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Investigation of Nd^{3+}-doped Lithium Sodium Bismuth Borate Glasses for NIR Applications

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ABSTRACT: The Nd^{3+} doped lithium sodium bismuth borate glasses in the $60B_2O_3$+20LiF+10NaF+$10Bi_2O_3$ system with various concentrations of 0.2, 0.4, 0.6, 0.8, 1.0, 1.5 and 2.0 mol% are prepared by melt quenching technique. The structural and elemental analysis is studied using XRD, SEM and EDS spectra respectively. FTIR and Raman Spectral analysis confirm the glass-rare earth complex formation and ion-glass interactions. Optical absorption spectroscopy is investigated and Judd-Ofelt analysis is performed. Radiative rates, branching ratio and lifetime of the $^4F_{3/2}$ level of Nd^{3+} are determined. NIR photoluminescence spectra show three emission bands $^4F_{3/2} \rightarrow {}^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ under the excitation of 808 nm. Among all the concentrations of Nd^{3+} ions, 0.6 mol% Nd^{3+} contained glass samples exhibit prominent emission at 1.06 μm. From the photoluminescence analysis, 0.6 mol% Nd^{3+} contained glass samples are suggested for potential NIR luminescent device applications.

KEYWORDS: Nd^{3+} doped glass, SEM, XRD, Raman spectra, Optical absorption, Photoluminescence, lifetime.

I. INTRODUCTION

A special attention has been focused on the rare earth ions doped glasses due to their wide variety of applications in various fields such as solid state lasers, flat panel displays, planar waveguides, optoelectronic devices and high density frequency domain optical data storage systems (Shanmugavelu et al., 2014). Among various glasses, borate glasses are excellent host matrices because boric oxide (B_2O_3) acts as a good glass former and flux material (Lee et al. 2009). Borate glasses are structurally more intricate as compared to silicate or phosphate glasses due to two types of coordination of boron atoms with oxygens (3 and 4) and the structure of vitreous B_2O_3 consists of a random network of boroxyl rings and BO_3 triangles connected by B-O-B linkages. Moreover, the addition of a modifier oxide causes a progressive change of some BO_3 triangles to BO_4 tetrahedra and results in the formation of various cyclic units like diborate, triborate, tetraborate or pentaborate groups (Lin et al. 2003).

The glass containing heavy metal ions like Bi_2O_3 in borate glasses, decreases the host phonon energy and thereby improves the effective fluorescence (Mori et al. 2002) and also the addition of alkali fluoride (NaF) minimizes the phonon energy of the host glass matrix (Tanabe et al. 2000). Moreover, bismuth oxide contained host glass matrix improves chemical durability of the glass (Stambuoli et al. 2013). Despite the fact that the Bi_2O_3 is not a classical network former, it exhibits some superior physical properties like high density, high refractive index and exhibits high optical basicity, large polarizability and large nonlinear optical susceptibility (Sidebottom et al. 1997). The presence of two network forming oxides such as classical B_2O_3 and the conditional Bi_2O_3 glass former, the possible participation in the glass structure of both boron and bismuth ions with more than one stable coordination and the capability of the bismuth polyhedral and of the borate structural groups to form independent interconnected networks has gained importance (Mariappan et al. 2005). Nd^{3+} doped glasses have been extensively studied for their applications in the development of solid state laser materials. The required properties, for good laser efficiency of $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+} ion are long fluorescence lifetime and high stimulated emission cross-sections. Hence, optimization of the best ion-host combination is important since the spectroscopic parameters of the rare earth (RE) ions are strongly affected by the local structure of the RE ions and their distribution in the host matrix (Arul Rayappan et al. 2012).

In the present work, the structural, optical and luminescence properties of Nd^{3+} ion doped lithium sodium bismuth borate glasses have been studied by using the XRD, SEM, EDS, Raman, absorption and emission measurements. From

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the absorption and emission spectra, spectroscopic parameters such as the Judd-Ofelt intensity parameters Ω_λ (λ = 2, 4, 6), spontaneous radiative transition probabilities (A_R), branching ratios (β_R) and radiative lifetimes (τ_R) are calculated for various concentrations of Nd^{3+} in prepared glasses. The stimulated emission cross-sections (σ_P) and effective bandwidths (λ_{eff}) of the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ laser transition at 1060 nm obtained from the emission spectra excited at 808 nm.

