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Luminescence study and Judd-Ofelt analysis of Nd^{3+} doped lithium lanthanum borate glass for green laser device

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Abstract

Lithium lanthanum borated glasses doped with Nd^{3+} (LiLaBNd) were synthesized to study in luminescence properties and laser potential. Refractive index, density and molar volume of glass increase with increasing of Nd^{3+} concentration. Glasses absorb photon in visible light and near- infrared region corresponding to energy transition from $^4I_{9/2}$ ground state of Nd^{3+}. Covalent nature and interaction between Nd^{3+} and ligand result to small red shift of Nd^{3+} energy level. The strongest emission with 1059 nm corresponds to $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition of Nd^{3+} under 808 nm excitation. The optimum concentration of Nd_2O_3 for this glass is 0.50 mol% which creates the maximum intensity of emission. Luminescence decay curve indicated cross relaxation process between $Nd^{3+}$with low probability and life time value as 114 microsecond for 0.50 mol% doped glass. Judd-Ofelt analysis was used to evaluate several radiative parameters. Narrow effective bandwidth, high stimulated emission cross section and branching ratio indicated the advantage potential to develop LiLaBNd glass for using as laser medium in green emitting laser device.

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Keywords: Borate glass; Neodymium; Luminescence

1. Introduction

In recent years, laser devices have been developed to use in variety of human activities. Since easy work as presentation pointer until complicated functional works in surgery, communication, holography, distance identification and movement sensor, laser devices encourage to rise performance of these works. A component that

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originates light in solid state laser device called laser gain medium, often made from crystal material doped with lanthanide ion (Ln^{3+}) such as well-known Nd:YAG crystal in commercial laser device [1]. But from complicated and long-time preparation of crystal material, production of crystal laser then consumes high cost. To solve this problem, it need to find out another material that can be doped with Ln^{3+} and high transparency like crystal, but take easier process and shorter time of synthesis. The preferential one is glass material. Lithium borate glass doped with Ln^{3+} is very attractive for using as laser medium because of its high strength atomic bond, high transparency, low melting point, high thermal stability, non-hygroscopic and good Ln^{3+} solubility [2,3]. This glass can be improved properties by taking some elements into glass network. It was found that lanthanum (La) addition can enhance density, hardness, melting point, refractive index and chemical durability of glass material [4-6]. Moreover, high weight of lanthanum can help to reduce the phonon vibration, that degrade luminescence property, in borate glass. From endurance characteristic, lithium lanthanum borate glass (LiLaB) should be developed for using as medium in laser or in another photonic device with stable luminescence in high pressure, temperature and humidity circumstance. LiLaB glass then were used as host material for $Dy^{3+}$to study in previous research [7,8]. Neodymium ion (Nd^{3+}) is one of the effective activator in Ln^{3+} group. Since its multiple strong absorption band in ultraviolet (UV) to near-infrared (NIR) range for efficient pumping, high stimulated emission cross-section and suitable energy level for low laser threshold, it was then used as luminescence center in solid state laser, temperature sensor and color display [9,10]. The significant transition of Nd^{3+} is $^4F_{3/2}\rightarrow{}^4I_{11/2}$corresponding to 1.06 μm emission. This NIR emitting can be applied to non-linear optical material for doubling frequency generation by second harmonic process [11], which reduce wavelength to 530 nm for using in green light laser. Therefore, Nd^{3+} doped glasses were study and developed widely to reach the best glass for laser application such as P_2O_5–K_2O–Al_2O_3–CaO–CaF_2:Nd^{3+} [9], P_2O_5–AlF_3–BaF_2–SrF_2–PbO–Li_2O:Nd^{3+} [12], TeO_2–Nb_2O_5–WO_3:Nd^{3+} [13], TeO_2–PbF_2–AlF_3:Nd^{3+} [14], P_2O_5-K_2O-Al_2O_3-BaO-PbO:Nd^{3+} [15], TeO_2-P_2O_5-Al_2O_3-K_2O-La_2O_3:Nd^{3+} [16], B_2O_3-SiO_2-Gd_2O_3-CaO:Nd^{3+} [17], SiO_2-Bi_2O_3-Li_2O:Nd^{3+} [18].

