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Spectral and Transmittance Properties of Ho^{3+} Ions Doped Lead Lithium Aluminosodium Phosphate Glasses

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ABSTRACT: Glass of the system: $(45-x)\text{P}_2\text{O}_5:10\text{PbO}:10\text{Li}_2\text{O}:15\text{Al}_2\text{O}_3:20\text{Na}_2\text{O}:x\text{Ho}_2\text{O}_3$. (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 Transmittance spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ ($\lambda=2, 4$ 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: LLASP Glasses, Optical Properties, Judd-Ofelt Theory, Transmittance Properties.

I. 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, medical diagnostics, lasers, photonics, fibers, solar cells, nuclear, and sensor fields [1-8]. Amorphous material is also known as super cooled liquid due to the rapid cooling of the molten mass into the rigid state and due to lack of its first order phase transition where volume, entropy and enthalpy are discontinuous. Rare earth doped phosphate glasses are being extensively studied due to their technological importance and their applications in solid state lasers, optical fibers, low glass transition temperature, high coefficient of thermal expansion and low melting temperature [9-12]. These glasses have high luminescence efficiency and they lack thermal lensing effects [13-15]. 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 [16-20]. Phosphate glasses possess unique and interesting optical, thermal and electrical properties which make them candidates for a wide range of application [21-24].

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 Optical absorption, Excitation, fluorescence and Transmittance properties of Ho^{3+} doped lead lithium aluminosodium phosphate 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 , $\lambda=2, 4$ and 6).

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Ho^{3+} doped phosphate glass samples $(45-x)\text{P}_2\text{O}_5:10\text{PbO}:10\text{Li}_2\text{O}:15\text{Al}_2\text{O}_3:20\text{Na}_2\text{O}:x\text{Ho}_2\text{O}_3$. (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 , PbO , Li_2O , Al_2O_3 , Na_2O and Ho_2O_3 . They were thoroughly mixed by using an agate pestle mortar. then melted at 985°C 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°C 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 %)
LLASP(UD)	45P ₂ O ₅ :10PbO:10Li ₂ O:15Al ₂ O ₃ :20Na ₂ O
LLASP (HO1)	44P ₂ O ₅ :10PbO:10Li ₂ O:15Al ₂ O ₃ :20Na ₂ O:1 Ho ₂ O ₃
LLASP(HO1.5)	43.5P ₂ O ₅ :10PbO:10Li ₂ O:15Al ₂ O ₃ :20Na ₂ O:1.5Ho ₂ O ₃
LLASP(HO2)	43P ₂ O ₅ :10PbO:10Li ₂ O:15Al ₂ O ₃ :20Na ₂ O:2Ho ₂ O ₃

LLASP (UD) -Represents undopedLead Lithium AluminoSodium Phosphate glass specimen.

LLASP(HO)-Represents Ho³⁺dopedLead Lithium AluminoSodium Phosphateglass specimens.

III.THEORY

3.1Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [25].

$$f_{\text{expt.}} = 4.318 \times 10^{-9} \int \epsilon(v) dv \quad (1)$$

where, $\epsilon(v)$ is molar absorption coefficient at a given energy v (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 [26], using the modified relation:

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

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

3.2. Judd-Ofelt Intensity Parameters

According to Judd[27] and Ofelt[28] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial J manifold $|4f^N(S, L) J\rangle$ level and the terminal J' manifold $|4f^N(S', L') J'\rangle$ is given by:

$$\frac{8\pi^2 m c \bar{\nu}}{3h(2J+1)n} \left[\frac{(n^2+2)^2}{9} \right] \times S(J, J') \quad (3)$$

Where, the line

strength $S(J, J')$ is given by the equation

$$S(J, J') = e^2 \sum \Omega_\lambda \langle 4f^N(S, L) J \| U^{(\lambda)} \| 4f^N(S', L') J' \rangle^2 \quad (4)$$

 $\lambda = 2, 4, 6$

In the above equation m is the mass of an electron, c is the velocity of light, $\bar{\nu}$ 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 ($\lambda=2, 4$ and 6) are known as Judd-Ofelt intensity.