II. EXPERIMENTAL STUDIES

Nd^{3+} doped lithium sodium bismuth borate (LSBiB) glass samples with compositions, (60-x) B_2O_3 + 20LiF+10NaF+10Bi_2O_3 + xNd_2O_3 (where x=0.2, 0.4, 0.6, 0.8, 1.0, 1.5 and 2.0 mol.%) are prepared by conventional melt quenching method using high purity precursor chemicals of boric acid (H_3BO_3), bismuth oxide (Bi_2O_3), lithium fluoride (LiF), sodium fluoride (NaF) and neodymium oxide (Nd_2O_3) powders. About 10 g batches of chemicals are mixed and grinded in an agate mortar to obtain homogeneous mixture. The mixture is taken into a porcelain crucible and put into an electric furnace at a temperature range of 1050-1100 °C for 45 min. Then the mixture is melted and air quenched by pouring it on a preheated brass plate. These samples are annealed at 300 °C for 3 h in order to remove strains. For all the glass samples, the physical parameters like density, thickness and refractive indices are calculated.

The XRD spectral profiles of prepared glassy samples are obtained using SEIFERT 303 TT X-ray diffractometer (with CuK_α line of 1.5405 Å), operating at 40 kV and 50 mA anode current. The SEM with EDS spectrum is measured for these prepared glasses. The FTIR spectrum is recorded using Thermo Nicolet IR200 spectrometer at room temperature (RT) in the wavenumber range 3000-400 cm^{-1}. Raman spectral profile is carried out at RT in the wavenumber range 1400-200 cm^{-1} using LabRam HR 800 confocal Raman Spectrometer with Nd: YAG laser source (532.15 nm). The optical absorption spectra are recorded for all Nd^{3+} doped glasses using JASCO V570 UV-VIS-NIR spectrophotometer. The emission spectra are recorded by exciting the sample at 808 nm with laser diode using FLS-980 Edinburgh instrument.

III. RESULTS AND DISCUSSION

A. X-ray Diffraction (XRD) and SEM with EDS Studies

Figure 3.1 illustrates the XRD pattern of lithium sodium bismuth borate glass. The presence of no sharp crystallization peak and a broad hump between 20˚ and 40˚ confirms the amorphous nature of the prepared glass. SEM image also confirms same nature and is shown Fig. 3.2. The EDS spectrum of lithium sodium bismuth borate glass is shown in Fig. 3.3. From this figure the elements that are present in the investigated glass are identified.

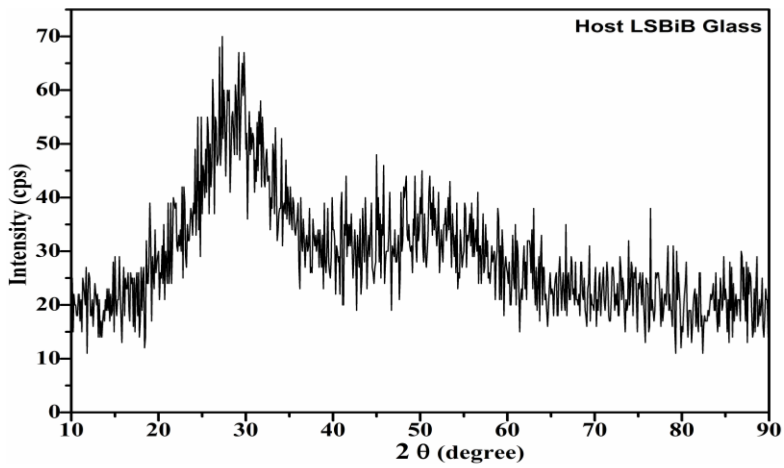

Fig. 3.1 XRD profile of the lithium sodium bismuth borate host glass matrix.

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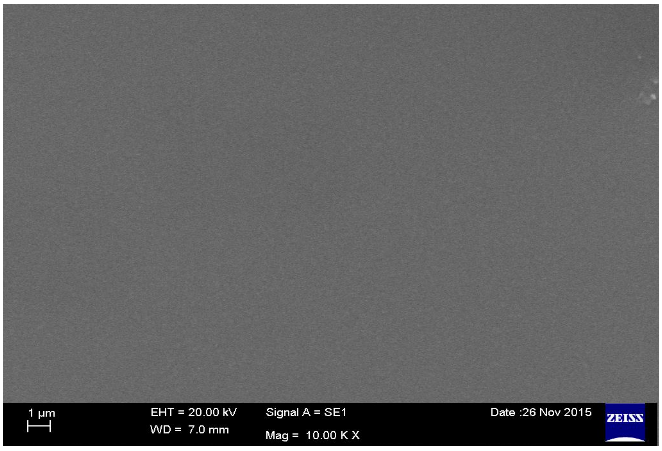

Fig. 3.2 SEM image of 0.6 mol% Nd^{3+} doped lithium sodium bismuth borate glass matrix.

