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Energy-transfer upconversion of rare earth ions in ionic crystals: Case of Tm^{3+}/Ho^{3+}-codoped $LiYF_4$ crystals

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Abstract

Up-conversion spectra of Tm^{3+} and Ho^{3+} ions in $LiYF_4$ crystals have been investigated at various temperatures between 10 and 320 K. The photoluminescence is investigated under excitation with Xe-lamp and laser diode. In addition to blue up-converted emission of Tm^{3+} and Ho^{3+} and green up-converted Ho^{3+} emission, anti-Stokes emission bands are observed at 687 and 703 nm under excitation in the $^3H_4$ state of Tm^{3+} with 785 nm laser diode. These bands are observed above 200 K, and their intensities increase exponentially with increasing temperature. They are attributed to endothermic Tm^{3+} emission due to the transition to the $^3H_6$ ground state from the upper $^3F_3$ state, which is thermally populated from the $^3H_4$ state. Discussion is given on the optical process of green up-converted Ho^{3+} emission, which is generated by the 785 nm laser diode excitation.

Keywords: Luminescence; Rare-earth ions; Upconversion; Tm^{3+}; Ho^{3+}; $LiYF_4$

1. Introduction

Tm^{3+}/Ho^{3+}-codoped crystals have been of interest because the infrared lasers of about 2 μm wavelength are important for medical applications [1–4]. Tm^{3+}-doped crystals show an additional infrared laser oscillation at about 1.47 μm. Optical communication has been currently performed using lasers operating in the C-band (1.53–1.67 μm) region where silica fibers have the ultra low loss. The wavelength region of 1.45–1.50 μm is also a low-loss region. This wavelength region will be important in near future as it will enable us to extend the optical communication bandwidth to S-band (1.48–1.53 μm) region [5,6]. Therefore, Tm^{3+}-doped materials are suggested to be necessary as optical amplification device in the future optical communications.

There are many kinds of host materials such as oxides, chlorides and fluorides for doping rare-earth ions. Of them the oxides have been intensively studied. Unlike the oxides, intense emission is obtained in fluorides because the oxides have much higher phonon energy than the fluorides. The high phonon energy easily gives rise to non-radiative multiphonon transition from the emitting state to the nearest low-energy state, resulting in degradation of photoluminescence. Additionally the fluorides are more transparent in the ultra violet spectral region than the oxides, i.e. not only the blue luminescence but also ultra violet luminescence is observed in fluorides. This indicates that the fluorides are more useful to obtain the blue and ultra violet emissions than the oxides. Such a short-wavelength luminescence is necessary for the optical memory, optical recording and full-color display. Therefore, we investigate the fluoride host material such as $LiYF_4$ (YLF) in the present study.

The Tm^{3+}- and Tm^{3+}/Ho^{3+}-codoped crystals are also of interest because they generate ultraviolet, blue, green and red up-converted emissions by infrared laser pumping. Of various Tm^{3+}-doped crystals, Tm^{3+}-doped and Tm^{3+}/Ho^{3+}-codoped YLF crystals are materials well known for the high efficient up-conversion process, indicated by blue and red

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emissions [7–9]. Laser action in the visible has been obtained under infrared laser diode (LD) excitation at room temperature. Spectroscopic study of the upconversion helps to achieve high efficient laser action. Here we report the optical properties of Tm^{3+}/Ho^{3+}-codoped YLF crystals under excitation with 785 nm LD. Taking into account the results obtained by excitation with 460, 680 and 780 nm light of Xe lamp, we suggest a new optical process for the anti-Stokes 687 and 703 nm emissions generated by 785 nm LD excitation.

There are various up-conversion processes in rare-earth-doped materials. The simple process is the two-, three- or multi-photon absorptions where two, three or *n* photons with the same photon energy $h\nu$ are absorbed by one rare-earth ion simultaneously and excited to the excited state at $2h\nu$, $3h\nu$ or $nh\nu$, respectively, and then luminescence with energy of $2h\nu$, $3h\nu$ or $nh\nu$ is generated by one-photon process. The second type of up-conversion process is via excited state absorption. For example, a Tm ion (Tm1) is excited to the $^3H_4$ state by 785 nm laser, followed the relaxation to the $^3H_5$ state non-radiatively, then the second 785 nm laser light gives rise to the transition from the $^3H_5$ state to the $^1G_4$ state (the excited state absorption). As a result the upconversion occurs by the transition from the $^1G_4$ state to the ground state (as shown in the left-hand side of Fig. 1). Alternatively, the nearby Tm ion (Tm2) is possible to be excited to the $^3H_4$ state, followed the photon emission by the reversed transition. The Tm1 is excited to the $^1G_4$ state by absorbing the emitted photon from Tm2 through the cross relaxation (see the right-hand side of Fig. 1). Such an energy transfer between the two Tm^{3+} ions is possible in heavily Tm^{3+}-doped crystals. In the case of co-doped materials (i.e. materials doped with different rare-earth ions like Tm^{3+} and Ho^{3+}), the cross relaxation process is frequently observed. In this paper we study which kind of up-conversion processes occurs in Tm/Ho-codoped YLF crystals.

