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Contributed Paper

Spectroscopic Properties of Lithium Borate Glass Material with Neodymium Ions

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ABSTRACT

We present spectroscopic properties of 81.4 B_2O_3 - 11 Li_2O - 5.1 Na_2O - 2.5 Al_2O_3 glass materials doped with different concentrations of neodymium ion. From the measured intensities of various absorption bands of these glasses, Judd-Ofelt parameters, Ω_λ, have been evaluated. The bonding environment surrounding the rare earth ion has also been discussed. With the help of Ω_λ parameters and luminescence data for various emission lines, radiative properties for different emission lines have been calculated and discussed.

The high value of σ_p for the $^4F_{3/2} \rightarrow {}^4I_{13/2}$ transition suggests that Nd^{3+} doped lithium borate glass specimen drawn in the form of a fiber with some suitable modifications (doping with high quality of Nd_2O_3) can be used as an efficient single mode fiber amplifier operating at 1.3 μm for telecommunication purposes.

Keywords: Neodymium lithium borate glass, Absorption Spectra, Fluorescence Spectra, Judd-Ofelt Parameters and Radaitive Properties

1. INTRODUCTION

Trivalent lanthanide ions doped optical glasses have been widely used in opto-electronic devices and optical communication systems [1-5]. Although glasses can be prepared by a wide variety of methods, the majority are still made by melting of batch components at high temperatures. The melting process mainly involves the selection and calculation of raw materials in a batch-basis, mixing of these materials, dehydration and thermal decomposition of the metallic salts, melting the batch materials to obtain homogeneous liquid, and finally quenching the liquid to obtain glasses. Conversion of the high temperature melt into a homogeneous liquid requires removing unmelted remnants, impurities, and bubbles. The melting process determines the quality of the glasses and should be treated as a key issue in the production of lanthanide doped optical glasses.

Systematic spectral investigation of a large number of inorganic glasses, viz., oxides [1, 6-20], Halides [1,7-8] and Chloride, Oxyhalides [21,15] and Chalcogenide [17-18] have shown that the stimulated emission cross-section, which in principal determinant of lasing action, can be varied over a large range by changing the glass network-forming and network-modifying ions. Due to the low melting point, high transparency, good solubility of rare earth ions, high thermal stabiltity and interesting spectral properties, there has been considerable interest in the study of borate based glasses over the past few years.

In the present work, a systematic study of Nd^{3+}doped lithium borate glass material has been carried out. The different dopant concentrations are expected to throw light on fluorescence quenching mechanism. It is worth mentioning that the spectroscopic properties of Nd^{3+}doped lithium borate glass materials have not been reported so far.

EXPERIMENTAL DETAILS:

The starting material of Lithium Borate glasses were used sodium carbonate, sodium borate, lithium carbonate and aluminium oxide of A. R. grade. The final composition of Neodymium doped lithium borate glass materials of the composition (in mol%) 81.4 B_2O_3 - 11 Li_2O - 5.1 Na_2O -2.5 Al_2O_3 - x Nd_2O_3, where x =0.0, 0.1, 0.3 and 0.5 were prepared by melt quenching technique [1] from reagents of analytical grade in 10 g batches. Nd_2O_3 added to the host glass was 99.99% pure. The glass materials were mixed in an agate pestle mortar for two hours and were thermally treated for 4 hours in a platinum crucible at 900 ± 25°C. Homogeneity of the melt was ensured by stirring the melt with a platinum rod from time to time. The melt was quenched by pouring it into rectangular shaped steel moulds placed on a preheated (300°C) heavy steel plate. The glass specimens so prepared were taken away after 24 hours and annealed for three hours at 250°C so as to remove stresses and to give them thermal stability and strength. Samples of the size 20×15×1.5 mm^3 were cut and polished on all sides to make the faces flat and parallel. The initial and final polishing of the samples was done with the help of fine powder of cerium oxide. These samples were again annealed at 200°C for further removing mechanical stresses developed during polishing. The glass samples so prepared were of good optical quality and were transparent.

The Characterization of the glass specimens was done to ensure the glass formation by X-ray diffraction. Optical absorption spectra were recorded at room temperature using a Hitachi double beam UV-VIS/NIR spectrophotometer model F-3010 with a resolution of 0.5 nm. The fluorescence spectra were recorded using Perkin Elmer Luminescence Spectrophotometer model LS50B. The refractive index of the glass specimens were measured on an Abbé refractometer (ATAGO3T). The densities of the materials were calculated using Archimede's principle with benzene as immersion liquid. Optical path lengths of the glass materials were measured using digital vernier calipers.

