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Spectral and Thermal properties of Ho^{3+} ions doped Zinc Lithium Alumino Potassiumniobate Borophosphate Glasses

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Abstract

Glass of the system: (35-x) P_2O_5: 10ZnO: 10Li_2O: 10Al_2O_3: 10K_2O: 10Nb_2O_3: 15B_2O_3: xHo_2O_3. (Where x=1, 1.5,2 mol %) have been prepared by melt-quenching method. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. Optical absorption, Excitation, fluorescence and Thermal spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=2, 4 and 6) are evaluated from the intensities of various absorption bands of optical absorption spectra. Using these intensity parameters various radiative properties like spontaneous emission probability (A), branching ratio (β), radiative life time (τ_R) and stimulated emission cross–section (σ_p) of various emission lines have been evaluated.

Keywords: ZLAPNBP Glasses, Optical Properties, Judd-Ofelt Theory, Transmittance Properties.

Introduction:

The rare earth ions doped glasses are used in making up conversion lasers due to their potential applications in variety of fields namely, optical data storage, display technology, undersea transmission and medical diagnostics [1-5].Phosphate glasses are known to be low melting, optical data transmission, detection, laser ,high homogeneity, good corrosion resistance, excellent chemical durability and low glass transition temperature (T_g), better thermal stability, high thermal expansion coefficient, low phonon energy, wide optical transmission region and high refractive index materials, therefore considered to be potential nonlinear materials [6-10]. The high gain density in phosphate glasses is due to high solubility of rare earth ions in phosphate network. These glasses have high luminescence efficiency and they lack thermal lensing effects. When compared with borate and silicate glasses these glasses have distinction optical properties such as large infrared transmission window, high gain density, low up conversion and wide bandwidth emission spectra [11-15]. Recently, phosphate glasses have attained great attention in synthesis, structure and physical properties due to their high density and high dielectric constant [16-18].

The present work reports on the preparation and characterization of rare earth doped heavy metal oxide (HMO) glass systems for lasing materials. I have studied on the Thermal, absorption, Excitation and fluorescence properties of Ho^{3+} doped zinc lithium alumino potassiumniobate borophosphate glasses. The intensities of the transitions for the rare earth ions have been estimated successfully using the Judd-Ofelt theory, The laser parameters such as radiative probabilities(A), branching ratio (β), radiative life

time(τ_R) and stimulated emission cross section(σ_p) are evaluated using J.O. intensity parameters (Ω_λ, λ=2,4 and 6).

Experimental:

Preparation of glasses:

The following Ho^{3+}doped borosilicate glass samples (35-x) P_2O_5:10ZnO:10Li_2O:10 Al_2O_3: 10K_2O: 10Nb_2O_3: 15B_2O_3: x Ho_2O_3. (where x=1,1.5 and 2 mol%) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of P_2O_5, ZnO, Li_2O, Al_2O_3, K_2O, Nb_2O_3, B_2O_3 and Ho_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1005^0C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould and annealed at temperature of 250^0C for 2h to remove thermal strains and stresses. Every time fine powder of cerium oxide was used for polishing the samples. The glass samples so prepared were of good optical quality and were transparent. The chemical compositions of the glasses with the name of samples are summarized in **Table 1.**

Table 1: Chemical composition of the glasses

Sample	Glass composition (mol %)
ZLAPNBP (UD)	35P_2O_5:10ZnO:10Li_2O:10Al_2O_3:10K_2O:10Nb_2O_3:15B_2O_3
ZLAPNBP (HO1)	34P_2O_5: 10ZnO: 10Li_2O: 10Al_2O_3: 10K_2O: 10Nb_2O_3: 15B_2O_3:1 Ho_2O_3
ZLAPNBP (HO1.5)	33.5P_2O_5: 10ZnO: 10Li_2O: 10Al_2O_3: 10K_2O:10Nb_2O_3:15B_2O_3:1.5Ho_2O_3
ZLAPNBP (HO2)	33P_2O_5: 10ZnO: 10Li_2O: 10Al_2O_3: 10K_2O:10Nb_2O_3:15B_2O_3: 2 Ho_2O_3

ZLAPNBP (UD) -Represents undoped Zinc Lithium Alumino Potassiumniobate Borophosphate glass specimen.
ZLAPNBP (HO)-Represents Ho^{3+} doped Zinc Lithium Alumino Potassiumniobate Borophosphate glass specimens.

