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Effects of yttrium codoping on fluorescence lifetimes of Er³⁺ ions in SiO₂-Al₂O₃ sol-gel glasses

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Abstract

In order to find a new glass host and optimize erbium doping for IR glass optical amplifiers in photonic applications, a study on the optimization of the emission of erbium ions in the SiO_2 – Al_2O_3 glass by codoping with Y_2O_3 is performed. It is first attempted to make a new sol–gel glass host based on SiO_2 , Al_2O_3 , and Y_2O_3 doped with Er^{3+} ions of the composition $(1-x)SiO_2-xAl_2O_3-yY_2O_3$:0.65 Er_2O_3 (in mol%), x varies from 0 to 65, and y from 0 to 4. The optimal proportion in mol% of SiO_2 and Al_2O_3 for the Er^{3+} emission (at a fixed optimal concentration of 0.65) was 65-35. The effect of Y_2O_3 content on photoluminescence, decay curve profiles and lifetime of the $^4I_{13/2}$ level of Er^{3+} in $SiO_2-Al_2O_3$ glass is observed. The largest quantum efficiency and the higher emission intensity are observed in the sample with $65Al_2O_3$ and $4Y_2O_3$. The emission intensity at 1530 nm is two times higher than in glasses without Y_2O_3 . A shift of 3 nm to shorter wavelengths is observed. The emission spectral profiles are flatter and broader for the glasses containing Al and Y (bandwidth of 59.5 nm). The decay curves show strong difference profiles for the different samples. The increase of the lifetime value τ (about ms) of the $^4I_{13/2}$ level of Er^{3+} in the $SiO_2-Al_2O_3$ with the Y_2O_3 is discussed. © 2006 Elsevier B.V. All rights reserved.

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1. Introduction

Sol–gel erbium-doped glasses are of interest for various applications including solid state micro-sphere lasers, optical waveguides, and optical amplifiers. The erbium-doped fiber amplifier (EDFA), utilizing the emission transition ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$ of Er^{3+} ions, is a key element of the 1.5 µm telecommunication systems. Due to the rapid increase of information capacity and the need for flexible networks,

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there is an urgent demand for optical amplifiers with a wide and flat gain spectrum in the telecommunication window, to be used in the dense-wavelength-division-multiplexing (DWDM) networks. Expansion of DWDM systems thus makes the capacity and bandwidth of fiber-optic amplifiers key aspects of the system. The EDFA is made by Er³⁺-doped silica glass, which shows a narrow emission band at 1.53 μm resulting in narrow gain spectra with a bandwidth around 30 nm. The study on spectral broadening of the 1.53 μm emission of Er³⁺-doped various glasses, therefore, has been paid great attention [1–5]. For the purpose of broadening the bandwidth, to flatten the gain, we realized a study on the properties of Er³⁺ ions in the novel sol–gel multi-component glass based on SiO₂–Al₂O₃–Y₂O₃.

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Up to now, there is not yet any systematic investigation on the glasses of the composition $(100-x)\mathrm{SiO}_2$ – $x\mathrm{Al}_2\mathrm{O}_3$ – $y\mathrm{Y}_2\mathrm{O}_3$:0.65 $\mathrm{Er}_2\mathrm{O}_3$, where x varies from 0 to 65 mol%, and y from 0 to 10 mol%, in comparison with the existing results; the sample up to 10 mol% of $\mathrm{Al}_2\mathrm{O}_3$ was investigated by Nogami et al. [6] and for 50 mol% by Nedelec et al. [7]. Recently, results on Er/Yb co-activated silica–alumina monolithic glasses increasingly were published, but the advantage of a specific $\mathrm{Er}/\mathrm{Yb}/\mathrm{Al}$ glass composition for the spectroscopy properties at 1.53 μ m is not yet identified [8].

In this work, at first we study the optimal proportion of SiO₂ and Al₂O₃ for the Er³⁺ emission in the glasses doped with 0.65 mol% of Er_2O_3 (≈ 4 wt%), which is considered as the optimal concentration in pure SiO₂ sol–gel glass [9,10]. The introduction of Al₂O₃ improves rare earth dilution and prevents the micro clustering for silica glass hosts with Er³⁺ and therefore the luminescence intensity can be increased. With high concentration of alumina in glass, an exceptionally flat gain characteristic can be obtained. Y₂O₃ is added to the glass to form the composition of $(100-x)SiO_2$ $xAl_2O_3-vY_2O_3:0.65Er_2O_3 \text{ (mol\%)}. Yttrium (Y^{3+}) is chosen$ since it has a similar ionic radius and the same valence as Er³⁺ ion, therefore it can easily substitute itself and can increase the distance between two Er³⁺ ions. In this study, with only very small amount of Y₂O₃ added to the glass, a good behavior of the decay time of Er³⁺ is observed. Therefore, we will emphasize on the analysis of the decay curves and lifetimes of ${}^4I_{13/2} - {}^4I_{15/2}$ transition of Er³⁺ ions and the role of Al₂O₃ and Y₂O₃ concentrations in the glass host. The non-exponential nature of the fluorescence decay will be discussed.