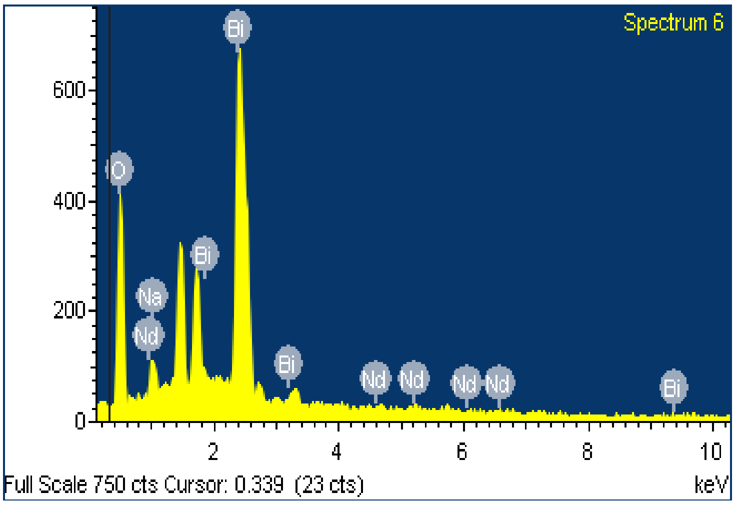

Fig. 3.3 EDS spectrum of 0.6 mol% Nd^{3+} doped lithium sodium bismuth borate glass matrix.

B. FTIR Analysis

The FTIR spectrum of host lithium sodium bismuth borate (LSBiB) glass is shown in Fig. 3.4. The spectrum reveals the characteristic peaks located at 516 cm^{-1}, 896 cm^{-1}, 1218 cm^{-1}, 1360 cm^{-1}, 1546 cm^{-1}, 1740 cm^{-1}, 2341 cm^{-1}, 3020 cm^{-1} and 3732 cm^{-1}. The broad bands are due to combination of several factors such as high degeneracy of vibrational state, thermal broadening of lattice dispersion and mechanical scattering of the sample. The infrared bands are mainly related to BO_3 and BO_4 groups. The FTIR transmission spectrum in the range of 500-520 cm^{-1} is assigned to B-O-B bending vibrations as well as borate ring deformation. The band centered at 896 cm^{-1} is assigned to B-O stretching vibrations of tetrahedral BO_4 units in different borate groups. The transmission band at 1218 cm^{-1} is specific principle signature to the B-O stretching vibrations of BO_3 triangular units with non-bridging oxygen atoms (Ivascu et al. 2011). The band at around 1360 cm^{-1} has been assigned to the stretching of trigonal BO_3 units in meta, ortho and pyro-borate groups (Elbatal et al. 2011). The FTIR spectral transmission peaks observed in the region of 2500-4000 cm^{-1} is attributed to water groups OH stretching vibrations.

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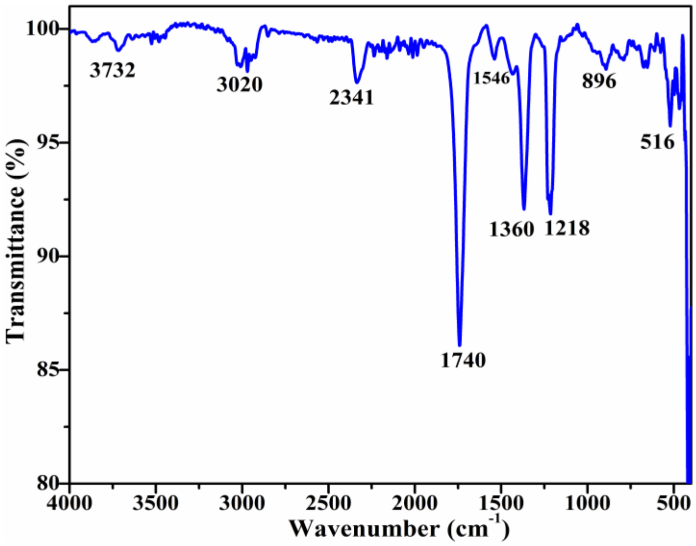

Fig. 3.4 FTIR spectrum of the lithium sodium bismuth borate host glass matrix.

C. Raman Studies

The Raman spectrum of host lithium sodium bismuth borate (LSBiB) glass is recorded and is shown in Fig. 3.5. The spectrum reveal the characteristic peaks located at 444, 747, 984 and 1273 cm^{-1}. The broad band 300-500 cm^{-1} is assigned to vibrations of Bi–O–Bi band of BiO_6 octahedral units. The band at 747 cm^{-1} is due to vibrations of chain type metaborate group. The band at 984 cm^{-1} is due to vibrations of orthoborate groups. The bands centered at 1273 cm^{-1} relate to the B–O stretching vibrations in various borate groups (Ivascu et al. 2011, Elbatal et al. 2011).

