

Mixture formation of $\text{Er}_x\text{Yb}_{2-x}\text{Si}_2\text{O}_7$ and $\text{Er}_x\text{Yb}_{2-x}\text{O}_3$ for broadening the C - band in an optical amplifier on Si

Hiroo Omi^{1,2,a)}, Yoshiyuki Abe^{1,3)}, Maria Anagnosti¹⁾, and Takehiko Tawara^{1,2)}

- ¹⁾ NTT Basic Research Laboratories, NTT Corporation,
3-1 Morinosato-Wakamiya, Atsugi, Kanagawa 243-0198, Japan
Phone: (+81) 46 240 3414, ^{a)}Email: omi.hiroo@lab.ntt.co.jp
- ²⁾ Nanophotonics Center, NTT Corporation,
3-1 Morinosato-Wakamiya, Atsugi, Kanagawa 243-0198, Japan
- ³⁾ Present affiliation: Toyohashi University of Technology

1. Introduction

Erbium silicates (Er_2SiO_5 and $\text{Er}_2\text{Si}_2\text{O}_7$) and erbium oxide (Er_2O_3) have great potential as optical amplifier materials in silicon photonics [1 - 9]. Recent research has shown optical gain in waveguide erbium doped amplifiers. However, luminescence is limited by concentration quenching mechanisms, such as energy migration and up conversion between the Er^{3+} ions. Therefore, recently, yttrium (Y), which has almost the same ionic radius as erbium (Y^{3+} of 0.9 Å, Er^{3+} of 0.89 Å) and is optically inactive, has been incorporated to increase the luminescence efficiency of the Er ions in the silicates and oxides by forming $\text{Er}_x\text{Y}_{2-x}\text{SiO}_5$ [5] and $\text{Er}_x\text{Y}_{2-x}\text{O}_3$ [6]. In addition, for further enhancement of the emission, ytterbium (Yb), which ionic radius is 0.99 Å in the Yb^{3+} , has been incorporated in silicates and oxides. The incorporation of Yb promotes effectively, energy transfer from the Yb ions to the Er ions, which results in high efficiency of the Er ion luminescence [2], when pumped at the 980 nm wavelength. Significant gain has already been demonstrated in an Yb - Er silicates slot waveguides [8].

The requirement for these materials to be used as a gain medium of an optical amplifier is the broadband luminescence in the region of C - band (1530 - 1565 nm). Flat regions and broad peaks in the photoluminescence spectrum are required for broadband amplification. Therefore one of the next steps for implementing an optical amplifier is to broaden the C - band using the aforementioned materials.

In this work, a mixture of $\text{Er}_x\text{Yb}_{2-x}\text{Si}_2\text{O}_7$ and $\text{Er}_x\text{Yb}_{2-x}\text{O}_3$ has been grown on a Si wafer. It is shown that this mixture formation enhances and broadens the photoluminescence of the Er ions in the C - band region of the optical spectrum.

2. Experiments

A 10 nm - thick Er oxide was sandwiched in-between 50 nm-thick Yb oxide on Si(111) substrates by rf - magnetron sputtering at room temperature. The samples were thereafter annealed in a furnace under Ar ambience for 1 - 3 hours at temperatures (T_a) in the range of 900 - 1100 °C. Prior to the photoluminescence (PL) measurements, synchrotron grazing incidence X - ray diffraction experiments were performed at the BL24 in SPring - 8 [7]. PL measurements were mainly performed on the annealed samples with a Ti: sapphire laser at 980 nm with an excitation power of around 10 mW and temperatures between 4 and 300 K.

3. Results and discussion

Figure 1 shows a typical X-ray powder diffraction pattern obtained from the samples annealed in Ar ambience at 900, 950, 1000, and 1100 °C. The peaks in **Fig. 1(a), (b)** and **(d)** are assigned to Yb oxide, according to the joint committee for powder diffraction standards (JCPDS) cards no. 41 - 1106, indicating that the thermal annealing actually produced polycrystalline Yb_2O_3 on Si(111). The additional peaks appear in **Fig. 1(c)** at 15.11, 18.46, 21.18, and 26.12, which correspond to the (001), (-111), (200), and (130) reflections of $\text{Yb}_2\text{Si}_2\text{O}_7$ (JCPDS card no. 25 - 1345), indicating the formation of $\text{Yb}_2\text{Si}_2\text{O}_7$ in addition to Yb_2O_3 . It is worth pointing out that we could not find any diffraction peaks from Er_2O_3 at all temperatures, suggesting that the Er ions of Er_2O_3 are incorporated into the lattice sites of $\text{Yb}_2\text{Si}_2\text{O}_7$ and Yb_2O_3 by the thermal annealing. Furthermore, the size of the Yb_2O_3 is estimated to be around 20 nm at a $T_a = 1000$ °C based on the analysis of the diffraction peak width.

