰ cm ²) | Ω_4/Ω_6 | Ref. |
|--------------------------------|--|--|--|---------------------|-----------|
| LiLuF ₄ | 3.25 | 4.45 | 3.81 | 1.17 | [8] |
| LiYF ₄ | 1.52 | 1.96 | 1.05 | 1.85 | [9] |
| Sc ₂ O ₃ | 6.56 | 1.67 | 0.52 | 3.21 | [10] |
| Lu ₂ O ₃ | 7.10 | 2.67 | 0.33 | 8.09 | [11] |
| CaGdAlO ₄ | 4.82 | 6.75 | 4.91 | 1.13 | [12] |
| YScO ₃ | 15.63 | 5.74 | 3.02 | 1.90 | This work |

Through the calculated intensity parameters, the calculated radiative transition rate, fluorescence branching ratio and radiative lifetimes of Ho³⁺ ions in YScO₃ crystal. were given in Table 3. We can see that the branching ratio of the ⁵I₆ → ⁵I₇ transition is about 13.89%. The radiative lifetime of ⁵I₆ energy level was calculated to be 1.70 ms, which is lower than that of Ho:LaF₃ (18.99 ms [29]), Ho,Pr:Sc₂O₃ (6.20 ms [10]) and Ho,Pr:Lu₂O₃ (7.97 ms [11]).

Table 3. The calculated radiative transition rates, luminescence branching ratios, and radiative lifetimes of Ho³⁺ ions in YScO₃ crystal.

| J→ | J' | λ (nm) | A _{ED} (s ⁻¹) | A _{MD} (s ⁻¹) | A _(J-J') (s ⁻¹) | β (%) | τ (ms) |
|-------------------------------|-----------------------------|-------------------|---------------------------------------|---------------------------------------|---|----------------|----------------|
| ⁵ I ₇ → | ⁵ I ₈ | 2044 | 211.719 | 36.0959 | 247.815 | 100 | 4.03 |
| ⁵ I ₆ → | ⁵ I ₈ | 1205 | 505.092 | 0 | 505.092 | 86.110 | 1.70 |
| | ⁵ I ₇ | 2913 | 59.757 | 21.7487 | 81.506 | 13.890 | |
| ⁵ I ₅ → | ⁵ I ₈ | 908 | 194.185 | 0 | 194.185 | 39.090 | 2.01 |
| | ⁵ I ₇ | 1660 | 267.465 | 0 | 267.465 | 53.840 | |
| | ⁵ I ₆ | 3901 | 24.472 | 10.6363 | 35.108 | 7.070 | |
| ⁵ I ₄ → | ⁵ I ₈ | 752 | 28.804 | 0 | 28.804 | 9.221 | 3.20 |
| | ⁵ I ₇ | 1223 | 141.071 | 0 | 141.071 | 45.165 | |
| | ⁵ I ₆ | 2153 | 115.536 | 0 | 115.536 | 36.989 | |
| | ⁵ I ₅ | 4578 | 21.528 | 5.4059 | 26.934 | 8.623 | |
| ⁵ F ₅ → | ⁵ I ₈ | 648 | 6561.315 | 0 | 6561.315 | 75.347 | 0.11 |
| | ⁵ I ₇ | 947 | 1754.322 | 0 | 1754.322 | 20.145 | |
| | ⁵ I ₆ | 1401 | 368.280 | 0 | 368.280 | 4.229 | |
| | ⁵ I ₅ | 2387 | 23.927 | 0 | 23.927 | 0.274 | |
| | ⁵ I ₄ | 4658 | 0.203 | 0 | 0.203 | 0.002 | |
| ⁵ S ₂ → | ⁵ I ₈ | 537 | 4173.168 | 0 | 4173.168 | 52.037 | 0.12 |
| | ⁵ I ₇ | 727 | 3034.947 | 0 | 3034.947 | 37.844 | |
| | ⁵ I ₆ | 1001 | 548.799 | 0 | 548.799 | 6.843 | |
| | ⁵ I ₅ | 1354 | 131.288 | 0 | 131.288 | 1.637 | |
| | ⁵ I ₄ | 1950 | 129.063 | 0 | 129.063 | 1.609 | |
| | ⁵ F ₅ | 3134 | 2.270 | 0 | 2.270 | 0.028 | |