RESULTS AND DISCUSSION:

The amorphous nature of the glass material was confirmed from XRD. The various physical properties of the present glass material were collected in Table 1. An increase in the average molecular weight influences significantly both the refractive index and density. Figure 1 shows the absorption spectra of Nd^{3+} doped lithium borate glass (LBG) material in the range 350-2200nm in terms of wavelength (nm) vs absorption. The observed bands are assigned on the basis of the reported energy levels of neodymium ions in different glass hosts. Table 2 shows experimental and calculated energy with their difference for various absorption bands in Nd^{3+} doped LBG materials along with partial derivatives and zero order energies E_{0j}. These absorption bands have been used to calculate energy interaction parameters.

Table 1. Various physical properties of Nd^{3+} doped LBG materials.

Physical Properties	LBG01	LBG02	LBG03
Refractive Index n_d	1.563	1.574	1.577
Density d (g/cm^3)	5.103	6.245	7.387
Thickness t (cm)	0.310	0.360	0.368
Average molecular weight (g)	59.055	59.720	60.385
Dielectric constant (°)	2.442	2.447	2.486
Optical dielectric constant (pdt/dp)	1.442	1.447	1.486
Molar Volume Vm (g/cm3)	11.572	9.562	8.174
Reflection losses (R)	0.047	0.049	0.049
Molar Refractivity (Rm)	3.576	3.111	2.707

LBG01 - Lithium Borate Glasses doped with 0.1% of Neodymium ions
LBG02 - Lithium Borate Glasses doped with 0.3% of Neodymium ions
LBG02 - Lithium Borate Glasses doped with 0.5% of Neodymium ions

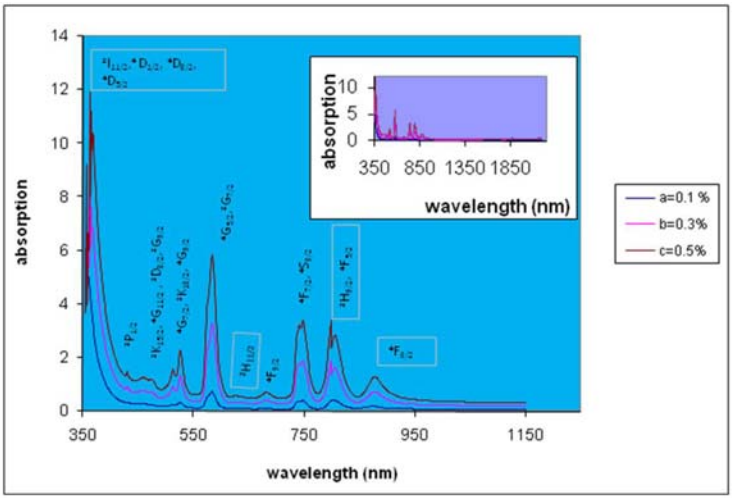

Figure 1. Absorption spectra of LBG materials with different concentration of Nd^{3+} ion

Table 2. Experimental and calculated energy E with their difference for various absorption levels in Nd^{3+} doped LBG materials along with zero order energies E_{0j}

