



Spectral and FTIR Analysis of Dy³⁺ Doped Zinc Lithium Lead Calcium Alumino Tellurite Glasses

S.L.Meena

*Ceramic Laboratory, Department of Physics,
Jai Narain Vyas University, Jodhpur 342001 (Raj.) India
E-mail address: shankardiya7@rediffmail.com*

Abstract

Glass sample of Zinc Lithium Lead Calcium Alumino Tellurite (40-x)TeO₂:10ZnO:10Li₂O:10PbO:10CaO:20Al₂O₃:xDy₂O₃. (where x=1, 1.5, 2 mol %) have been prepared by melt-quenching method. (where x=1, 1.5 and 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, Excitation, fluorescence and FTIR spectra have been 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, branching ratio, radiative life time and stimulated emission cross-section of various emission lines have been evaluated.

Keywords: ZLLCAT Glasses, Optical Properties, Judd-Ofelt Theory, Transmittance Properties

I. Introduction

Glasses are receiving considerable attention due to their potential application in optical devices such as frequency-conversion materials, laser action and optical fiber amplifiers [1-5]. Among different host matrices, tellurite glasses have wide range of applications in the field of glass ceramics, with the advantages such as low non-linear refractive index, good physical and chemical stability and high transparency [6-10]. Tellurite glasses have relatively low phonon energy and good thermal stability. Additionally, such glasses are characterized by a high capacity for dissolving rare earth elements [11-14]. Recently, glass-ceramics containing dysprosium oxides have been found in applications for several different purposes. Dy³⁺ doped glasses have attracted much interest due to their important optical

properties used in lasers, optical amplifiers, network formers, photonic devices and as infrared sensors [15-18].

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 and emission properties of Dy³⁺ doped zinc lithium calciummagnesium 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).

II. Experimental Techniques

Preparation of glasses

The following Dy³⁺ doped tellurite glass samples (40-x)TeO₂:10ZnO:10Li₂O:10PbO:10CaO:20Al₂O₃: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 TeO₂, ZnO, Li₂O, PbO, CaO, Al₂O₃ and Dy₂O₃. They were thoroughly mixed by using an agate pestle mortar. then melted at 1052⁰C 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 250⁰C 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 %)
ZLLCAT (UD)	40TeO ₂ :10ZnO:10Li ₂ O:10PbO:10CaO:20Al ₂ O ₃
ZLLCAT (DY1)	39TeO ₂ :10ZnO:10Li ₂ O:10PbO:10CaO:20Al ₂ O ₃ :1Dy ₂ O ₃ .
ZLLCAT (DY1.5)	38.5TeO ₂ :10ZnO:10Li ₂ O:10PbO:10CaO:20Al ₂ O ₃ :1.5Dy ₂ O ₃ .
ZLLCAT (DY2)	38TeO ₂ :10ZnO:10Li ₂ O:10PbO:10CaO:20Al ₂ O ₃ :2Dy ₂ O ₃ .
ZLLCAT (UD)	-Represents undoped Zinc Lithium Lead Calcium Alumino Tellurite glass specimen.
ZLLCAT (DY)	-Represents Dy ³⁺ doped Zinc Lithium Lead Calcium Alumino Tellurite 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, l is the optical path length, $\log I_0/I$ 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 $|4f^N(S, L) J\rangle$ level and the terminal J' manifold $|4f^N(S', L') J'\rangle$ is given by:

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

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

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

$\lambda=2, 4, 6$

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 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:

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

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

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

S, L, J

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

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

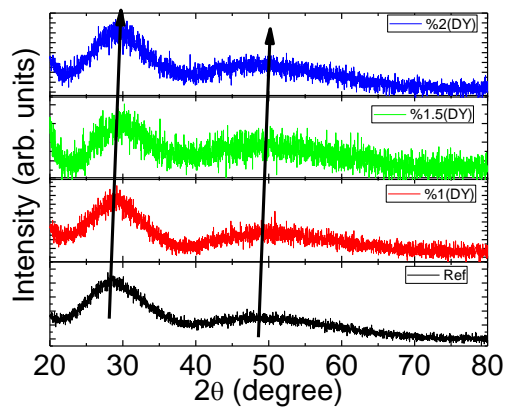
where, λ_p the peak fluorescence wavelength of the emission band and $\Delta\lambda_{eff}$ is the effective fluorescence line width.

IV. Result and Discussion

4.1 XRD Measurement

Figure 1 presents the XRD pattern of the sample contain $-TeO_2$ 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 $\text{TeO}_2:\text{ZnO}:\text{Li}_2\text{O}:\text{PbO}:\text{CaO}:\text{Al}_2\text{O}_3:\text{Dy}_2\text{O}_3$.

4.2 FTIR Transmission spectra

The FTIR spectrum of ZLLCAT (DY 01) glass is in the wave number range $400\text{-}1600\text{cm}^{-1}$ is presented in Fig.2 and the possible mechanism bands are tabulated in Table 2.

