



Spectral and Raman Analysis of Ho^{3+} ions doped Zinc Lithium Tungsten Aluminoborosilicate Glasses

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ABSTRACT: Glass of the system: $(40-x)\text{SiO}_2:10\text{ZnO}:10\text{Li}_2\text{O}:10\text{WO}_3:10\text{Al}_2\text{O}_3:20\text{B}_2\text{O}_3:x\text{Ho}_2\text{O}_3$ (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 Raman 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: ZLTABS Glasses, Optical Properties, Judd-Ofelt Theory, Raman Analysis.

I. INTRODUCTION

Rare-earth oxide doped glasses have drawn a lot attention due to their particular extensive applications such as sensor, optical fiber, solid-state laser and energy storage [1-5]. In general, the optical and spectroscopic properties of rare earth ions are strongly dependent on host materials. The host glass materials should have high refractive index with good chemical and thermal stability [6-10]. Due to a substantially higher operational performance, ceramic glasses might be a relatively inexpensive alternative to the epoxy materials. In recent decade an increasing interest in rare earth doped glasses from the viewpoint of their spectroscopic properties and technological applications in many fields [11-15]. Silicate glasses are chemically durable, thermally stable and optically transparent at the excitation and lasing wavelength [16-20]. Borosilicate glasses are more suitable due to its high refractive index, low phonon energy and good chemical suitability [21-23]. Silicate glasses can be a good matrix for fiber lasers because they exhibit good mechanical property and relatively high doping concentration ability [24-26].

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 absorption and emission properties of Ho^{3+} doped zinc lithium tungsten aluminoborosilicate 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 borosilicate glass samples $(40-x)\text{SiO}_2:10\text{ZnO}:10\text{Li}_2\text{O}:10\text{WO}_3: 10\text{Al}_2\text{O}_3:20\text{B}_2\text{O}_3:x\text{Ho}_2\text{O}_3$ (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 $\text{SiO}_2, \text{ZnO}, \text{Li}_2\text{O}, \text{WO}_3, \text{B}_2\text{O}_3$ 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 %)
ZLTABS(UD)	40SiO ₂ :10ZnO:10Li ₂ O:10WO ₃ :10Al ₂ O ₃ :20B ₂ O ₃
ZLTABS (HO1)	39SiO ₂ :10ZnO:10Li ₂ O:10WO ₃ :10Al ₂ O ₃ :20B ₂ O ₃ :1 Ho ₂ O ₃
ZLTABS(HO1.5)	38.5SiO ₂ :10ZnO:10Li ₂ O:10WO ₃ :10Al ₂ O ₃ :20B ₂ O ₃ :1.5Ho ₂ O ₃
ZLTABS(HO2)	38 SiO ₂ :10ZnO:10Li ₂ O:10WO ₃ :10Al ₂ O ₃ :20B ₂ O ₃ :2Ho ₂ O ₃

ZLTABS (UD) -Represents undoped Zinc Lithium Tungsten AluminoBorosilicate glass specimen.

ZLTABS(HO)-Represents Ho³⁺doped Zinc Lithium Tungsten AluminoBorosilicate glass specimens.

III. THEORY

A. Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [27].

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

where, $\epsilon(\nu)$ 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 [28], using the modified relation:

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

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 $\Delta\nu_{1/2}$ is half band width.

B. Judd-Ofelt Intensity Parameters

According to Judd[29] and Ofelt[30] 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:

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

Where, the line

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

$\lambda = 2, 4, 6$

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 ($\lambda=2, 4$ and 6) are known as Judd-Ofelt intensity.

C. 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:

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

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

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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.

D. 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 [31, 32]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{v_g}{v_a} \quad (9)$$

where, v_a and v_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

A. XRD Measurement

Figure 1 presents the XRD pattern of the sample contain - SiO₂ 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.

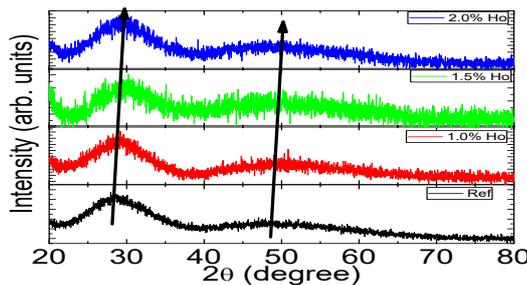


Fig. 1 X-ray diffraction pattern of SiO₂:ZnO:Li₂O:WO₃:Al₂O₃:B₂O₃:Ho₂O₃

B. Raman spectra

The Raman spectrum of Zinc Lithium Tungsten AluminoBorosilicate(ZLTABS) glass specimens is recorded and is shown in Fig. 2. The spectrum peaks located at 600, 797 and 1205 cm^{-1} . The band at 600 and 797 cm^{-1} assigned to Si-O-Si symmetric stretching and bending vibration, respectively. The band at 1205 cm^{-1} assigned to Si-O-Si asymmetric stretching.

