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Luminescence Properties of Nd^{3+} Ions doped Lithium Zinc Borosilicate Glasses for Photonic Applications

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Abstract:** To investigate the luminescence properties of lithium zinc borosilicate (LZBS) glasses with concentration variation of Nd_2O_3 have been prepared by melt – quench technique. The optical absorption spectra of the LZBS Ndx (x=0.1, 0.5, 1.0 and 2.0) glasses exhibited thirteen peaks in UV-VIS-NIR regions. Form absorption bands the J-O intensity parameters Ω_2, Ω_4, and Ω_6 were evaluated. These parameters are found to follow the trend as $\Omega_6 > \Omega_2 > \Omega_4$. The luminescence spectra revealed three emission bands due to $^4F_{3/2,} \rightarrow {}^4I_{9/2,11/2,\&13/2}$ transitions of Nd^{3+}ions. It was observed that, the laser transition $^4F_{3/2} \rightarrow {}^4I_{11/2}$ at 1056 nm is more intense than the other two transitions. The intensities of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition increases with increase of Nd^{3+} ions concentration from 0.1 to 0.5 mol% and decreases at higher concentration due to the quenching effect. Radioactive transition probabilities (A_R), peak stimulated emission cross-sections (σ_e), experimental (β_{exp}) and calculated (β_R) branching ratios were determined for different emission transitions. The nature of decay curves of $^4F_{3/2}$ level for different Nd^{3+} ions concentrations in the LZBS Ndx glasses have been analyzed and the lifetimes (τ_{exp}) were found to decrease with increase of Nd^{3+} ions concentrations. The emission decay curve of the$^4F_{3/2} \rightarrow {}^4I_{11/2}$transitions single exponential for all the glasses was determined. Hence these glasses are useful as laser materials.
***Keywords:** Neodymium, Borosilicate, Luminescence, Laser materials*

I. INTRODUCTION

Rare earth ions doped glasses are most efficient activators for versatile applications in the development of optical and optoelectronic devices which include color display, sensors, solid state lasers, optical amplifiers, solid state lighting [1-7]. It is widely used in different application due to its chemical, thermal resistance and good optical transparency. Rare-earth ion doped glasses are studied in the present research due to their multi applications in the field of lasers, optical detectors, fiber amplifiers, and fluorescent display devices. Rare earth ions emit intense radiations in the UV - VIS and NIR regions due to the 4f →4f and 4f →5d transitions under different excitation wavelengths. In the present work borosilicate glasses possess excellent optical properties and physical properties include higher chemical durability and better mechanical properties. Borosilicate glass is a type of glass with silica and boron trioxide as the main glass-forming constituents. Borosilicate glasses are known for having very low coefficients of thermal expansion (~ 3×10^{-6} K^{-1} at 20 °C). The lithium zinc borosilicate glasses consists of heavy metals, such as lithium and zinc in this glass system lithium is more electropositive and ZnO play the role of modifier or glass former, hence borosilicate glasses are more attractive hosts of rare-earth ions doped glasses for photonic applications due to its low transition temperature, high refractive index and low phonon energy. Among the different rare-earth ions doped glasses Nd^{3+} ion have been extensively investigated [8-12] because of its high efficient $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition near 1060 nm. Especially Nd^{3+} ion are more attractive of its non-linear optical properties and excellent broadband laser amplifiers due to a long fluorescent lifetime, broader effective band width high optical gain bandwidth. In this paper borosilicate glasses were prepared by conventional melt quenching method. We investigated optical and physical properties of Nd^{3+} doped borosilicate glasses. The main spectroscopic parameters of lithium zinc borosilicate (LZBS) glasses is proposed for laser amplification by using J-O intensity parameters Ω_λ (λ= 2,4 and 6) were found from the experimental oscillator strengths. The radiative transition probability (A_R), experimental branching ratio (β_R), stimulated emission cross-section (σ_e), gain band width (σ_ex $\Delta\lambda_p$) , optical gain ($\sigma_e x \tau_R$) were evaluated. The quenching phenomenon of concentration variation of Nd^{3+} ions and involved mechanism have been discussed. The aim of the present work is to develop lithium zinc borosilicate glasses doped with Nd^{3+} and investigate their luminescence properties through absorption, photoluminescence (PL) for different application in the solid state lighting field.

