

Spectral Investigations of Er³⁺ Doped Lead Bismuth Borophosphate Glasses

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Abstract: Different concentrations of Er³⁺ doped lead bismuth magnesium borophosphate (LBMBPH) glasses with chemical composition of (in mol %) 30 PbO–20Bi₂O₃–25MgHPO₄–(25-x) B₂O₃–xEr₂O₃ (x=1.0, 1.5 and 2.0 mol %) were prepared by the melt quenching method. The amorphous nature of these glass samples were confirmed with XRD studies. The spectral data from the optical absorption studies were employed to compute the spectroscopic parameters such as Racah (E¹, E² and E³), spin-orbit (ξ_{4f}), configurational interaction (α), (γ) and Judd-Ofelt intensity parameters (Ω_λ, λ= 2, 4 and 6). The spectral properties were investigated using the absorption. The experimental oscillator strengths were calculated from the area under the absorption bands. Applying Judd-Ofelt theory, the intensity parameters Ω₂, Ω₄ and Ω₆ were calculated, by the least square fit approach from which the radiative transition rates and branching ratios were determined. The radiative parameters such as radiative transition probabilities (A), the total radiative transitional probabilities (A_T), radiative life times (τ_R) and branching ratios (β) were computed for certain lasing levels. The effect of compositional changes on the optical band gap is also reported. The glass systems thus developed indicate their potential lasing candidature.

Keywords: Melt quenching, Radiative lifetimes, optical band gap, J-O parameters, Oscillator strengths

1. Introduction

Rare-earth ions doped solid-state materials are very attractive in making fluorescent display devices, optical detectors, bulk lasers, optical fibers, waveguide lasers and optical amplifiers [1, 2]. Useful information towards device fabrication is obtained from the Er³⁺ doped glasses [3]. Comparing with halide and sulfide glasses, oxide glasses are considered to be with more stable chemical durability and higher phonon energies [4, 5]. In recent years, the heavy metal glasses based on Bi₂O₃ and PbO have attracted considerable attention due to their interesting physical properties leading to different applications [6–8]. First, these glasses are ideal candidates for infrared transmission because of their long infrared cut-off wavelengths in excess of 9 μm [9]. In recent years, heavy metal oxides such as bismuth (Bi), lead (Pb) and tellurium (Te) based glasses have attracted more attention because of their low phonon energies, extended infrared transmission and high non-linear optical properties which are applicable in non-linear optoelectronics including optical fiber and optical switches. Bismuth in glasses plays a dual role as a glass network former (NWF) at high concentration and a modifier (NWM) at low concentration [10].

The present paper deals with the absorption properties of erbium doped Lead bismuth magnesium borophosphate [LBMBPE] glass systems. Absorption spectral intensities of varied Er³⁺ concentration in LBMBPE glasses are analyzed by using Judd-Ofelt theory. The results are examined with respect to the concentration effects, and are compared with the other reported glass systems.

2. Glass Preparation

Er₂O₃ doped glass samples (LBMBPE) with the molar composition of 30PbO–20Bi₂O₃–25MgHPO₄–(25-x) B₂O₃–xEr₂O₃ (where x = 1.0, 1.5 and 2 mol %) were prepared by melt quenching technique. These glasses are labeled as LBMBPE1, LBMBPE2 and LBMBPE3. Approximately 10 gm batches of chemicals were mixed and grinded in required proportions in an agate mortar. The mixture was taken into a porcelain crucible and preheated at 200 °C then melted at a temperature range 1025–1050 °C in high temperature furnace at ambient atmosphere. Finally quenched to room temperature in a brass mould. The obtained glass melts were annealed at 400 °C and cooled to room temperature gradually, and then were cut and well polished for optical measurements. The glass sample density was obtained based on the Archimede's principle using pure water as an immersion liquid and the measurement accuracy was ±0.001 g/cm³. Absorption spectra were recorded for all the samples by taking the undoped glasses as references at room temperature on JASCO UV–vis–NIR V-670 spectrometer. The refractive index 'n' of the samples was determined using conventional methods [11]. The recorded XRD pattern of present glasses has confirmed their amorphous nature.