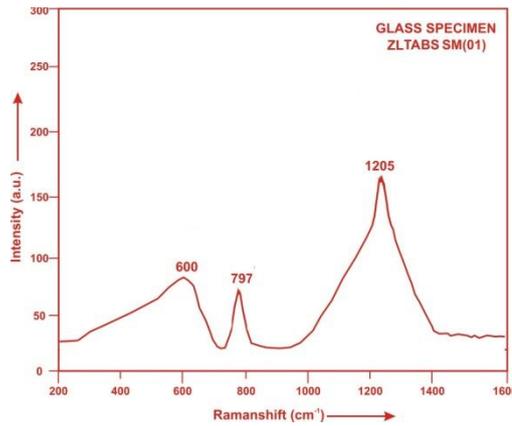


Fig. (2) Raman spectrum of ZLTABS HO (01) glass.

C. Absorption Spectrum

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

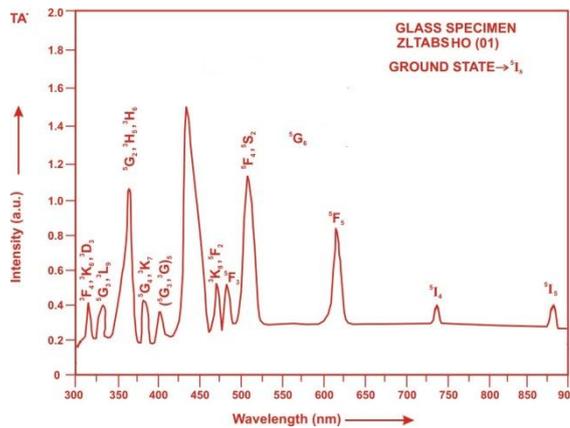


Fig. (3) Absorption spectrum of ZLTABS HO (01)glass.

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

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

Energy level from 5I_8	Glass ZLTABS(HO01)		Glass ZLTABS(HO1.5)		Glass ZLTABS(HO02)	
	P_{exp}	P_{cal}	P_{exp}	P_{cal}	P_{exp}	P_{cal}
5I_5	0.44	0.24	0.41	0.24	0.36	0.24
5I_4	0.07	0.02	0.06	0.02	0.04	0.02
5F_5	3.68	2.81	3.62	2.79	3.56	2.76
5F_4	4.68	4.35	4.64	4.32	4.58	4.27
5F_3	1.57	2.41	1.54	2.40	1.49	2.37
3K_8	1.44	1.97	1.41	1.96	1.37	1.93
5G_6	24.88	24.86	24.81	24.81	23.96	23.99
$(^5G, ^3G)_5$	3.86	1.72	3.81	1.71	3.77	1.69
5G_4	0.09	0.61	0.08	0.60	0.06	0.59
5G_2	5.69	5.31	5.63	5.30	5.58	5.14
5G_3	1.50	1.38	1.46	1.38	1.42	1.35
3F_4	1.40	4.21	1.37	4.18	1.32	4.14
r.m.s. deviation	± 1.1102		± 1.1019		± 1.1020	

Computed values of F_2 , Lande' parameter (ξ_{4f}), Nephelauxetic ratio (β') and bonding parameter ($b^{1/2}$) for Ho^{3+} ions in ZLTABS 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 Zinc Lithium Tungsten AluminoBorosilicate glasses (ZLTABS) 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 0.618 and 0.619 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 ZLTABS glass specimens.

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4/Ω_6
ZLTABS (HO01)	5.196	1.176	1.901	0.6186
ZLTABS (HO1.5)	5.188	1.168	1.887	0.6189
ZLTABS (HO02)	4.985	1.155	1.865	0.6193

D. Excitation Spectrum

The Excitation spectra of Ho^{3+} doped ZLTABS 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 349, 420, 452, 475 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 452nm. So this is to be chosen for excitation wavelength.

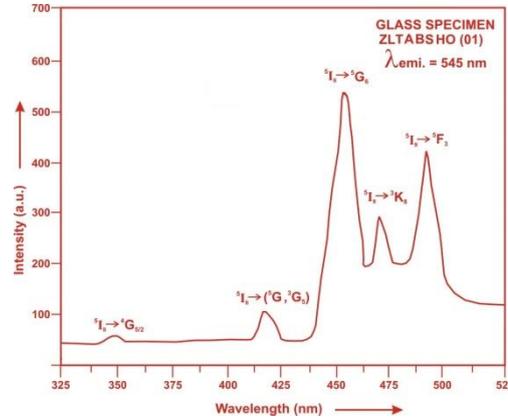


Fig. (4) Excitation spectrum of ZLTABS HO (01) glass.