Theory:

Oscillator Strength:

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [21].

$$f_{expt.} = 4.318 \times 10^{-9} \int \varepsilon(\nu)\, d\nu \qquad (1)$$

where, $\varepsilon(\nu)$ is molar absorption coefficient at a given energy ν (cm^{-1}), to be evaluated from Beer–Lambert law.

Under Gaussian Approximation, using Beer–Lambert law, the observed oscillator strengths of the absorption bands have been experimentally calculated [22], using the modified relation:

$$P_m = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta\upsilon_{1/2} \qquad (2)$$

where, c is the molar concentration of the absorbing ion per unit volume, I is the optical path length, $\log I_0/I$ is optical density and $\Delta\upsilon_{1/2}$is half band width.

Judd-Ofelt Intensity Parameters

According to Judd [23] and Ofelt [24] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial Jmanifold $|4f^N (S, L) J>$ level and the terminal J' manifold $|4f^N (S', L') J'>$ is given by:

$$\frac{8\Pi^2 mc\bar{\upsilon}}{3h(2J+1)}\frac{1}{n}\left[\frac{(n^2+2)^2}{9}\right]\times S(J,J') \qquad (3)$$

Where, the line strength S (S', L') is given by the equation

$$S(J, J') = e^2 \sum \Omega_\lambda <4f^N(S, L) J \| U^{(\lambda)} \| 4f^N(S', L')J'>^2 \qquad (4)$$

$\lambda = 2, 4, 6$

In the above equation m is the mass of an electron, c is the velocity of light, ν is the wave number of the transition, h is Planck's constant, n is the refractive index, J and J' are the total angular momentum of the initial and final level respectively, Ω_λ ($\lambda = 2, 4, 6$) are known as Judd-Ofelt intensity parameters.

Radiative Properties

The Ω_λ parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability (A) and radiative life time (τ_R), and laser parameters like fluorescence branching ratio (β_R) and stimulated emission cross section (σ_p).

The spontaneous emission probability from initial manifold $|4f^N(S', L') J'>$ to a final manifold $|4f^N (S,L) J >|$ is given by:

$$A[(S', L') J'; (S,L)J] = \frac{64\pi^2\nu^3}{3h(2J'+1)}\left[\frac{n(n^2+2)^2}{9}\right]\times S(J',J) \qquad (5)$$

where, $S(J', J) = e^2 [\Omega_2 \| U^{(2)} \|^2 + \Omega_4 \| U^{(4)} \|^2 + \Omega_6 \| U^{(6)} \|^2]$

The fluorescence branching ratio for the transitions originating from a specific initial manifold $|4f^N (S', L') J'>$ to a final many fold $|4f^N (S,L)J >$ is given by

$$\beta[(S', L') J'; (S, L) J] = \sum \frac{A[(S' L)]}{A[(S' L')J'(\bar{S} \bar{L})]} \qquad (6)$$

S L J where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} = \sum A[(S', L') J'; (S,L)] = A^{-1}_{Total} \qquad (7)$$

S L J where, the sum is over all possible terminal manifolds. The stimulated emission cross - section for a transition from an initial manifold $|4f^N (S', L') J'>$ to a final manifold $|4f^N (S,L)J >|$ is expressed as

$$\sigma_p(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}}\right]\times A[(S', L')J'; (\bar{S}, \bar{L})\bar{J}] \qquad (8)$$

where, λ_p the peak fluorescence wavelength of the emission band and $\Delta\lambda_{eff}$ is the effective fluorescence line width.