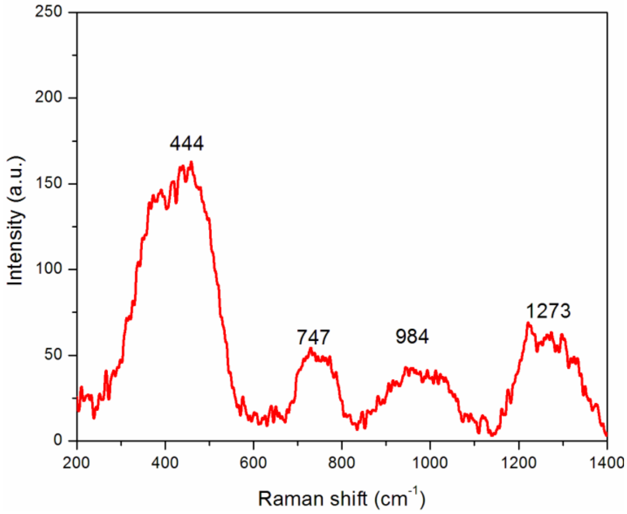

Fig. 3.5 Raman spectrum of the lithium sodium bismuth borate host glass matrix.

D. Optical absorption Spectral Analysis

Optical absorption spectra of various concentrations of Nd^{3+} doped lithium sodium bismuth borate glasses are recorded in the region 400-850 nm and are shown in Fig. 3.6. As can be seen, the spectra consist of 9 bands Nd^{3+} ion due to transitions from the ground state $^4I_{9/2}$ to various excited states, $^2P_{1/2}$, $^2D_{3/2}$+$^2G_{9/2}$, $^4G_{9/2}$+$^2K_{13/2}$, $^4G_{7/2}$, $^4G_{5/2}$, $^2H_{11/2}$, $^4F_{9/2}$, $^4F_{7/2}$+$^4S_{3/2}$ and $^4F_{5/2}$+$^2H_{9/2}$ appear at 417, 476, 511, 524, 583, 624, 680, 745 and 803 nm respectively. The spectral intensities both experimental (f_{exp}) and calculated (f_{cal}) of different absorption bands of Nd^{3+} are obtained using the formulae given in the cited reference (Carnall et al. 1968). These values are presented for 0.6 mol% of Nd^{3+} doped

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glass matrix in Table 3.1. $^4I_{9/2}\rightarrow{}^4G_{5/2}$ is the hypersensitive transition for Nd^{3+} ion (Xue et al. 2015). It follows the selection rules $\Delta J\leq 2$, $\Delta L\leq 2$ and $\Delta S=0$. The position and intensity of the hypersensitive transition are very sensitive to the environment of the rare earth ion. From Table 3.1, it is observed that the hypersensitive transition ($^4I_{9/2}\rightarrow{}^4G_{5/2}$) centered at 17153 cm^{-1} (583 nm) is the most intense one among the observed absorption bands. This band also exhibits large values of double reduced matrix elements. The accuracy of fit between the experimental and calculated spectral intensities is given by root mean square (rms) deviations. It is noticed from the table that the experimental intensities are in good agreement with the calculated values for most of the transitions indicating the validity of Judd-Ofelt theory.

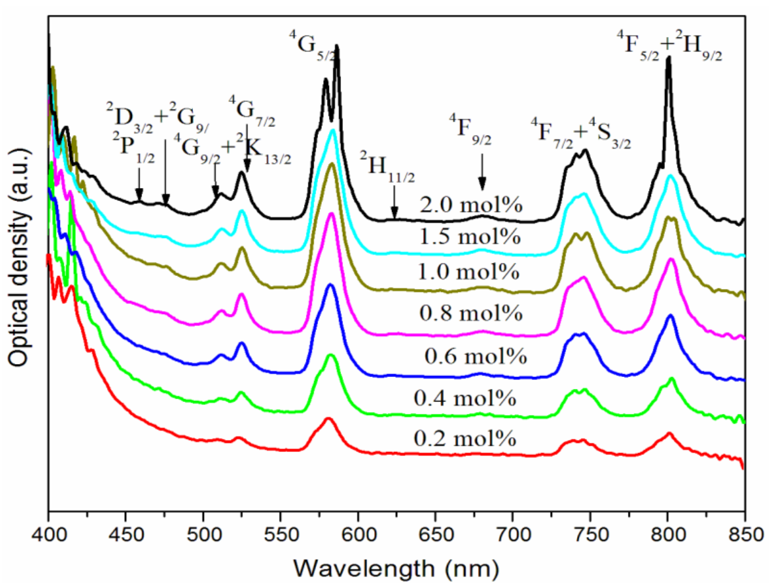

Fig. 3.6 Optical absorption spectra for various concentrations of Nd^{3+} doped lithium sodium bismuth borate glasses.