II. EXPERIMENTAL PROCEDURE

A. Glass Compositing

LZBS glasses composition are (30-x)H_3BO_3 : 25SiO_2 : 10Al_2O_3 : 30LiF : 5ZnO : xNd_2O_3 mol% were prepared by conventional melt quenching technique with different Nd_2O_3 concentration of 0.1, 0.5, 1.0, and 2.0 mol % labeled as LZBS Nd0.1, LZBS Nd0.5, LZBS Nd1.0, and LZBS Nd2.0 as listed in **Table I**, about10g of the batch materials thoroughly mixed to get homogeneous. This mixture was taken by porcelain crucible to heat at 1200 ^{0}C in an electric furnace for 3h then the melt was poured on pre heated brass plate. After casting the glass sample was annealed at 350^{0}C for 7h to remove thermal strains and stress and it is cooled to room temperature. This glass sample was polished for good transference and spectroscopic analysis.

TABLE I
Glass compositions and coding of Ndx^{3+}: LZBS glass

B	H_3BO_3 (mol %)	SiO_2 (mol%)	Al_2O_3 (mol%)	LiF (mol%)	ZnO (mol%)	Nd_2O_3 (mol%)
LZBSNd0.0	30	25	10	30	5	0.0
LZBSNd0.1	29.9	25	10	30	5	0.1
LZBSNd0.5	29.5	25	10	30	5	0.5
LZBSNd1.0	29	25	10	30	5	1.0
LZBSNd2.0	28	25	10	30	5	2.0

B. Physical and Optical Measurements

The refractive indices (n) of the LZBS Ndx (x = 0.0, 0.1, 0.5, 1.0 and 2.0) glasses were measured using Abbe refract meter using sodium vapour lamp. Through the Archimedes'^{s} principle the densities (ρ) were determined and the other physical parameters such as ionic concentration (N), inter ionic distance (r_i) etc were determined [13] for LZBS Nd1.0 glasses which is shown in Table II.
The optical absorption spectra were recorded from 200 to 2200 nm using JASCO V-770 UV-VIS-NIR spectrophotometer and the photoluminescence, excitation and decay measurements were recorded using the FLS-3 spectrofluorimeter.

TABLE II
Physical properties such as density (d), refractive index (n), total molecular weight (w_m), concentration of Nd^{3+} ions etc., for $Nd_{1.0}$:LZBS glasses

Parameters	LZBS Nd0.1
Density (ρ)	2.650
Refractive Index(n)	1.650
Total molecular weight(w_m)	58.36
Concentration of Nd^{3+} ions (c) /cc	0.453
Oxygen packing density (O)	7.95
Polaron radius (r_P)	4.675
Field strength (F) in 10^{24}	13.72
Molar volume (V_m)	22.022
Molar refractivity (R_m)	8.032
Reflection loss (R)	6.01
Electronic polarizability (α) X 10^{-24}	3.186
Metalizaion factor (M)	0.635

III. RESULTS AND DISCUSSION

A. Optical Absorption Spectrum and Judd-Ofelt Analysis

The absorption bands of LZBS Ndx (x = 0.1, 0.5, 1.0 and 2.0) ion doped glasses are recorded in the region 300-1000 nm originated from $^4I_{9/2}$ ground state and it is observed twelve absorption bands are at 350, 357,430,462,476,512,525,583,626,681,747,803 and 875nm corresponding to $^4D_{5/2}$, $^4D_{3/2}$, $^2P_{1/2}$, $^4G_{11/2}$, $^2K_{15/2}$, $^4G_{9/2}$, $^4G_{7/2}$, $^4G_{5/2}$, $^2H_{11/2}$, $^4F_{9/2}$, $^4F_{7/2}$+ $^4S_{3/2}$, $^4F_{5/2}$ and $^4F_{3/2}$ transitions respectively shown in Fig 1.