In this work, lithium lanthanum borate glasses doped with neodymium ion (LiLaBNd) were prepared to investigate potential for using this glass as laser medium. Physical, optical and luminescence properties of glass were studied under influence of Nd_2O_3 concentration. Finally, Judd-Ofelt theory was use to analyzed laser potential through various parameters.

2. Experiment

LiLaBNd glasses were synthesized by melt quenching technique with system $60Li_2O$-$10La_2O_3$-$(30-x)B_2O_3$-xNd_2O_3 where x is 0.05, 0.10, 0.50, 1.00 and 1.50 mol%. The high purity chemicals composing ofLi_2CO_3, La_2O_3, H_3BO_3 and Nd_2O_3were mixed thoroughly in alumina crucible for 10 g total weight. Mixture chemicals were melted at 1100 °C for 3 hours in an electrical furnace. Then, glassy melts were air quenched by pouring on preheated 300 °C stainless steel mold to arrange rectangular shape and subsequently annealed at 300 °C for 3 hours, to reduce thermal strain. Obtained glasses were cut and polished to be high transparency and smooth surfaces for comfortable measurement. All samples were study density (ρ) by using 4-digit sensitive microbalance (A&D, HR-200) with Archimedes principle via immersion water. The molar volume (V_M) was calculated using the relation, $V_M = M_T/\rho$, where M_T is total molecular weight of the chemical multi-component in glass. Refractive index (n) of glass was measured by using an Abbe refractometer at sodium wavelength (589.3 nm) with mono-bromonapthalene ($C_{10}H_7Br$) as a contact liquid.

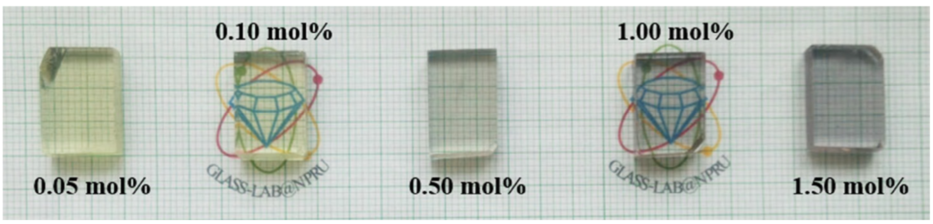

Fig. 1. The LiLaBNd glasses with different Nd_2O_3 concentration.

Absorption spectra of glass were investigated by UV-VIS-NIR spectrophotometer (Shimadzu, UV-3600). Emission spectra and decay curve were monitored by using phosphorescence/fluorescence spectrometer (Edinburgh, FLS980) with 808 diode laser as a excitation source. Judd-Ofelt (J-O) theory were used to evaluated oscillator strength (f), J-O intensity parameter (Ω_2, Ω_4, Ω_6), radiative transition possibility (A_R), stimulated emission cross section (σ_R) and branching ratio (β_R). These parameters led to laser potential analysis.

3. Results and discussion

3.1. Physical properties

LiLaBNd glass samples are shown in Fig. 1. All glasses are transparence and show variation of color. Light yellow color is dominant with 0.05 - 0.10 mol% of Nd_2O_3 concentration and it becomes to be more intense purple in 0.50 - 1.50 mol% range. Density, refractive index, and molar volume of glass increase with increasing of Nd_2O_3 concentration as show in Fig. 2. Since Nd_2O_3 molecular weight (336.48 g/mol) is higher than B_2O_3 (69.62 g/mol), more B_2O_3 substitution by Nd_2O_3 results to higher total molecular weight of glass that leads to increase intensity. Refractive index variation is in the same trend with density. Since $n = c/v_g$ (where c and v_g is speed of light in vacuum and in glass material, respectively), more adding heavy Nd_2O_3 results to the reduction of light velocity in glass sample. For molar volume expansion, it probably cause by modifier behavior of Nd^{3+} which create interstitial space with non-bridging oxygen (NBO) in glass network.