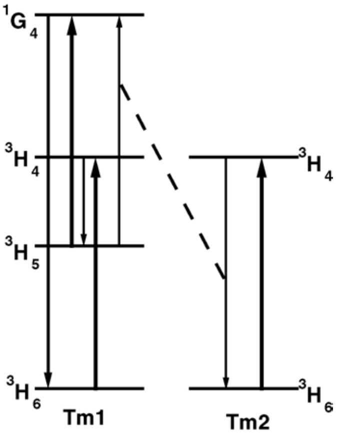

Fig. 1. Two kinds of up-conversion processes by two-photon absorption via (1) the excited state absorption in a Tm^{3+} ion (Tm1) and (2) the cross-relaxation between two Tm^{3+} ions (Tm1 and Tm2) and the excited state absorption in Tm1.

2. Experimental procedure

Single crystals of Ho^{3+}/Tm^{3+}-codoped YLF crystals were grown in a standard RF-heating Czochralski furnace. An iridium crucible was put in the furnace. Two kinds of crystals were studied here; one is a very-lightly Ho^{3+}-doped crystal (called TmYLF, hereafter) with 6% Tm^{3+} and 0.01% Ho^{3+} ions, the other is YLF crystal with 8% Tm^{3+} and 0.5% Ho^{3+} ions (called HoYLF). The concentrations of Tm^{3+} and Ho^{3+} ions are 1.12×10^{21} and $7.00 \times 10^{19}\ cm^{-3}$ in HoYLF, respectively. The emission and excitation spectra were measured with a Spex Fluorolog-3 fluorophotometer in 350–1600 nm spectral region. The excitation was undertaken by 450 W Xe-lamp. The spectral resolution was set to be 0.2 nm. Crystals were also excited with 785 nm LD with maximum power of 42 mW to investigate the upconversion. Absorption spectra of the crystal were measured with a Cary-5E spectrophotometer. Emission and absorption spectra were measured at various temperatures between 10 and 320 K.

3. Experimental results and discussion

Tm^{3+} ions have absorption bands at about 1600–1780, 1120–1230, 770–800, 680–700, 645–670, 450–480, 350–365, 285–290, 280–284, 270–275 and 255–264 nm, which are due to the $4f^{12} \rightarrow 4f^{12}$ electronic transitions from the $^3H_6$ ground state to the $^3F_4$, $^3H_5$, $^3H_4$, $^3F_3$, $^3F_2$, $^1G_4$, $^1D_2$, $^1I_6$, $^3P_0$, $^3P_1$ and $^3P_2$ states of Tm^{3+}, respectively [9–12]. Tm^{3+} gives rise to many emission bands in infrared to ultra violet spectral region. Of them, there is a broad emission band with several components at 1400–1500 nm [13], as observed in various Tm^{3+}-doped materials [5,6]. Fig. 2 shows the excitation spectrum for 1451 nm emission of TmYLF at 285 K, which is measured in 250–850 nm region. Many excitation bands are observed in Fig. 2. All the excitation bands correspond to the absorption bands of Tm^{3+}. The excited states are indicated for each excitation band in Fig. 2.

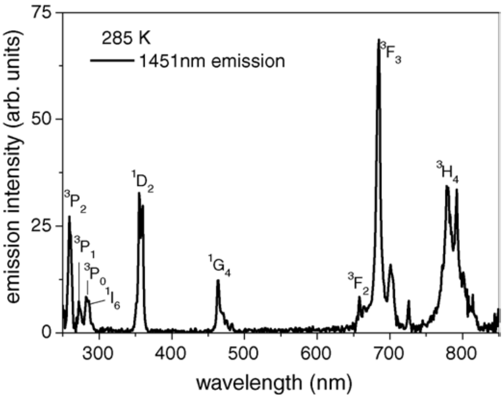

Fig. 2. Excitation spectrum for 1451 nm Tm^{3+} emission in TmYLF crystal at 285 K.

