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Control of the energy transfer between Tm^{3+} and Yb^{3+} ions in Tm , Yb -codoped ZnO grown by sputtering-assisted metalorganic chemical vapor deposition

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We report on the epitaxial growth and the optical properties of Tm,Yb -codoped ZnO (ZnO:Tm,Yb) thin films by sputtering-assisted metalorganic chemical vapor deposition. The characteristic light emission due to the intra-4f shell transitions of Tm^{3+} and Yb^{3+} ions in the ZnO host is observed at 4 K by using photoluminescence (PL) characterization. The time-resolved PL measurements on the Tm^{3+} and Yb^{3+} emission reveals that the carrier decay time of each ion can vary by changing the concentration of both Tm^{3+} and Yb^{3+} ions, which depends on a mixing ratio of Tm_2O_3 and Yb_2O_3 in a sputtering target. The energy transfer from Tm^{3+} to Yb^{3+} ions occurs in the sample with the high mixing ratio, and the reversed energy transfer process occurs with the lower mixing ratio. These results indicate that the concentration, and thus the carrier dynamics of Tm^{3+} and Yb^{3+} ions in the ZnO:Tm,Yb host, can be controlled by altering the mixing ratio of the sputter target and the frequency power. *Published by AIP Publishing.* <https://doi.org/10.1063/1.5011270>

I. INTRODUCTION

Silicon (Si) solar cell has a problem in that its energy conversion efficiency is limited to $\sim 30\%$ (known as the Shockley-Queisser limit).¹ This is owing to the mismatch between the solar spectrum, with a wide emission regime, and the absorption properties of Si, where the excessive energy of photons with higher energy than the bandgap of Si is wasted as heat. Down-conversion (DC) is a promising approach to improving the energy conversion efficiency by converting one high energy photon to some lower energy photons.^{2,3} It has been reported that the theoretical upper limit of conversion efficiency of Si solar cells can be improved from 30% to $\sim 40\%$ by employing the DC scheme.⁴ Such wavelength conversion is achieved by using rare earth (RE) ion pairs. Materials co-doped with thulium (Tm) and ytterbium (Yb) have received much attention due to their potential as downconverters, since Tm^{3+} - Yb^{3+} pair is one of the suitable combinations for the Si solar cells. In this configuration, Tm^{3+} is excited first with a 489 nm photon at the $^1\text{G}_4$ - $^3\text{H}_6$ transition, and then the energy is transferred to the $^2\text{F}_{5/2}$ - $^2\text{F}_{7/2}$ transition where the radiative recombination emits photon at the near-infrared emission at 980 nm. This is effectively absorbed by the Si solar cells, and thus the quantum efficiency over 100% can be realized. There have been many reports on the demonstration of the DC using various RE ion pairs including Tb^{3+} - Yb^{3+} ,^{3,5-7} Pr^{3+} - Yb^{3+} ,⁸ and Tm^{3+} - Yb^{3+} .^{9,10} Some reports have already exhibited almost an ideal efficiency near 200%.^{9,10} For most Tm^{3+} - Yb^{3+} DC materials, the host materials of RE ions are insulating materials or fluorescent materials.¹¹ However, Tm^{3+} ions cannot efficiently absorb sunlight because the absorption of Tm^{3+} ions is

characterized by the intra-4f transitions, which have low excitation cross section and narrow absorption bandwidth.

In order to circumvent these problems, our group has proposed Tm,Yb -codoped ZnO (ZnO:Tm,Yb) as a material for photonic downconverters. At ambient conditions, the semiconductor ZnO crystallizes in the wurtzite structure and has a direct bandgap of 3.4 eV (Ref. 12) and thus has widespread applications in light-emitting diodes or lasers operating in the visible or ultraviolet (UV) regimes,¹³ solar cells,¹⁴ liquid crystal displays,¹⁵ gas sensors,^{16,17} transistors,¹⁸ and transparent conductive films.¹⁹ Furthermore, it has been reported that the emission of RE-doped ZnO , which is characterized by an indirect excitation mechanism from the ZnO host to RE ions, has been observed.^{20,21} In our DC model using ZnO:Tm,Yb , the ZnO host can absorb photons with energy in a wide range from UV to violet light with a large excitation cross section. The RE intra-4f shell is excited via an energy transfer from the host material, followed by light emission in the near-infrared regime from the dopants. The broad light absorption region and the large absorption coefficient of ZnO are expected to give a highly efficient photonic conversion in this material. In this article, we establish the growth technique for the ZnO:Tm,Yb , control the composition of both Tm and Yb, and study their carrier dynamics to investigate the effects of Tm,Yb -codoping on the energy transfer between Tm^{3+} and Yb^{3+} ions.