3. Results and Discussion

3.1. Physical Properties

Physical properties such as erbium ion concentration (N), inter-ionic distance (r_i), polaron radius (r_p) field strength (F), molar refractivity (R_M), electronic polarizability (α) are evaluated using relevant expressions [12]. The number of luminescent centers is found to be densely distributed

because of the fact that the glass density (d) and refractive index (n) values are higher. The polaron radius (r_p) is smaller than inter ionic distance (r_i) and hence such a smaller r_p value has resulted in a higher field strength (F) shown in Table 1. The polaron radius, inter ionic distance, electronic polarizability and molar refractivity are observed to be increased with the Er_2O_3 content. But the field strength increases with the increment of Er^{3+} .

Table 1: Physical properties of Er^{3+} ions doped with 1.0, 1.5 and 2.0 mol% LBMBPE glass matrices

Physical parameter	LBMBPE1.0	LBMBPE1.5	LBMBPE2.0
Average molecular weight	222.4	224.0	225.6
Density (gm/cm^3)	4.565	4.599	4.632
Refractive index 'n'	1.979	1.981	1.998
Concentration 'N' (10^{22})	1.24	1.855	2.474
Polaron radius ' R_p ' (Å^0)	3.74	3.26	2.96
Inter ionic distance ' r_i ' (Å^0)	4.32	3.77	3.43
Field strength 'F' (10^{15})	2.15	2.82	3.41
Molar refractivity ' R_M '	24.02	24.04	24.33
Electronic polarizability ' α ' (10^{-24})	9.52	6.35	4.82
Molar Volume ' V_m '	48.7	48.7	48.68
Dielectric constant ' ϵ '	2.917	2.924	2.995

3.2. Absorption spectra

The absorption spectra of various mol % of Er_2O_3 doped LBMBPE glasses, consists of absorption bands corresponding to the absorptions from the ground state $^4\text{I}_{15/2}$ of Er^{3+} ions. The inhomogeneous broadening of bands are assigned in accordance with the transitions occurred from the $^4\text{I}_{15/2}$ ground state to the various excited states due to the f-f interaction of Er^{3+} ion.

The observed seven distinct and sharp bands (Fig 1) of LBMBPE1-3 glasses at 487nm, 521nm, 543nm, 651nm, 796nm, 974nm and 1531nm corresponding to the absorption transitions from the ground state $^4\text{I}_{15/2}$ to the excited states $^4\text{I}_{13/2}$, $^4\text{I}_{11/2}$, $^4\text{I}_{9/2}$, $^4\text{F}_{9/2}$, $^4\text{S}_{3/2}$, $^2\text{H}_{11/2}$, $^4\text{F}_{7/2}$. The energy levels lying below 450 nm could not be observed in the present glass system due to intrinsic inter band absorption of the host matrices. All these transitions are found to occur almost at the same place for the three present glass systems. The $^4\text{I}_{15/2} \rightarrow ^2\text{H}_{11/2}$ transition possesses higher intensity compare to the other transitions and follows the selection rules $\Delta L \leq 2$ and $\Delta J \leq 2$. This transition is known as hypersensitive transition

The Racah coefficients (E^1 , E^2 and E^3), Spin-orbit coupling (ξ_{4f}), and Configuration interaction (α , β and γ) factors are evaluated by least square fit method [13] and are shown in Table 2 along with other spectroscopic parameters. The observed and calculated values of the energy levels of these glass systems are also presented in Table 3. The

low rms deviation values between the experimental and calculated energy levels reflect the validity of full matrix diagonalization [14].

