

e-ISSN: 2319-8753 | p-ISSN: 2347-6710

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# International Journal of Innovative Research in SCIENCE | ENGINEERING | TECHNOLOGY

# INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

Volume 13, Issue 1, January 2024

INTERNATIONAL STANDARD SERIAL NUMBER INDIA

# Impact Factor: 8.423

9940 572 462

6381 907 438

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|e-ISSN: 2319-8753, p-ISSN: 2347-6710| www.ijirset.com | Impact Factor: 8.423| A Monthly Peer Reviewed & Referred Journal |

Volume 13, Issue 1, January 2024

DOI:10.15680/IJIRSET.2024.1301032

# Structural and Luminescent Properties of Dy<sup>3+</sup> doped in Zinc Lithium Lead Tungsten Aluminophosphate glasses

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**ABSTRACT:** Glass of the system :( 40-x)P<sub>2</sub>O<sub>5</sub>:10ZnO:10Li<sub>2</sub>O:10PbO:10WO<sub>3</sub>:20Al<sub>2</sub>O<sub>3</sub>: x Dy<sub>2</sub>O<sub>3</sub>. (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  $\Omega_{\lambda}$  ( $\lambda$ =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: ZLLTAP Glasses, Optical Properties, Judd-Ofelt Theory, FTIR Spectra.

## I. INTRODUCTION

Rare earth ions doped in different ceramic glasses to achieve favorable potential applications in a variety of optical devices such as lasers, fiber amplifiers, optical, liner, frequency-conversion materials, laser action and optical fiber amplifiers[1-5]. Among different host matrices, phosphate glasses have wide range of applications in the field of glass ceramics, with the advantages such as low non-linear refractive index, high refractive index, good physical and chemical stability and high transparency [6-10]. Phosphate glasses have excellent properties such as low phonon energy, thermal stability, high transparency, chemical durability, high refractive index, high density and optical stability [11-14]. The high gain density in phosphate glasses is due to high solubility of rare earth ions in phosphate network. Phosphate glasses can be a good matrix for fiber lasers because they exhibit good mechanical property. The upconversion of phosphate glasses is also compressed because of their relatively large phonon energy[15-17].Dy<sup>3+</sup> doped glasses have attracted much interest due to their important optical properties used in lasers, optical amplifiers, photonic devices and as infrared sensors [18-20].

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  $Dy^{3+}$  doped in zinc lithium lead tungsten aluminophosphate 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 ( $\beta$ ), radiative life time( $\tau_R$ ) and stimulated emission cross section( $\sigma_p$ ) are evaluated using J.O.intensity parameters( $\Omega_{\lambda}, \lambda=2,4$  and 6).

#### **II. EXPERIMENTAL TECHNIQUES**

#### **Preparation of glasses**

The following  $Dy^{3+}doped$  phosphate glass samples :(40-x)P\_2O\_5:10ZnO:10Li\_2O:10PbO:10WO\_3:20Al\_2O\_3: xDy\_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, PbO, WO\_3, Al\_2O\_3 and Dy\_2O\_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1052°C by an electrical muffle furnace for 2h., After complete melting, the melts were quickly poured in to a preheated stainless steel mould



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### Volume 13, Issue 1, January 2024

# | DOI:10.15680/IJIRSET.2024.1301032 |

and annealed at temperature of  $250^{\circ}$ 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.

ZLLTAP (UD) -Represents undoped zinc lithium lead tungsten aluminophosphate glass specimen. ZLLTAP (DY)-Represents Dy<sup>3+</sup> doped zinc lithium lead tungsten aluminophosphate glass specimens.

#### **III. THEORY**

#### 3.1 Oscillator Strength

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

$$f_{\text{expt.}} = 4.318 \times 10^{-9} [\varepsilon (v) \, \mathrm{d} \, v$$
 (1)

where,  $\varepsilon(v)$  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 [22], using the modified relation:

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

where c is the molar concentration of the absorbing ion per unit volume, I is the optical path length,  $logI_0/I$  is optical density and  $\Delta v_{1/2}$  is half band width.