3.3 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 $|4f^N(S', L') J'\rangle$ to a final manifold $|4f^N(S, L) J\rangle$ is given by:

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

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

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

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

S, L, J

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{rad} = \sum A[(S', L') J'; (S, L) J] = A_{total}^{-1} \quad (7)$$

S, L, J

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

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

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

3.4 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 [29, 30]. The Nephelauxetic Ratio is given by

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

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

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

IV. RESULT AND DISCUSSION

4.1 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.

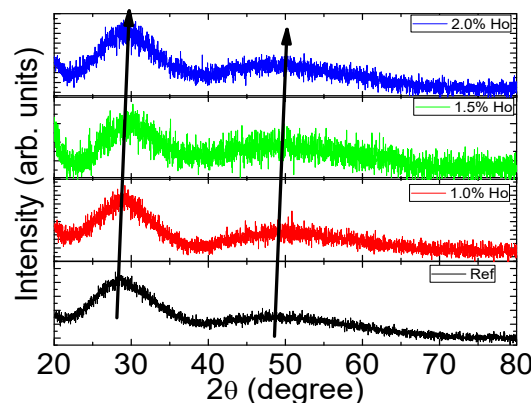


Fig. 1 X-ray diffraction pattern of $\text{P}_2\text{O}_5:\text{PbO}:\text{Li}_2\text{O}:\text{Al}_2\text{O}_3:\text{Na}_2\text{O}:\text{Ho}_2\text{O}_3$

4.2 Transmittance Spectrum

The Transmittance spectrum of Ho^{3+} doped in leadlithium aluminosodium phosphate glass is shown in Figure 2.

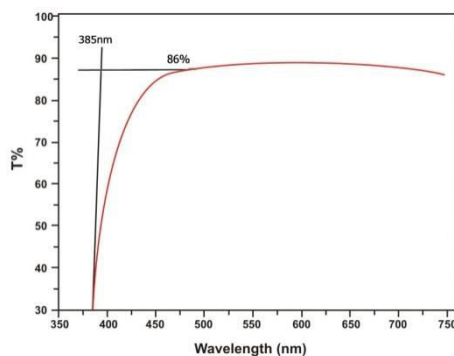


Fig. (2) Transmittance spectrum of Ho^{3+} doped LLASP glasses.

4.3 Absorption Spectrum

The absorption spectra of Ho^{3+} doped LLASP 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 $^5\text{I}_8$ to excited states $^5\text{I}_5$, $^5\text{I}_4$, $^5\text{F}_5$, $^5\text{F}_4$, $^5\text{F}_3$, $^3\text{K}_8$, $^5\text{G}_6$, $(^5\text{G}, ^3\text{G})_5$, $^5\text{G}_4$, $^5\text{G}_2$, $^5\text{G}_3$, and $^3\text{F}_4$ for Ho^{3+} doped LLASP glasses.

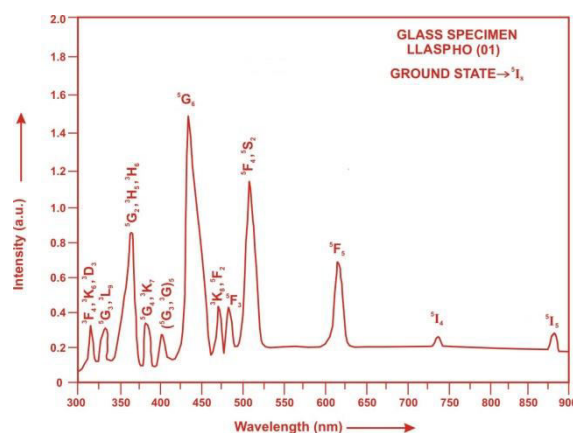


Fig. (3) Absorption spectrum of LLASP HO (01) glass.

The experimental and calculated oscillator strength for Ho^{3+} ions in LLASP glasses are given in Table 2.

Table 2: Measured and calculated oscillator strength ($P_m \times 10^{-6}$) of Ho^{3+} ions in LLASP glasses.