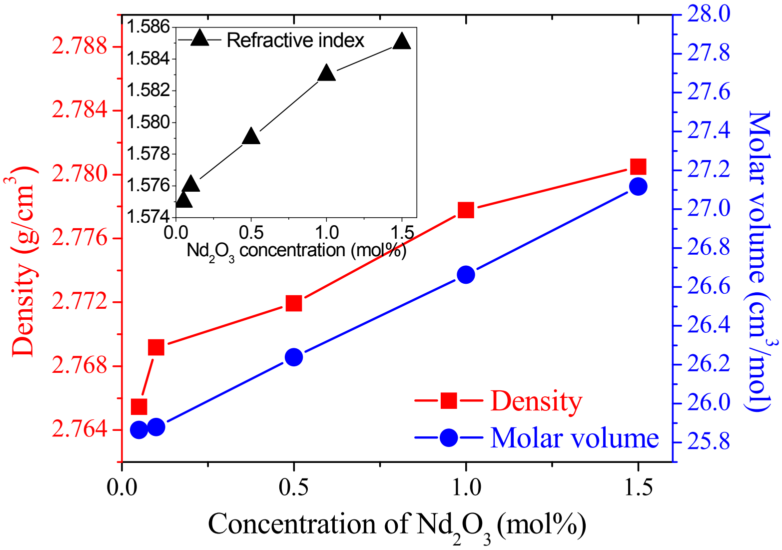

Fig. 2. The density, molar volume and refractive index (inset) of LiLaBNd glasses.

3.2. Optical absorption, oscillator strength and J-O parameter

Absorption spectra of LiLaBNd glasses are shown in Fig. 3 with eleven wavelengths of photon those can be absorbed by Nd^{3+}. Energy transition of Nd^{3+} from $^4I_{9/2}$ ground state to higher states such as $^4I_{9/2}\rightarrow{}^2P_{1/2}$, $^4I_{9/2}\rightarrow{}^4G_{11/2}$, $^4I_{9/2}\rightarrow{}^2K_{15/2}$, $^4I_{9/2}\rightarrow{}^4G_{9/2}$, $^4I_{9/2}\rightarrow{}^4G_{7/2}$, $^4I_{9/2}\rightarrow{}^4G_{5/2}$, $^4I_{9/2}\rightarrow{}^2H_{11/2}$, $^4I_{9/2}\rightarrow{}^4F_{9/2}$, $^4I_{9/2}\rightarrow{}^4F_{7/2}$, $^4I_{9/2}\rightarrow{}^4F_{5/2}$ and $^4I_{9/2}\rightarrow{}^4F_{3/2}$ corresponding to photon absorption with 434, 460, 476, 513, 528, 584, 630, 684, 748, 806 and 877 nm wavelength, respectively [19]. First, second and third absorption band is combination between absorbing of Nd^{3+} and glass host. Absorption ability of glass is better with increasing of Nd_2O_3 concentration. Visible light (VIS) with 584 nm were mostly absorbed by Nd^{3+} via hypersensitive transition $^4I_{9/2}\rightarrow{}^4G_{5/2}$, corresponding to selection rule, $|\Delta S| = 0$, $|\Delta L| \leq 2$, $|\Delta J| \leq 2$ [14, 17]. While, the second one, near-infrared (NIR) with 806 nm is often used for pumping system in Nd^{3+} laser device [15, 20]. The comparison between energy transition from absorption spectrum of Nd^{3+} in this glass (υ_c) and Nd^{3+} in aquo-ion (υ_a) [19] was exhibited by nephelauxetic ratio (β), $\beta = \upsilon_c/\upsilon_a$. [21]. It performs energy level shift of Nd^{3+} under influence of host complex. The average value of β is $\bar{\beta}$ which was then taken to evaluate the bonding parameter (δ) via relation

$$\delta = \left(\frac{1-\bar{\beta}}{\bar{\beta}}\right) \times 100, \quad (1)$$