Table 2: Racah (E^1 , E^2 and E^3), spin-orbit (ξ_{4f}) and configuration interaction (α) parameters in (cm^{-1}) of Er^{3+} ions doped with 1.0, 1.5 and 2.0 mol% LBMBPE glass matrices

parameter	LBMBPE1.0	LBMBPE1.5	LBMBPE2.0
E^1 (Racah parameters)	6050	5886	6123
E^2	25.2	24.2	25.4
E^3	609.2	611.3	616.1
ξ_{4f} (spin Orbit Coupling)	2409	2418	2408
α (Configurational Interaction)	-35.4	-40.9	-26.8
β	675.0	964.0	510.2
γ	649.6	649.4	649.7
E^1/E^3 (hydrogenic ratios)	9.93	9.62	9.94
E^2/E^3	0.041	0.039	0.041

The spectral oscillator strengths of the absorption bands were calculated using Beer-Lambert's principle and presented in Table 4. By performing a least squares fit analysis [15, 16], a set of best-fit intensity parameters (Judd-Ofelt Q_λ parameters) for the absorption spectrum of the above glass were computed. The measured and calculated spectral oscillator strengths and Judd-Ofelt parameters are given in Table 5, and these results are compared with other glasses [17, 18]. It is shown that Judd-Ofelt factor Ω_2 is larger than Ω_4 and Ω_6 . The unit tensor operators U^k for the observed absorption bands were obtained from the work of Carnall et al. [19]. Based on the spectral intensities of the measured absorption levels, the transitions can be arranged as follows.

$$(^2\text{H}, ^4\text{G})_{11/2} > ^4\text{I}_{13/2} > ^4\text{F}_{9/2} > ^4\text{S}_{3/2} > ^4\text{F}_{7/2} > ^4\text{I}_{11/2} > ^4\text{I}_{9/2}$$

The Ω_2 parameter is sensitive to the symmetry of the rare-earth site and the covalency between rare-earth ions and ligand anions; the rigidity of the network surrounding the rare-earth ion is supposed to influence the magnitude of Ω_4 and Ω_6 . Ω_2 is strongly dependent on the hypersensitive transitions. Hypersensitivity is related to the covalency through affects the polarizability of the ligands around the rare earth ions [20]. The increasing of erbium ions in the glass network does not lead to a significant effect on the symmetry at RE sites or the covalency between Er^{3+} and ligands. Er^{3+} ions may have similar environment around them whatever erbium content increases [21]. In the present glass, the Ω_2 parameter is in decreasing trend with increasing Er^{3+} concentration up to 2 wt % and later shows increasing trend [20].

Table 3: Experimental and calculated spectral intensities (in cm^{-1}) of observed absorption bands of Er^{3+} ions doped with 1.0, 1.5 and 2.0 mol% LBMBPE glass matrices

	LBMBPE1.0		LBMBPE1.5		LBMBPE2.0	
Level	E_{exp}	E_{cal}	E_{exp}	E_{cal}	E_{exp}	E_{cal}
$^4\text{I}_{13/2}$	6531	6532	6540	6545	6534	6533
$^4\text{I}_{11/2}$	10251	10250	10266	10263	10240	10240
$^4\text{I}_{9/2}$	12554	12552	12562	12553	12547	12547

$^4F_{9/2}$	15325	15334	15325	15365	15325	15324
$^4S_{3/2}$	18433	18433	18348	18349	18399	18399
$^2H_{11/2}$	19175	19175	19175	19176	19175	19175
$^4F_{7/2}$	20512	20506	20576	20550	20492	20492
rms dev	±4.24		±18.3		±0.65	

3.3. Radiative properties:

Radiative transition probabilities (A), branching ratios (β) and radiative lifetimes (τ_R) for stimulated emission for certain excited states $^4G_{11/2}$, $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ of Er^{3+} doped LMBBPE glasses have been calculated [22]. These results are presented in Table 6 and Table 8. The radiative lifetimes of the excited states $^4F_{5/2}$, $^4F_{7/2}$, $^4S_{3/2}$ and $^4I_{13/2}$ are decreasing with the increment of Er^{3+} in the above glass matrices. It is observed that the magnitudes of lifetimes are slightly higher in the case of LMBBPE1 glass than glasses. It is observed from the table that the branching ratios of the transitions $^4F_{7/2} \rightarrow ^4I_{15/2}$ and $^4S_{3/2} \rightarrow ^4I_{15/2}$ are increasing with Er^{3+} content.