Figure 2(a) shows the PL spectra obtained from the samples annealed at $T_a = 900, 1000$, and 1100 °C measured at 4 K. For the sample annealed at 900 °C, the PL spectrum obtained at 4 K have two sharp peaks at the wavelengths of 1535 and 1548 nm, which are due to the transition between the $^4I_{15/2} - ^4I_{13/2}$ levels at the lattice sites of C_2 and C_{3i} . It should be noted that the spectrum is similar but not identical to that of the Er_2O_3 [9]. These results indicate that the thermal annealing at 900 °C causes Er ions to be incorporated into the two different lattice sites (C_2 and C_{3i}) of Yb_2O_3 . At $T_a = 1000$ °C, the PL spectrum has two additional peaks at the wavelengths of 1529 and 1530 nm in comparison to the sample annealed at $T_a = 900$ °C. The peaks are therefore attributed to the incorporation of the Er into the $\text{Yb}_2\text{Si}_2\text{O}_7$ based on the X - ray diffraction pattern in **Fig. 1**.

To check the origin of the two additional peaks depicted at the 1529 and 1530 nm, PL measurements were performed as a function of temperature. The behavior of the 1529/1530 nm and 1535/1548 nm peaks is quite different (not shown). The intensity at the 1529/1530 nm peak reaches a maximum point at around 100 K and then decreases rapidly with a further increase in the temperature, while the intensity at the 1535/1548 nm decreases smoothly with increasing temperature. These results possibly indicate that the emissions at 1529/1530 nm are due to the Er ions in the ytterbium silicate and the emissions at 1535/1548 nm are due to the ytterbium oxide.

Fig. 2(b) shows the PL spectra obtained from the

samples at 300 K. We plot the PL intensities and effective bandwidth (λ_{eff}) as a function of T_a in **Fig. 2(c)** and **(d)**.

The effective bandwidth was defined as:

$$\lambda_{eff} = \int I(\lambda) / I_{max},$$

Where $I(\lambda)$ is the PL intensity at the wavelength λ . As can be seen in these figures, the intensity of the PL at the main peaks of 1529, 1530 nm and 1535 nm but also the effective bandwidth is maximum when annealed at $T_a = 950$ °C. The PL is not only strong but also broad at this temperature, where the mixtures of $Er_xYb_{2-x}Si_2O_7$, and $Er_xYb_{2-x}O_3$ are formed. The two different maxima of the main peaks (1529/1530 nm in $Er_xYb_{2-x}Si_2O_7$, and 1535/1548 nm in $Er_xYb_{2-x}O_3$) should induce a considerable broadening of the PL peak in the C - band at room temperature. As a reference, we also plot the effective bandwidth as a function of T_a at 4 K in **Fig. 2(d)**. The bandwidth of the sample annealed at $T_a = 950$ °C measured at 300 K is about three times larger than that at 4 K.

We also obtained a cross-sectional high-angle annular dark field scanning transmission electron microscopy (HAADF - STEM) image of the sample annealed at 950 °C and analyzed specific regions in the HAADF - STEM image by energy dispersive X-ray spectrometer (EDS). The HAADF - STEM image shows relatively bright contrast near the interface region of the film and the EDS analysis shows that this region is Si rich. This indicates interdiffusion of the Silicon from the Si substrate into the film, which can cause silicate formation near the interface region. We on the other hand observed the preferential formation of Yb_2O_3 at the upper side of the film of the samples annealed at 950 °C.

As for the Er distribution in the film, EDX profiles show that the Er ions of the Er_2O_3 sandwiched between Yb_2O_3 films are inhomogeneously distributed in the film after the annealing at $T_a = 950$ °C. This supports the incorporation of Er into $Yb_2Si_2O_7$ and Yb_2O_3 , which produces $Er_xYb_{2-x}Si_2O_7$, and $Er_xYb_{2-x}O_3$ formations.