Energy level from $^5\text{I}_8$	Glass LLASP(HO01)		Glass LLASP(HO1.5)		Glass LLASP(HO02)	
	P_{exp}	P_{cal}	P_{exp}	P_{cal}	P_{exp}	P_{cal}
$^5\text{I}_5$	0.39	0.24	0.34	0.24	0.29	0.23
$^5\text{I}_4$	0.08	0.02	0.06	0.02	0.05	0.02
$^5\text{F}_5$	3.66	2.83	3.61	2.81	3.56	2.68
$^5\text{F}_4$	4.68	4.37	4.62	4.34	4.57	4.19
$^5\text{F}_3$	1.60	2.42	1.56	2.40	1.48	2.33
$^3\text{K}_8$	1.48	1.98	1.41	1.97	1.36	1.91
$^5\text{G}_6$	25.28	25.26	24.86	24.88	23.75	24.02

$(^5G, ^3G)_5$	3.94	1.75	3.88	1.74	3.81	1.60
5G_4	0.09	0.61	0.08	0.61	0.07	0.59
5G_2	5.54	5.38	5.48	5.31	5.42	5.13
5G_3	1.52	1.39	1.48	1.38	1.48	1.34
3F_4	1.39	4.27	1.32	4.23	1.26	3.96
r.m.s. deviation	± 1.1227		± 1.1234		± 1.1189	

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

Table 3: 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 Lead Lithium Aluminosodium Phosphate Glasses (LLASP) 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 0.627 and 0.628 in the present glasses.

The values of Judd-Ofelt intensity parameters are given in Table 4.

Table 4: Judd-Ofelt intensity parameters for Ho^{3+} doped LLASP glass specimens.

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4/Ω_6	Ref.
LLASP (HO01)	6.098	1.384	2.202	0.6285	P.W.
LLASP (HO1.5)	5.993	1.371	2.184	0.6277	P.W.
LLASP (HO02)	5.677	1.355	2.157	0.6281	P.W.
SBT	3.17	0.36	0.54	0.6667	[31]
PHOSPHATE	6.28	1.03	1.39	0.7410	[32]

4.4 Excitation Spectrum

The Excitation spectra of Ho^{3+} doped LLASP 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 545 nm having different excitation band centered at 349, 419, 452, 473 and 486 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 452 nm. So this is to be chosen for excitation wavelength.

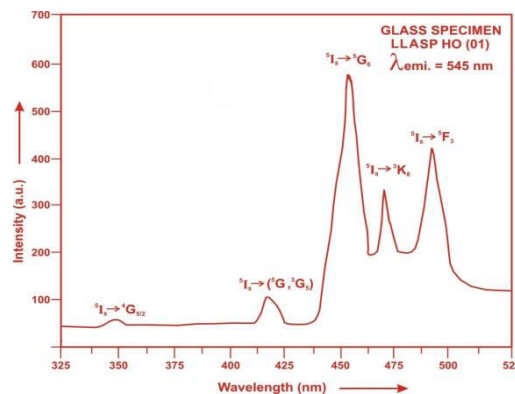


Fig. (4) Excitation spectrum of LLASP HO (01) glass.

4.5 Fluorescence Spectrum

The fluorescence spectrum of Ho^{3+} doped in leadlithium aluminosodium phosphateglass is shown in Figure 5. There are nine broad bands observed in the Fluorescence spectrum of Ho^{3+} doped leadlithium aluminosodium phosphateglass. The wavelengths of these bands along with their assignments are given in Table 5. The peak with maximum emission intensity appears at 555nm and corresponds to the ($^5\text{F}_4 \rightarrow ^5\text{I}_8$) transition.