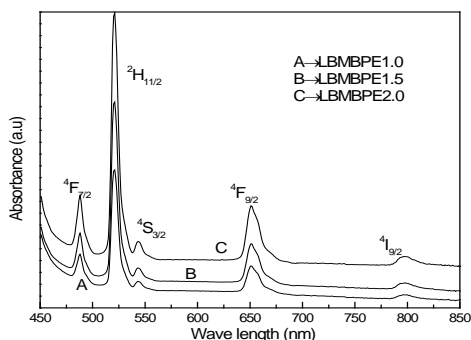


Figure 1 (a): Visible absorption spectrum of LMBBPE1, LMBBPE1.5 and LMBBPE2 glasses.

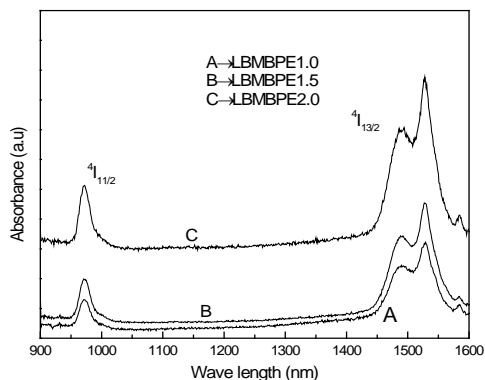


Figure 1 (b): NIR absorption spectrum of LMBBPE1, LMBBPE1.5 and LMBBPE2 glasses.

3.4 Optical band gap

The study of optical absorption edge gives more important information. The principle of the technique is that a photon with energy greater than the band gap energy will be absorbed. There are two kinds of optical transitions at the fundamental absorption edge: direct and indirect transitions, both of which involve the interaction of an electromagnetic wave with an electron in the valence band. The optical band

gaps (E_{opt}) for both indirect and direct transitions of present glasses are measured [3] given in Fig. 2a and 2b.

Table 4: Experimental and calculated spectral intensities ($f \times 10^{-6}$) of observed absorption bands of Er^{3+} ions doped with 1.0, 1.5 and 2.0 mol% LMBBPE glass matrices.

level	LMBBPE1.0		LMBBPE1.5		LMBBPE2.0	
	f_{exp}	f_{cal}	f_{exp}	f_{cal}	f_{exp}	f_{cal}
$^4F_{7/2}$	3.56	5.04	3.46	5.59	3.62	5.61
$^2H_{11/2}, ^4G_{11/2}$	17.1	17.1	16.6	16.6	17.5	17.5
$^4S_{3/2}$	0.41	1.36	0.40	1.61	0.36	1.65
$^4F_{9/2}$	4.55	4.39	4.34	4.17	4.04	3.94
$^4I_{9/2}$	0.51	0.58	0.49	0.41	0.54	0.33
$^4I_{11/2}$	1.02	1.58	1.21	1.83	1.32	1.90
$^4I_{13/2}$	3.75	3.41	4.39	3.93	4.45	4.01
rms dev	±0.71		±0.97		±0.94	

Table 5: Judd-Ofelt intensity parameters ($\Omega_\lambda \times 10^{-20}$) ($\lambda=2, 4, 6$) (cm^2) of Er^{3+} ions doped with 1.0, 1.5 and 2.0 mol% LMBBPE glass matrices.

