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Photoluminescence quantum efficiency and energy transfer of ErRE silicate (RE = Y, Yb) thin films

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Abstract
Photoluminescence quantum efficiency and energy transfer were studied in ErRE (RE = Y, Yb) silicate thin films. For the first time, we experimentally determined the radiative/non-radiative decay rates of ErYb and ErY silicates and obtained photoluminescence quantum efficiencies of 0.28 ± 0.08 and 0.39 ± 0.13, respectively. Energy transfer between Yb and Er in ErYb silicate was investigated by comparing the effective excitation cross sections of ErYb and ErY silicate. At different pumping wavelengths, forward and backward energy transfer caused by the presence of Yb was experimentally demonstrated.

1. Introduction
Yttrium (Y) silicates (Y₂SiO₅ and Y₂Si₂O₇) have been widely studied specially when doped with rare-earth (RE) ions for applications in solid-state lasers [1], information storage [2], for high-temperature thermometry [3], and as red, green and blue phosphors for display applications [4, 5]. Yttrium silicates doped with erbium (Er) as well as pure Er silicates (Er₂SiO₅ and Er₂Si₂O₇) have recently received considerable attention due to their efficient luminescence at the telecommunication wavelength of 1.53 µm [6–14]. Compared with the conventional Er-doped materials such as silica or alumina, Er-doped silicates have up to two orders of magnitude higher Er concentrations (~10²² cm⁻³) due to their stoichiometric nature. They are potential light source materials that provide higher gain or stronger emission for the emerging field of silicon photonics. However, at such high Er concentrations, strong Er–Er interactions result in up-conversion and concentration quenching effects, which are detrimental to optical gain [8]. It is thus necessary to dilute the high Er concentration in order to reduce the negative effects originating from interactions between neighbouring Er ions.

To dilute the Er concentrations in pure Er silicates, yttrium and ytterbium (Yb) can be co-doped to substitute part of Er ions in silicates without significantly affecting the crystal structures because of the similar ionic radius (Y: 0.90 Å, Er: 0.88 Å, Yb: 0.86 Å) and similar crystal lattice sizes [15]. It is found that ErY disilicates (ErₓY₂₋ₓSi₂O₇) are good photon energy down-converters to cut high-energy photons into infrared photons whose cutting efficiencies can be up to 400% [14]. Waveguide amplifiers using ErₓY₂₋ₓSiO₂ were reported but the gain was limited by cooperative up-conversion and no net gain was achieved [12].

Different from Y which is optically inactive and only acts as a disperser for Er, Yb is well known for its optical transition at 980 nm that is resonant with the second excited state (⁴I₁₁/₂ level) of Er. Compared with Er, Yb has an almost one order of magnitude larger absorption cross section...
at around 980 nm, which makes Yb a good sensitizer for Er. Yb is commonly co-doped in Er-doped fibre amplifiers and lasers to enhance the absorption of Er and reduce the pumping threshold [16–19]. Thus, it is expected that co-doping Yb into Er silicates may dilute and sensitize Er at the same time. Recently, we have demonstrated that these co-doped samples show extraordinary 1.53 µm photoluminescence (PL) efficiency of Er, sensitization and dilution effect of Yb for Er, and optical amplification in Er1Yb2−xSiO5 thin films and waveguides [20–23]. Miritello et al reported the efficient coupling between Er and Yb in disilicate [24].

However, some of the fundamental properties such as the PL quantum efficiency, radiative and non-radiative decay rates of ErRE silicates are still unknown. The energy transfer between Yb and Er should also be investigated in a wider range using more pump wavelengths. A comparison of ErYb and ErY silicates is useful since ErY silicate provides a reference having similar material properties while Er remains the only optically active element. In this work, we fabricated ErRE silicate (RE = Y, Yb) thin films on Si substrates by magnetron sputtering. The material compositions and PL properties at 1.53 µm are characterized. The PL quantum efficiencies and the radiative/non-radiative decay rates of ErY and ErYb silicates are determined, for the first time, by comparing the measured variation in lifetime for samples in contact with different refractive indices with the calculated variation in radiative lifetime using the local optical density of states (LDOS). The effective excitation cross-sections are then obtained by comparing the quantum efficiencies and PL intensities, at pumping wavelengths of 488, 654 and 974 nm. The forward and backward energy transfer between Yb and Er are explained by an energy level model.