3.3. Fluorescence spectrum

Under 640 nm excitation, the fluorescence spectrum of Ho,Pr:YScO₃ was recorded. The stimulated emission cross section can be calculated from the measured fluorescence spectrum via the Füchtbauer Ladenburg (F-L) formula:

$$\sigma_{em}(\lambda)_{JJ'} = \frac{\lambda^5 A(J'' \rightarrow J')}{8\pi c n^2} \frac{I(\lambda)}{\int \lambda I(\lambda) d\lambda}$$

Where $A(J'' \rightarrow J')$ stands for the radiative transition rates, $I(\lambda)$ is the emission intensity at the wavelength λ , c is the speed of light, n stands for the refractive index. The spectra of the stimulated emission cross section for the $^5I_7 \rightarrow ^5I_8$ and $^5I_6 \rightarrow ^5I_7$ transitions are presented in Fig. 4. The peak emission cross sections at 2040 nm and 2890 nm were calculated to be $0.80 \times 10^{-20} \text{ cm}^2$ and $1.22 \times 10^{-20} \text{ cm}^2$, with a FWHM of 187.0 nm and 197.4 nm, respectively. The value of emission cross section at 2890 nm is lower than that of Ho,Pr:LiLuF₄ ($1.91 \times 10^{-20} \text{ cm}^2$ at 2944 nm [8]) and Ho,Pr:CaGdAlO₄ ($2.38 \times 10^{-20} \text{ cm}^2$ at 2860 nm [12]), but higher than that of Ho,Pr:LiLiF₄ ($0.68 \times 10^{-20} \text{ cm}^2$ at 2950 nm [9]), Ho,Pr:Sc₂O₃ ($0.54 \times 10^{-20} \text{ cm}^2$ at 2864 nm [10]), Ho,Pr:Lu₂O₃ ($0.42 \times 10^{-20} \text{ cm}^2$ at 2893 nm [11]) and Ho,Pr:CaF₂ ($1.04 \times 10^{-20} \text{ cm}^2$ at 2851 nm [18]). The FWHMs are larger than those of Ho,Pr:Sc₂O₃ (159.0 nm at 2110 nm, 144.6 nm at 2864 nm [10]), which displays the Ho,Pr:YScO₃ crystal may be used as a gain medium for tunable and ultrashort pulse lasers.

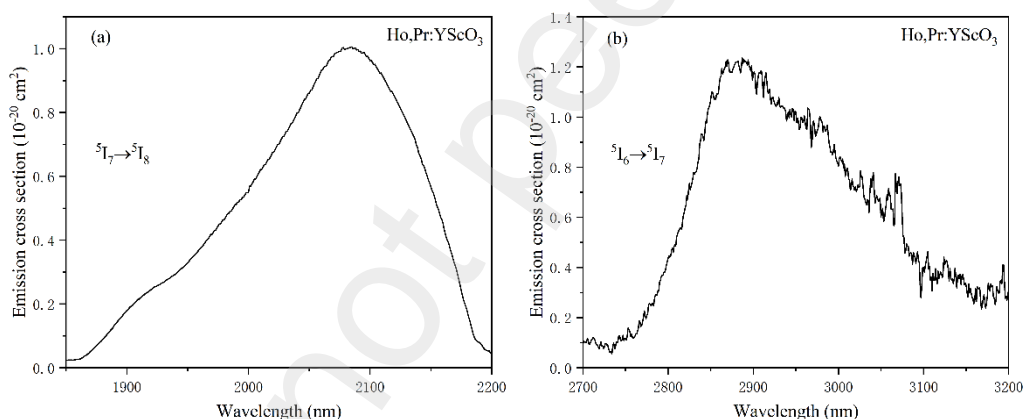


Fig. 4. Emission cross section around (a) 2 μm and (b) 3 μm of the Ho,Pr:YScO₃ crystal.