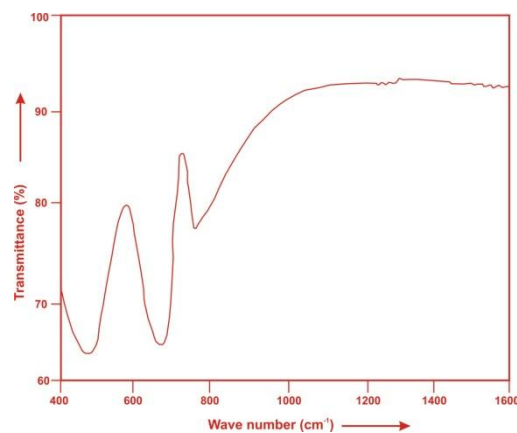


Fig. (2) FTIR spectrum of ZLLCAT DY (01) glass.

The band observed at 472 cm^{-1} is attributed to the symmetric vibration of the Zn-O bond [23]. The observed band around at 680 cm^{-1} is due to the stretching vibration of Te-O bonds in trigonalbipyramidal units TeO_4 [24].while the occurrence of band around 771cm^{-1} is assigned to the vibration of the continuous network composed of TeO_4 and Te-O stretching vibration of TeO_{3+1} polyhedra [25].

Table2. Assignment of infrared transmission bands ofZLLCAT(DY 01) glass.

Peak position(cm ⁻¹)	Band Assignment
~ 472	Symmetric stretching vibration of Zn-O bond
~680	Stretching vibration of Te-O bonds in TeO ₄
~771	Stretching vibration of TeO ₃₊₁ polyhedra

4.3 Absorption Spectrum

The absorption spectra of Dy³⁺ doped ZLLCAT DY (01) 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 ZLLCA DY(01)glass.

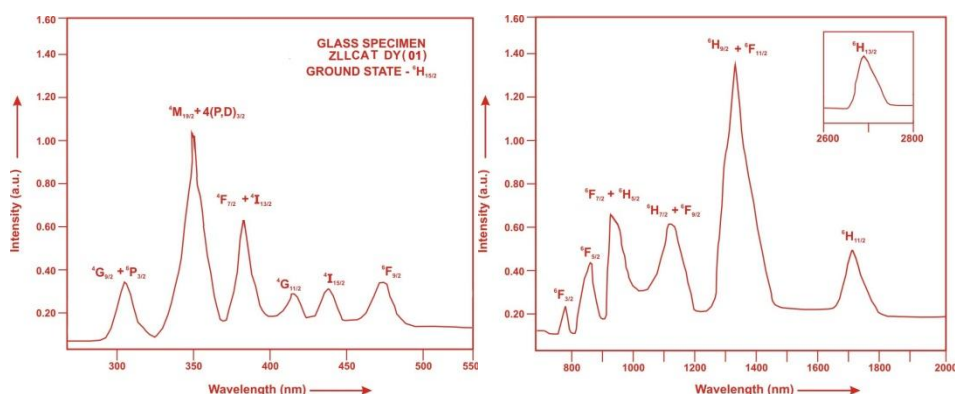


Fig. (3) Absorption spectrum of ZLLCAT DY (01) glass.

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

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

Energy level from ⁶ H _{15/2}	Glass ZLLCAT(DY01)		Glass ZLLCAT(DY1.5)		Glass ZLLCAT(DY02)	
	P _{exp.}	P _{cal.}	P _{exp.}	P _{cal.}	P _{exp.}	P _{cal.}
⁶ H _{13/2}	3.16	2.94	3.13	2.92	3.09	2.90
⁶ H _{11/2}	2.42	2.88	2.38	2.85	2.35	2.81
⁶ H _{9/2} + ⁶ F _{11/2}	11.44	11.43	11.41	11.40	11.37	11.36
⁶ H _{7/2} + ⁶ F _{9/2}	6.52	6.29	6.48	6.26	6.43	6.21
⁶ F _{7/2} + ⁶ H _{5/2}	5.74	5.28	5.71	5.23	5.66	5.17
⁶ F _{5/2}	2.54	2.60	2.50	2.57	2.46	2.54

${}^6F_{3/2}$	0.96	0.49	0.94	0.49	0.91	0.48
${}^6F_{9/2}$	1.03	0.41	1.00	0.40	0.98	0.40
${}^4I_{15/2}$	0.98	1.02	0.96	1.013	0.93	1.00
${}^4G_{11/2}$	0.88	0.14	0.85	0.14	0.82	0.14
${}^6F_{7/2}+{}^4I_{13/2}$	4.59	4.76	4.56	4.72	4.52	4.68
${}^6M_{19/2}+4(P,D)3/2$	8.95	10.28	8.92	10.28	8.87	10.24
${}^4G_{9/2}+{}^6P_{3/2}$	2.68	2.85	2.64	2.83	2.60	2.80
r.m.s. deviation	0.5201		0.5203		0.5165	

In the Zinc Lithium Lead Calcium Alumino Tellurite glasses 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 0.6315 and 0.6552 in the present glasses.