Nephelauxetic Ratio (β) and Bonding Parameter ($b^{1/2}$)

The nature of the R-O bond is known by the Nephelauxetic Ratio (β') and Bonding

Parameters ($b^{1/2}$), which are computed by using following formulae [25, 26]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \qquad (9)$$

where, ν_a and ν_g refer to the energies of the corresponding transition in the glass and free ion, respectively. The values of bonding parameter $b^{1/2}$ are given by

$$b^{1/2} = \left[\frac{1-\beta'}{2}\right]^{1/2} \qquad (10)$$

Results and Discussion:

XRD Measurement

Figure 1 presents the XRD pattern of the sample contain – P_2O_5 which is show no sharp Bragg's peak, but only a broad diffuse hump around low angle region. This is the clear indication of amorphous nature within the resolution limit of XRD instrument.

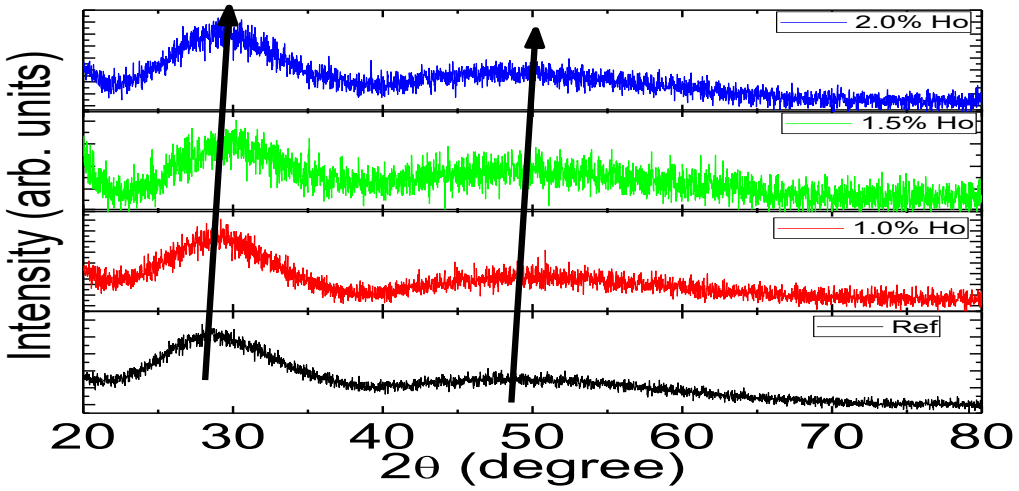

Fig. 1: X-ray diffraction pattern of P_2O_5: ZnO: Li_2O: Al_2O_3: K_2O: Nb_2O_3:B_2O_3: Ho_2O_3

Thermal Properties

Fig. 2 depicts the DTA thermogram of powdered ZLAPNBP sample show an endothermic peak corresponding to glass transition event followed by an exothermic peak related to crystallization event. The glass transition temperature (T_g), onset crystallization temperature (T_x), crystallization temperature (T_c) were estimated to be 516^0C, 582^0C and 595^0C respectively. From the measured value of T_g, T_x and T_c, the glass stability factor ($\Delta T = T_x - T_g$) has been determined to be 66^0C indicating the good stability of the glass.

Obtained results indicate that by increasing the amount of mol% Ho_2O_3, the T_g of the samples also increases, the small increase of T_g in these glasses shows that the structure is strongly and progressively modified. The thermal stabilities ΔT of the ZLAPNBP reference glass and Ho^{+3}doped ZLAPNBP glass has been evaluated from their T_g, T_c and T_c values, the results are listed out in Table 2. Hruby's parameter also calculated by using eq. (11), the greater values of the Hruby's parameter indicate higher glass forming tendency, the values of Hin our glasses increased with the addition of the Ho_2O_3.Eqs. (12) and (13) present the GS parameter of Weinberg [25]and Lu and Liu [26], respectively.

$$H=\frac{T_X-T_g}{T_c-T_X} \quad (11)$$

$$K_W=\frac{T_X-T_g}{T_c} \quad (12)$$

$$K_{LL}=\frac{T_X}{T_g+T_c} \quad (13)$$

Table 2: Thermal parameters determined from the DTA traces of ZLAPNBP (HO) glasses.