Positive and negative value of δ indicated the covalent and ionic nature of Nd^{3+} with ligand, respectively [21]. Comparison of energy transition, β, $\bar{\beta}$ and δ of 0.5 mol% doped glass (LiLaBNd0.50) are show in Table 1. It was found small energy red shift in most transition and $\bar{\beta}$ is 0.995. That means Nd^{3+} energy level taking small effect from interaction with surround ligand. Moreover, δ is positive value as 0.491 that shows the covalent nature between Nd^{3+} and ligands.

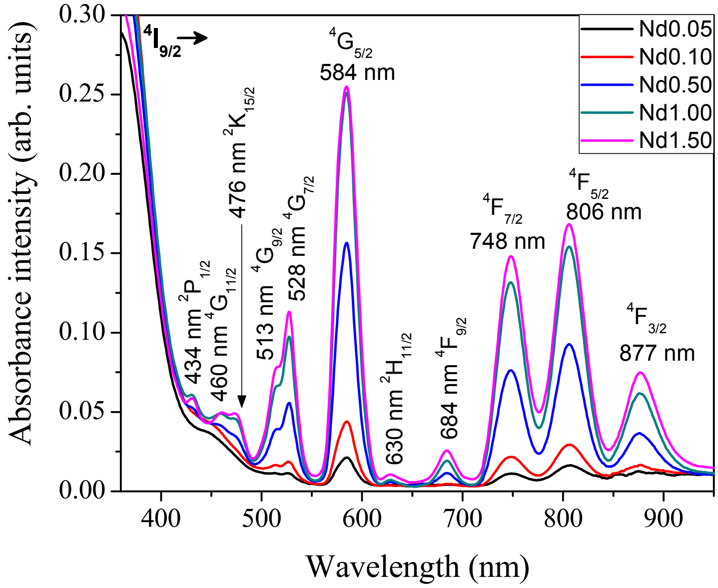

Fig. 3. The absorption spectra of LiLaBNd glasses.

Table 1. Absorption'stransition, wavelength (λ, nm), Nd^{3+} transition energy in LiLaB glass (υ_c, cm^{-1}), transition energy of Nd^{3+} aquo-ion (υ_c,cm^{-1}), nephelauxetic ratio (β), average nephelauxetic ratio ($\bar{\beta}$), bonding parameter (δ), experimental oscillator strength (f_{exp}, $x10^{-6}$), calculated oscillator strength (f_{cal}, $x10^{-6}$), root mean square (rms, $x10^{-6}$) and J-O intensity parameter (Ω_2, Ω_4, Ω_6, $x10^{-20}$ cm^2) of LiLaBNd0.50 glass.

Transition $^4I_{9/2}\rightarrow$	λ	υ_c	υ_a	β	f_{exp}	f_{cal}
$^2P_{1/2}$	434	23041	23250	0.991	0.26	-
$^4G_{11/2}$	460	21739	21650	1.004	1.17	-
$^2K_{15/2}$	476	21008	21000	1.000	0.28	0.20
$^4G_{9/2}$	513	19493	19550	0.997	1.07	0.64
$^4G_{7/2}$	528	18939	19160	0.988	1.39	1.30
$^4G_{5/2}$	584	17123	17300	0.990	6.45	6.45
$^2H_{11/2}$	630	15873	15870	1.000	0.04	0.11
$^4F_{9/2}$	684	14620	14700	0.994	0.19	0.39
$^4F_{7/2}$	748	13369	13500	0.990	2.58	1.94
$^4F_{5/2}$	806	12407	12480	0.994	2.91	3.33
$^4F_{3/2}$	876	11416	11460	0.996	0.94	0.65
$\bar{\beta}$ = 0.995					rms = 0.319	
δ = 0.491					Ω_2 = 2.267	
					Ω_4 = 0.647	
					Ω_6 =3.993	