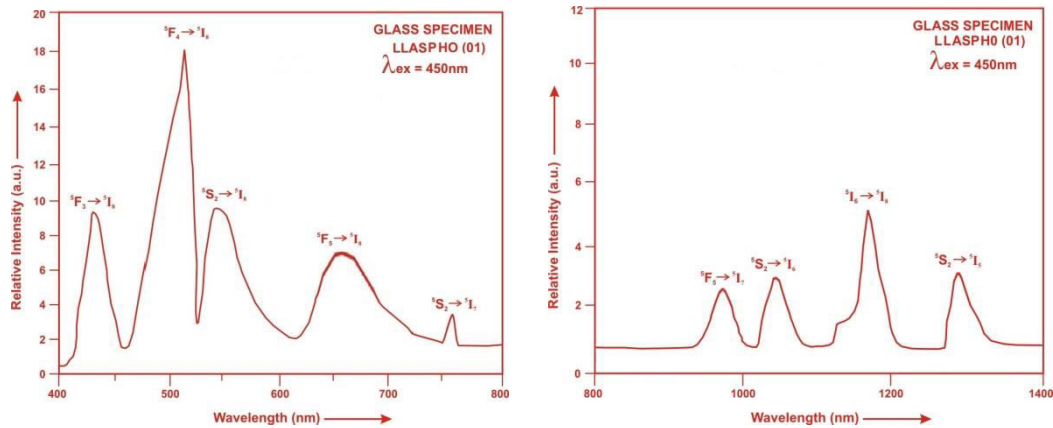


Fig. (5). Fluorescence spectrum of LLASP HO (01) glass.

Table 5: 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 LLASP glasses.

Transition	λ_{max} (nm)	LLASP (HO 01)				LLASP (HO 1.5)				LLASP (HO 02)			
		$A_{\text{rad}}(\text{s}^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(\mu\text{s})$	$A_{\text{rad}}(\text{s}^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(\mu\text{s})$	$A_{\text{rad}}(\text{s}^{-1})$	β	σ_p (10^{-20} cm^2)	$\tau_R(10^{-20}$ cm^2)
$^5\text{F}_3 \rightarrow ^5\text{I}_8$	435	4206.25	0.249 5	0.617	5932.3 2	4177.73	0.2495	0.608	5971.6 9	4142.0 5	0.249 5	0.586	6022.66
$^5\text{F}_4 \rightarrow ^5\text{I}_8$	501	6698.75	0.397 4	1.273		6656.07	0.3975	1.248		660.06 5	0.397 5	1.219	
$^5\text{S}_2 \rightarrow ^5\text{I}_8$	555	1755.75	0.104 2	0.442		1744.86	0.1042	0.437		1730.1 4	0.104 2	0.427	
$^5\text{F}_5 \rightarrow ^5\text{I}_8$	652	1918.06	0.113 8	0.750		1903.66	0.1137	0.741		1888.0 3	0.113 7	0.723	
$^5\text{S}_2 \rightarrow ^5\text{I}_7$	761	1330.91	0.078 9	1.147		1323.18	0.7902	1.126		1312.0 2	0.790 2	1.096	
$^5\text{F}_5 \rightarrow ^5\text{I}_7$	995	446.49	0.026 5	1.225		443.25	0.0265	1.207		438.22 4	0.026 4	1.176	
$^5\text{I}_6 \rightarrow ^5\text{I}_8$	1032	204.77	0.012 1	0.705		203.47	0.0122	0.691		201.83 2	0.012 2	0.670	
$^5\text{S}_2 \rightarrow ^5\text{I}_5$	1195	233.55	0.013 9	1.229		232.03	0.0139	1.207		229.71 8	0.013 8	1.179	
$^5\text{S}_2 \rightarrow ^5\text{I}_6$	1310	62.27	0.003 7	0.635		61.90	0.0037	0.621		61.38 7	0.003 7	0.601	

V. CONCLUSION

In the present study, the glass samples of composition $(45-x)\text{P}_2\text{O}_5:10\text{PbO}:10\text{Li}_2\text{O}:15\text{Al}_2\text{O}_3:20\text{Na}_2\text{O}:x\text{Ho}_2\text{O}_3$. (where $x = 1, 1.5$ and 2 mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition ($^5\text{F}_4 \rightarrow ^5\text{I}_8$) for glass LLASP(HO 01), suggesting that glass LLASP (HO



01) is better compared to the other two glass systems LLASP (HO1.5) and LLASP(HO02). On the basis of spectrophotometric, transmittance reaches about 86% for all phosphate glasses doped with Ho³⁺ ions.

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