Sample Name	% Sm_2O_3	$T_g{}^0C$	$T_X{}^0C$	$T_C{}^0C$	ΔT	H	K_W	K_{LL}
ZLAPNBP (HO 1.0)	1	516	582	595	66	5.076	0.1109	0.5231
ZLAPNBP (HO 1.5)	1.5	518	585	598	67	5.153	0.1120	0.5242
ZLAPNBP (HO 02)	2	522	592	605	70	5.231	0.1157	0.5253

Absorption Spectrum

The absorption spectra of Ho^{3+}doped ZLAPNBP glass specimens have been presented in Figure 3 in terms of optical density versus wavelength. Twelve absorption bands have been observed from the ground state $^5I_8$ to excited states $^5I_5$, $^5I_4$, $^5F_5$, $^5F_4$, $^5F_3$, $^3K_8$, $^5G_6$, $(^5G,^3G)_5$, $^5G_4$, $^5G_2$, $^5G_3$, and$^3F_4$ for Ho^{3+} doped ZLAPNBP glasses.

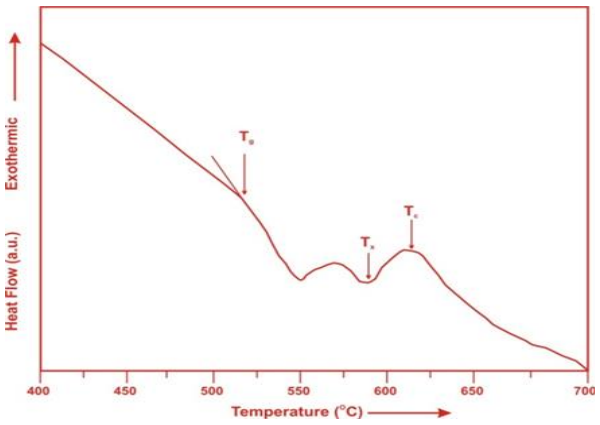

Fig. 2. DTA thermogram of powdered ZLAPNBP sample.

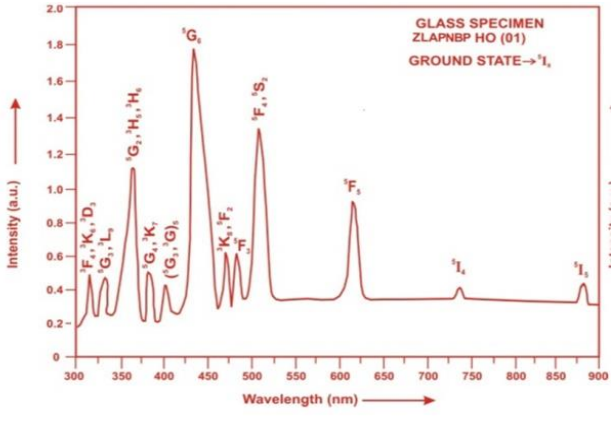

Fig. 3: Absorption spectrum of Ho^{3+}doped ZLAPNBP HO (01) glass.

The experimental and calculated oscillator strength for Ho^{3+} ions in ZLAPNBP glasses are given in **Table 3.**

Table 3: Measured and calculated oscillator strength ($P_m \times 10^{+6}$) of Ho^{3+}ions in ZLAPNBPglasses.