All bands in absorption spectra were taken to find the experimental oscillator strength (f_{exp}) by using relation

$$f_{exp} = 4.318 \times 10^{-9} \int \alpha(\upsilon) d\upsilon, \quad (2)$$

where $\alpha(\upsilon)$ is molar absorption coefficient at each energy υ[17,22]. Obtained f_{exp} are shown in Table 1. The highest value of f_{exp} belongs to $^4I_{9/2}\rightarrow{}^4G_{5/2}$ (582 nm) hypersensitive transition which significantly senses with asymmetric environment of Nd^{3+}. The f_{exp} were fitted with calculated oscillator strength (f_{cal}) via equation from J-O theory

$$f_{cal} = \frac{8\pi^2 mc\upsilon}{3h(2J+1)} \frac{(n^2+2)^2}{9n} \sum_{\lambda=2,4,6} \Omega_\lambda \left(\psi J \left\| U^\lambda \right\| \acute{\psi}\acute{J}\right)^2, \quad (3)$$

where m is electron mass, c is the velocity of light, υ is transition energy, n is refractive index, h is Plank's constant, J and J′ is a total angular momentum of initial ψ and final $\acute{\psi}$ state, respectively [17,22] and $\left\|U^\lambda\right\|$ is doubly reduced matrix element of the unit tensor operator from Carnal paper [19]. With at least square fitting method, f_{cal} and Ω_2, Ω_4, Ω_6 values were obtained and shown in Table 1. Since $^4I_{9/2}\rightarrow{}^2P_{1/2}$ and $^4I_{9/2}\rightarrow{}^4G_{11/2}$transition of Nd^{3+} were effectively combined with glass host absorption, both transitions then were not taken to calculate. Fitting result gave the close value between f_{exp} and f_{cal}, except for $^4I_{9/2}\rightarrow{}^4G_{9/2}$ (513 nm) and $^4I_{9/2}\rightarrow{}^2H_{11/2}$ (630 nm) transition. Former poor fitting is a result from bands convolution with $^4I_{9/2}\rightarrow{}^4G_{9/2}$ transition, while later cause by its small and non Gaussian shape band. However, overall result is a good fitting observed from low value of root mean square as 0.319×10^{-6}. Ω_2 value from this fitting is often used to indicate the level of asymmetry environment surround Nd^{3+} and covalency between Nd^{3+} and ligand. For Ω_4 and Ω_6, both are used to introduce rigidity and viscosity of host glass [12,19].

3.3. Emission and Radiative properties

Since strong absorption at 808 nm is often used for pumping in Nd^{3+} based laser, this wavelength was used to investigate emission spectra of Nd^{3+} as show in Fig. 4. Excitation with 808 nm rise energy state of Nd^{3+} by $^4I_{9/2}\rightarrow{}^4F_{5/2}$ transition. Nd^{3+} then was decayed down to luminescence level, $^4F_{3/2}$, by non-radiative relaxation (NR) which release energy as phonon vibration, not photon emitting. After that, 3 NIR with 877, 1059 and 1333 nm were emitted by $^4F_{3/2}\rightarrow{}^4I_{9/2}$, $^4F_{3/2}\rightarrow{}^4I_{11/2}$ and $^4F_{3/2}\rightarrow{}^4I_{13/2}$ transition of Nd^{3+}, respectively [15,16,18]. All energy transition processes of emission spectra are shown Fig. 5. The emission intensity is varied as a function of Nd^{3+} amount in glass. Intensity of emission with 1059 nm increases with increasing of Nd_2O_3 concentration from 0.05 - 0.50 mol% after that it decreases via concentration quenching as show in inset of Fig. 4.