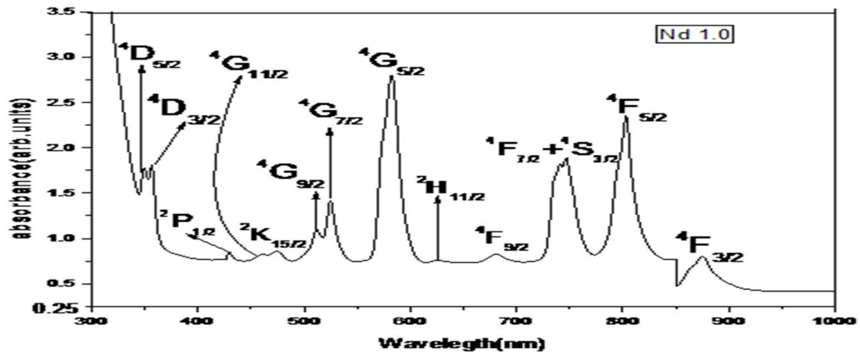

Fig 1: Optical absorption spectrum of $Nd_{0.5}$:LZBS glass in the UV-VIS-NIR regions

All above transitions are based on the assignments of Ln^{3+} spectra reported by Carnell *et al* [14]. The intensity of absorption bands is determined by using oscillation strength which is directly proportional to the area under the absorption band. These absorption spectra were assigned by comparing the band positions in the absorption spectra with those reported in previous the literature[20]. The oscillation strength of LZBS Ndx glasses are abstained in both experimental (f_{exp}) and calculated (f_{cal}). To find the nature of the covalence between the rare earth ion and its ligands as well as the radiative properties of the Nd^{3+} ions in the prepared glasses, the experimental oscillator strength (f_{exp}) of the absorption bands were determined by using the formula[13].

$$f_{\exp} = 4.32\times10^{-9}\int\varepsilon(\nu)\,d\nu$$

Where $\varepsilon(\nu)$ is the molar absorptivity of a band at a wavenumber ν (cm^{-1}) which can be calculated from the Beer-Lambert's law. The calculated oscillator strengths (f_{cal}) and the Judd – Ofel. [15,16] (J-O) intensity parameters (Ω_λ = 2, 4 and 6) are obtained from the experimental oscillator strength (f_{exp}) by the least square fitting method using the relation

$$f_{cal}(\Psi J,\Psi' J') = \frac{8\pi^2 mc\nu}{3h(2J+1)}\left[\frac{(n^2+2)^2}{9n}S_{ed}(\Psi J,\Psi' J') + nS_{md}(\Psi J,\Psi' J')\right]$$

where c is the speed of light, ν is the wavenumber, h is the Planck's constant, n is refractive index of the glass, $(n^2+2)^2/9n$ is the Lorentz local field correction factor and accounts for dipole-dipole correction and (2J+1) is the degeneracy of the ground state. The electric (S_{ed}) and magnetic (S_{md}) dipole line strengths are given by

$$S_{md}(\Psi J,\Psi' J') = \frac{e^2h^2}{16\pi^2 m^2 c^2}\left|\langle\Psi J\|(L+2S)\|\Psi' J'\rangle\right|^2 \quad S_{ed}(\Psi J,\Psi' J') = e^2\sum_{\lambda=2,4,6}\Omega_\lambda\left|\langle\Psi J\|U^\lambda\|\Psi' J'\rangle\right|^2$$

where e is the charge of electron, Ω_λ (λ= 2,4 and 6) are the J–O intensity parameters and $\|U^\lambda\|$ are the doubly reduced matrix elements of rank 'λ'. While calculating the intensity parameters in order to know the quality of fit between the f_{exp} and f_{cal} values, it is necessary to find the root mean square deviation (δ_{rms}).

