

Spectral and Thermal Properties of Dy³⁺ ions Doped Phosphate Glasses for White LED Technology

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ABSTRACT: Glass of the system: (20-x)P₂O₅: 10ZnO: 10Li₂O 10Na₂O: 10CaO: 10WO₃: 10Sb₂O₃:20Y₂O₃:xDy₂O₃. (where x=1, 1.5,2 mol %) have been prepared by melt-quenching technique. The amorphous nature of the prepared glass samples was confirmed by X-ray diffraction. Optical absorption and fluorescence spectra were recorded at room temperature for all glass samples. DTA curve was analysed to evaluate the glass transition temperature, crystallization temperature and melting temperature. Judd-Ofelt intensity parameters Ω_λ ($\lambda=2, 4$ and 6) are evaluated from the intensities of various absorption bands of optical absorption spectra. The radiative properties like spontaneous emission probability (A), branching ratio (β), radiative life time(τ_R),stimulated emission cross-section(σ_p)and thermal properties have been evaluated.

KEYWORDS: YZLSLTAP Glasses, Spectral Properties, Judd-Ofelt Theory, Thermal Properties.

I. INTRODUCTION

Rare earth doped ceramic glasses have many attractive applications such as optical waveguide, optoelectronic devices, solid state ionic devices, optical data transmission, laser action, thermionic emission and optical amplifiers [1-5]. Among different host matrices, phosphate glasses have wide range applications in the field of glass ceramics such as high transparency from near Ultra Violet to mid-Infrared region, good physical and chemical stability [6-10]. Phosphate glasses have a best thermo-optical performance with considerable with weak upconversion, chemical durability and high gain [11-13]. They present superior properties that include high transparency, high gain density, high thermal expansion coefficient low melting point and high thermal stability [14-16]. Phosphate glasses are very well known for their suitable chemical homogeneity. The addition of network modifier (NWF) Li₂O is to improve both electrical and mechanical properties of such glasses. Phosphate glasses also exhibit high RE ions solubility [17,18].

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 absorption, emission and thermal properties of Dy³⁺ doped yttrium zinc lithium sodalime tungsten antimony 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).The glass transition temperature(T_g), crystallization temperature(T_c) and melting temperature(T_m) are calculated using DTA curve.

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Dy³⁺doped phosphate glass samples (20-x)P₂O₅: 10ZnO: 10Li₂O 10Na₂O: 10CaO: 10WO₃: 10Sb₂O₃:20Y₂O₃:xDy₂O₃. (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 P₂O₅, ZnO, Li₂O,Na₂O,CaO, WO₃, Sb₂O₃, Y₂O₃ and Dy₂O₃. They were thoroughly mixed by using an agate pestle mortar. then melted at 1025⁰C 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 350⁰C 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**.

Table1. Chemical composition of the glasses

Sample	Glass composition (mol %)
YZLSLTAP (UD)	20P ₂ O ₅ : 10ZnO: 10Li ₂ O 10Na ₂ O: 10CaO: 10WO ₃ : 10Sb ₂ O ₃ :20Y ₂ O ₃
YZLSLTAP DY (1.0)	19P ₂ O ₅ : 10ZnO: 10Li ₂ O 10Na ₂ O: 10CaO: 10WO ₃ : 10Sb ₂ O ₃ :20Y ₂ O ₃ : 1 Dy ₂ O ₃ .
YZLSLTAP DY (1.5)	18.5P ₂ O ₅ : 10ZnO: 10Li ₂ O 10Na ₂ O: 10CaO: 10WO ₃ : 10Sb ₂ O ₃ :20Y ₂ O ₃ :1.5 Dy ₂ O ₃ .
YZLSLTAP DY (2.0)	18P ₂ O ₅ : 10ZnO: 10Li ₂ O 10Na ₂ O: 10CaO: 10WO ₃ : 10Sb ₂ O ₃ :20Y ₂ O ₃ :2Dy ₂ O ₃ .

YZLSLTAP (UD) -Represents undoped Yttrium Zinc Lithium Sodalime Tungsten Antimony Phosphate glass specimens.

YZLSLTAP (DY) -Represents Dy³⁺ doped Yttrium Zinc Lithium Sodalime Tungsten Antimony Phosphate glass specimens.

III. THEORY

3.1 Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [19].

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

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 [20], 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, I is the optical path length, logI₀/I is optical density and $\Delta\nu_{1/2}$ is half band width.

3.2. Judd-Ofelt Intensity Parameters

According to Judd [21] and Ofelt [22] 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> level and the terminal J' manifold |4f^N(S', L') J'> is given by:

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

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

$$S(J, J') = e^2 \sum_{\lambda=2,4,6} \Omega_{\lambda} \langle 4f^N(S, L) J \| U^{(\lambda)} \| 4f^N(S', L') J' \rangle^2 \quad (4)$$

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 parameters .