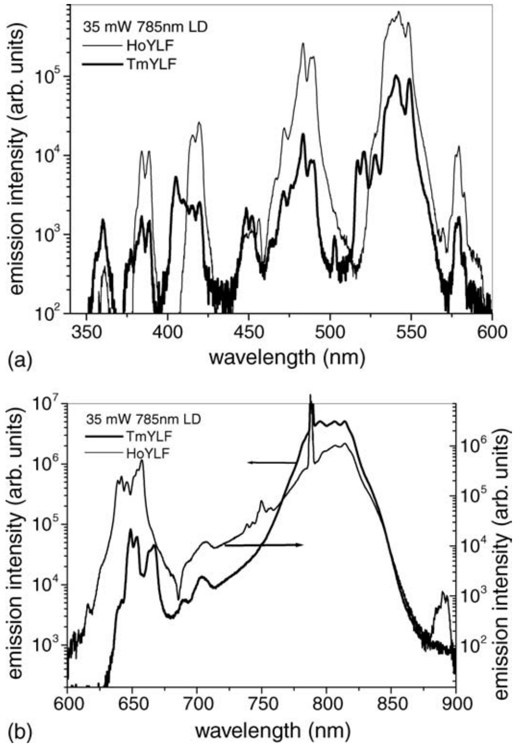

Fig. 3. (a) Up-conversion emission spectra, in 340–600 nm spectral region, of TmYLF and HoYLF crystals excited with 785 nm laser diode with 35 mW at 290 K. (b) Up-conversion emission spectra, in 600–900 nm spectral region, of TmYLF and HoYLF crystals excited with 785 nm laser diode with 35 mW at 290 K. An intense line at 785 nm is due to the excitation laser diode.

Fig. 3(a) and (b) shows the ultraviolet to near-infrared emission spectra of TmYLF and HoYLF crystals excited in the $^3H_4$ state of Tm^{3+} with 785 nm LD at room temperature. No upconversion are observed at 300–350 nm, but many up-converted emission bands are observed at wavelength longer than 350 nm. Fig. 4 shows the enlarged spectrum of Fig. 3(a) in the blue region. Nguyen et al. reported the blue emission bands appearing at 443–458 nm region in Tm^{3+}-doped YLF crystal by two-color excitation with 780.78 and 648.77 nm lights at room temperature [14]. The spectrum is consistent with the spectrum of TmYLF shown in Fig. 4. Comparing the TmYLF spectrum with the HoYLF spectrum, we can attribute the emission bands at 456 nm to Ho^{3+} and the other bands to Tm^{3+} as shown in Fig. 4.

The emission bands with peaks at 360, 449, 477, 666, 687, 703, 806 and 814 nm are due to Tm^{3+} ions, while the emission bands at 383.5, 385, 406, 418, 456, 520, 541, 549, 579, 649.5, and 750 nm are due to Ho^{3+} ions. The 360, 449, 477, 483.5, 666, 806 and 814 nm Tm^{3+} bands are attributed to the transitions $^1D_2 \rightarrow {}^3H_6$, $^1D_2 \rightarrow {}^3F_4$, $^1G_4 \rightarrow {}^3H_6$, $^1G_4 \rightarrow {}^3H_6$, $^3H_4 \rightarrow {}^3H_6$, and $^3H_4 \rightarrow {}^3H_6$, respectively. The 385, 406, 418, 456, 483.5, 520, 541, 549, 579, 649.5, and 750 nm Ho^{3+} bands are attributed to the transitions $^5G_4 \rightarrow {}^5I_8$, $^3D_3 \rightarrow {}^5I_6$, $^5G_5 \rightarrow {}^5I_8$, $^5D_3 \rightarrow {}^5I_5$, $^5D_3 \rightarrow {}^5I_4$, $^3H_5 \rightarrow {}^5I_6$, $^5D_2 \rightarrow {}^5I_8$, $^5S_2 \rightarrow {}^5I_8$, $^5G_4 \rightarrow {}^5I_6$, $^5F_5 \rightarrow {}^5I_8$, and $^5I_4 \rightarrow {}^5I_8$, respectively. These assignments are made by comparison of the up-conversion spectrum with the energy levels of Tm^{3+} and Ho^{3+} ions as shown in Fig. 5. For example, if the energy of emission band peak coincides with that of excited state of Tm^{3+} or Ho^{3+}, the emission band is assigned to the transition from the excitated state to the ground state. The assignments are also made by comparison with the usual photoluminescence (down-conversion) measurement using Xe lamp light with different excitation wavelengths such as 260, 360, 460 and 680 nm.