The experimental spectral intensity is shown with rms deviation between experimental (f_{exp}) and calculated (f_{cal}) spectral intensities in Table III. The RMS of LZBS Ndx are very small indicating the validity of the J-O theory and it is observed that the obtained δ_{rms} is ± 0.154×10^{-6} for LZBS Nd05. By using least –squares fit method the J-O intensity parameters Ω_λ (λ= 2,4 and 6) are obtained from experimental intensities. The J-O intensity parameters of NdX: LZBS (x=0.1, 0.5, 1.0 and 2.0) trend followed $\Omega_6>\Omega_2>\Omega_4$.The parameter $\Omega_2$is related to the covalency and structural changes in the Nd^{3+} ion, the Ω_4 and Ω_6 value for LZBS NdX: (x=0.1, 0.5, 1.0 and 2.0) glasses are large indicates the stronger rigidity in nature.

In the present study the maximum fluorescence measure occurs at 0.5 mol % of Nd_2O_3 as observed for the $^4F_{3/2} \rightarrow ^4I_{11/2}$ transitions. The evaluated J-O intensity parameters for the LZBS Nd0.5 glass are $\Omega_2 = 6.85 \times 10^{-20}$, $\Omega_4 = 0.49 \times 10^{-20}$, $\Omega_6 = 10.73 \times 10^{-20}$, Similar trend has been observed for other Nd^{3+} doped host matrices such as, 33.3 Zno-66.6TeO_2 [17], 97[30 Bi_2: 70 B_2O_3] [18], Nd: KLTB [19], $Nd_{1.0}$ LBS [20], Oxy fluoroborate [21] and as compared in Table IV .

TABLE III
Assignments of absorptions transitions, their energies (cm^{-1}), experimental (f_{exp}) and calculated (f_{cal}) oscillator strengths of absorption bands of Nd^{3+} ions in $Nd_{0.5}$:LZBS glass.

Transitions	Energy	LZBS Nd0.1		LZBS Nd0.5		LZBS Nd1.0		LZBS Nd2.0	
		f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}
$^4F_{3/2}$	11428.57	2.08	2.16	0.78	0.88	1.83	1.73	2.30	2.38
$^4F_{5/2}$	12453.30	21.79	21.76	8.44	8.42	20.07	20.28	18.48	18.49
$^4F_{7/2}+^4S_{3/2}$	13386.88	14.30	14.32	5.52	5.50	13.98	13.58	11.61	11.72
$^4F_{9/2}$	14684.29	2.13	2.80	1.01	1.07	2.48	2.63	2.94	2.31
$^2H_{11/2}$	16000.00	0.76	0.76	0.33	0.28	0.73	0.70	0.63	0.62
$^4G_{5/2}$	17182.13	56.67	56.69	17.75	17.74	41.68	41.67	42.42	42.42
$^4G_{7/2}$	19047.62	3.61	3.17	1.36	1.18	2.83	2.91	2.50	2.54
$^4G_{9/2}$	19531.25	4.20	3.61	1.26	1.39	3.14	3.22	3.66	3.20
$^2K_{15/2}$	21052.63	0.88	1.34	0.39	0.51	1.24	1.25	1.40	1.12
$^4G_{11/2}$	21739.13	0.95	0.75	0.06	0.29	0.70	0.70	0.86	0.64
$^2P_{1/2}$	23255.81	0.53	0.22	0.00	0.06	0.15	0.31	1.15	0.01
$^4D_{3/2}$	28011.2	0.52	0.70	0.76	0.40	0.23	0.01	1.64	1.90
$^4D_{5/2}$	28653.3	0.53	0.85	0.11	0.29	0.72	0.65	0.97	0.74
		RMS=0.387		RMS=0.154		RMS=0.201		RMS=0.407	

