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Enhanced 1057 nm Luminescence Peak and Radiative Properties of Laser Pump Nd 3+ -doped Sodium Borate Glasses M. Djamal 1 J. Rajagukguk 1,2 , R. Hidayat 1, Suprijadi 1 and J. Kaewkhao 3 1Department of Physics, Institut Teknologi Bandung, Bandung, Indonesia (Tel : +62-22-2515032; E-mail: mitra@fi.itb.ac.id) 2Department of Physics, State University of Medan, Indonesia 20221 (Tel : +62-61-6628970; E-mail: juniastel@yahoo.com) 3Center of Excellence in Glass Technology and Materials Science, Nakhon Pathom Rajabhat University, Nakhon Pathom, Thailand 73000 (E-mail: gmink110@hotmail.com) Abstract- This paper presents a study on the spectroscopic and radiative properties of Nd 3+ ions in sodium-borate glasses (B 2O3- Na 2O-PbO-ZnO-Li 2O).

The thermal behaviors of the sample glasses were observed using thermogravimetric analysis (DTA) and a -12% total mass changes was recorded during a thermal treatment from 75 oC to 900 oC. The Judd-Ofelt parameters (?2,4,6) have been computed from the experimental and theoretical oscillator strengths, as well as from the optical absorption spectra.

Several important radiative properties such as radiative transition probability (A R), branching ratio (ßR), emission cross section (se) and fluorescence lifetime (t) of the 4F3/2 ? 4I11/2 transition have been obtained and compared with other glasses from previous studies. The calculations of emission cross section and quantum efficiency have shown that the 1.0 mol.%

Nd 3+ -doped glass makes a good potential laser candidate at the 4F3/2 ? 4I11/2 transition. Keywords: radiative properties, borate glasses, 1057 nm. I. I NTRODUCTION Physical properties of glass compositions that are used as host matrices of trivalent rare-earth neodymium (Nd3+) ions have been widely investigated in recent years [1-2].

Radiative properties such as lifetime, branching ratio, radiative transition probability and emission cross section of Nd 3+ -doped sodium borate glasses have become important parameters to determine the suitability of a laser medium and optical waveguide. The behavior of surrounding neodymium ions can in turn be affected by the chemical compositions of the glass host matrix, a change in the glass modifier on the host matrix can influence the absorption and fluorescence transition of Nd 3+ ions [3].

For example, emission bands of Nd 3+ ions in bismuth-zinc-borate glasses [1] produce 4F3/2 ? 4I11/2 transitions at 1063 nm, which is shifted slightly to 1058 nm when Nd 3+ -doped lithium fluoro- borate glasses use MgO-CaO as modifier oxides [4]. The Judd-Ofelt theory can be used to determine the effects of modifier compositions and Nd 3+ ions concentration on the spectroscopic properties of glass systems [5-6].

Judd-Ofelt calculation requires the spectroscopic properties to be obtained from quantitative characteristics of the absorption bands of glasses, analysis of the associated covalent bonding and rigidity of the medium around the Nd 3+ ions. It is well known that the radiative properties of neodymium ions depend significantly on the environment of the host glass with which the active ions are doped.

Many rare-earth (RE) ions-doped borate glass systems have been successfully developed using a materials with high fluorescence efficiency and solid-state lasers in the near-infra- red (NIR) spectral range [7-8]. In particular, borate glasses are widely used as hosts of Nd 3+ ions for several photonic applications, such as optical amplifier, solid state laser, optical waveguide and storage data [9]. The utilization of borate composition as a glass former also has advantages in optical applications.

Several of the unique properties of borate glasses include thermal stability, fully amorphous structure and the ability to generate efficient radiative transitions [10]. The effects of network modifier and glass former on the radiative properties of lead-based borate glasses [11], and fluoro-borate glasses [12] have been reported. There are three fluorescence bands that can be obtained from these glasses when doped with Nd 3+ ions, namely 4F3/2 ? 4I9/2 , 4F3/2 ? 4I11/2 , and 4F3/2 ? 4I13/2 transitions. In addition, the pattern and intensity of fluorescence spectra are found to be affected by an excitation from light sources.

A number of fluorescence experiments using Nd 3+ -doped glasses reported the use of several excitation sources such as light pulses from a laser diode (785 nm) [13], the third harmonic generation of Nd:YAG laser (355 nm) [14], a He-Cd laser at 325 nm [15], a laser diode at 800 nm to 808 nm [16-17] and a 514.5 nm argon-ion laser line [18]. In the present work, the spectroscopic and radiative properties of Nd^{3+} -doped sodium borate glass systems are investigated.