2. Experiment

ErRE (RE = Y, Yb) silicate films were fabricated by the radio frequency (RF) magnetron sputtering method. Two mixed targets made of fully mixed RE oxides and silicon dioxide powders (molar ratio: Er2O3 : RE2O3 : SiO2 = 0.1 : 0.7 : 2.0 : 7.2). This is off the stoichiometric value of RE disilicate RE2Si2O7 and Er2Y1.8Si1O7, were separately sputtered by argon ions at a pressure of 3 mTorr and deposited on (1 0 0) oriented p-type silicon wafers for 40 min. The RF powers applied on the targets were 120 W. The thicknesses of the deposited films were ~100 nm in both cases, determined from cross-sectional scanning electronic microscope (SEM) images. The as-deposited films were then annealed in a furnace at 1200 °C for 1 h in N2 ambient. Spectroscopic ellipsometry was used to measure the refractive index of the annealed films, and the layer thickness values evaluated from ellipsometry data are consistent with SEM observations.

The compositions of the films were determined by Rutherford backscattering spectroscopy (RBS) using 2.022 MeV He+ ions with a detection angle of 165°, plus x-ray fluorescence spectra measurement for the discrimination of Er and Yb. The PL spectra were obtained using a tripling-grating monochromator equipped with a thermoelectrically cooled InGaAs detector, plus a lock-in amplifier taking the reference frequency from a mechanical chopper. The 488 nm line of an argon ion laser, and semiconductor lasers operating at 654 and 974 nm were used as the excitation sources and were modulated on–off at 34 Hz by the mechanical chopper. A digital oscilloscope was used to record the PL decay curves. Three liquids whose refractive indices are 1.3, 1.5 and 1.7 covered the surface of the films to change the LDOS, and the thickness of the liquid was kept constant at 1.5 mm in all experiments.

3. Results and discussion

3.1. Compositional and optical properties

Figure 1 shows the RBS spectra of the annealed films. For ErY silicate, the signals corresponding to Er, Y, Si and O are clearly visible. The composition was estimated to be Er : Y : Si : O = 0.1 : 0.7 : 2.0 : 7.2. This is off the stoichiometric value of RE disilicate (RE : Si : O = 2 : 2 : 7, RE = Er + Y). The ratio of Er and Y in the sputtered film is close to the ratio in the target (Er : Y = 0.1 : 0.9). For ErYb silicate, the RBS spectrum shows well-distinguished signals corresponding to Si and O, but only one peak at the position of Er, which is much higher than that of ErY silicate. The energy resolution of the RBS measurement (~30 keV) is insufficient to distinguish the contributions from Yb (atom number 70) and Er (atom number 68) and the two elements have almost identical scattering cross-sections. Therefore, we sum the contribution of the Yb and Er ions in the RBS spectrum and estimate the composition to be (Er + Yb) : Si : O = 1 : 2 : 7.0, very similar to the composition of ErY silicate. As an additional method, x-ray fluorescence spectra measurement was used to identify the ratio of Er : Yb. The result demonstrates that Er : Yb is 1 : 8.5, quite close to the designed composition. Both sputtered films are silicon and oxygen rich and the system is expected to be phase-separated in stoichiometric RE disilicate RE2Si2O7 and SiO2 [15]. The atomic volume concentrations of Er in the two films are calculated by dividing the Er areal densities derived from RBS by the film thicknesses. The values are
 Decay fitting.