TABLE IV
Comparison of Judd-Ofelt intensity parameters (Ω_λ x 10^{-20} cm^2) and their trend of Nd^{3+} ions in LZBS Nd0.5glass along with some other reported glasses

System	Ω_2	Ω_4	Ω_6	Trend	Ω_4/Ω_6	Reference
LZBS Nd0.5	6.85	0.49	10.73	$\Omega_6 > \Omega_2 > \Omega_4$	0.04	Present work
33.3 Zno-66.6TeO_2	4.24	0.88	7.05	$\Omega_6 > \Omega_2 > \Omega_4$	0.124	[17]
97[30 Bi_2: 70 B_2O_3]	4.65	2.89	5.85	$\Omega_6 > \Omega_2 > \Omega_4$	0.490	[18]
Nd: KLTB	6.83	5.39	7.29	$\Omega_6 > \Omega_2 > \Omega_4$	0.730	[19]
$Nd_{1.0}$ LBS	10.96	1.62	19.87	$\Omega_6 > \Omega_2 > \Omega_4$	0.082	[20]
Oxy fluoroborate	1.71	1.70	2.05	$\Omega_6 > \Omega_2 > \Omega_4$	0.82	[21]

B. Photoluminescence Studies

The luminescence spectra of LZBS Ndx (x = 0.1, 0.5, 1.0 and 2.0 mol %) glasses recorded by exciting with 805 nm wavelength are shown in Fig 2 in the region 800-1500 nm.

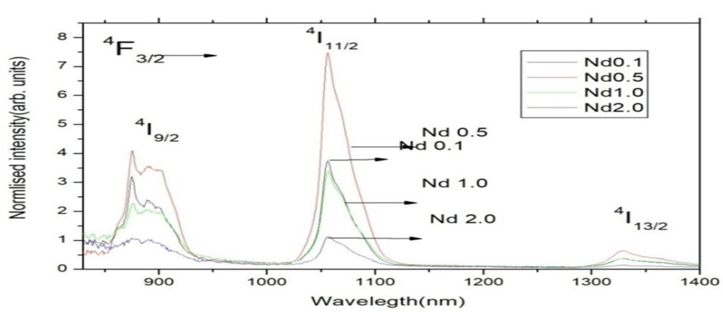

Fig 2: Photoluminescence spectra of Nd^{3+} ions in Nd_x:LZBS glasses

Due to the excitation energy the Nd^{3+} ions of ground state $^4I_{9/2}$ are excited to $^4F_{5/2}$ energy level and returns as a non-radiative to $^4F_{3/2}$ further it returns to lower levels $^4I_{9/2}$, $^4I_{11/2}$, $^4I_{13/2}$, with emitting radiation at 890, 1056 and 1332 nm respectively. From these emission spectra it is found that the transition between $^4F_{3/2}$ that is the metastable state and lower level $^4I_{11/2}$ at 1056 nm is more intense than the other two transitions. The intensity of emission bands are increased from 0.1 to 0.5 mol% then decreased with increase of Nd^{3+} ions concentrations. From this, it is concluded that, the optimum concentration as well as efficient luminescence of Nd^{3+} ions is obtained for LZBSNd0.5 glass. The concentration quenching exhibited by Nd^{3+} ions at higher concentrations could be due to the energy transfer among the excited Nd^{3+} ions [22-23].