For Nd^{3+}, Ω_2 value is independent on emission with $^4F_{3/2}\rightarrow{}^4I_J$ ($\Delta J \geq 2$) because matrix element $\|U^2\|$ of these transitions is zero. Ratio of Ω_4/Ω_6 called spectroscopic quality parameter (χ) is then the significant factor to identify competition efficiency of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ (1059 nm) and $^4F_{3/2}\rightarrow{}^4I_{9/2}$ (877 nm) emission. In lithium borate glass, lower in χ value exhibits higher strength of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ than $^4F_{3/2}\rightarrow{}^4I_{9/2}$ transition [23]. Obtained χ value of LiLaBNd0.50 glass is small as 0.162. It is very consistent with stronger emission band at 1072 nm than emission band at 877 nm.Ω_2, Ω_4, Ω_6 and n value of LiLaBNd0.50 glass were used to evaluate radiative transition probability (A_R), calculated branching ratio (β_R) of each emission band and radiative lifeime (τ_R) by using these relations

$$A_R = \frac{64\pi^4\upsilon_p^3}{3h(2J+1)} \left[\frac{n(n^2+2)^2e^2}{9} \sum_{\lambda=2,4,6} \Omega_\lambda \left| \langle \psi J \left\| U^\lambda \right\| \acute{\psi}\acute{J} \rangle \right|^2 + \frac{n^3e^2h^2}{16\pi^2m^2c^2} \left| \langle \psi J \| L + 2S \| \psi' J' \rangle \right|^2 \right], \quad (4)$$

$$\beta_R = \frac{A_R}{\sum A_R}, \quad (5)$$

$$\tau_R = \frac{1}{\sum A_R}, \quad (6)$$

where $\upsilon_p$is emission energy [16,24,25]. Area per height of each emission band called effective bandwidth ($\Delta\lambda_{eff}$) and

A_R value then used to calculate stimulate emission cross section (σ_R) via equation

$$\sigma_R = \frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}} A_R, \tag{7}$$

where λ_p is emission wavelength [16,25].

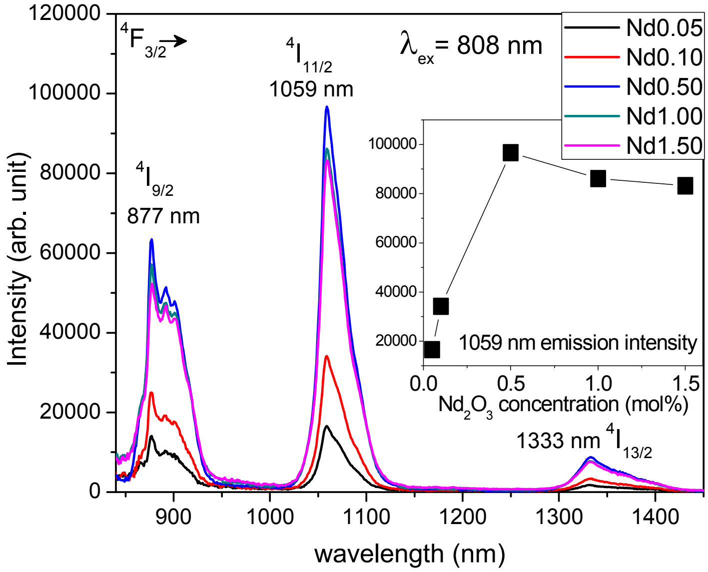

Fig. 4. The emission spectra of LiLaBNd glasses and intensity of 1059 nm emission as a function of Nd_2O_3 concentration (inset).