Glass matrix	Ω_2	Ω_4	Ω_6	Ω_4/Ω_6
LMBBPE1.0	5.89	1.857	2.506	0.741
LMBBPE1.5	5.95	1.251	2.965	0.421
LMBBPE2.0	6.41	0.956	3.002	0.318
BOTNZER[17]	4.60	0.74	1.26	0.59
B1TNZER[17]	6.15	1.08	1.22	0.88
0.1[18]	2.715	2.340	1.262	
1.0[18]	0.505	0.348	0.149	

As given in Table 7, the direct and indirect optical band gaps decrease from 3.133 and 2.745eV to 2.974 and 2.515eV, respectively, as the concentration of Er^{3+} ions is increased up to 2 mol %. The fundamental absorption edges of the spectra shift towards the higher wave lengths with increasing Er_2O_3 content. Decrement in optical band gap is due to decrease in the average of bond energy. The increment of erbium content in the present glass hosts cause a decrease in the compactness of the network and formation of the non-bridging oxygens(NBO) [3].

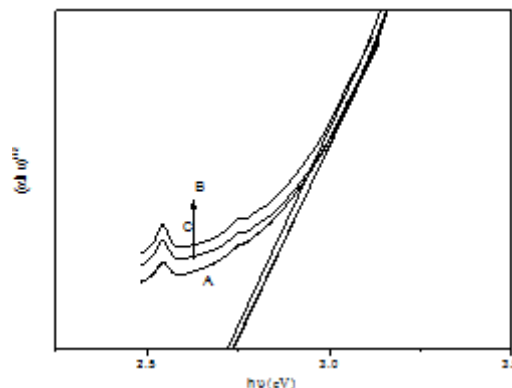


Figure 2 (a): Indirect band gap energy profile of (A) LMBBPE1, (B) LMBBPE1.5 and (C) LMBBPE2 glasses

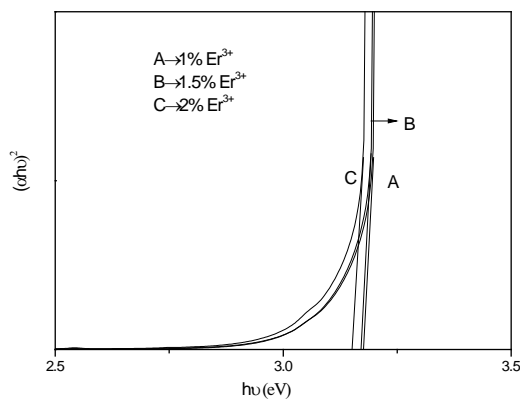


Figure 2 (b): Indirect band gap energy profile of (A) LBBMBPE1, (B) LBBMBPE1.5 and (C) LBBMBPE2 glasses.

Table 7: Optical band gap of indirect transition and direct transition of Er³⁺ ions doped with 1.0, 1.5 and 2.0 mol% LBBMBPE glass matrices.

Band gap	LBBMBPE1.0	LBBMBPE1.5	LBBMBPE2.0
Indirect transition	2.745	2.651	2.515
Direct transition	3.133	3.016	2.974

Table 6: Estimated values of spontaneous emission probabilities (A), and branching ratios (β) of emission transitions of Er³⁺ ions doped with 1.0, 1.5 and 2.0 mol% LBBMBPE glass matrices.