C. Laser Characteristics Studies

Different radiative parameters determined for the $^4F_{3/2} \rightarrow {}^4I_{9/2}$, $^4F_{3/2} \rightarrow {}^4I_{11/2}$, and $^4F_{3/2} \rightarrow {}^4I_{11/2}$ emission transitions are summarized in Table V. The efficiency of laser transition mainly depends on the stimulated emission cross-section (σ_e), gain bandwidth ($\sigma_e \times \Delta\lambda_{eff}$) and optical gain parameters ($\sigma_e \times \tau_{exp}$) [23]. The experimental branching ratios (β_{exp}) are compared with the calculated branching ratios (β_R) obtained from the Judd-Ofelt analysis and there exists a good agreement between experimental (β_{exp}) and calculated (β_R) branching ratios and also laser characteristic parameters such as radiative transition probability (A_R), experimental branching ratio (β_R), stimulated emission cross-section (σ_e), gain band width (σ_ex $\Delta\lambda_p$) , optical gain (σ_exτ_R), the fluorescence quantum efficiency (η) , non-radiative relaxation rate (W_{NR}) are calculated for the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transitions in the LZBSNd0.5 glass are calculated compared with those of Nd^{3+} doped other hosts [24-30] in Table VI.

TABLE V
Laser characteristic for the emission transition of the LZBS Ndx (X=0.1,0.5,1.0 and 2.0 mol%) glasses**.**

Transitions	Lasing parameters	LZBS Nd0.1	LZBS Nd0.5	LZBS Nd1.0	LZBS Nd 2.0
$^4F_{3/2} \rightarrow {}^4I_{9/2}$	λ_P	875	875	875	875
	$\Delta\lambda_{eff}$	8.48	11.57	13.47	9.15
	σ_e	4.42	6.92	3.538	4.55
	τ_R	325	225	102	97
	σ_ex$\Delta\lambda_{eff}$	3.74	8.00	4.75	41.16
	σ_ex τ_R	14.36	15.57	3.60	4.41
$^4F_{3/2} \rightarrow {}^4I_{11/2}$	λ_P	1056	1056	1055	1057
	$\Delta\lambda_{eff}$	26.06	25.85	26.37	32.98
	σ_e	12.61	20.37	14.58	11.11
	τ_R	325	225	102	97
	σ_ex$\Delta\lambda_{eff}$	32.86	52.65	38.46	36.64
	σ_ex τ_R	40.98	45.83	14.87	10.78
$^4F_{3/2} \rightarrow {}^4I_{13/2}$	λ_P	1330	1328	1325	1331
	$\Delta\lambda_{eff}$	36.06	41.08	39.16	41.18
	σ_e	5.13	10.07	6.28	5.79
	τ_R	325	225	102	97
	σ_ex$\Delta\lambda_{eff}$	18.49	41.36	24.46	23.84
	σ_ex τ_R	16.67	22.65	6.41	5.61

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TABLE VI
Comparison of peak positions λ_p (nm), effective line widths ($\Delta\lambda_{eff}$ nm) etc., for the transitions $^4F_{3/2} \rightarrow {}^4I_{11/2}$

Glasses	λp (nm)	Δλeff (nm)	σ_e	τ_R(μs)	τ_{exp} (μs)	A_R (s^{-1})	β_R	β_{exp}	σ_ex$\Delta\lambda_{eff}$ x10^{-26}	σ_ex τ_R x10^{-25}	n	W_{nR}
[P] LZBSNd0.5	1056	25.85	20.37	225	198	8722	0.66	0.66	52.65	45.83	0.88	7638
[24]TZN Nd	1061	25.88	3.69	187	144	----		------			0.77	--------
[25] Oxyfluoride (Pb(Cd)F2) GC	----	20.00	6.88	300	------	-------	---------	--------			1.48	------
[26] PKCFAN10	-------	-------	3.42	254	237	------	0.47	0.65	--------	81.05	0.93	------
[27]silicate	1061	48.41	2.23	748	355	----	------	-------			0.47	------
[28] BINLAB1	1063	14.02	9.47	409	------	-----	0.52	0.72			------	-------
[29] PbO–2O3–Al2O3–WO3	1061	28.00	3.58	233	86	-----	-----	------			0.37	-----
[30]LBTAFNd 05	1070	34	2.60	470	230	1184	0.56	0.62	8.77	5.98	0.50	-------

D. Luminescence decay analysis

The decay profiles of $^4F_{3/2}$ excited level in LZBSNdx (x = 0.1, 0.5, 1.0 and 2.0 mol %) glasses are obtained by exciting with 806 nm wavelength and monitoring the emission at 1060 nm and it shown in Fig3.