II. EXPERIMENTAL DETAILS

ZnO:Tm,Yb films were grown on *c*-plane sapphire substrates with a resistive heater at 550 °C by a sputtering-assisted metalorganic chemical vapor deposition (SA-MOCVD). The ZnO was grown in the same chamber at 550 °C by a thermal reaction of diethylzinc (DEZn) in oxygen (O_2) with flow rates

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of 1 and 24 sccm, respectively. The growth duration was fixed to 180 min for all the samples. The detailed growth conditions of ZnO by our MOCVD system has been reported in our previous publications.^{22,23} Tm and Yb were doped during the growth of ZnO by radio-frequency (RF) magnetron sputtering of a Tm_2O_3 and Yb_2O_3 mix-sintered targets with a diameter of 5 cm, which were fixed on the top of the substrate at a 90° angle and at a distance of 10 cm. Several targets with different mixing ratios of Tm_2O_3 and Yb_2O_3 , 1:1, 1:2, and 1:5, were prepared to control the concentration ratio of Tm and Yb in the ZnO host. The Tm_2O_3 and Yb_2O_3 target was sputtered at a RF power between 30 and 60 W, in order to control the Tm and Yb concentration. Tm-doped or Yb-doped ZnO films were also prepared for comparison by using a sinter Tm_2O_3 or Yb_2O_3 target, respectively, instead of the mix-sintered targets. The Tm and Yb concentration was subsequently measured by the energy dispersive X-ray (EDX) spectroscopy equipped with a scanning electron microscope (JEOL Ltd., EX-64175JMV) for higher RE concentration (>0.1 at. %), and an X-ray fluorescence (XRF) spectroscopy for lower RE concentration below the detection limit of EDX (~ 0.1 at. %). Several positions on each sample were randomly chosen for EDX or XRF characterization, and the obtained values of the RE concentration were averaged. After the growth, the films were annealed at 600°C for 30 min in O_2 ambient to activate the luminescence from Tm^{3+} ions in ZnO, then cooled down by turning off the resistive heater, and ceasing the gas flows of both DEZn and O_2 . The samples were finally unloaded from the chamber when the temperature dropped from 600 to $<200^\circ\text{C}$ (>1 h).²⁴ The thickness of ZnO films were characterized by first coating a portion of the as-grown ZnO films with photoresists, then etching the exposed ZnO films with hydrochloric acid, and finally analyzing using a surface roughness measuring instrument (Kosaka Laboratory Ltd., Surfcomer SE500). The structural characterization were performed by using an X-ray diffractometer (Rigaku HyPix-3000, 45 kV acceleration voltage and 200 mA current) in the θ - 2θ configuration at the (002) geometry. Photoluminescence (PL) measurements were carried out at 4 K with an excitation by a He-Cd laser (325 nm), a diode laser (980 nm), or an optical parametric oscillator (OPO) laser (797 nm), and the PL was detected by an electrically cooled Si photodiode array. The time-resolved PL (TR-PL) measurements were also performed with an excitation by a pulsed He-Cd laser modulated by a function generator through an acousto-optic modulator and detection by a Silicon avalanche photodiode. The spectrometer resolution was below 0.05 nm for the TR-PL measurements.