Absorption levels	E_{Oj}	Partial derivatives				Lithium Borate glass		
		E_j/F_2	E_j/F_4	E_j/F_6	E_j/ξ_{4f}	$E_{mes}(cm^{-1})$	$E_{cal}(cm^{-1})$	ΔE
$^4F_{3/2}$	11523.3	35.27	39.90	-588.9	1.02	11455	11419.53	35.47
$^4F_{5/2}$	12606.7	34.93	39.86	-631.4	2.06	12516	12513.42	2.58
$^2H_{9/2}$	12612.0	12.59	121.28	238.2	1.30	12612	12605.61	6.39
$^4F_{7/2}$	13453.7	35.02	41.04	-602.5	3.24	13477	13374.37	101.63
$^4S_{3/2}$	13611.0	33.53	48.07	-598.6	3.54	13611	13529.64	81.36
$^4F_{9/2}$	14902.4	28.58	58.06	-382.8	5.06	14663	14877	-214.00
$^2H_{11/2}$	15980.0	9.26	123.31	406.0	5.22	16051	16059.91	-8.91
$^4G_{5/2}$	17353.6	30.09	133.23	-368.3	2.82	17123	17173.23	-50.23
$^2G_{7/2}$	17357.5	540.98	63.01	-991.2	1.29	17153.6	17241.77	-88.17
$^2K_{13/2}$	18977.7	24.99	137.34	236.8	3.01	19011	18999.9	11.1
$^4G_{7/2}$	19288.9	41.95	101.66	-620.8	4.13	19288.93	19179.26	109.67
$^4G_{9/2}$	19718.0	43.14	88.67	-723.4	45.12	19718	19610.1	107.9
$^2K_{15/2}$	2027.2	26.31	132.96	235.2	5.04	21186	21082.3	103.70
$^2G_{9/2}$	21254.0	28.18	132.02	-215.4	70.44	21254	21239.32	14.68
$^2D_{3/2}$	21248.2	40.74	85.49	239.8	2.47	21248	21329.96	-81.76
$^4G_{11/2}$	21825.0	52.28	70.75	-940.8	6.52	21825	21713.48	111.52
$^2P_{1/2}$	23147.0	42.63	93.71	226.5	3.56	23256	23236.63	19.37
$^2D_{5/2}$	23878.2	35.38	165.56	-93.8	4.80	27933	28380.64	-447.64
$^2P_{3/2}$	26349.9	41.46	78.98	329.1	7.56	———	———-	———
$^4D_{3/2}$	28640.1	85.69	112.82	-1382.8	2.13	———	———	———

RMS deviation(σ)=+143.2795

Various energy interaction parameters such as Slater Condon (F_2, F_4, F_6), Racah (E^1, E^2, E^3), Lande, (ζ_{4f}), β and $b^{1/2}$ have been computed and collected in Table 3. The relation between different F_k parameters is found to be $F_2 > F_4 > F_6$. Small values of r.m.s. deviation 'σ' between experimental energy (E_{exp}) and calculated energy (E_{cal}) of absorption bands in neodymium ion doped LBG materials justifies the suitability of the use of Taylor series expansion method.

Ten absorption bands have been observed in Nd^{3+} doped LBG materials. The assignment of these bands from ground state $^4I_{9/2}$ to the various excited states is observed in the prepared materials. From the absorption spectra, experimental oscillator strength and line strength have been calculated for all the absorption bands. The experimental and calculated line strengths along with their U matrix are given in Table 4.

Table 3. Slater-Condon, Lande, Racah, Nephlexautic and Bonding parameters forNd□ z doped LBG materials.

	Slater-Condon parameters					Lande Para-Meter	Racah parameters					Nephlau-xetic Ratio	Bonding parameter
	F_2 cm^{-1}	F_4 cm^{-1}	F_6 cm^{-1}	F_4/F_2	F_6/F_2	ζ_{4f} (cm^{-1})	E^1 cm^{-1}	E^1 cm^{-1}	E^1 cm^{-1}	E^1 cm^{-1}	E^1 cm^{-1}	β	b^{12}
Free ions	331.16	50.72	5.15	0.15	0.015	884.00	5024.00	5024.00	5024.00	5024.00	5024.00	----	----
Lithium borate	332.07	48.95	5.35	0.147	0.016	906.42	5030.94	5030.94	5030.94	5030.94	5030.94	0.988	0.035

Table 4 (a). Measured values of wavelength, U matrix and oscillator strength of Nd^{3+} doped LBG materials along with their matrix elements.

Absorption levels	Waveleng-th (nm)	U Matrix			Experimental O.S. $(P_{exp})10^{-6}$		
		$\|\|U^2\|\|^2$	$\|\|U^4\|\|^2$	$\|\|U^6\|\|^2$	0.1 %	0.3%	0.5%
^{4}F3/2	873	0.0000	0.2239	0.0549	0.343	0.35	0.385
^{2}H9/2, ^{4}F5/2	799	0.0102	0.2439	0.5124	0.839	0.841	0.881
^{4}F7/2, ^{4}S3/2	742	0.0010	0.0449	0.6597	0.834	0.839	0.846
^{4}F9/2	682	0.0009	0.0092	0.0417	0.097	0.098	0.101
^{2}H11/2	623	0.0001	0.0027	0.0104	0.094	0.097	0.099
^{4}G5/2,^{2}G7/2	584	0.9736	0.5941	0.0673	2.82	2.93	2.98
^{4}G7/2, ^{2}K13/2, ^{4}G9/2	526	0.0664	0.2180	0.1271	0.661	0.672	0.691
^{2}K15/2, ^{4}G11/2, ^{2}D3/2,^{2}G9/2	472	0.0010	0.0441	0.0364	0.003	0.003	0.003
^{2}P1/2	430	0.0000	0.0367	0.0000	0.174	0.175	0.182
^{2}I11/2, ^{4}D1/2, ^{4}D3/2, ^{4}D5/2	358	0.0050	0.5257	0.0479	0.191	0.195	0.205

Table 4 (b). Experimental and calculated line strengths of Nd^{3+} doped LBG materials along with their matrix elements.