3.4. Fluorescence lifetime

Fig. 5 depicts the fluorescence decay curves of the 5I_6 and 5I_7 multiplets of Ho,Pr:YScO₃ crystal under excitation at 640 nm. The measured decay curves show a single exponential decaying behavior. The fluorescence lifetime of the 5I_6 and 5I_7 levels was fitted to be 0.31 ms and 5.89 ms, leading to a fluorescence quantum efficiency of 7.6% and 54.2%, respectively. The fluorescence of 5I_6 multiplet is much higher than that of Ho,Pr:CaGdAlO₄ (0.194 ms [12]), but lower than that of Ho,Pr:LiLuF₄ (1.47 ms [8]), Ho,Pr:Sc₂O₃ (1.64 ms [10]), Ho,Pr:Lu₂O₃ (0.55 ms [11]) and Ho,Pr:CaF₂ (2.73 ms [18]). It is noticed that the fluorescence lifetime of the 5I_6 state is much shorter than that of 5I_7 state in Ho,Pr:YScO₃ crystal, which can cause the 2.9 μm laser transition to self-terminate. Unfortunately, we have not obtained the laser output around 2.9 μm from Ho,Pr:YScO₃ crystal. Pr³⁺ ion has been proved to be an effective deactivation ion for 5I_7 level of Ho³⁺ ion, population inversion and practical laser

operation could be realized by optimizing the concentration of Ho^{3+} and Pr^{3+} ions in YScO_3 crystal.

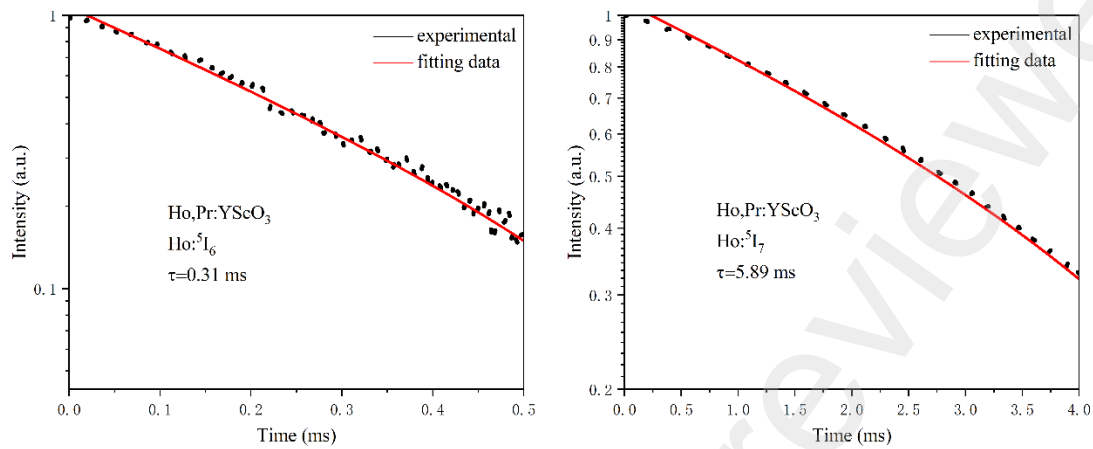


Fig. 5. Fluorescence decay curves of the $^5\text{I}_6$ and $^5\text{I}_7$ multiplets of Ho,Pr:YScO_3 crystal.

4. Conclusion

In summary, Ho,Pr:YScO_3 crystal has been successfully grown by the Czochralski method. Room temperature absorption spectrum, fluorescence spectrum and fluorescence decay curve were measured and analyzed. The peak absorption cross section of Ho,Pr:YScO_3 at 649 nm and 1130 nm was calculated to be $0.93 \times 10^{-20} \text{ cm}^2$ and $0.32 \times 10^{-20} \text{ cm}^2$, with a FWHM of 16.7 nm and 16.4 nm, respectively. The intensity parameters Ω_2 , Ω_4 and Ω_6 were obtained to be $15.63 \times 10^{-20} \text{ cm}^2$, $5.74 \times 10^{-20} \text{ cm}^2$, $3.02 \times 10^{-20} \text{ cm}^2$, respectively. The calculated emission cross sections is $0.80 \times 10^{-20} \text{ cm}^2$ at 2040 nm and $1.22 \times 10^{-20} \text{ cm}^2$ at 2890 nm, with a FWHM of 187.0 nm and 197.4 nm, respectively. The fluorescence lifetimes of the $^5\text{I}_6$ and $^5\text{I}_7$ levels were measured to be 0.31 ms and 5.89 ms. All the results indicate that Ho,Pr:YScO_3 crystal could be a potential laser material for mid-infrared lasers.

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