In addition, in order to get a better structure and optical quality of the borate glass systems, several network modifiers such as Na_2O, PbO, ZnO and Li_2O have been used in this work. These elements have been known to modify the behavior of the glass system, while also creating non-bridging oxygens (NBO) [19]. The focus of this study is to investigate the NIR fluorescence parameters, including the radiative transition probability (A_R), branching ratio (β_R), life time (τ_R) and stimulated emission cross section (σ_e) of Nd^{3+} -doped borate glass systems at the transition $^4F_{3/2}$ level. II.

EXPERIMENTS 248 2015 4th International Conference on Instrumentation, Communications, Information Technology, and Biomedical Engineering (ICICI-BME) Bandung, November 2-3, 2015 9 7 8 -1 -4 67 3 -7 8 00 -0 /1 5 /$ 3 1 .0 0 _ 2 0 1 5 IEEE The molar compositions of the glasses used in this study were $64.5B_2O_3$-$15Na_2O$-10PbO-5ZnO-$5Li_2O$-$(0.5)Nd_2O_3$ (BNPZLiN3), $64B_2O_3$-$15Na_2O$-10PbO-5ZnO-$5Li_2O$- $(1.0)Nd_2O_3$ (BNPZLiN4) and $63B_2O_3$-$15Na_2O$-10PbO-5ZnO- $5Li_2O$-$(2.0)Nd_2O_3$ (BNPZLiN5). Preparation and characterization procedures used corresponded to those of our earlier work [9].

Thermogravimetric analysis (TGA) with STA 449 F1 Jupiter model was employed in order to investigate the thermal behavior of the glasses. The scanning temperature ranged from 35 to 1000 ^oC with a heating rate of 24 ^oC/min under nitrogen (40 mL/min) and helium (20 mL/min) gas flow. A photo pumped laser diode at 805 nm was used in this experiment to excite electrons from the ground state to higher energy levels.

Fluorescence spectra of the glasses were monitored using UV- NIR fiber optic spectrometer in the region 350-1100 nm (Aurora4000), equipped with a 3648 pixels CCD linear array detector. Fluorescence decay time was recorded using Hamamatsu photomultiplier tube (PMT R928P) from PTI QM- 300 which was excited by a light source at 805 nm.

The absorption coefficients in cm^{-1} were determined from the optical transmission measurements by means of Beer's formula: d T T 1 2 1 ln × -= ¸ ¸ · ¨ ¨ § a (1) where T1 and T2 are relative and background transmissions (%) respectively, and d is the thickness of the glass samples (cm). The Judd-Ofelt parameters were obtained by analyzing the absorption intensity and oscillator strength as defined by the following expression [20].

Oscillator strength is dimensionless and provides an explanation regarding the intensity of the absorption band which is associated with the probability of quantum transitions. ( ) ( ) ? ?e ? ?e p d d eN mc f ³ - × = ³ = 9 10 32.4 2 2 exp (2) where fexp is the experimental oscillator strength, e and m are the electric charge and mass of the electron respectively, c is the velocity of light in vacuum, N is the concentration of dopant ion and e(?) is the coefficient of molar absorption.

The theoretical oscillator strength (fcal) along a (S,L)J manifold with a final excited level along a (S′,L′) J′ manifold is given by: ¦ = ? ? ? + + = 6,4,2 2 ''||)(|| 9 2)2 2 ( )1 2(3 2 8 ? ? ? ? p J U J n n Jh mc cal f (3) where (2 J + 1) is the diversity of the lower state, ?? (? = 2, 4, 6) are the Judd-Ofelt parameters, n is the refractive index and 2 ''||)(|| J UJ ? ? ? are the squared reduced matrix elements of the unit tensor || U(?)|| relating the initial and final states [21]. The Judd-Ofelt parameters (?2, ?4, and ?6) were then obtained using a least square program on the experimental and theoretical oscillator strengths.