Table 3.1. Transition assignments, experimental (f_{exp}, x10^{-6}) and calculated (f_{cal}, x10^{-6}) spectral intensities of 0.6 mol% Nd^{3+} doped lithium sodium bismuth borate glass matrix.

Transition	0.6 mol%	
$^4I_{9/2}\rightarrow$	f_{exp}	f_{cal}
$^4D_{1/2}+{}^4D_{3/2}$	9.14	10.31
$^2P_{1/2}$	0.71	1.13
$^2D_{3/2}+{}^2D_{9/2}$	1.49	0.52
$^4G_{9/2}+{}^2K_{13/2}$	2.88	3.25
$^4G_{7/2}$	4.95	6.30
$^4G_{5/2}$	23.64	23.54
$^2H_{11/2}$	0.25	0.24
$^4F_{9/2}$	1.16	0.87
$^4F_{7/2}+{}^4S_{3/2}$	10.94	11.17
$^4F_{5/2}+{}^2H_{9/2}$	9.97	9.41
δ_{rms}	± 0.84	

The three Judd-Ofelt intensity parameters, the Ω_2 parameter is related with hypersensitive transition and the covalency degree of RE-O bonds (Ramachari et al. 2014), the Ω_4 and Ω_6 intensity parameters indicate the rigidity of the material (Jlassi et al. 2010). Mainly, Ω_6 parameter gives information on the electron-phonon coupling intensity between the RE^{3+} ion and anion ligands (Atul et al. 2013). Higher Ω_6 parameter indicates strong interaction between 4f and 5d orbitals. This strong electron-phonon coupling also enlarges the emission band widths (Linganna et al. 2015). In the present work, the Judd-Ofelt parameters (Ω_2, Ω_4 and Ω_6) are obtained for 0.6 mol% of Nd^{3+} doped glass matrix and are presented in Table 3.2. From the table, it is observed that among all glass matrices, 0.6 mol% Nd^{3+} doped lithium

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sodium bismuth borate glass matrix has high Ω_2 value ($5.62x10^{-20}$ cm^2) indicating higher asymmetry of Nd^{3+} sites and strong covalency of the Nd-O bonds (Ivascu et al. 2011). On the other hand, the Ω_6 parameter is also high ($6.12x10^{-2}$ cm^2) for 0.6 mol% Nd^{3+} doped lithium sodium bismuth borate glass matrix indicating strong electron-phonon coupling of the ligand field (Takebe et al. 1994) and direct influence on luminescence intensity of the $^4F_{3/2}$ to $^4I_{11/2}$ transition. The magnitudes of Ω_2 and Ω_6 parameters are higher when compared with the other reported glasses such as borate (Zou et al. 1996), phosphate (Compbell et al. 2000), tellurite (Hufner. 1978) and silicate (Dieke. 1968).

Table 3.2. Judd-Ofelt parameters (Ω_2, Ω_4 and Ω_6, x 10^{-20} cm^2) and spectroscopic quality factor ($\chi = \Omega_4/\ \Omega_6$) of 0.6 mol%$Nd^{3+}$ doped lithium sodium bismuth borate glass matrix.

Glass	Ω_2	Ω_4	Ω_6	χ	Reference
0.6 mol%	5.62	5.03	6.12	0.82	Present work
Borate	2.67	3.31	3.98	0.83	[20]
Phosphate	2.90	3.30	3.80	0.90	[21]
Tellurite	2.13	3.29	3.83	0.85	[22]
Silicate	4.23	1.04	0.61	1.70	[23]

E. Photoluminescence Studies

Figure 3.7 shows the NIR photoluminescence spectra of the Nd^{3+} doped lithium sodium bismuth borate glasses with varying Nd_2O_3 concentration in the region, 850-1500 nm with excitation wavelength 808 nm. The spectra exhibit three emission bands at 893, 1060 and 1326 nm (or 0.89, 1.06 and 1.32 μm) corresponding to $^4F_{3/2}\rightarrow{}^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ transitions, respectively. Among these three bands, the band at 1060 nm attributed to $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition is a potential transition with higher intensity than the rest of the bands. The intensity of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition increases with increasing Nd_2O_3 concentration up to 0.6 mol% and then decreased with the increase of Nd_2O_3 concentration showing quenching due to the enhanced interaction between Nd^{3+} ions and host defects leading to energy transfer through cross-relaxations between the active ions (Sontakke et al. 2010). The absorption, excitation, emission mechanisms and cross-relaxation channels are shown in Fig. 3.8 along with the possible cross-relaxation channel of Nd^{3+} doped lithium sodium bismuth borate glass matrix.