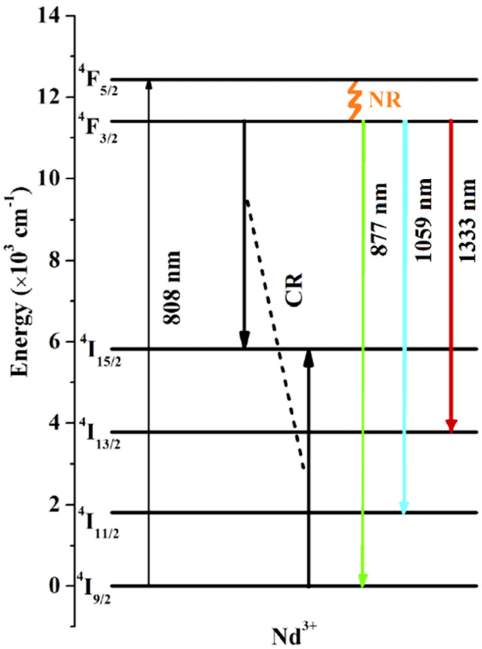

Fig. 5. Energy level diagram and energy transfer mechanism of Nd^{3+}. [18]

Table 2. Emission's transition, wavelength (λ_p, nm), Nd^{3+} energy transition (υ_p, cm^{-1}), effective bandwidth ($\Delta\lambda_{eff}$, nm), radiative transition probability (A_r, s^{-1}), stimulated emission cross section (σ_r, x10^{-20} cm^2), calculated branching ratio (β_r), experimental branching(β_{exe}) and radiative life time (τ_r, μs).

Transition $^4F_{3/2}\rightarrow$	λ_p	υ_p	$\Delta\lambda_{eff}$	A_r	σ_r	β_{exp}	β_r
$^4I_{9/2}$	877	11403	39.58	356	0.28	0.39	0.23
$^4I_{11/2}$	1059	9443	34.01	955	1.88	0.52	0.61
$^4I_{13/2}$	1333	7502	59.30	234	0.66	0.08	0.15
τ_R = 642							

Table 3. Comparison of effective bandwidth ($\Delta\lambda_{eff}$, nm), stimulated emission cross section (σ_R, x10^{-20} cm^2), calculated branching ratio (β_R) for $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition and radiative lifeime (τ_R) between LiLaNd0.50 and another Nd^{3+} doped glass from previous research.

Glass	$\Delta\lambda_{eff}$	σ_R	β_R	τ_R	Ref
LiLaBNd0.50	34.01	1.88	0.61	642	in this work
NaF-Na_2O-B_2O_3	57.20	0.50	0.43	1138	[26]
Nd: BPG	30.00	1.10	0.50	-	[27]
Phosphate	30.00	1.30	-	952	[28]
BSGdCaNd	32.87	1.39	0.45	600	[17]
TeNd25	37.60	1.67	-	258	[29]
SPB1	43.00	1.80	0.55	426	[30]
SBiLNd	34.00	2.33	0.33	290	[31]
BBONd	39.40	2.49	0.51	318	[32]
TAKLNP20	35.00	2.96	0.50	272	[16]
BiZNd	37.00	3.36	0.48	169	[33]

Value of all radiative parameters, including experimental branching ratio (β_{exp}) evaluated from area of each emission band/total area of emission band, are shown in Table 2. From lowest Δλeff and highest A_R, σ_R, β_R, and β_{exp} value compared with other emissions, 1059 nm is the best emission wavelength of this glass. This glass is then very suitable for using as laser medium that work with nonlinear optical material to generate strong green emission (~530 nm) in laser device. Some radiative parameters of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition (1059 nm) were compared with another Nd^{3+} doped glass as show in Table 3. Since lower $\Delta\lambda_{eff}$ indicates more precise in wavelength of laser, LiLaBNd0.50 glass exhibits more precise lasing wavelength about 1059 nm than most glass in Table 3. High value of σ_R performs low threshold energy to generate laser [17, 18]. Our glass then can be generated laser by pumping with less energy than most of compared glass. Since β_R value of our glass is larger than 0.50 and perhaps is the largest value in Table 3, laser created from this glass performs the highest lasing power at 1059 nm compared with other glasses in Table.