The matrix elements of ||U ?|| for Nd 3+ as published by Carnall [22] was used in the calculation to determine the Judd-Ofelt parameters and oscillator strength. Using the Judd-Ofelt parameters, the oscillator strengths of electric and magnetic dipoles, corresponding to Sed and Smd respectively, can be obtained from the following equations: ( ) ( ) 2 '',')(, 6,4,2 2 JL S U JLS e ed S ? ? ? ¦ = ? = (4) and ( ) ( ) 2 '',')(, 6,4,2 222 16 22 JL S U JLS cm he md S ? ? ? p ¦ = ? = (5) The radiative transition probability (AR) from the initial level (S,L)J to the excited level (S′,L′)J′ is given by: [ ] ( ) » » º « « ª + + + = md Sn ed S nn J h c JLS JLS R A 3 9 2 2 2)1 2(3 3 34 64)')''(,),(? p (6) The radiative lifetime (tR) and fluorescence branching ratio (ßR) at the excited level (S′,L′)J′ are given by: [ ] ¦ =)')''(,),(1 JLS JLS R A R t (7) and [ ] R J LS JLS R A R t ß)')''(,),(= (8) The stimulated emission cross section (se) between the fluorescence levels (S,L)J and (S′,L′)J′ related to radiative transition probability, AR[(S,L)J, (S′,L′)J′], can be obtained from the following equation: [ ])')''(,) (2 8 4 J LS J SL R A eff cn p e ? p ? s ? = (9) where ?? eff is the effective emission bandwidth which can be calculated from the relation: ³ = ? ? ? ? ? ? ? d I I eff)( )((10) where I(?) is the emission spectrum intensity as a function of wavelength IV. RESULTS AND DISCUSSION A.

Thermal analysis Thermogravimetric analysis for all glass samples is represented by the BNPZLiN4 sample, as all samples show a similar pattern. From the thermogravimetric curve in Fig. 1, it could be deduced that the thermal treatment influences mass change. During the heating process from 68 to 1000 oC, we recorded a -12% total mass change of the BNPZLiN4 glass occurring in ten stages as shown in Table 1.

Table 1 The mass change as function of temperature of 1.0 Nd 3+ in sodium borate glass system Onset Temp. (oC) 75 91 99 117 124 146 193 204 306 357 Change mass (%) -0.84 -1.91 -1.02 -0.49 -0.39 -0.42 -0.38 -1.06 -0.76 -5.09 249 2015 4th International

Conference on Instrumentation, Communications, Information Technology, and Biomedical Engineering (ICICI-BME) Bandung, November 2-3, 2015 100 200 300 400 500 600 700 800 900 88 90 92 94 96 98 100 Mass DTG/(%/min) Temperature (oC) Mass change (%) -4.0 -3.5 -3.0 -2.5 -2.0 -1.5 -1.0 -0.5 0.0

DTG (%/min) Fig. 1. Thermogravimetric analysis of BNPZLiN4 glass 300 400 500 600 700 800 900 100 0 0.0 0.5 1.0 1.5 2.0 2.5 3.0 Absorption Int.(a.u) Wavelength (nm) 1.0 mol% Nd_Borate 4F3/2 4F5/2 4F7/2 4F9/2 2H11/2 2G7/2 ,4G5/2 4G7/2 2G9/2 2P1/2 2D5/2 4I9/2 ? Fig. 2 Optical absorption spectra of BNPZLiN4 glass at room temperature B.

Judd-Ofelts Parameters In our experiment, there were ten absorption bands that have been recorded from all of the Nd 3+ -doped glass samplesacross the wavelength range of 300 – 1000 nm as shown in Fig. 2. The optical absorption spectra occured from the 4I9/2 band as the ground state to 2D5/2 , 2P1/2 , 2G9/2 , 4G7/2 , 4G5/2 , 2H11/2 , 4F9/2 , 4F7/2 , 4F5/2 , 4F3/2 transitions associated with 354 nm, 430 nm, 475 nm, 525 nm, 582 nm, 626 nm, 680 nm, 751 nm, 804 nm and 875 nm excitations respectively.

Table 3 shows the Judd-Ofelt parameters of Nd 3+ -doped sodium borate glasses featured in this study and elsewhere [4,23,26,27,28]. It should be noted, ?6>?2>?4 for BNPZLiN3 and BNPZLiN4 glasses, whereas ?6>?4>?2 for BNPZLiN5. Higher ?6 values indicate higher polarizability results and higher covalence degree of the ligands around the Nd 3+ ions.

Higher ligand polarizability in the environment of the host matrix may in turn be caused by the presence of PbO compositions in glass structures [29]. In general, the ?2 parameter value explains the covalency bond and asymmetry of the rare-earth environment. The values of ?2 of the glass systems used in this study are higher than those of other reported glass systems, including fluoroborate and chloroborate host glasses.