The decay times are 2.1 ms (ErYb) and 1.1 ms (ErY), using single exponential films, recorded at 1538 nm for ErYb and 1530 nm for ErY. The $1/\tau$ curves of ErY silicate (circles) and ErYb silicate (square dot) thin films, normalized to ErY silicate, and the intensity ratios of the former versus the latter are 0.6 and 0.25 for the 488 nm and 654 nm pumping, respectively. For ErY silicate, the peak centred at 1530 nm is constituted by two peaks at 1528 and 1533 nm. The integrated PL intensity of ErYb silicate shows a weaker PL intensity than ErY silicate, and the intensity ratios of the former versus the latter are 0.6 and 0.25 for the 488 nm and 654 nm pumping, respectively. Figure 2(b) shows the PL decay curves measured at 1.53 $\mu$m under 974 nm pumping. Both curves are well fitted by single exponential decay, which is consistent with the low excitation level. The fitted $1/\tau$ times are 2.1 ms and 1.1 ms for ErYb and ErY silicates, respectively. The total decay rate $1/\tau_{\text{tot}}$ can be expressed as $1/\tau_{\text{tot}} = 1/\tau_{\text{r}} + 1/\tau_{\text{nr}}$, where $\tau_{\text{tot}}$ is the measured total PL lifetime, $\tau_{\text{r}}$ is the radiative lifetime and $\tau_{\text{nr}}$ is the non-radiative lifetime.

### 3.2. PL quantum efficiency

The PL quantum efficiency at 1.53 $\mu$m is defined as $\eta = \tau_{\text{nr}}/(\tau_{\text{rad}} + \tau_{\text{nr}})$. The total decay rate is obtained by the inversion of PL lifetime $\tau$. The radiative decay rate, $1/\tau_{\text{rad}}$, can be varied in a controllable way by placing the ions close to a metal mirror [25], a dielectric interface [26, 27, 29], or in a thin film on a semiconductor substrate [28, 29]. The decay rate is proportional to the atomic transition matrix element and the LDOS according to the Fermi’s golden rule [26, 27]:

$$
\frac{1}{\tau_{\text{rad}}(z)} = \frac{\pi \omega}{\hbar \varepsilon(z)} |D|^2 \rho(\omega, z).
$$

The transition matrix element $|D|^2$ is determined by the microscopic field around Er$^{3+}$ and is not influenced by the optical properties of the outer environment. $\varepsilon(z)$ is the position-dependent dielectric constant. The position-dependent LDOS $\rho(\omega, z)$ can be calculated at the transition frequency of Er ions using the method developed previously [28, 29]. By changing the refractive index on the surface of the film, the LDOS and consequently the radiative decay rate are changed [27, 29]. In addition, a non-radiative decay that is independent of the variation in the LDOS may be present, and the radiative decay rate in the silicate film and quantum efficiency can be determined by comparing the measured and calculated variations in decay rate using the relation [27]

$$
\frac{1}{\tau_{\text{tot}}(n)} = \frac{1}{\tau_{\text{nr}}(n)} + f_{\text{bulk}}(n) \frac{1}{\tau_{\text{rad}}(n)}.
$$

The factor $f_{\text{bulk}}(n)$ is the average effect of the LDOS on the decay rate for a film in contact with a liquid with refractive index $n$, normalized to the LDOS of bulk silicate material. Using this principle, the radiative decay rate and quantum efficiency of Er and other RE ions in different dielectrics have been determined experimentally [29, 30].

Figure 3 shows the calculated LDOS of a 100 nm-thick silicate film on a Si substrate ($n = 3.45$) in contact with a medium with different refractive indices. The LDOS is normalized to bulk silicate material with a refractive index $n = 1.6 (f_{1.6} = 1)$. The transition wavelength used for the LDOS calculation was 1538 nm, corresponding to the peak PL.