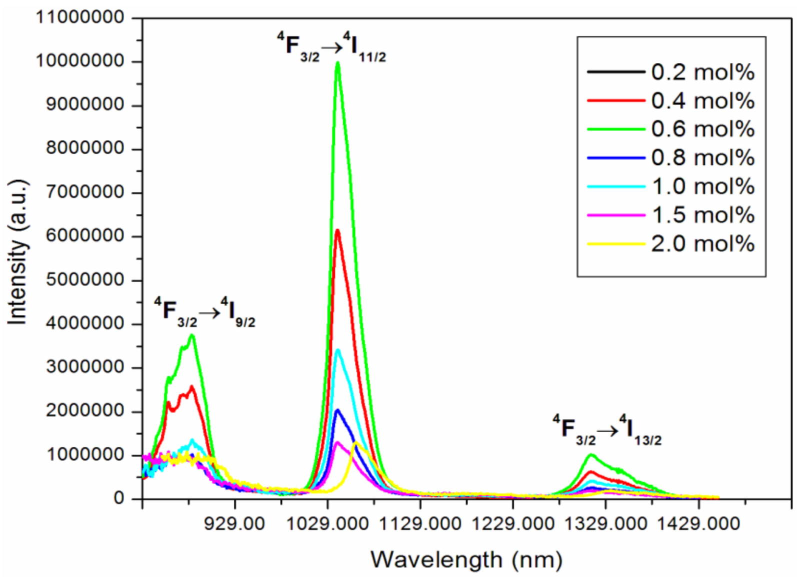

Fig. 3.7 Emission spectra for different concentrations of Nd^{3+} doped lithium sodium bismuth borate glasses.

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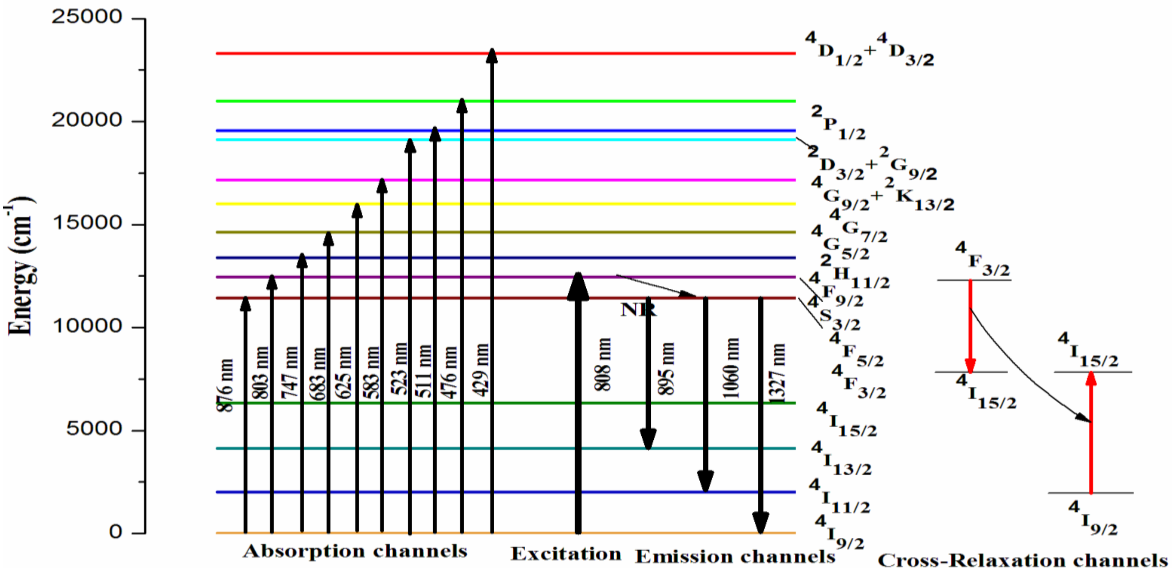

Fig. 3.8 The absorption, excitation, emission mechanisms and cross relaxation channel.