This indicates the presence of covalent and asymmetry bonding in the site of rare earth ions. It is clear that for BNPZLiN3, BNPZLiN4 and BNPZLiN5 glasses, the bond of Nd 3+ ions with ligand anions in covalency is stronger than those of the other glasses as listed in Table 3.

The spectroscopic quality factor, ?, of the glasses can be determined by comparing the ?4 to ?6 values in order to analyze the emission intensity of Nd 3+ ions [19]. Smaller ? values indicate that the emission intensity of the 4F3/2 ? 4I11/2 transition is stronger than that of the 4F3/2 ? 4I9/2 transition. ? values of the BNPZLiN glasses suggest that the laser action at the 4F3/2 ? 4I11/2 transition (around 1.06 μm) maypotentially result from these glasses. Table 2.

Experimental (fexp) and theoretical (fcal) oscillator strength values (x 10 -6) for various Nd 3+ -doped borate glass systems Transitions BNPZLiN3 Nd:glass A[23] Glass B [24] Glass C [24] 4I9/2 ? ?abs (p) Energy fexp f cal f exp f cal f exp f cal f exp f cal 4D3/2 353 28,329 19.65 18.01 - - - - - - 2P1/2 430 23,256 2.47 1.64 - - 1.85 0.85 1.76 0.57 2G9/2 ,4G11/2 475 21,053 2.889 1.56 1.47 2.74 3.73 0.75 6.32 0.97 4G7/2 524 19,084 13.31 11.79 6.22 9.43 3.73 8.23 4.17 5.38 4G5/2 582 17,182 55.82 55.90 52.78 51.48 6.00 9.06 7.43 10.59 2H11/2 627 15,949 0.49 0.59 0.28 0.99 36.03 35.85 51.78 51.6

4F9/2 682 14,663 2.33 2.17 1.37 1.65 1.85 2.48 1.91 3.44 4F7/2 746 13,405 19.56 18.56 21.29 22.24 16.19 12.09 17.62 16.99 4F5/2 805 12,422 19.70 20.88 19.87 19.42 19.25 21.66 28.56 28.66 4F3/2 875 11,429 6.63 7.03 3.67 3.82 8.21 4.97 6.37 5.12 rms dev iat ion ±0.99 ±1.58 ±2.98 ±2.25 250 2015 4th International Conference on Instrumentation, Communications, Information Technology, and Biomedical Engineering (ICICI-BME) Bandung, November 2-3, 2015 Table 3. Judd-Ofelt parameters (x10 -20 cm 2) and quality factor of spectroscopic for Nd3+ doped borate glass Glass ?2 ?4 ?6 ?(?4/?6) Ref. BNPZLiN3 17.96 9.91 22.9

0.43 This work BNPZLiN4 16.56 12.36 19.41 0.64 This work BNPZLiN5 10.47 11.83 12.09 0.98 This work Pb-Glass 4.27 6.12 6.49 0.94 [4] LCB glass 10.24 5.53 10.05 0.55 [27] LSG glass 7.57 7.01 8.86 0.79 [28] Fluoroborate 4.69 5.09 6.50 0.78 [30] Chloroborate 4.84 5.31 6.32 0.84 [30] C. Radiative Properties and Decay Time In this section, the radiative properties of BNPZLiN3 are not shown because it does not produce emission when excited by LD pumping at 805 nm. Table 4 shows that the emission peak wavelengths are observed to be similar at 1057 nm and 1056 nm for BNPZLiN4 and BNPZLiN5 respectively.

In addition, the radiative transition probability of BNPZLiN4 glass is much higher than those of the other glass types, including sodium- aluminum-borate glass [31] and fluorophosphates glass [32]. This condition is also true for the fluorescence intensity, where the fluorescence intensity of BNPZLiN4 is higher than that of BNPZLiN5, as shown in Fig. 3.

The magnitude of the branching ratio, ßR, of BNPZLiN4 glass also shows a good value, being more than 50%, as a potential laser candidate for the 4F3/2 ? 4I11/2 transition. The branching ratio enhancement is in accordance with the achieved emission cross section se (?p) as presented in Table 4, where that of BNPZLiN4 is also higher than those of the other glasses. 800 850 900 950 1000 1050 1100 115 0 0 10 20 30 40 50 60 Exc. by 805 nm 4F3/2 ? 4I11/2 Fluorescence Intensity (a.u) Wavelen gth (nm) BNPZLiN4

BNPZLiN5 Fig. 3.