---

**Table 1. Normalized integrated PL intensity and effective excitation cross section.**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Er$<em>{2}$Y$</em>{1.8}$Si$<em>{2}$O$</em>{7}$</th>
<th>Er$<em>{2}$Y$</em>{1.8}$Si$<em>{2}$O$</em>{7}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Er]</td>
<td>$6 \times 10^{20}$ cm$^{-3}$</td>
<td>$5 \times 10^{20}$ cm$^{-3}$</td>
</tr>
<tr>
<td>$\eta$</td>
<td>$\sim 0.28$</td>
<td>$\sim 0.39$</td>
</tr>
<tr>
<td>$I(488 \text{ nm})$</td>
<td>0.6</td>
<td>1</td>
</tr>
<tr>
<td>$I(654 \text{ nm})$</td>
<td>0.25</td>
<td>1</td>
</tr>
<tr>
<td>$I(974 \text{ nm})$</td>
<td>3.2</td>
<td>1</td>
</tr>
<tr>
<td>$\sigma_{\text{eff}}(488 \text{ nm})$</td>
<td>$\sim 0.7$</td>
<td>1</td>
</tr>
<tr>
<td>$\sigma_{\text{eff}}(654 \text{ nm})$</td>
<td>$\sim 0.3$</td>
<td>1</td>
</tr>
<tr>
<td>$\sigma_{\text{eff}}(980 \text{ nm})$</td>
<td>$\sim 3.7$</td>
<td>1</td>
</tr>
</tbody>
</table>

---

Figure 2. (a) PL spectra of Er ions in ErY silicate (dashed–dotted line) and ErYb silicate (solid line). The samples are excited with 600 mW of 974 nm laser light in a ~1 mm$^2$ spot. (b) PL decay curves of ErY silicate (circles) and ErYb silicate (square dot) thin films, recorded at 1538 nm for ErYb and 1530 nm for ErY. The $1/\tau$ times are 2.1 ms (ErYb) and 1.1 ms (ErY), using single exponential decay fitting.

$5 \times 10^{20}$ cm$^{-3}$ and $6 \times 10^{20}$ cm$^{-3}$ for ErY and ErYb silicates, respectively.
The interface of the film and the Si substrate is positioned at \( z = 0 \). The blue, red and black curves refer to LDOS calculations of the film in contact with different refractive index media. Clearly, the LDOS in the film is increased by the increase in refractive index of the medium. The oscillations on both sides of the film are due to the interference of the incident and reflected fields.

Figure 3. Calculated LDOS for a wavelength of 1538 nm of a thin film with refractive index \( n = 1.6 \) and thickness \( t = 100 \) nm on a p-Si substrate \( (n = 3.45) \). The interface of the film and the Si substrate is positioned at \( z = 0 \). The blue, red and black curves refer to LDOS calculations of the film in contact with different refractive index media. Clearly, the LDOS in the film is increased by the increase in refractive index of the medium. The oscillations on both sides of the film are due to the interference of the incident and reflected fields.

The experimental decay rates increase with increasing wavelength of ErYb silicate. Calculations are shown for a film in air (black line), for an index-matched medium \( (n = 1.6, \) red line) and for a medium with \( n = 2.0 \) (blue line). Clearly, the LDOS of the film is increased compared with that in air by covering the surface with a higher refractive index medium.

Figure 4 shows the measured variation of the PL decay rate versus the refractive index of the liquids for the two films. The experimental decay rates increase with increasing \( n \) due to the increase in LDOS. The dashed lines are the variation in radiative decay rate obtained from the LDOS calculation. Here the radiative decay rate is just a scaling factor to compare the LDOS with the measured decay rates. To calculate the LDOS for the two silicate films, the film thickness and refractive index values used were obtained from the ellipsometry measurement: \( n(\text{Er}) = 1.57, t(\text{Er}) = 100 \) nm, and \( n(\text{ErYb}) = 1.613, t(\text{ErYb}) = 98.5 \) nm, which are slightly different from the parameters used in the calculation of figure 3. The LDOS was then integrated along the depth of each film. Since the Er ions are uniformly distributed along the film’s depth, the LDOS effect on the decay rate can be accounted for by a simple average. Clearly, the measured decay rates show a weaker dependence on the refractive index \( n \) than the calculated LDOS for both films. By taking into account the non-radiative decay, the total decay rate given by equation (2) can be fitted with the experimental data. This results in radiative and non-radiative decay rates of 1/\( \tau_{\text{rad}}(\text{ErYb}) = 128 \pm 33 \) s\(^{-1} \), 1/\( \tau_{\text{rad}}(\text{ErY}) = 322 \pm 54 \) s\(^{-1} \), and 1/\( \tau_{\text{rad}}(\text{ErY}) = 313 \pm 84 \) s\(^{-1} \), 1/\( \tau_{\text{rad}}(\text{ErY}) = 479 \pm 128 \) s\(^{-1} \), corresponding to quantum efficiencies of \( \eta(\text{ErYb}) = 0.28 \pm 0.08 \) and \( \eta(\text{ErY}) = 0.39 \pm 0.13 \).