Weak emission band is observed at 885–895 nm in HoYLF in Fig. 3(b). It consists of three components at 887, 890 and 893 nm. This emission band is due to the transition $^5I_5 \rightarrow {}^5I_8$

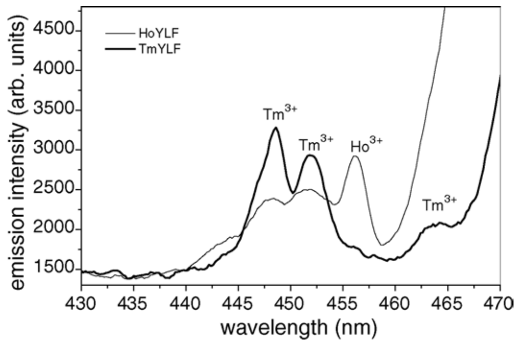

Fig. 4. Enlarged spectrum of Fig. 3(a) in a 430–470 nm spectral region.

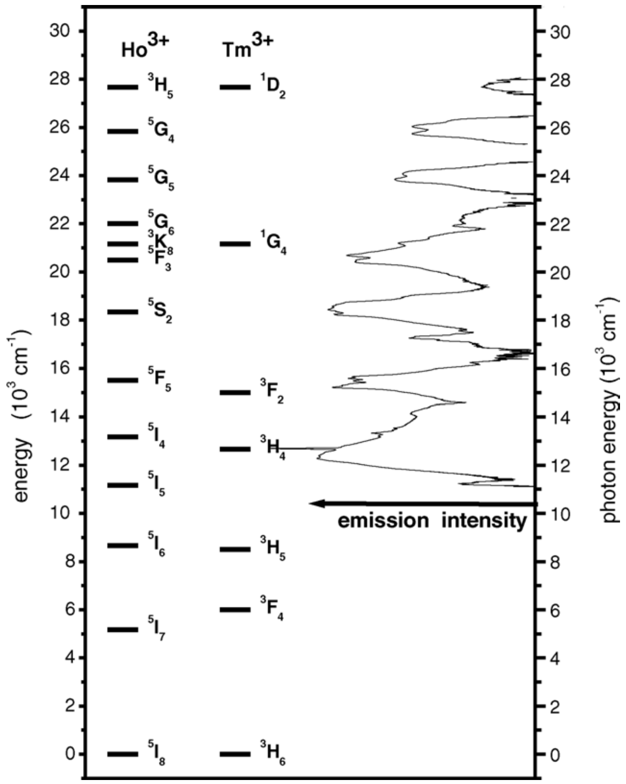

Fig. 5. Comparison of up-conversion spectrum of HoYLF excited by 35-mW 785 nm LD (right-hand side of the figure) with the energy levels of Tm^{3+} and Ho^{3+} ions. The up-conversion intensity is shown in log-scale. A sharp peak at 12739 cm^{-1} is due to the 785 nm LD used for excitation.

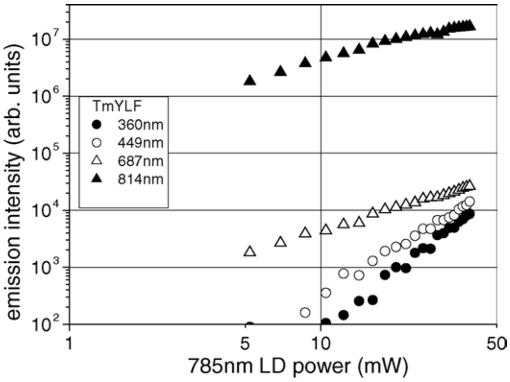

Fig. 6. Peak intensities of the 360, 449, 687 and 814 nm Tm^{3+} emission bands in TmYLF crystal plotted against the 785 nm laser diode intensity at 290 K.

of Ho^{3+}. In Fig. 3(b), two weak emission bands are observed at 687 and 703 nm by 785 nm LD excitation. These emission bands were not observed by the down-conversion measurement with, e.g. 360, 460 and 680 nm light from Xe lamp. We discuss the 687 and 703 nm emission bands later. In TmYLF the 549 nm emission band is observed as the Ho^{3+} emission in TmYLF. The intensity is comparable with the 650–670 nm Tm^{3+} emission intensity although the Ho^{3+} concentration is much smaller than the Tm^{3+} one in TmYLF, indicating efficient energy transfer from Tm^{3+} to Ho^{3+}.