Fluorescence spectra of BNPZLiN4 and BNPZLiN5 glasses under excitation at 805 nm LD 150 200 250 300 350 400 450 50 0 0.0 0.2 0.4 0.6 0.8 1.0 BNPLiN3 BNPZLiN4 BNPZLiN5 Normalized Intensity (a.u) Time (μs) Fig. 4. Luminescence decay time BNPZLiN glasses for various Nd 3+ concentrations with exc. 805 nm and Em. 1060 nm From the luminescence decay curve in Fig.

4, the fluorescence lifetime (texp) can be determined using the one to four exponentials power-fit-10 lifetime. The experimental lifetime, radiative lifetime and fluorescence quantum efficiency of various Nd 3+ -doped sodium borate glass systems are listed in Table 5. The experimental life times for BNPZLiN4 and BNPZLiN5 glasses were obtained to be 56.19 μs and 40.67 μs respectively.

It is inversely proportional to the calculated lifetime (tR), which is lower for BNPZLiN4 than BNPZLiN5. The fluorescence quantum efficiency of BNPZLiN4 glass is also found to be relatively high at 62.43% compared with other glasses, suggesting high suitability for laser applications [34]. All these indicates that the 1.0 mol.%

Nd 3+ -doped 64B 2O3- 15Na 2O-10PbO-5ZnO-5Li 2O glass can be applied to generate intense fluorescence at 1.05 μm. 251 2015 4th International Conference on Instrumentation, Communications, Information Technology, and Biomedical Engineering (ICICI-BME) Bandung, November 2-3, 2015 Table 4.

Radiative properties, such as emission peak wavelength (?p), effective bandwidth (?? eff), emission cross-section (se(?p) x 10 -20), branching ratio (ßR) and radiative transition probability (A R), of various Nd 3+ -doped sodium borate glasses for the 4F3/2 ? 4I11/2 transition Glass ?p (nm) ?? eff (nm) se (?p) (cm 2) ßR (%) tR (μs) texp (μs) AR (s -1) Ref. BNPZLiN4 1057 22.93 16.03 52.18 90 56.19 5753 This work BNPZLiN5 1056 16.18 15.89 48.12 119 48.4

4041 This work NaAB 1057 - 3.1 44.0 295 59 3386 [31] Fluorophosphate 1054 28.50 4.51 36.50 - 271 1801 [32] BINLAB1 1063 14.02 9.47 52.03 409 - - [33] Table 5. Radiative lifetime (tR), experimental lifetime (texp) and fluorescence quantum efficiency (?) of various Nd 3+ -doped sodium borate glasses for the 4F3/2 ? 4I11/2 transition Glass tR (μs) tex p(μs) ?(%) Ref. BNPZLiN4 90 56.19 62.43 This work BNPZLiN5 119 48.4 40.67 This work BZBNd10 145 62.0 42.75 [1] NaAB 295 59.0 20.0

[31] Bi-5 256 67.0 26.0 [35] Bi-30 169 83.3 49.0 [35] Bi-60 131 95.8 73.0 [35] V. CONCLUSIONS The spectroscopic and radiative properties of three Nd 3+ - doped

sodium borate glass systems have been studied and analyzed. The experimental oscillator strengths for the sample glasses were investigated from absorption transitions in the wavelength range of 300 – 1000 nm.

The highest experimental and theoretical oscillator strength value is obtained at the 4I9/2 ? 4G5/2 transition and usually referred to as hypersensitive transition. This value was subsequently used to determine the ?2, ?4 and ?6 parameters. From the calculated Judd-Ofelt parameters, it can be concluded that the sample glasses are able to produce higher emission transition intensity at the 4F3/2 ? 4I11/2 transition as compared with the 4F3/2 ? 4I9/2 transition. The spectroscopic quality factors of these glasses range from 0.43-0.98.

Moreover, the calculated emission cross section and quantum efficiency indicates that the 1.0 mol.% Nd^{3+} -doped sodium borate glass system can serve as a potential laser candidate at a wavelength of 1057 nm. A CKNOWLEDGMENT The authors would like to thanks to Center of Excellence in Glass Technology and Materials Science, Nakhon Pathom Rajabhat University (NPRU) for facilities J. Rajagukguk to J-O training. REFERENCES [1] B. Shanmugavelu, V. Venkatramu, and V.V.R.K.

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