Absorption levels	Line Strengths					
	0.1%		0.3%		0.5%	
	S_{exp} 10^{-20}	S_{cal} 10^{-20}	S_{exp} 10^{-20}	S_{cal} 10^{-20}	S_{exp} 10^{-20}	S_{cal} 10^{-20}
^{4}F3/2	0.197	0.051	0.200	0.052	0.219	0.055
^{2}H9/2, ^{4}F5/2	0.440	0.292	0.440	0.292	0.459	0.299
^{4}F7/2, ^{4}S3/2	0.406	0.432	0.408	0.431	0.409	0.437
^{4}F9/2	0.043	0.018	0.044	0.018	0.045	0.018
^{2}H11/2	0.038	0.004	0.039	0.004	0.040	0.004
^{4}G5/2,^{2}G7/2	1.086	1.049	1.120	1.082	1.135	1.095
^{4}G7/2, ^{2}K13/2, ^{4}G9/2	0.228	0.182	0.231	0.185	0.237	0.190
^{2}K15/2, ^{4}G11/2, ^{2}D3/2,^{2}G9/2	0.001	0.012	0.0009	0.012	0.001	0.0126
^{2}P1/2	0.049	0.011	0.049	0.012	0.051	0.011
^{2}I11/2, ^{4}D1/2, ^{4}D3/2, ^{4}D5/2	0.044	0.093	0.046	0.093	0.048	0.101

According to the Judd-Ofelt Theory, the values of Judd-Ofelt intensity (Ω_2, Ω_4 and Ω_6) parameters were computing by using the fitting approximation of the experimental oscillator strengths to the calculated oscillator strengths with respect to their electric dipole contributions of Nd^{3+} doped LBG materials. The computation of Ω_λ parameters is very important since they have been used in the calculation of laser parameters. The Values of Judd-Ofelt parameters were collected in Table 5. These parameters show the general tendency $\Omega_4 < \Omega_6 < \Omega_2$. Similar trend has been observed in Nd^{3+} doped lithium cesium mixed alkali borate glasses [22] and also in Sm^{3+}: CaO-Li_2O-B_2O_3-BaO glass and codoped Sm^{3+}:Eu^{3+} [23]. From table it is clear that Ω_2, Ω_4 and Ω_6 increases with increasing Nd^{3+} concentration. In this paper omega parameters have been compared with other reported glasses and were collected in Table 6. Table 6 shows similar trend in omega parameters.

Table 5. Judd-Ofelt intensity parameters for Nd^{3+} doped lithium borate glass materials.

Lithium borate glass with	$\Omega_2 10^{-20})$	$\Omega_4(10^{-20})$	$\Omega_6(10^{-20})$	Ω_4/Ω_6
LBG01	0.8472127	0.1332411	0.3860727	0.34512
LBG02	0.8793065	0.133623	0.3854247	0.34669
LBG03	0.879378	0.1489517	0.3892799	0.38263

Table 6. Comparison of Judd-Ofelt Parameters with other Reported Glasses.

Glass Matrix	$\Omega_2 10^{-20})$	$\Omega_4(10^{-20})$	$\Omega_6(10^{-20})$
LBG01(present)	0.8472127	0.1332411	0.3860727
LBG02 (present)	0.8793065	0.133623	0.3854247
LBG03 (present)	0.879378	0.1489517	0.3892799
NGBNd [33]	5.75(0.30)	3.44(0.17)	3.73(0.19)
Alumino-borosilicate glass [34]	5.52	3.45	4.85
Nd^{3+}:GC[35]	5.2	3.7	4.0

The fluorescence spectrum of Nd^{3+} (0.5%) doped LBG material has been presented in Figure 2 in terms of wavelength (nm) vs relative fluorescence in arbitrary units (a.u).