III. RESULTS AND DISCUSSION

Figure 1 shows the RF power dependence of Tm and Yb concentration in the ZnO host. The mixing ratio of Tm_2O_3 and Yb_2O_3 in the target was set at 1:1, which exhibits an increase in the concentration of both Tm and Yb with RF powers due to the increased etching rate of the target. In all cases, the concentration of Tm is higher than that of Yb, indicating that the Tm is more easily incorporated into ZnO than Yb. The concentration of both Tm and Yb shows almost a linear increase with an increased RF power since more Tm

FIG. 1. RF power dependence of Tm and Yb concentration in the ZnO host, showing an increase in the concentration of both Tm and Yb with RF power.

and Yb ions are sputtered from the target with higher power. This result enables the control of both Tm and Yb concentrations in the ZnO host by adjusting the RF power accordingly.

Figure 2 shows XRD patterns of the ZnO:Tm,Yb films using a two-dimensional time delay and integration (2D-TDI) mode and their rocking curves at ZnO (002) with various sputtered RF powers ranging from 30 to 60 W. The thickness and, hence, the growth rate of ZnO:Tm,Yb films displayed in Table I show a gradual decrease with the increase in RF powers due to an increase in the etching rate. The XRD peaks of ZnO (002) and (004) as well as other single peaks from the sapphire substrate (006) are observed for all the samples, while no other phases, such as Tm_2O_3 or Yb_2O_3 , were observed within the detection limit. This pattern corresponds to a typical growth mode of undoped ZnO on a c-plane sapphire substrate, and thus these results indicate that the c-axis-oriented ZnO:Tm,Yb films are successfully grown on sapphire substrates. By contrast, the β direction of the peak spread shows the corresponding rocking curve of the reflection as shown in the bottom of Fig. 2. The full-width at half-maximum (FWHM) of (002) ZnO rocking curves, indicating the crystal quality of ZnO films, shows a reduction from 3.27° for the sample at 30 W to $\sim 2.5^\circ$ for the samples at 40 W and 50 W. It is explained by the increase in oxygen pressure introduced into the reactor at a lower RF power at 30 W to suppress the desorption of oxygen from the ZnO host, which deters the crystal quality. The FWHM shows again an increase of up to 3.34° for the sample at 60 W due to the degradation of crystal quality of the ZnO host by sputtering with high RF power. These results suggest that a moderate RF power is required to obtain the highest crystal quality of ZnO:Tm,Yb films, and there is a trade-off between the RF power, which determines the Tm/Yb concentration and the crystal quality of the ZnO films indicating that it is difficult to obtain simultaneously high crystal quality and high Tm/Yb concentration.

In the ZnO:Tm,Yb sample, a He-Cd laser pumps the ZnO host, yielding an electron-hole pair, which can photoexcite the RE ions through the energy transfer from the ZnO host. An

FIG. 2. XRD patterns in ZnO:Tm,Yb films at various RF powers ranging from 30 to 60 W (from left to right). (top) 2D diffraction images using 2D-TDI mode measured from $2\theta = 20^\circ$ to 80° (bottom) (002) ZnO rocking curves.

energy exchange between Tm^{3+} and Yb^{3+} is possible, which can be utilized as DC (or down-shift). Therefore, the optical characterization of the ZnO:Tm,Yb with different mixing ratios is carried out to clarify the carrier dynamics. Figure 3(a) shows the PL spectra of ZnO:Tm,Yb samples, fabricated using different targets, with an excitation by the He-Cd laser. As depicted in band diagrams of Figs. 3(b) and 3(c), the light emission due to the $^3\text{H}_4\text{--}^3\text{H}_6$ transition of Tm^{3+} ions [Fig. 3(b)], and the light emission due to the $^2\text{F}_{5/2}\text{--}^2\text{F}_{7/2}$ transition of Yb^{3+} ions [Fig. 3(c)] are simultaneously observed at 797 nm and 980 nm, respectively. However, the PL intensity of 980 nm light emission of Yb^{3+} gradually enhances with an increase in Yb_2O_3 composition from 1:1 to 1:5 due to the increase in Yb^{3+} mixing ratio in the targets, while the PL intensity at 797 nm exhibits an opposite trend. This result suggests that the concentration ratios between Tm^{3+} and Yb^{3+} in the ZnO host can be controlled by changing the mixing ratio of the sintered targets. It is noted here that the thickness of ZnO films, shown in Table I, is much thicker than