Using the Judd-Ofelt intensity parameters, different radiative parameters such as radiative transition probabilities (A_R), emission cross sections (σ_P) and branching ratios (β) (both experimental (β_{exp}) and calculated (β_R)) of the observed emission transitions are calculated and presented for 0.6 mol% of Nd^{3+} doped glass matrix in Table 3.3. In general, the stimulated emission cross-section (σ_P) is independent on the J-O parameters and effective band widths ($\Delta\nu_{eff}$) of the emission bands. From the table, it is observed that among the three emission transitions, $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition shows higher A_R values (1957 s^{-1}) than other transitions. From table 3.3, it is also observed that the 0.6 mol% of Nd^{3+} doped glass matrix shows higher branching ratio (71%) for the transition $^4F_{3/2}\rightarrow{}^4I_{11/2}$. The emission cross section (σ_P) is an important parameter in predicting the potential laser transition and its value signifies the energy extraction from the lasing material. From table 3.3, it is observed that 0.6 mol% of Nd^{3+} doped lithium sodium bismuth borate glass matrix shows higher σ_P (5.12×10^{-20} cm^2) value for the transition $^4F_{3/2}\rightarrow{}^4I_{11/2}$ than the remaining transitions. Hence in the present work, Nd^{3+} doped lithium sodium bismuth borate glass matrix with 0.6 mol% of Nd^{3+} is recommended as a suitable host for lasing emission at 1.06 µm.

Table 3.3. Emission band positions (λ_P, nm), effective band widths ($\Delta\nu_{eff}$, cm^{-1}), radiative transition probabilities (A_R, s^{-1}), Peak stimulated emission cross-sections (σ_P, x10^{-20} cm^2), experimental (β_{exp}) and radiative (β_R) branching ratios of $^4F_{3/2}$ state of 0.6 mol%Nd^{3+} doped lithium sodium bismuth borate glass matrix.

Transition	Parameters	0.6 mol%
	λ_P	893
	$\Delta\nu_{eff}$	367
	A_R	1767
	σ_P	1.87
	β_{exp}	0.09
$^4F_{3/2}\rightarrow{}^4I_{9/2}$	β_R	0.07
	λ_P	1060
	$\Delta\nu_{eff}$	240
	A_R	1957
$^4F_{3/2}\rightarrow{}^4I_{11/2}$	σ_P	5.12
	β_{exp}	0.48
	β_R	0.71

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$^4F_{3/2}\rightarrow{}^4I_{13/2}$	λ_P	1326
	$\Delta\nu_{eff}$	198
	A_R	351
	σ_P	1.52
	β_{exp}	0.43
	β_R	0.22

F. Decay Analysis

The fluorescence lifetime is an important parameter for the characterization of laser gain medium. Excited at 808 nm laser diode and monitored the emission at 1.06 µm, the decay profiles of Nd^{3+} are measured and calculated. Figure 3.9 shows the decay curves of lithium sodium bismuth borate glasses with different doping concentrations of neodymium ion. As seen from figure, the decay curves resolved with single exponential function at lower concentrations (0.2, 0.4 and 0.6 mol%) and changed into bi-exponential function for higher concentrations (>0.6 mol%). The lifetimes are obtained by e folding times for the studied glasses. The obtained lifetimes are found to be 91, 83, 69, 45, 36, 31 and 28 µs for 0.2, 0.4, 0.6, 0.8, 1.0, 1.5 and 2.0 mol% of Nd^{3+} doped lithium sodium bismuth borate glasses respectively. It is obviously observed that the decay times show the regular decrease. As can be seen from the magnitudes of lifetimes, the 0.2 mol% Nd^{3+} doped sample had the longest decay time and 2.0 mol% Nd^{3+} doped sample had the shortest decay time. The higher lifetime of $^4F_{3/2}$ level indicates the higher population inversion between upper laser level ($^4F_{3/2}$) and lower laser level ($^4I_{11/2}$), possibility of attaining stimulated emission. With the rise in dopant concentration, decay time values gradually decreased. It is expected that, with an increase in the Nd^{3+}-ion concentration, the decay time should decrease as a result of concentration quenching. The test results of Nd^{3+} emission intensities and decay times are consistent with the phenomenon called concentration quenching. The non-exponential nature of the decay curves at higher concentration indicate existence of cross relaxation channels in between Nd^{3+} atoms which is caused by the relatively higher doping level in the lithium sodium bismuth borate glasses.