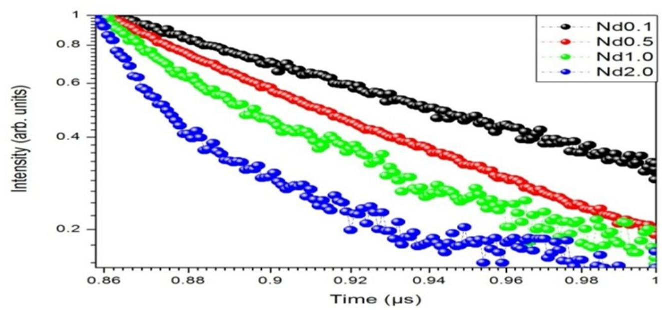

Fig3: The decay profiles for the $^4F_{3/2}$ excited level of Nd^{3+} ions in LZBS Ndx glasses

The experimental (τ_{exp}) lifetime values for the LZBSNd0.1, LZBSNd0.5, LZBSNd1.0 and LZBSNd2.0 glasses are summarized in Table VII. It is observed that the fluorescence decay curve for all Nd^{3+} doped LZBS glasses exhibits single exponential nature .The τ_{exp} was decrease with increase of Nd^{3+} ion concentration the decrease of τ_{exp} values of $^4F_{3/2}$ emission level with the increase of Nd^{3+} ions concentrations could be due to the energy transfer through non-radiative decay rates (W_{NR}) at higher concentrations. One way of evaluating the W_{NR} is given below:

$$W_{NR} = \frac{1}{\tau_{exp}} - \frac{1}{\tau_R}$$

Where τ_R and τ_{exp} are the radiative and experimental lifetimes, respectively. The evaluated values of τ_R, τ_{exp} and W_{NR} values for LZBSNdx glasses are compared with other host materials in table 7. Moreover, the fluorescence quantum efficiency (η) of an emission level $^4F_{3/2}$ can be obtained using the formula.

$$\eta = \frac{\tau_{exp}}{\tau_R}$$

TABLE VII

Experimental (τ_{exp}), radiative lifetime (τ_R), quantum efficiency (η) and non-radiative relaxation rate (W_{NR}) for the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transitions in Nd^{+3} doped LZBS glasses.

Glass	τ_{exp} (ms)	τ_R (ms)	η (%)	W_{NR} (s^{-1})
LZBSNd0.1	0.293	0.325	90	3360
LZBSNd0.5	0.192	0.225	85	7638
LZBSNd1.0	0.096	0.102	94	6127
LZBSNd2.0	0.086	0.110	78	2536

IV. CONCLUSIONS

Nd^{3+} ion doped lithium zinc borosilicate glasses (LZBS) were prepared by conventional melt quenching method, and characterized by different analytical and spectroscopic measurements. The absorption and photoluminescence spectra recorded at room temperature were analyzed with the Judd- Ofelt theory and the evaluated intensity parameters (Ω_2, Ω_4 and Ω_6) were found to be in the order $\Omega_6 > \Omega_2 > \Omega_4$ for all the LZBS Ndx: glasses. The radiative properties such as transition probabilities (A_R), total transition probabilities (A_T), branching ratios (β_R), radiative lifetimes (τ) and peak stimulated emission cross section (σ_e) were calculated for the emission transitions. The large stimulated emission cross sections observed for the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transitions suggests that, the present glasses are excellent hosts for laser active materials. The decay profiles of the $^4F_{3/2}$ excited level exhibited single exponential nature for all glasses. From these results it is conclude that 0.5mol%Nd^{3+} doped LZBS glass could be potential host for laser emission.

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