Energy level from $^5I_8$	Glass ZLAPNBP (HO01)		Glass ZLAPNBP (HO1.5)		Glass ZLAPNBP (HO02)	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^5I_5$	0.53	0.24	0.48	0.24	0.45	0.24
$^5I_4$	0.06	0.02	0.05	0.02	0.04	0.02
$^5F_5$	3.66	2.78	3.62	2.75	3.58	2.72
$^5F_4$	4.60	4.29	4.56	4.25	4.52	4.22
$^5F_3$	1.53	2.37	1.49	2.36	1.45	2.34
$^3K_8$	1.45	1.97	1.42	1.94	1.38	1.92
$^5G_6$	26.68	26.63	25.28	25.26	24.78	24.78
$(^5G,^3G)_5$	3.82	1.72	3.76	1.68	3.72	1.67
$^5G_4$	0.08	0.61	0.06	0.60	0.05	0.59
$^5G_2$	5.62	5.61	5.58	5.36	5.54	5.27
$^5G_3$	1.50	1.40	1.46	1.37	1.42	1.35
$^3F_4$	1.38	4.20	1.32	4.12	1.27	4.08
r.m.s. deviation	±1.1031	--	±1.0975	--	±1.0977	--

Computed values of F_2, Lande′ parameter (ξ_{4f}**),** Nephlauxetic ratio (β') and bonding parameter ($b^{1/2}$) for Ho^{3+} ions in ZLAPNBP glass specimen are given in Table 4.

Table 4: F_2, ξ_{4f}, β' and $b^{1/2}$ parameters for Holmium doped glass specimen.

Glass Specimen	F_2	ξ_{4f}	β'	$b^{1/2}$
Ho^{3+}	358.82	1258.16	0.9337	0.1821

In the Zinc Lithium Alumino Potassiumniobate Borophosphate glasses (ZLAPNBP) Ω_2, Ω_4 and Ω_6 parameters decrease with the increase of x from 1 to 2 mol%. The order of magnitude of Judd-Ofelt intensity parameters is $\Omega_2 > \Omega_6 > \Omega_4$ for all the glass specimens. The spectroscopic quality factor (Ω_4 / Ω_6)

related with the rigidity of the glass system has been found to lie between 0.6181 and 0.6294 in the present glasses. The values of Judd-Ofelt intensity parameters are given in **Table 5.**

Table 5: Judd-Ofelt intensity parameters for Ho^{3+} doped ZLAPNBP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(pm^2)$	Ω_4/Ω_6	Ref.
ZLAPNBP (HO01)	6.576	1.369	2.175	0.6294	[P.W.]
ZLAPNBP (HO1.5)	6.186	1.338	2.160	0.6194	[P.W.]
ZLAPNBP (HO02)	6.051	1.324	2.142	0.6181	[P.W.]

Excitation Spectrum

The Excitation spectra of Ho^{3+}doped ZLAPNBP glass specimens have been presented in Figure 4 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 325–525 nm fluorescence at 545nm having different excitation band centered at 350,419, 452, 472and 485 nm are attributed to the $^5G_3$, $(^5G, ^3G)_5$, $^5G_6$, $^3K_8$ and $^5F_3$ transitions, respectively. The highest absorption level is $^5G_6$ and is at 452nm.So this is to be chosen for excitation wavelength.

Fluorescence Spectrum

The fluorescence spectrum of Ho^{3+}doped in zinc lithium alumino potassiumniobate borophosphate glass is shown in Figure 5. There are five broad bands observed in the Fluorescence spectrum of Ho^{3+}doped zinc lithium alumino potassiumniobate glass. The wavelengths of these bands along with their assignments are given in Table 6. The peak with maximum emission intensity appears at 555 nm and corresponds to the ($^5F_4 \rightarrow {}^5I_8$) transition.