3. Conclusion

Mixtures of polycrystalline $Er_xYb_{2-x}Si_2O_7$ and $Er_xYb_{2-x}O_3$ films were synthesized on a Si(111) by means of rf - sputtering and subsequent thermal annealing up to 1100 °C in an Ar ambience. It is shown that the mixtures of the silicates and oxides formed at 950 °C produce a strong and broad PL spectrum in the C - band of the telecommunications wavelength.

Acknowledgement

We thank Dr. Toshiki Makimoto for useful discussions.

References

- [1] C. P. Michael *et al.*, Optics Express **16**, 19649 (2008).
- [2] X. J. Wang *et al.*, Appl. Phys. Lett. **98**, 071903 (2011).
- [3] M. Miritello *et al.*, Optics Express **19**, 20761 (2011).
- [4] J. Zheng *et al.*, Materials Letters **65**, 860 (2011).
- [5] K. Suh *et al.*, Optics Express **18**, 7724 (2010).
- [6] R. L. Savio *et al.*, J. Appl. Phys. **106**, 043512 (2009).
- [7] H. Omi *et al.*, AIP Adv. **2**, 012141 (2012).

[8] R. Guo *et al.*, Optics Letters **37**, 1427 (2012).

[9] H. Omi *et al.*, Jap. J. Appl. Phys. **51**, 02BG07 (2012).

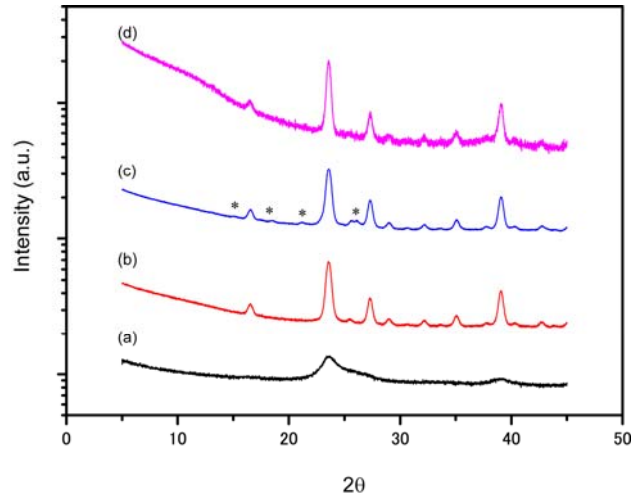


Figure 1: 0–20 X - ray powder diffraction pattern obtained at the incidence angle of 1.0° from the samples (a) as grown at room temperature annealed at (b) 900, (c) 1000 and (d) 1100 °C in an Ar ambience. The X-ray wavelength was 0.124 nm. The peaks from the di - silicate are marked by asterisks.

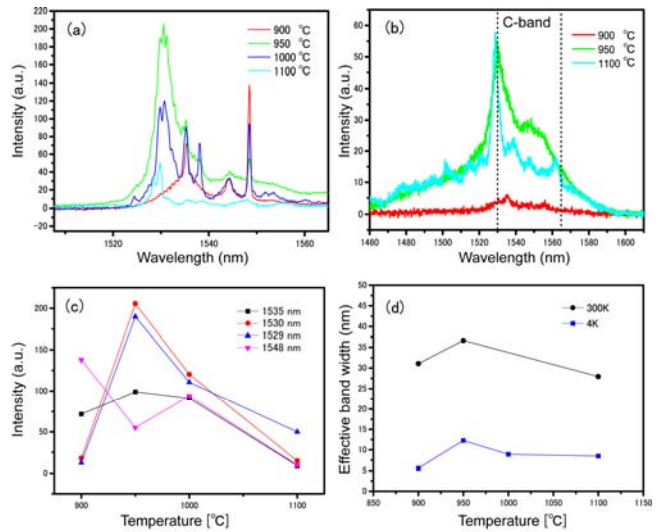


Figure 2: Photoluminescence spectra from samples annealed at $T_a = 900, 950, 1000$, and 1100 °C, obtained at (a) 4 K and (b) 300 K excited with 980 nm wavelength. (c) PL intensity from the sample annealed at $T_a = 950$ °C as a function of temperature. (d) Effective bandwidth as a function of temperature at 4 and 300 K.