Transition		LBBMBPE1.0		LBBMBPE1.5		LBBMBPE2.0	
SLJ	SL'J'	A	β	A	β	A	β
⁴ I _{13/2}	⁴ I _{15/2}	488.1	0	3.956	0	4.079	0
⁴ I _{11/2}	⁴ I _{13/2}	101.4	0	0.056	0	0.042	0
	⁴ I _{15/2}	570.4	0.00	40.16	0.00	39.51	0.00
⁴ I _{9/2}	⁴ I _{11/2}	6.385	0.03	599.1	0.04	595.5	0.04
	⁴ I _{13/2}	231.2	0.05	654.2	0.04	594.9	0.04
	⁴ I _{15/2}	345.8	0.08	852.9	0.05	650.2	0.04
⁴ F _{9/2}	⁴ I _{9/2}	15.62	0.81	12726	0.85	12562	0.86
	⁴ I _{11/2}	293.6	0	0.077	0	0.061	0
	⁴ I _{13/2}	222.8	0.00	86.65	0.00	91.03	0.00
	⁴ I _{15/2}	4429	0.01	99.57	0.01	95.96	0.00
⁴ S _{3/2}	⁴ F _{9/2}	2.531	0.05	966	0.05	957.7	0.05
	⁴ I _{9/2}	234.9	0.93	16927	0.93	17567	0.93
	⁴ I _{11/2}	163	0.00	2.762	0.00	2.786	0.00
	⁴ I _{13/2}	2125	0.09	245.1	0.08	240.6	0.08
	⁴ I _{15/2}	78.03	0.06	183.1	0.06	183.6	0.06
² H _{11/2}	⁴ S _{3/2}	0.079	0.81	2464	0.82	2487	0.82
	⁴ F _{9/2}	86.23	0.02	91.33	0.03	92.17	0.03
	⁴ I _{11/2}	108.9	0.00	15.68	0.00	16.31	0.00
	⁴ I _{13/2}	978.6	0.05	336	0.07	340.8	0.07
	⁴ I _{15/2}	17447	0.04	201	0.04	185.6	0.04
⁴ F _{7/2}	² H _{11/2}	3.117	0.89	4194	0.88	3886	0.87
	⁴ S _{3/2}	0.067	0.01	6.537	0.01	6.421	0.01
	⁴ F _{9/2}	40.36	0.39	273.2	0.46	275.4	0.58
	⁴ I _{9/2}	531.8	0.59	244.2	0.41	192	0.40
	⁴ I _{11/2}	714.7	0.15	112.5	0.14	112.3	0.14
	⁴ I _{13/2}	1247	0.84	666.1	0.85	677.4	0.85
	⁴ I _{15/2}	11344	1	554.2	1	555.4	1

Table 8: Estimated values of radiative lifetimes (τ_R) of emission transitions of Er³⁺ ions doped with 1.0, 1.5 and 2.0 mol% LBBMBPE glass matrices.

SLJ	SL'J'	τ _R (μs)	τ _R (μs)	τ _R (μs)
⁴ I _{13/2}	⁴ I _{15/2}	2049	1804	1800
⁴ I _{11/2}	⁴ I _{13/2} , ⁴ I _{15/2}	1488	1284	1266
⁴ I _{9/2}	⁴ I _{11/2} , ⁴ I _{13/2} , ⁴ I _{15/2}	1714	1908	2110
⁴ F _{9/2}	⁴ I _{9/2} , ⁴ I _{11/2} , ⁴ I _{13/2} , ⁴ I _{15/2}	202	211	226
⁴ S _{3/2}	⁴ F _{9/2} , ⁴ I _{9/2} , ⁴ I _{11/2} , ⁴ I _{13/2} , ⁴ I _{15/2}	384	335	333
² H _{11/2}	⁴ S _{3/2} , ⁴ F _{9/2} , ⁴ I _{11/2} , ⁴ I _{13/2} , ⁴ I _{15/2}	53.7	55.3	53.4
⁴ F _{7/2}	² H _{11/2} , ⁴ S _{3/2} , ⁴ F _{9/2} , ⁴ I _{11/2} , ⁴ I _{13/2} , ⁴ I _{15/2}	72	67.2	69.2

4. Conclusions

A novel multi component Erbium doped borophosphate glasses were developed by varying the mol% of Er³⁺ constituent heavy metal oxides PbO and Bi₂O₃. The inter electronic repulsion parameter ratios E¹/E³ and E²/E³ observed in all the glasses do not deviate much from the hydrogenic ratios and indicate that the radial properties (radial distribution function) of all these glasses are similar. The higher values obtained for Ω₂ in all glasses indicate that the Er³⁺ ion is subjected to higher covalency with low symmetry. The band gap of all the glasses studied shows that decreasing trend with the increment of Er³⁺ content. It appears that the LBBMBPE1 glass among other glasses exhibits significant values in its radiative life times for all transitions reported. Further the optical band gaps evaluated for these glasses both for direct and indirect transitions showed lower values reflecting their goods witching action. From our analysis it is suggested that the LBBMBPE1 glass is a good lasing candidate.

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