Fig. 6 plots the intensity of the 360, 449, 687 and 814 nm Tm^{3+} emissions observed in TmYLF against the 785 nm LD power. The 360, 449, 687 and 814 nm emissions take slopes of 3.0, 3.0, 1.2 and 1.0 in the log–log scale. This indicates that the 360 and 449 nm emission is due to the upconversion by three-photon process. The 360 and 449 nm emission bands arise from the transition from the $^1D_2$ state to the $^3H_6$ ground state and $^3F_4$ state, respectively, as mentioned above. To excite into the $^1D_2$ state by 785 nm photon, the following processes are necessary. The first photon excites Tm^{3+} ion from the ground state to the $^3H_4$ state from which Tm^{3+} relaxes to the $^3H_5$ state. The second 785 nm photon excites from the $^3H_5$ state into the $^1G_4$ state from which Tm^{3+} relaxes to the $^3F_3$ state. The third 785 nm photon excites from the $^3F_3$ state into the $^1D_2$ state. On the other hand, the 814 nm emission is confirmed to arise from the one-photon down conversion. It was observed that the up-converted 687 and 703 nm emissions take the same slope of 1.0.

We plot the intensities of the other emission bands, which were observed in TmYLF and HoYLF crystals, against LD power in Figs. 7 and 8, respectively. The slope in the log–log plotted power dependence is 1.98, 1.87 and 1.86 for the 477, 483 and 489 nm emissions, 1.93, 1.93, 1.91, 1.96 and 2.02 for the 517, 520, 528, 541 and 549 nm emissions, 1.59, 1.52, 1.56 and 1.59 for the 648, 653, 663 and 666 nm emissions, 1.41 and 1.42 for the 642 and 656 nm emissions, 1.8, 1.9, 2.0 and 2.1 for the 690, 705, 740 and 750 nm emissions, respectively. These up-conversion emission bands have the slope values of much higher than 1.0. The results of Fig. 7 are the same as the results of Fig. 8 except for the 360 and 386 nm emissions. The 360 nm Tm^{3+} and 386 nm Ho^{3+} emissions take slope of 3 in TmYLF, while both of the two emissions take slope of 2 at low LD power range and slope of 3 at high power range in HoYLF as shown in Fig. 8. This indicates that different optical process occurs to give rise to these up-converted emissions between the TmYLF and HoYLF crystals. We consider this optical process as follows.

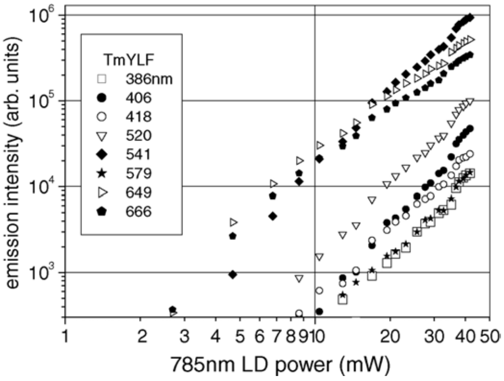

Fig. 7. Peak intensities of various emission bands in TmYLF crystal plotted against the 785 nm laser diode intensity at 290 K.

The concentration of Ho^{3+} ions is higher in HoYLF than in TmYLF. The 360 nm Tm^{3+} emission arises from the transition from the $^1D_2$ state at about 27800 cm^{-1} above the ground state. Therefore, at least three 785 nm photons (with photon energy of 12740 cm^{-1}) are needed to excite into the $^1D_2$ state as shown in Fig. 9(b). This interpretation is consistent with the slope of 3 obtained for the 360 nm emission in TmYLF. In HoYLF, however, the 360 nm emission was obtained by two-photon absorption process at low 785 nm LD power range. Why only two photons are possible to excite into the $^1D_2$ state in HoYLF?

The first 785 nm photon excites Tm^{3+} ion from the ground state to the $^3H_4$ state. Ho^{3+} ion has the $^5I_4$ state which is quite close to the $^3H_4$ state. Therefore, the energy transfer occurs easily from Tm^{3+} to Ho^{3+} because Tm^{3+} ions are more possible to locate near Ho^{3+} sites in heavily Tm^{3+}-doped

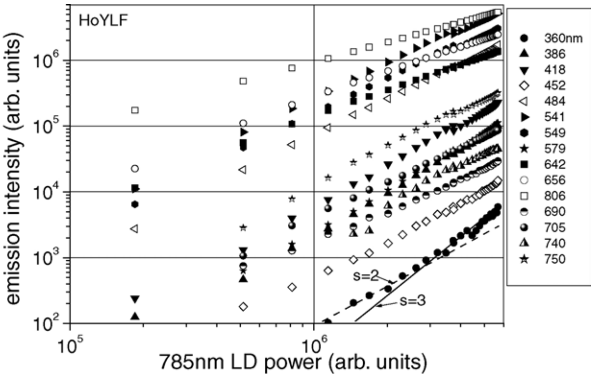

Fig. 8. Peak intensities of various emission bands in HoYLF crystal plotted against the 785 nm laser diode intensity at 290 K. A letter "*s*" means the value of slope for a straight line drawn in the log–log scale.