The experimentally determined 1.53 \( \mu \)m PL quantum efficiencies of ErYb and ErY silicates are similar, and the difference is within the error bars. This is reasonable since neither Yb nor Y has a direct effect on the Er radiative transition of \( ^4I_{13/2} \) to \( ^4I_{15/2} \). Yb interacts with Er only through the coupling between \( ^2F_5/2 \) and \( ^4I_{11/2} \) levels [16]. It is interesting to note that ErY silicate with a higher total decay rate has a similar quantum efficiency to ErYb silicate. This is due to the high radiative decay rate of ErY silicate, which is about 2.5 times the radiative rate of ErYb silicate. The difference in radiative rate could be due to a difference in local environment for the Er ions, which is also reflected by the different PL spectra. Compared with Er-doped silica or alumina, the non-radiative decay rates of silicates are higher. This may be induced by the non-stoichiometric composition of silicates, which introduces defects or other luminescence quenching centres. In a recent study, using multi-target co-sputtering instead of a single mixed target, the sputtering yield was controlled more accurately, and close to stoichiometric samples have been realized [24]. The improvement of material quality has increased the total PL lifetime and we expect that the quantum efficiency could also be improved.

### 3.3. Energy transfer between Yb and Er

At the low excitation level, the PL process can be simplified to a two-level model. In steady state, the PL intensity can be expressed as the following equation [14]:

\[
I = \sigma_{\text{eff}} \varphi N_{\text{Er}} \frac{\tau_{\text{tot}}}{\tau_{\text{rad}}}
\]

where \( \sigma_{\text{eff}} \) represents the effective excitation cross section, \( \varphi \) the incident photon flux and \( N_{\text{Er}} \) the Er ion concentration. The \( \tau_{\text{tot}}/\tau_{\text{rad}} \) is the PL quantum efficiency determined in the last section. With the knowledge of the values of PL intensities \( I \), \( \varphi, N_{\text{Er}}, \tau_{\text{tot}}/\tau_{\text{rad}} \) and the ratio of the effective excitation cross section \( \sigma_{\text{eff}} \) can be determined using equation (3). Table 1 shows the normalized \( \sigma_{\text{eff}} \) at the three pumping wavelengths. \( \sigma_{\text{eff}} \) of ErYb silicate was normalized to that of ErY silicate for each pump wavelength.

To explain the dependence of \( \sigma_{\text{eff}} \) on pumping wavelength, the energy diagram is shown in figure 5. Because Y is optically inactive, the Y ion is omitted from the energy level model. Yb has only two levels \( ^2F_{5/2} \) and \( ^2F_{7/2} \). The excited state level \( ^2F_{5/2} \) is resonant with the second excited state level \( ^4I_{11/2} \) of Er.
consistent with recent studies [20, 22–24].

Proof of the sensitization effect of Er by Yb in silicates and is ErYb silicate compared with ErY silicate. This is a direct results in the larger effective excitation cross section of Er in photons directly. The difference in the excitation mechanism no such energy transfer exists and Er ions absorb the pump photons more efficiently than the Er ions, both due to its large absorption cross section and high concentration. The energy is then resonantly transferred from Yb to Er, making the excitation of Er ions more efficient. In this case, Er ions act as luminescence quenching centres for the excited Yb ions. In ErY silicate, no such energy transfer exists and Er ions absorb the pump photons directly. The difference in the excitation mechanism results in the larger effective excitation cross section of Er in ErYb silicate compared with ErY silicate. This is a direct proof of the sensitization effect of Er by Yb in silicates and is consistent with recent studies [20, 22–24].