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:

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

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

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

$$\beta[(S', L') J'; (S, L) J] = \sum_{S, L, J} \quad (6)$$

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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.

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.

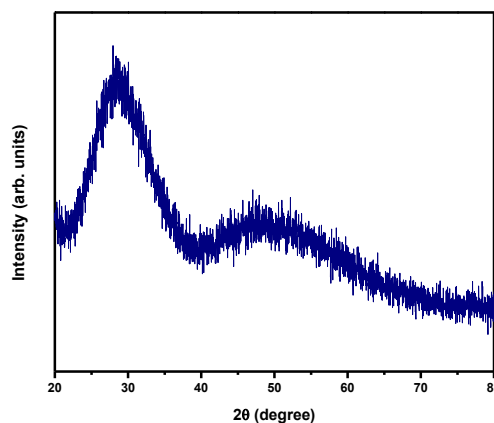


Fig. 1 X-ray diffraction pattern of $P_2O_5: ZnO: Li_2O: Na_2O: CaO: WO_3: Sb_2O_3: Y_2O_3: Dy_2O_3$.

4.2 Thermal Property

Differential thermal analysis checks the heat absorbed by glass samples during heating or cooling. Fig. 2 depicts the DTA thermogram of powdered YZLSLTAP DY(1.0) sample. The glass transition temperature (T_g), onset crystallization temperature (T_c), crystallization temperature (T_p), melting temperature (T_m), thermal stability (T_s), Balaji Parameter (B_p), Hurby's criterion (H_R) and reduced glass transition temperature (T_{rg}) were calculated. Shankar's parameter also calculated by using eq. (13). All the determined thermal parameters are given in table 2.

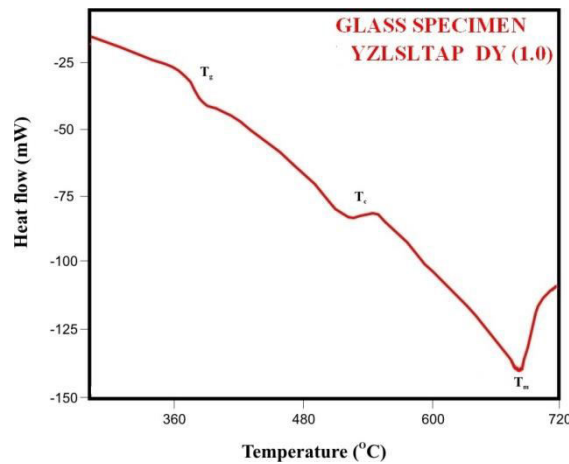


Fig.2 DTA curve of YZLSLTAP DY (1.0) glass.

Table 2: Thermal parameters determined from the DTA traces of YZLSLTAP DY glasses.

Sample Name	T_g ($^{\circ}\text{C}$)	T_c ($^{\circ}\text{C}$)	T_p ($^{\circ}\text{C}$)	T_m ($^{\circ}\text{C}$)	T_s ($^{\circ}\text{C}$)	B_p ($^{\circ}\text{C}$)	H_R ($^{\circ}\text{C}$)	K_s ($^{\circ}\text{C}$)	T_{rg} ($^{\circ}\text{C}$)
YZLSLTAP DY (1.0)	375	510	548	685	135	3.553	0.217	34.489	0.545
YZLSLTAP DY (1.5)	380	512	550	688	132	3.385	0.222	33.767	0.552
YZLSLTAP DY (2.0)	385	514	555	694	129	3.146	0.218	34.945	0.555

The thermal stability of the glass samples can be calculated by difference between onset crystallization temperature and transition temperature [23].

$$\text{Thermal Stability } (T_s) = T_c - T_g \quad (9)$$

Balaji Parameter can be calculated using [23].

$$\text{Balaji Parameter } (B_p) = [(T_c - T_g) / (T_p - T_c)] \quad (10)$$

Hruby's criterion is calculated using the Hruby's relation [23].

$$\text{Hruby's criterion } (H_R) = [(T_p - T_c) / (T_m - T_c)] \quad (11)$$

Reduced glass transition temperature is given as [23].

$$\text{Reduced glass transition temperature } (T_{rg}) = T_g / T_m \quad (12)$$

Thermal Parameter is given as [23].