2. Experimental

Using the sol–gel method as reported in previous studies [8,11], we have prepared a series of glass samples of the composition $(100-x)\mathrm{SiO}_2$ – $x\mathrm{Al}_2\mathrm{O}_3$ – $y\mathrm{Y}_2\mathrm{O}_3$:0.65 $\mathrm{Er}_2\mathrm{O}_3$ (mol%), x varies from 0 to 65, y=0, 2, 4, to 10. Volume ratios of reactants were chosen in order to realize a known total of Al (mol% SiO_2 + mol% $\mathrm{Al}_2\mathrm{O}_3$ = 1). The used chemical precursors were $\mathrm{Si}(\mathrm{OC}_2\mathrm{H}_5)_4$ (TEOS) 98% and $\mathrm{Al}(\mathrm{OC}_4\mathrm{H}_9^{\mathrm{sec}})_3$ 98% from Merk. The rare earth salts were $\mathrm{ErCl}_3 \cdot 6\mathrm{H}_2\mathrm{O}_3$, $\mathrm{Y}_2(\mathrm{CO}_3)$ 99.9% from Aldrich. The hydrolyzed mixture solution was stirred for 1 h, and then left for about 10 days to form a stiff gel of 26×230 mm. The stiff gel was dried at room temperature for 15 days, after that, it was heated in air for 2 h at 900 °C to form glass. All used glass samples in this study are transparent.

The glass samples were excited by a diode laser in the near IR range \approx 980 nm, with an output power up to 200 mW. For the decay analysis, the laser diode was pulsed with a generator at a frequency of 10 Hz and 100 μ s in width with a power energy up to 340 mW (34 μ J per pulse). The rise time is of the order of 6 μ s and the fall time of the order of 4 μ s. The IR emitted light from the sample, collected by an optical fiber located at 10 mm perpendicular

to the surface, was analyzed on the same side as the excitation with a TRIAX 320 Spectrometer and a near IR array detector Hamamatsu (256 pixel). This setup has a resolution of 1 nm/point for a slit \leq 50 μ m. The decays were analyzed by an InGaAs detector and a Nicolet 400 scope. This experimental setup has a time constant of the order of 50 μ s. All measurements in this study were performed at $T=300~\rm K$.

3. Results on the effect of Y₂O₃ concentration on the lifetime of the ⁴I_{13/2} level of Er³⁺ ions

In our previous work [11], we have analyzed the influence of the different glass compositions on the emission of Er³⁺ ions in the Near-IR region. In the glasses of (100-x)SiO₂-xAl₂O₃, with a fixed Er³⁺ concentration at 0.65 mol%, the higher emission intensity of ${}^4I_{13/2} - {}^4I_{15/2}$ transition at 1530 nm is reached for the samples containing 50 to 65 mol% in Al₂O₃. We observed also an increase in emission intensity in all glass samples containing a low amount of Y₂O₃ compared with the samples without Y₂O₃. The highest emission intensity of Er³⁺ ions is observed in the glass sample with $Al_2O_3 = 65 \text{ mol}\%$ and $Y_2O_3 = 4 \text{ mol}\%$. Fig. 1 presents the influence on the Er³⁺ emission of the Al₂O₃ concentration for an Y₂O₃ content y = 4 mol%. We analyzed the emission intensities at 1530 nm for the different compositions of Al₂O₃ and Y₂O₃. The strongest emission intensity is observed for the sample $35SiO_2-65Al_2O_3-4Y_2O_3:0.65Er_2O_3$. The emission intensity at 1530 nm is two times higher than in the glass without Y₂O₃. A shift of 3 nm to shorter wavelengths is observed. The emission spectral profiles are flatter and broader than those in SiO₂ glass. The spectral bandwidth is 59.5 nm. The same situation is observed for the series of glass samples with 2, 6, 8 and 10 mol\% Y₂O₃.

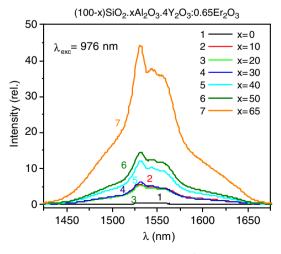


Fig. 1. Photoluminescence spectra (PL) of Er^{3+} in the glasses of the composition of $(100-x)SiO_2-xAl_2O_3-4Y_2O_3$: 0.65 Er_2O_3 x=0, 10, 20, 30, 40, 50, 65 in mol%) under 976 nm excitation at 300 K, for comparison of relative fluorescence intensity of Er^{3+} ions at 1530 nm.

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