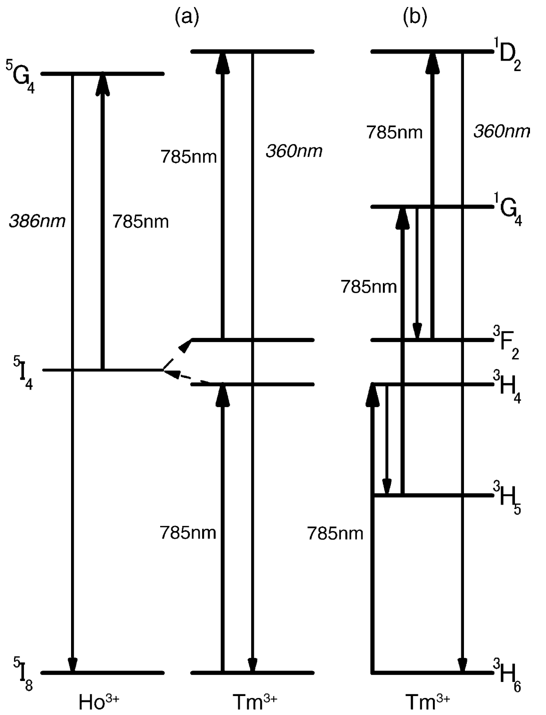

Fig. 9. (a) The 360 nm Tm^{3+} up-converted emission and 386 nm Ho^{3+} up-converted emission by two-photon process with 785 nm LD photon by energy transfer between Tm^{3+} and Ho^{3+} ions. (b) The 360 nm Tm^{3+} up-converted emission by three-photon process (see text).

(8%) HoYLF crystal than in the very lightly Ho^{3+}-doped (0.01%) TmYLF crystal. The second 785 nm photon excites Ho^{3+} from the excited $^5I_4$ state to the $^3G_4$ state of Ho^{3+}, from which the 386 nm Ho^{3+} emission is generated (see Fig. 9(a)). At the same time the $^3F_2$ state of Tm^{3+} locates close to the $^5I_4$ state. Thus, the energy transfer occurs to the $^3F_2$ state, followed the excitation from the $^3F_2$ state to the $^1D_2$ state of Tm^{3+} by the second 785 nm photon (as shown in Fig. 9(a)). In this way we can understand the presence of the 386 and 360 nm upconversions by two-photon absorption process.

The 687 and 703 nm emission bands show the slope of almost 1.00 as mentioned above. The slope of 1 means that excitation with one-photon absorption process gives rise to emission. Why the up-converted 687 and 703 nm emissions are possible by the one-photon excitation process? We explain this unusual result as follows.

Fig. 10 shows the temperature dependence of the 687 and 703 nm emission bands. It is observed when crystal is heated from 15 K up to 320 K that these bands appear above 200 K. The band intensities increase exponentially with temperature in a form of $a_i \exp(-\Delta E/kT)$ as shown in Fig. 11. The best fit was observed at $\Delta E = 1700\ \mathrm{cm}^{-1}$ and $a_2/a_1 = 1.77$ where a_1 and a_2 are parameters for the 687 and 703 nm emission bands, respectively. This indicates that the two emission bands arise from the thermally excited states.

Fig. 12 shows the $^3H_6 \rightarrow {}^3H_4$, $^3F_3$ and $^3F_2$ absorption spectra of 655–805 nm region at 15 and 290 K, together with the spectrum of the 687 and 703 nm emission bands.

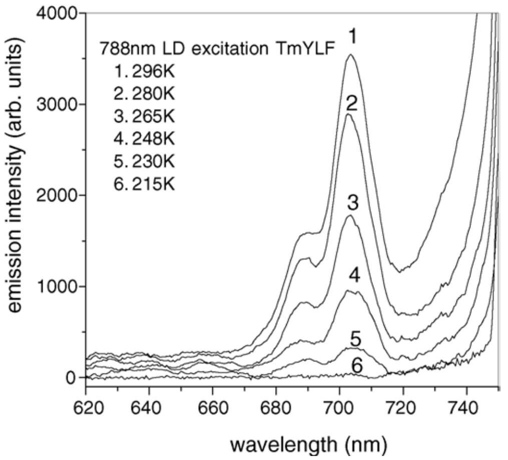

Fig. 10. The 687 and 703 nm emission bands of TmYLF, which was excited with 785 nm laser diode, at various temperatures between 215 and 296 K.