E. Fluorescence Spectrum

The fluorescence spectrum of Ho³⁺ doped in zinc lithium tungsten aluminoborosilicate glass is shown in Figure 5. There are nine broad bands observed in the Fluorescence spectrum of Ho³⁺ doped zinc lithium tungsten aluminoborosilicate glass. 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 ($^3F_4 \rightarrow ^5I_8$) transition.

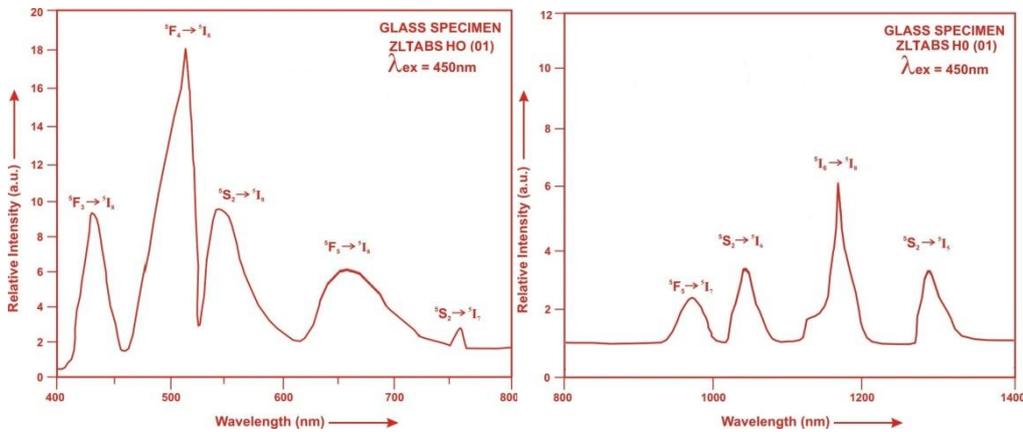


Fig. (5). Fluorescence spectrum of ZLTABS HO (01) glass.

Table5: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 ZLTABS glasses.

Transition	ZLTABS(HO 01)					ZLTABS(HO 1.5)				ZLTABS (HO 02)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	$\sigma_p(10^{-20} cm^2)$	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p(10^{-20} cm^2)$	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p(10^{-20} cm^2)$	$\tau_R(10^{-20} cm^2)$
$^5F_3 \rightarrow ^5I_8$	435	5121.46	0.2499	0.580	4879.28	5093.40	0.2499	0.562	4905.38	5043.59	0.2499	0.547	4955.33
$^5F_4 \rightarrow ^5I_8$	501	8143.34	0.3973	1.204		8099.36	0.3973	1.189		8015.93	0.3972	1.163	
$^5S_2 \rightarrow ^5I_8$	555	2136.61	0.1043	0.422		2126.05	0.1043	0.417		2104.12	0.1043	0.408	
$^5F_5 \rightarrow ^5I_8$	652	2323.64	0.1134	0.727		2321.13	0.1134	0.716		2289.10	0.1134	0.702	
$^5S_2 \rightarrow ^5I_7$	761	1620.49	0.0791	1.119		1612.25	0.0791	1.099		1596.48	0.0791	1.071	
$^5F_5 \rightarrow ^5I_7$	995	540.52	0.0264	1.186		537.73	0.0264	1.165		531.56	0.0263	1.137	
$^5I_6 \rightarrow ^5I_8$	1032	248.95	0.0121	0.684		247.68	0.0121	0.675		245.27	0.0122	0.656	
$^5S_2 \rightarrow ^5I_5$	1195	284.03	0.0139	1.192		282.61	0.0139	1.178		279.59	0.0139	1.146	
$^5S_2 \rightarrow ^5I_6$	1310	75.80	0.0037	0.619		75.39	0.0037	0.606		74.65	0.037	0.591	

V. CONCLUSION

In the present study, the glass samples of composition $(40-x)SiO_2:10ZnO:10Li_2O:10WO_3:10Al_2O_3:20B_2O_3:xHo_2O_3$. (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 ($^5F_4 \rightarrow ^5I_8$) for glass ZLTABS (HO 01), suggesting that glass ZLTABS (HO 01) is better compared to the other two glass systems ZLTABS (HO1.5) and ZLTABS (HO02). The increasing stability of the glass samples shows that they are thermally resistant.

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