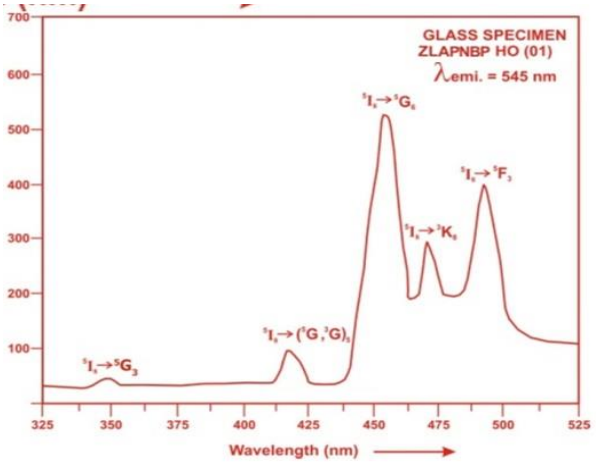

Fig. 4: Excitation spectrum of doped with Ho^{3+} ZLAPNBP glasses.

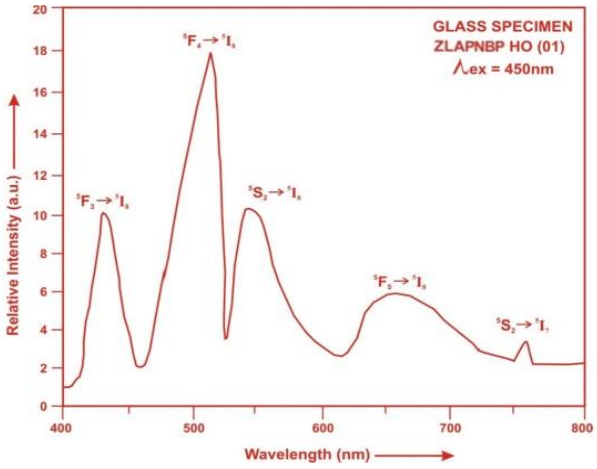

Fig. 5: Fluorescence spectrum of doped with Ho^{3+} ZLAPNBP glasses

Conclusion:

In the present study, the glass samples of composition(35-x) P_2O_5: 10ZnO:10Li_2O:10 Al_2O_3: 10K_2O: 10Nb_2O_3: 15B_2O_3:xHo_2O_3. (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^5F_4 \rightarrow {}^5I_8$) for glass ZLAPNBP (HO 01), suggesting that glass ZLAPNBP (HO 01) is better compared to the other two glass systems ZLAPNBP (HO1.5) and ZLAPNBP (HO02). The greater values of the Hruby's parameter indicate higher glass forming tendency.

Conflict of Interest:

The authors declared that they have no conflict of interest.

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Table 6: Emission peak wave lengths (λ_p), radiative transition probability (A_{rad}), branching ratio (β), stimulated emission cross-section (σ_p) and radiative life time (τ_R) for various transitions in Ho^{3+} doped ZLAPNBP glasses.

Transition		ZLAPNBP (HO 01)				ZLAPNBP (HO 1.5)				ZLAPNBP (HO 02)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(\mu s)$	λ_{max} (nm)	A_{rad} (s^{-1})	β	σ_p (10^{-20} cm^2)	$A_{rad}(s^{-1})$	λ_{max} (nm)	A_{rad} (s^{-1})	β
$^5F_3 \rightarrow {}^5I_8$	435	4086.30	0.2641	0.5233	6464.10	4069.01	0.2648	0.509	6506.51	4040.35	0.2646	0.490	6549.07
$^5F_4 \rightarrow {}^5I_8$	501	6517.41	0.4213	1.146		6467.20	0.4208	1.124		6428.04	0.4210	1.083	
$^5S_2 \rightarrow {}^5I_8$	555	1706.85	0.1103	0.396		1698.46	0.1105	0.389		1686.75	0.1105	0.381	
$^5F_5 \rightarrow {}^5I_8$	652	1864.64	0.1205	0.710		1846.06	0.1201	0.693		1833.92	0.1201	0.675	
$^5S_2 \rightarrow {}^5I_7$	761	1294.87	0.08370	0.948		1288.50	0.08384	0.934		1280.31	0.08385	0.910	