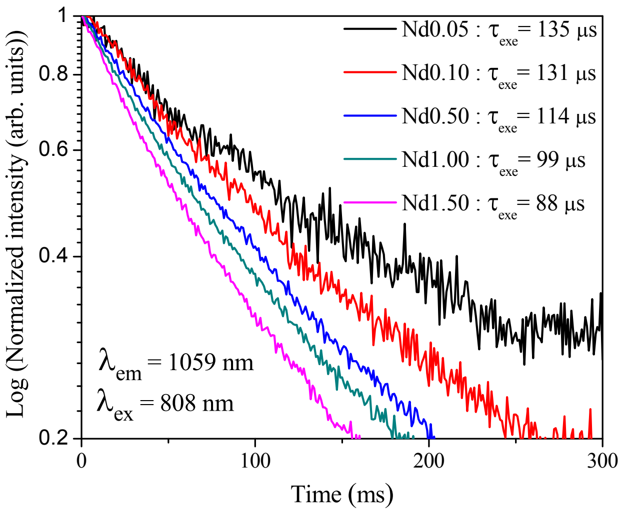

Fig. 6. The decay curve for $^4F_{3/2}$ luminescence level of Nd^{3+} in the LiLaBNd glass under 1054 nm emission and 808 nm excitation wavelength.

3.4. Decay curve

Luminescence decay curve of the LiLaBNd glass in Fig. 6 exhibit de-excitation character of Nd^{3+} from $^4F_{3/2}$ excited state. Decay curveof 1.50 mol% Nd_2O_3 doped glass (LiLaBNd1.50) shows a few behavior of non-single exponential. Obtained experimental lifetime of glass decreases from 135 – 88 µs with increasing of Nd_2O_3 concentration in 0.05 – 1.50 mol% range. From little non-single exponential behavior and lifetime reduction, it can be said that emission of Nd^{3+} in this glass is quenched by energy transfer between Nd^{3+} called cross relaxation (CR) in low probability [17,18]. Experiment lifetime value of the LiLaBNd0.50 glass is 114 µs which is lower than radiative life time, 634 µs. This lifetime different come from multi-phonon relaxation (MPR) with high phonon energy in borate glass [34]. This life time gap can be reduced by adding more heavy elements into glass network for MPR reduction. It will extend the lifetime of Nd^{3+} on $^4F_{3/2}$ luminescence level that is preferred for lasing based on stimulated emission.

4. Conclusion

LiLaBNd glasses with different Nd^{3+} concentration were prepared by melt quenching technique. Refractive index, density and molar volume of glass increase with increasing of Nd^{3+} amount in glass. Nd^{3+} probably acts as modifier in this glass network. Nd^{3+}absorbs photon in visible light and near- infrared region corresponding to energy transition from $^4I_{9/2}$ ground state. Visible light with 584 nm were mostly absorbed by hypersensitive transition $^4I_{9/2} \rightarrow {}^4G_{5/2}$. Covalent nature and interaction between Nd^{3+} and ligand result to small red shift of Nd^{3+} energy level. Emission spectra of LiLaBNd glasses were study via 808 nm laser excitation. The strongest emission belongs to photon with 1059 nm corresponding to $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition of Nd^{3+}. The optimum concentration of Nd_2O_3 for this glass is 0.50 mol% which results to the maximum intensity of emission. Decay curve indicates cross relaxation process between Nd^{3+} with low probability and microsecond order of luminescence life time value. Judd-Ofelt analysis was used to evaluate several parameter that point out the laser potential of glass. Obtained Ω_2/Ω_4 ratio is too small that is good relation with stronger 1059 nm emission ($^4F_{3/2}\rightarrow{}^4I_{11/2}$) than 877 nm emission ($^4F_{3/2}\rightarrow{}^4I_{9/2}$). Value of low effective bandwidth, high stimulated emission cross section and branching ratio of LiLaBNd0.50 glass performs the advantage potential to develop this glass for using as laser medium in green emitting solid state laser.

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