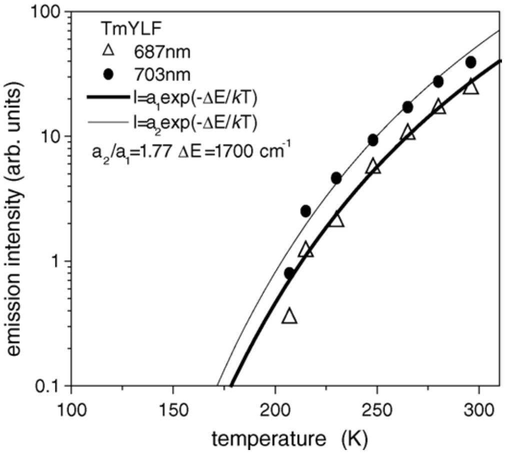

Fig. 11. Temperature dependence of the intensities of the 687 and 703 nm emission bands observed in Fig. 10. Solid curves are the best fit to the experimental points, which were derived using a form of $a_i \exp(-\Delta E/kT)$ (see text).

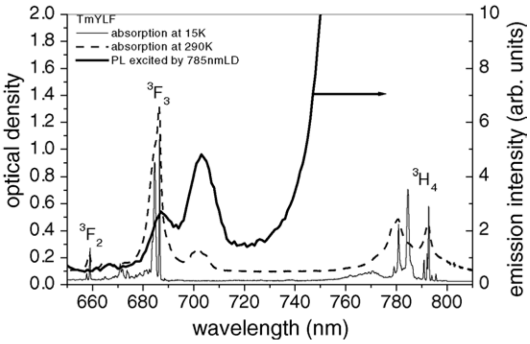

Fig. 12. Absorption spectra of TmYLF at 15 and 290 K, together with the spectrum of the 687 and 703 nm emission bands obtained by 785 nm laser diode excitation at 290 K.

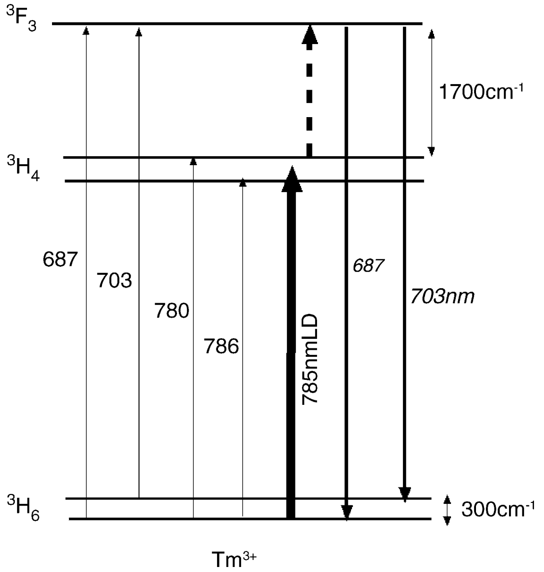

Fig. 13. Schematic energy level diagram of the $^3F_3$, $^3H_4$ and $^3H_6$ states of Tm^{3+} and the optical transitions of the absorption bands at 687, 703, 780 and 786 nm at 290 K and the 687 and 703 nm emission bands obtained by 785 nm laser diode excitation.

The 687 nm band peak wavelength coincides with the wavelength of the $^3H_6 \rightarrow {}^3F_3$ absorption band peak. Absorption band at 703 nm is observed at room temperature. Its intensity decreases with decreasing temperature and the 703 nm absorption band is not observed at low temperatures such as 15 K. This means that the 703 nm absorption band is a hot band which is due to transition from a thermally excited Stark substate of the $^3H_6$ ground state (see Fig. 13). The wavelength of the 703 nm absorption band peak coincides with that of the 703 nm emission band peak. This coincidence allows us to attribute the 703 nm emission to the reverse electronic transition of the 703 nm absorption (Fig. 13).

The $^3F_3$ state is at about 1700 cm^{-1} above the $^3H_4$ state which is excited by the 785 nm LD. The energy gap between the $^3F_3$ and $^3H_4$ states agrees with ΔE value derived from the experimental results. Taking into account these results and considering that the LD irradiation gives rise to heating of crystal, it is confirmed that the 687 and 703 nm emission bands are caused by transition from the $^3F_3$ state which is thermally populated from the $^3H_4$ state as shown in Fig. 11. The 687 and 703 nm emissions are attributed to the transition from the $^3F_3$ state to the Stark levels at 0 and 300 cm^{-1} in the $^3H_6$ ground state, respectively. The presence of the 300 cm^{-1} Stark level is confirmed by the appearance of the 703 nm hot absorption band.