that of the penetration depth of light at 325 nm (<65 nm),²⁵ and the excited volume should therefore be the same for all ZnO films with various growth/sputter conditions. Figures 3(b) and 3(c) show the PL spectra of Tm^{3+} ions at ~ 800 nm and Yb^{3+} ions at ~ 980 nm in the ZnO:Tm,Yb film with an excitation by a diode laser and an OPO laser, respectively. Several sharp emission peaks from the $^3\text{H}_4\text{--}^3\text{H}_6$ transition of Tm^{3+} ions can be observed by photopumping the Yb^{3+} ions with a diode laser, indicating the presence of an energy transfer mechanism from Yb^{3+} to Tm^{3+} , as suggested in Fig. 3(b). On the other hand, several sharp emission peaks from the $^2\text{F}_{5/2}\text{--}^2\text{F}_{7/2}$ transitions of Yb^{3+} ions can be observed by photopumping the Tm^{3+} ions with an OPO laser, indicating a possible energy transfer route between Tm^{3+} and Yb^{3+} ions.

In this configuration, it is possible for a direct energy transfer from Yb^{3+} to Tm^{3+} ions to occur at lower Yb^{3+} concentration and vice versa at higher Yb^{3+} concentration. Therefore, the TR-PL characterization is performed for several ZnO:Tm,Yb samples with different Tm/Yb concentrations in order to elucidate the carrier dynamics between Yb^{3+} and Tm^{3+} ions in the ZnO host. Carrier decay times are obtained by fitting the decay curves with a double exponential. This is justified by the presence of two components; one is a faster carrier decay time originated from the emission from defects, and the other is a slower carrier decay time from rare-earth ions, as shown in Fig. 4(a). Figures 4(b)–4(d) plot the carrier decay times of the Tm^{3+} emission, with an error bar corresponding to the standard deviation, as

TABLE I. Thicknesses and growth rates of ZnO films at various RF powers from 30 to 60 W.

RF power (W)	Thickness (nm)	Growth rate (nm/min)
30	690	3.8
40	670	3.7
50	560	3.1
60	500	2.8

FIG. 3. (a) PL spectra at 4 K of ZnO: Tm,Yb sputtered by sintered targets with various mixing ratios of Tm₂O₃: Yb₂O₃ from 1:1 to 1:5 with an excitation by a He-Cd laser and the energy level diagram of the ZnO:Tm,Yb (b) PL spectrum at 4 K of the ZnO:Tm,Yb film under resonant excitation of Tm³⁺ using a diode laser at 980 nm, and the suggested energy transfer model from Yb³⁺ to Tm³⁺ in ZnO:Tm,Yb. (c) PL spectrum at 4 K of the ZnO: Tm,Yb film under resonant excitation of Yb³⁺ using an OPO laser at 797 nm, and the derived energy transfer model from Tm³⁺ to Yb³⁺ in the ZnO:Tm,Yb.

a function of the Tm concentration in ZnO:Tm,Yb samples prepared using several targets with different mixing ratios. The decay times of ZnO:Tm are also plotted for comparison (black solid squares), which exhibit a gradual decrease with an increase in the Tm concentration due to the concentration quenching of rare-earth ions in the ZnO host. An incorporation of Yb ions into the ZnO:Tm with the mixing ratio of 1:1 tends to make the carrier decay time larger (a few to ~20%) [Fig. 4(b)]. With an increase in the composition of Yb₂O₃ from 1:1 to 1:5, the carrier decay time of the Tm³⁺ ion in ZnO:Tm,Yb tends to decrease: almost comparable with the mixing ratio of 1:2 as shown in Fig. 4(c), and shorter with the mixing ratio of 1:5 [Fig. 4(d)]. By contrast, the relation of the carrier decay times between ZnO:Tm,Yb and ZnO:Yb exhibits the opposite trend. Figures 5(a)–5(c) show the carrier decay times of the Yb³⁺ emission, with an error bar corresponding to the standard deviation, as a function of Yb concentration in ZnO:Tm,Yb prepared using several targets with different mixing ratios. The carrier decay time of the Yb³⁺ ion in ZnO:Tm,Yb tends to be comparatively shorter