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

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

Glass Specimen	$\Omega_2(\text{pm}^2)$	$\Omega_4(\text{pm}^2)$	$\Omega_6(\text{pm}^2)$	Ω_4/Ω_6	Ref.
ZLLCAT(DY01)	3.509	1.417	2.244	0.6315	P.W.
ZLLCAT(DY1.5)	3.491	1.428	2.215	0.6447	P.W.
ZLLCAT(DY02)	3.476	1.431	2.184	0.6552	P.W.
ZP(DY)	2.483	0.950	0.673	1.412	[26]
LLCTMBB(DY)	2.457	1.471	1.116	1.318	[27]

4.4 Excitation Spectrum

The Excitation spectra of Dy³⁺ doped ZLLCAT glass specimen has 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 322, 353, 365, 385, 425, 454 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.

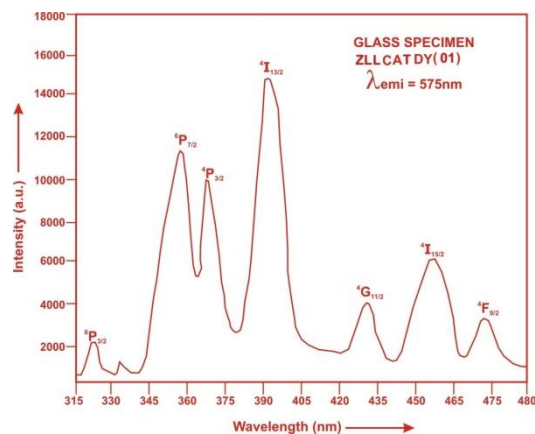


Fig. (4) Excitation spectrum of ZLLCATDY (01) glass.

4.5 Fluorescence Spectrum

The Fluorescence spectrum of Dy^{3+} doped in Zinc Lithium Lead Calcium Alumino Tellurite glass is shown in Figure 5. There are four broad bands observed in the Fluorescence spectrum of Dy^{3+} doped Zinc Lithium Lead Calcium Alumino Tellurite glass. The wavelengths of these bands along with their assignments are given in Table 4. The peak with maximum emission intensity appears at 485nm, 575nm, 665nm and 752 nm corresponds to the ($^4F_{9/2} \rightarrow ^6H_{15/2}$), ($^4F_{9/2} \rightarrow ^6H_{13/2}$), ($^4F_{9/2} \rightarrow ^6H_{11/2}$) and ($^4F_{9/2} \rightarrow ^6H_{9/2}$) transition.

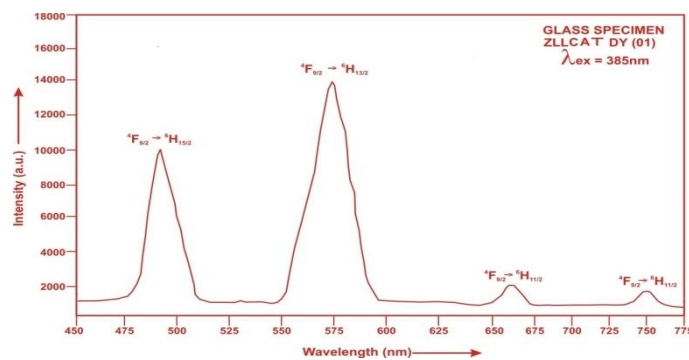


Fig. (5). Fluorescence spectrum of ZLLCATDY (01) glass.

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 Dy^{3+} doped ZLLCAT glasses.



†

Transition	λ_{em} (nm)	ZLLCAT(DY01)				ZLLCAT(DY1.5)				ZLLCAT(DY02)			
		$A_{em}(s^{-1})$	β	σ_e (10^{-20} cm^2)	$\tau_e(\mu s)$	$A_{em}(s^{-1})$	β	σ_e (10^{-20} cm^2)	$\tau_e(\mu s)$	$A_{em}(s^{-1})$	β	$\sigma_e(10^{-20}$ cm^2)	$\tau_e(10^{-20}$ cm^2)
$^4F_{9/2} \rightarrow ^6H_{13/2}$	485	111.12	0.2192	0.247	1971.40	110.14	0.2185	0.241	1983.88	109.05	0.2176	0.233	1995.80
$^4F_{9/2} \rightarrow ^6H_{13/2}$	575	336.13	0.6626	1.564		334.21	0.6630	1.516		332.53	0.6637	1.469	
$^4F_{9/2} \rightarrow ^6H_{11/2}$	665	34.01	0.0670	0.181		33.88	0.0672	0.178		33.75	0.0673	0.173	
$^4F_{9/2} \rightarrow ^6H_{9/2}$	752	25.90	0.0511	0.164		25.83	0.0512	0.162		25.73	0.0513	0.159	

V. Conclusion

In the present study, the glass samples of composition (40-x) $TeO_2:10ZnO:10Li_2O:10PbO:10CaO:20Al_2O_3: xDy_2O_3$ (where x =1, 1.5 and 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 ($^4F_{9/2} \rightarrow ^6H_{13/2}$) for all glass specimens. This shows that ($^4F_{9/2} \rightarrow ^6H_{13/2}$) transition is most probable transition. The FTIR of glasses revealed the presence of characteristic bonding vibrations of different functional groups.

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