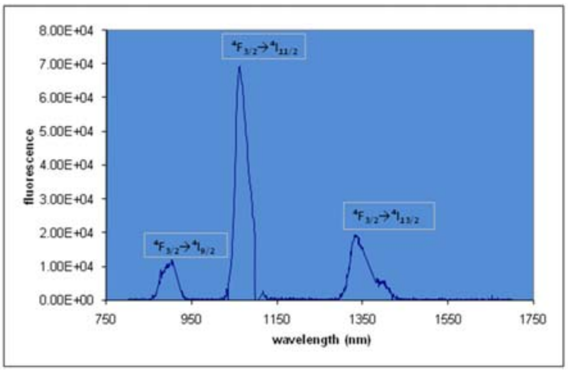

Figure 2. Fluorescence Spectrum of Nd3+(0.5%) doped LBG material in wavelength range (650-2050) nm.

The radiative properties of neodymium ion have been theoretically studied by Krupke [27] and Other [28]. These properties often called laser parameters like spontaneous emission probability (A), branching ratio (β), radiative life time (τ), and stimulated emission cross-section (σ_p) and calculated with the help of emission wave length and reduced matrix elements [29,30]. These properties were collected in Table 7.

Table 7. Spontaneous emission probability (A), fluorescence branching ratio (β), radiative life time(τ), effective line width (Δλ) and emission cross section (σ_p) for lithium borate glass materials with different concentration of Nd^{3+}.

Lithium borate glass with	Transitions	(λ) nm	A (sec-1)	B	τ (μ sec)	$\Delta\lambda_{eff}$ (nm)	σ_p (10^{-22}) (cm^2)
LBG01	$^4F_{3/2}\rightarrow{}^4I_{9/2}$	906	43.18	0.5182	23159	29	5.45
	$^4F_{3/2}\rightarrow{}^4I_{11/2}$	1063	26.73	0.3210	37406	19	9.76
	$^4F_{3/2}\rightarrow{}^4I_{13/2}$	1338	13.41	0.161	74595	29	8.05
LBG02	$^4F_{3/2}\rightarrow{}^4I_{9/2}$	906	43.61	0.517	22928	29	5.48
	$^4F_{3/2}\rightarrow{}^4I_{11/2}$	1063	27.04	0.320	37032	20	9.32
	$^4F_{3/2}\rightarrow{}^4I_{13/2}$	1333	13.69	0.162	73024	24	9.74
LBG03	$^4F_{3/2}\rightarrow{}^4I_{9/2}$	910	43.17	0.518	23240	21	5.50
	$^4F_{3/2}\rightarrow{}^4I_{11/2}$	1065	27.2	0.322	37510	27	9.80
	$^4F_{3/2}\rightarrow{}^4I_{13/2}$	1341	13.4	0.162	75500	21	9.74

The values of A and β are maximum for $^4F_{3/2} \rightarrow {}^4I_{9/2}$ transition and is closely followed by $^4F_{3/2} \rightarrow {}^4I_{11/2}$ for all the neodymium doped lithium borate glasses, suggesting that, these transitions are probable laser transitions. Stimulated emission cross-section σ_p is the most important laser parameter. Its value signifies the rate of energy extraction from the laser material. From table 6, it is clear that $^4F_{3/2} \rightarrow {}^4I_{11/2}$ and

$^4F_{3/2} \rightarrow {}^4I_{13/2}$ are the most probable laser transition and $^4F_{3/2} \rightarrow {}^4I_{9/2}$ is also a laser transition.

The high value of σ_p for the $^4F_{3/2} \rightarrow {}^4I_{13/2}$ transition suggests that Nd^{3+} doped lithium borate glasses drawn in the form of a fiber with some suitable modifications (doping with high quality of Nd_2O_3) can be used as an efficient single mode fiber amplifier operating at 1.3 μm for telecommunication purposes [31]. The figure-of-merit for gain ($\sigma_p\ \tau_R$) is of the order of 6.85×10^{-23} cm^2 sec in the lithium borate glasses, which is quite comparable with the value recently reported for Nd^{3+} doped fluoroaluminate glasses [32] developed for the 1.3 μm amplifier. Such optical amplifiers easily compensate for the losses in the processing and distribution of optical signals while maintaining the high band width and low cross talk [33].

The product of FWHW (nm) and σ_p (pm^2) is observed to be ~ 344.5×10^{-28} cm^3. This is quite high as compared to those observed in silicate and phosphate glasses [34, 35]. Therefore the present glass system is a suitable optical material to be used as broad band fiber amplifier such materials have been widely used [35] in increasing the transmission capacity of wavelength division-multiplexing (WDM) systems.

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