than that in ZnO:Yb, if we use the target with the mixing ratio of 1:1 as can be seen in Fig. 5(a). These values become longer than that of the Yb³⁺ ion in the ZnO:Yb, comparable with the mixing ratio of 1:2 as shown in Fig. 5(b), and longer with the mixing ratio of 1:5 [Fig. 5(c)]. These results can be explained as follow. At lower Yb³⁺ concentration, with the target concentration of 1:1, the direct energy transfer from Yb³⁺ to Tm³⁺ ions produces extra carriers in Tm³⁺, which renders the carrier decay time of Tm³⁺ longer while that of Yb³⁺ becomes shorter due to the consumption of carriers for the energy transfer. By contrast, at higher Yb³⁺ concentration, with the target concentration of 1:5, the direct energy transfer from Tm³⁺ to Yb³⁺ ions produces extra carriers in Yb³⁺, which renders the carrier decay time of Yb³⁺ longer while that of Tm³⁺ becomes shorter due to the consumption of carriers for the energy transfer. Therefore, these results indicate that the energy transfer from the ¹G₄ state of Tm³⁺ ions to the ²F_{5/2} state of Yb³⁺ ions is enhanced in the sample prepared with high Yb₂O₃ ratio and vice versa. A possible suggestion could be that the wavelength conversion process

FIG. 4. (a) Decay curve of Tm^{3+} emission in ZnO:Tm,Yb with a mixing ratio ($\text{Tm}_2\text{O}_3:\text{Yb}_2\text{O}_3$) of 1:2. Red solid lines are double exponential fits to the data. (b)–(d) Decay lifetimes of Tm^{3+} emission in ZnO:Tm (black square) and ZnO:Tm,Yb (colored circles) as a function of Tm concentration. Tm_2O_3 and Yb_2O_3 in the target were mixed with different ratios of (b) 1:1, (c) 1:2, and (d) 1:5.

FIG. 5. Decay lifetimes of Yb^{3+} emission in ZnO:Yb (black squares) and ZnO:Tm,Yb (colored circles) as a function of Yb concentration. Tm_2O_3 and Yb_2O_3 in the target were mixed with the ratios of (a) 1:1, (b) 1:2, and (c) 1:5.

between Tm^{3+} and Yb^{3+} ions is controllable by changing both the Tm_2O_3 and Yb_2O_3 ratio of the sputtering target. However, further details regarding the carrier dynamics are still unclear and thus will be a subject of future research. Since the energy of the $\text{Yb}^{3+} {}^2F_{5/2} \rightarrow {}^2F_{7/2}$ transition is matched well with the bandgap of Si, the ZnO-based down-converter discussed in our work may have prospects of realizing the high efficiency Si solar cells.

IV. CONCLUSION

In conclusion, we report on the growth and optical properties of ZnO:Tm,Yb thin films by SA-MOVPE. The characteristic light emission due to the intra- $4f$ shell transitions of

Tm³⁺ and Yb³⁺ ions in the ZnO host are observed at 4 K by using the PL characterization, and the PL intensity of each peak can be controlled by changing the concentration ratio of Tm³⁺ and Yb³⁺. The TR-PL measurements of the Tm³⁺ and Yb³⁺ emission implies the existence of direct energy transfer between Tm³⁺ and Yb³⁺ ions, which depends on a mixing ratio of Tm₂O₃ and Yb₂O₃ in a sputtering target. The energy transfer from Tm³⁺ to Yb³⁺ ions occurs in the sample with the high mixing ratio and the reversed energy transfer process occurs with the lower mixing ratio. These results indicate that the concentration, and thus the carrier dynamics of Tm³⁺ and Yb³⁺ ions in the ZnO:Tm,Yb host, can be controlled by altering the mixing ratio of the sputtering targets, along with a radio-frequency sputtering power.

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