$$K_s = [(T_m - T_c) (T_c - T_g) / T_m] \tag{13}$$

4.3 Absorption Spectrum

The absorption spectra of Dy³⁺-doped YZLSLTAP DY(1.0) glass specimen has been presented in Figure 3 in terms of Intensity versus wavelength. Thirteen absorption bands have been observed from the ground state ⁶H_{15/2} to excited states ⁶H_{13/2}, ⁶H_{11/2}, ⁶H_{9/2}+⁶F_{11/2}, ⁶H_{7/2}+⁶F_{9/2}, ⁶F_{7/2}+⁶H_{5/2}, ⁶F_{5/2}, ⁶F_{3/2}, ⁶F_{9/2}, ⁴I_{15/2}, ⁴G_{11/2}, ⁶F_{7/2}+⁴I_{13/2}, ⁶M_{19/2}+⁴(P,D)_{3/2} and ⁴G_{9/2}+⁶P_{3/2} for Dy³⁺-doped YZLSLTAP DY(1.0) glass.

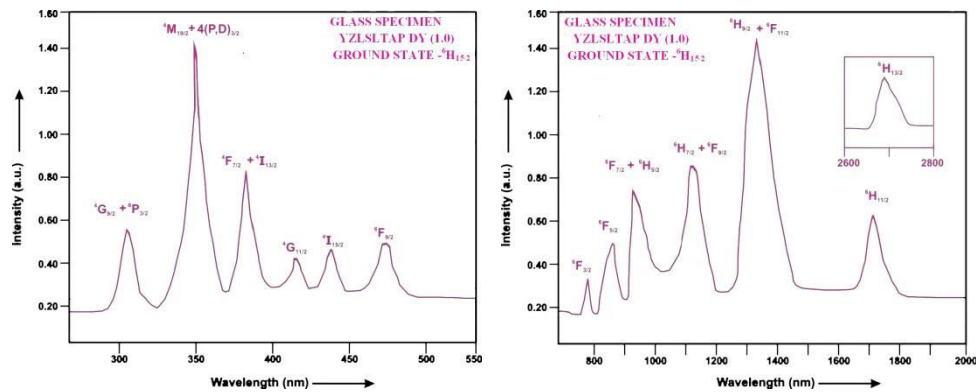


Fig (3) Absorption spectrum of YZLSLTAP DY(1.0) glass.

The experimental and calculated oscillator strength for Dy³⁺ ions in YZLSLTAP glasses are given in Table 3.

Table 3: Measured and calculated oscillator strength (P_m × 10⁺⁶) of Dy³⁺ ions in YZLSLTAP glasses.

Energy level from ⁶ H _{15/2}	Glass YZLSLTAP DY(1.0)		Glass YZLSLTAP DY(1.5)		Glass YZLSLTAP DY(2.0)	
	P _{exp.}	P _{cal.}	P _{exp.}	P _{cal.}	P _{exp.}	P _{cal.}
⁶ H _{13/2}	3.48	3.14	3.46	3.13	3.43	3.14
⁶ H _{11/2}	2.72	3.17	2.70	3.15	2.87	3.21
⁶ H _{9/2} + ⁶ F _{11/2}	11.92	11.97	11.89	11.94	11.85	11.90
⁶ H _{7/2} + ⁶ F _{9/2}	6.98	6.70	6.95	6.68	6.92	6.65
⁶ F _{7/2} + ⁶ H _{5/2}	5.99	5.81	5.96	5.78	5.93	5.85
⁶ F _{5/2}	2.92	2.91	2.89	2.89	2.86	2.95
⁶ F _{3/2}	1.10	0.55	1.08	0.55	1.06	0.56
⁶ F _{9/2}	1.13	0.45	1.11	0.45	1.09	0.45
⁴ I _{15/2}	1.17	1.14	1.13	1.13	1.11	1.15
⁴ G _{11/2}	0.99	0.14	0.96	0.14	0.93	0.13
⁶ F _{7/2} + ⁴ I _{13/2}	4.92	5.14	4.90	5.13	4.86	5.16
⁶ M _{19/2} + ⁴ (P,D) _{3/2}	8.99	10.53	8.96	10.52	8.93	10.30
⁴ G _{9/2} + ⁶ P _{3/2}	2.85	3.12	2.82	3.11	2.78	3.15
r.m.s. deviation	0.5843		0.5819		0.5319	

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

Table 4: Judd-Ofelt intensity parameters for Dy³⁺ doped YZLSLTAP glass specimens

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4 / Ω_6
YZLSLTAP DY(1.0)	3.652	1.322	2.460	0.5374
YZLSLTAP DY(1.5)	3.639	1.325	2.443	0.5424
YZLSLTAP DY(2.0)	3.668	1.245	2.492	0.4996

4.4 Excitation Spectrum

The Excitation spectra of Dy³⁺doped YZLSLTAP glass specimens have been presented in Figure 4 in terms of Excitation Intensity versus wavelength. The excitation spectrum was recorded in the spectral region 315–465 nm fluorescence at 575nm having different excitation band centered at 321,354, 365, 385, 425, 455 and 473 nm are attributed to the ⁶P_{3/2}, ⁶P_{7/2}, ⁴P_{3/2}, ⁴I_{13/2}, ⁴G_{11/2}, ⁴I_{15/2} and ⁴F_{9/2} transitions, respectively. The highest absorption level is ⁴I_{13/2} and is at 385nm. So this is to be chosen for excitation wavelength.