An intense Ho^{3+} up-conversion band is observed at 549 nm in TmYLF by 785 nm LD excitation as shown in Fig. 3(a). Their slope values in the log–log plotted power dependence are quite close to 2.0, indicating these emissions are due to the two-photon excitation. The LD light excites Tm^{3+} to the $^3H_4$ state. Ho^{3+} is not excited by the 785 nm LD because Ho^{3+} has no absorption band around 785 nm. Therefore, the 549 nm Ho^{3+} emission band is caused by energy transfer from Tm^{3+} to Ho^{3+}. Two processes are conceivable for the energy transfer, which are due to the non-resonant energy transfer [8] assisted by creation or annihilation of phonons. The energies of the $^5I_5$ and $^5I_4$ states of Ho^{3+} are close to that of the $^3H_4$ state of Tm^{3+} as shown in Fig. 14. Therefore, the energy in the $^3H_4$ state of Tm^{3+} excited by the first 785 nm LD photon is transferred to the $^5I_5$ and $^5I_4$ state, subsequently excited to the upper states $^5G_5$ and $^5G_4$ of Ho^{3+} by the second 785 nm photon, respectively (see Fig. 14(a) and (b)). In fact, two-photon excited up-converted 419 and 386 nm emission bands, which are due to the $^5G_5 \rightarrow {}^5I_8$ and $^5G_4 \rightarrow {}^5I_8$ transitions of Ho^{3+}, are observed in HoYLF crystal, respectively, although these emission intensities are not as intense as the other upconversion. Therefore, the two processes of Fig. 14(a) and (b) occur in HoYLF. It is difficult to determine at this moment which process occurs more strongly and more efficiently.

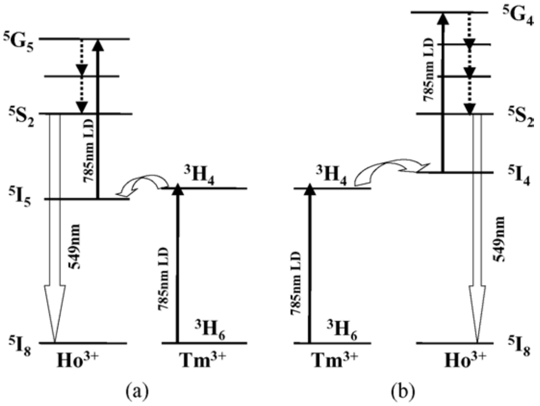

Fig. 14. Two possible energy transfers from Tm^{3+} to Ho^{3+} by non-resonant process in the up-converted 549 nm Ho^{3+} emission generated by excitation in the $^3H_4$ state of Tm^{3+} with 785 nm laser diode. The dotted arrow means the non-radiative relaxation.

4. Summary

Photoluminescence and up-conversion spectra of Tm^{3+} and Ho^{3+} ions in $LiYF_4$ crystals with 6% Tm^{3+} and 0.01% Ho^{3+} ions and with 8% Tm^{3+} and 0.5% Ho^{3+} ions were investigated in 350–1600 nm spectral region at various temperatures between 10 and 320 K. The photoluminescence was investigated using excitation with 460, 680 and 780 nm light of Xe-lamp, while up-conversion and down-conversion was investigated by 785 nm laser diode with maximum power of 35 mW. The 1150–1250 nm $^1G_4 \rightarrow {}^3H_4$ emission of Tm^{3+} was observed under the 460 nm excitation but not observed under the 680 and 780 nm excitation. In addition to the 386 and 419 nm Ho^{3+}, 477–489 nm Tm^{3+}, 541–549 nm Ho^{3+}, 648–666 nm Tm^{3+} up-converted emissions due to two-photon excitation, anti-Stokes emission bands were observed at 687 and 703 nm under excitation in the $^3H_4$ state of Tm^{3+} with 785 nm laser diode. The 687 and 703 nm bands were observed to appear above 200 K, and their intensities increased exponentially with increasing temperature. They were attributed to endothermic Tm^{3+} emission due to the transition to the $^3H_6$ ground state from the upper $^3F_3$ state, which is thermally populated from the $^3H_4$ state. Optical processes by energy transfer from Tm^{3+} to Ho^{3+} were suggested for the green up-converted Ho^{3+} emission.

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