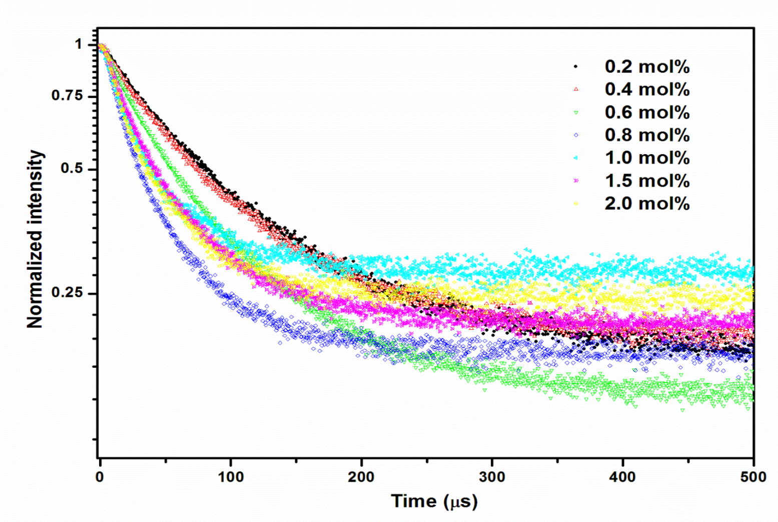

Fig. 3.9 Decay profiles for various concentrations of Nd^{3+} doped lithium sodium bismuth borate glasses.

IV. CONCLUSIONS

Nd^{3+} doped lithium sodium bismuth borate (LSBiB) glass samples with compositions, (60-x) B_2O_3 + $20LiF+10NaF+10Bi_2O_3$ + xNd_2O_3 (where x=0.2 mol.% , 0.4 mol.%, 0.6 mol.%, 0.8 mol.%, 1.0 mol.% , 1.5 mol.% and 2.0 mol.%) are prepared by conventional melt quenching method. XRD pattern and SEM of lithium sodium bismuth borate glass confirms the amorphous nature of the prepared glass. The spectral intensities both experimental (f_{exp}) and calculated (f_{cal}) of different absorption bands of Nd^{3+} are obtained for Nd^{3+} doped lithium sodium bismuth borate (LSBiB) glasses. Among all glass matrices 0.6 mol% Nd^{3+} doped lithium sodium bismuth borate glass matrix has higher Ω_2 value (5.62×10^{-20} cm^2) indicating higher asymmetry of Nd^{3+} sites and strong covalency of the Nd-O bonds. Among three emission bands, the band at 1060 nm attributed to $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition is a potential transition with

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higher intensity than the rest of the bands. Intensity of $^4F_{3/2}\rightarrow{}^4I_{11/2}$ transition increased with increasing Nd_2O_3 concentration up to 0.6 mol% and then decreased with the increase of Nd_2O_3 concentration showing concentration quenching. Magnitudes of lifetimes, the 0.2 mol% Nd^{3+} doped samples had the longest decay time and 2.0 mol% Nd^{3+} doped sample had the shortest decay time which is observed from decay profiles. 0.6 mol% of Nd^{3+} doped lithium sodium bismuth borate glass matrix shows higher σ_P ($5.12\text{x}10^{-20}$ cm^2) value than the remaining concentrations for the transition $^4F_{3/2}\rightarrow{}^4I_{11/2}$. Hence, 0.6 mol% glasses is suggested for lasing emission at 1.06 μm.

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Dr. S. Venkatramana Reddy received Ph.D. degree in 2001 from Sri Venkateswara University, Tirupati, Andhra Pradesh, India. He taught the subjects Electronics and Materials Science to the students of M.Sc. Electronics/ M.Tech. Energy Management/ M.Sc. Physics/ 5yr Integrated M.Sc. Physics from the year 1995. He has been actively involved in designing M.Sc. Electronics Course in S.V. University. He has published about 70 research papers in internationally reputed Journals and presented more than 70 papers at National/International Conferences/Symposia. He is Fellow, Institution of Electronics and Telecommunication Engineers, New Delhi and Life member (LM) in many Professional Bodies like the Instrument Society of India, Bangalore, Semiconductor Society (India), New Delhi, Indian Physics Association, BARC, Mumbai, Indian Association of Physics Teachers, Kanpur, Uttar Pradesh, Senior Member in International Association of Computer Science and Information Technology, Member in International Association of Engineers (IAENG). He was Vice-President for Institution of Electronics and Telecommunication Engineers (IETE) Tirupati Centre during 2013-2016. At present 10 students are working for Ph.D. under his guidance. Presently he is working as an Assistant Professor (senior scale).

Dr. A. V. Chandrasekhar has been working as Lecturer in Physics in Sri Venkateswara Arts College, Tirumala Tirupati Devasthanams. He was awarded doctorate degree in Physics in 1997 by Sri Venkateswara University, Tirupati, Andhra Pradesh, India. He also obtained M.Phil. Degree from the same University in 1992. He has been teaching Physics to the graduate students and quantum mechanics to the Post graduate students. He is involved in research in the field of Solid State Spectroscopy. He has published about 40 research papers in internationally reputed Journals. At present 4 students are working for Ph.D. under his guidance.

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