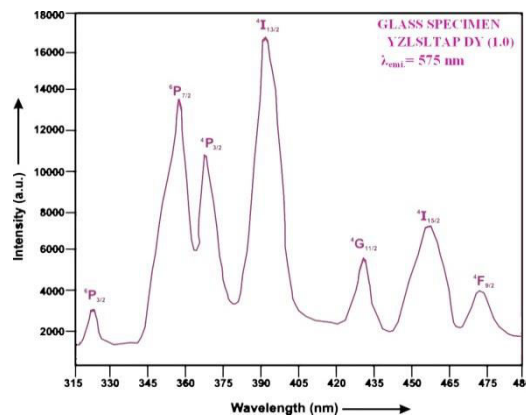


Fig (4) Excitation spectrum of YZLSLTAP DY(1.0)glass.

4.5. Fluorescence Spectrum

The fluorescence spectrum of Dy³⁺doped in yttrium zinc lithium sodalime tungsten antimony phosphate glass is shown in Figure 5. There are four broad bands observed in the Fluorescence spectrum of Dy³⁺doped yttrium zinc lithium sodalime tungsten antimony phosphate glass. The wavelengths of these bands along with their assignments are given in Table 5. The peak with maximum emission intensity appears at 485nm, 575 nm, 665 nm,752nm and corresponds to the (⁴F_{9/2}→⁶H_{15/2}), (⁴F_{9/2}→⁶H_{13/2}),(⁴F_{9/2}→⁶H_{11/2}) and (⁴F_{9/2}→⁶H_{9/2}), transition.

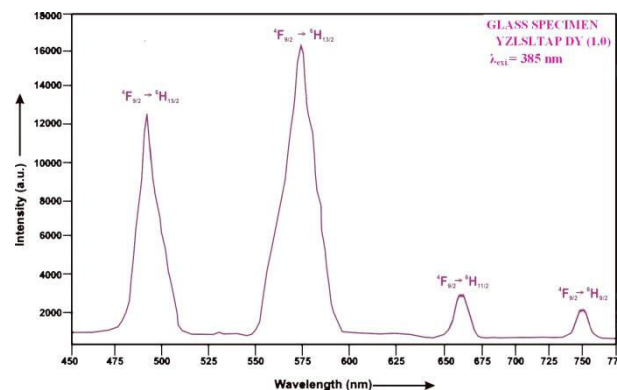


Fig (5) Fluorescence spectrum of doped YZLSLTAP DY(1.0).

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 Dy³⁺ doped YZLSLTAP glasses.

Transition	YZLSLTAP DY(1.0)					YZLSLTAP DY(1.5)				YZLSLTAP DY(2.0)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p ($10^{20}cm^2$)	$\tau_r(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p ($10^{20}cm^2$)	$\tau_r(\mu s)$	$A_{rad}(s^{-1})$	β	σ_p ($10^{20}cm^2$)	τ_r ($10^{20}cm^2$)
$^4F_{9/2} \rightarrow ^6H_{15/2}$	485	126.48	0.2240	0.219	1770.95	125.95	0.2237	0.211	1775.94	127.93	0.2247	0.206	1756.61
$^4F_{9/2} \rightarrow ^6H_{13/2}$	575	372.62	0.6599	1.332		371.68	0.6601	1.292		375.53	0.6597	1.263	
$^4F_{9/2} \rightarrow ^6H_{11/2}$	665	37.33	0.0661	0.164		37.26	0.0662	0.158		37.58	0.0660	0.157	
$^4F_{9/2} \rightarrow ^6H_{9/2}$	752	28.24	0.0500	0.170		28.20	0.0501	0.168		28.24	0.0496	0.164	

V. CONCLUSION

In the present study, the glass samples of composition (20-x)P 2 O 5 : 10ZnO: 10Li 2 O 10Na 2 O: 10CaO: 10WO 3 : 10Sb 2 O 3 :20Y 2 O 3 :xDy 2 O 3. (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The large value

of Balaji parameter indicate that the prepared glass samples have good thermal stability.The value of stimulated emission cross-section (σ_p) is found to be maximum for the transition (4 F 9/2 → 6 H 13/2) for all glass specimens. This shows that (4 F 9/2 → 6 H 13/2) transition is most probable transition.

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