At 488 nm and 654 nm pumping, neither Yb nor Y ions show optical absorption. In these cases, the Er ions are excited directly to the \(4F_{7/2}\) level (488 nm pumping) or the \(4I_{15/2}\) level (654 nm pumping) and successively relaxed to the lower levels until the ground level \(4I_{13/2}\), either by radiative or non-radiative decay. It was found that in Er, Y2–Si2O7 films, Er ions can act as a self-sensitizer and cut high-energy incident photons into infrared photons [14]. The cutting efficiency can be up to 400% and depends dramatically on the Er concentration. Essentially this is a cross-relaxation process between neighbouring Er ions. In our samples, the Er concentrations are much lower than the critical value for self-sensitization. Checking the PL intensity under 488 and 654 nm pumping, no dependence on the pump photon energy was found, which excludes the self-sensitization of Er ions. The smaller \(\sigma_{\text{eff}}\) of Er in ErYb silicate compared with that in ErY silicate may be explained by the energy back transfer from the excited Er ions on the \(4I_{13/2}\) level to the Yb ions on the \(2F_{7/2}\) level. At 654 nm pumping, the ratio of \(\sigma_{\text{eff}}(\text{ErYb})/\sigma_{\text{eff}}(\text{ErY})\) is 0.25, which is smaller than the ratio of 0.6 at 488 nm pumping. The non-radiative decay path of Er at the two pump wavelengths may explain the difference. As shown in figure 5, at 488 nm pumping, the \(2H_{11/2}\) and \(2S_{3/2}\) levels of Er ion are populated by non-radiative decay from \(4F_{7/2}\) level before the successive decay to the lower lying \(4F_{9/2}\) level, from where the subsequent transitions are equivalent to the 654 nm pumping. The Er ion on the \(2S_{3/2}\) level may populate the \(1I_{13/2}\) level by transfer of the extra energy to excite a Yb ion on its ground level. Thus, at 488 nm pumping, the ratio of \(\sigma_{\text{eff}}(\text{ErYb})/\sigma_{\text{eff}}(\text{ErY})\) is not as significant as that at 654 nm pumping. In addition to the energy transfer induced difference in the \(\sigma_{\text{eff}}\) of Er in ErYb/Y silicate, other possibilities may also exist. For example, the radiative transition rates of the considered transitions may vary in ErYb/Y silicate due to the change in the crystal field when changing from Y to Yb in the host.

The finding that the \(\sigma_{\text{eff}}\) of ErYb silicate is larger than that of ErY silicate only at 974 nm indicates that the efficient energy transfer between Yb and Er should be engineered by optimizing the forward process (Yb to Er) and prohibiting the reverse process (Er to Yb). The non-radiative decay rate of Er from \(1I_{13/2}\) to \(1I_{11/2}\) level should be maximized to prohibit the energy back transfer from Er to Yb. Since this non-radiative decay process is dominated by multiphonon emission, an increase in the phonon energy will increase the decay rate. For instance, a lighter RE element may be co-doped to increase the phonon energy or to act as an intermediate non-radiative decay assistant [31, 32].

4. Conclusion

PL quantum efficiency and energy transfer in ErY and ErYb silicate thin films were investigated and compared. The obtained PL quantum efficiencies of 0.28 and 0.39, for ErYb and ErY silicates respectively, can be further improved by optimizing the fabrication and post-thermal processes. The forward energy transfer from Yb to Er is characterized by a four times larger effective excitation cross section at 974 nm in ErYb silicate compared with that in ErY silicate. At pumping

Figure 5. Energy diagrams showing the energy transfer between Er and Yb for different pump wavelengths. At 974 nm pumping, the Yb absorbs incident photons and transfers the excitation to the second excited state of Er ions, resulting in a larger effective absorption cross section. For pump wavelengths of 654 and 488 nm the light is absorbed by the higher excited states of Er ions. From these higher lying levels, the excitation decays sequentially to the lower lying states and the branching is affected by energy transfer from the second excited state of Er to the Yb ions.
wavelengths of 488 nm and 654 nm, backward energy transfer from Er to Yb occurs and results in smaller effective excitation cross sections.

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