#### **3.2. 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 J manifold  $|4f^N(S, L) J\rangle$  level and the terminal J' manifold  $|4f^N(S', L') J\rangle$  is given by:

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

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

$$S (J, J') = e^{2} \sum \Omega_{\lambda} < 4f^{N}(S, L) J \| U^{(\lambda)} \| 4f^{N}(S', L') J' > 2$$
(4)  
  $\lambda = 2, 4, 6$ 

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

## **3.3 Radiative Properties**

The  $\Omega_{\lambda}$  parameters obtained using the absorption spectral results have been used to predict radiative properties such as spontaneous emission probability (A) and radiative life time ( $\tau_R$ ), and laser parameters like fluorescence branching ratio ( $\beta_R$ ) and stimulated emission cross section ( $\sigma_p$ ).

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:

(3)



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

Where, S (J', J) =  $e^{2} \left[ \Omega_{2} \| U^{(2)} \|^{2} + \Omega_{4} \| U^{(4)} \|^{2} + \Omega_{6} \| U^{(6)} \|^{2} \right]$ 

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

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

$$S L J$$

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

where,  $\lambda_p$  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 ZLLTAP DY (01)



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### 4.2 FTIR Transmission spectra

The FTIR spectrum of ZLLTAP glass samples are in the wave number range 450-4000 cm<sup>-1</sup> is presented in Fig.2 and the possible mechanism bands are tabulated in Table 2





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

Peak position(cm <sup>-1</sup> )	Band Assignment
~740	symmetric stretching vibration of P-O-P bonding [25]
~892	Asymmetric stretching vibration of P-O-P bonds [25]
~1270	Asymmetric stretching vibration of P=O and O-P-O bonds [25]

## 4.3 Absorption Spectrum

The absorption spectra of  $Dy^{3+}$  doped ZLLTAP glass specimens have been presented in Figure 3 in terms of Intensity versus wavelength. Thirteen absorption bands have been observed from the ground state  ${}^{6}H_{15/2}$  to excited states  ${}^{6}H_{13/2}$ ,  ${}^{6}H_{11/2}$ ,  ${}^{6}H_{9/2} + {}^{6}F_{11/2}$ ,  ${}^{6}H_{7/2} + {}^{6}F_{9/2}$ ,  ${}^{6}F_{5/2}$ ,  ${}^{6}F_{5/2}$ ,  ${}^{6}F_{3/2}$ ,  ${}^{6}F_{9/2}$ ,  ${}^{4}I_{15/2}$ ,  ${}^{6}F_{7/2} + {}^{4}I_{13/2}$ ,  ${}^{6}M_{19/2} + 4(P,D)_{3/2}$  and  ${}^{4}G_{9/2} + {}^{6}P_{3/2}$  for Dy<sup>3+</sup>doped ZLLTAP glasses.



Fig. (3): Absorption spectrum of ZLLTAP DY (01) glass.

The experimental and calculated oscillator strength for Dy<sup>3+</sup> ions in ZLLTAP glasses are given in Table 3.



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DOI:10.15680/IJIRSET.2024.1301032

**Table 3:** Measured and calculated oscillator strength ( $P_m \times 10^{+6}$ ) of Dy<sup>3+</sup>ions in ZLLTAP glasses.

Energy level from	Glass	8	Glass	5	Glass			
<sup>6</sup> H <sub>15/2</sub>	ZLLTAP	<b>(DY01)</b>	ZLLT	AP (DY1.5)	ZLLTAP (DY02)			
	P <sub>exp</sub> .	P <sub>cal</sub> .	P <sub>exp</sub> .	P <sub>cal</sub> .	P <sub>exp</sub> .	P <sub>cal</sub> .		
<sup>6</sup> H <sub>13/2</sub>	2.12	2.42	2.08	2.39	2.02	2.46		
<sup>6</sup> H <sub>11/2</sub>	1.48	2.04	1.45	2.01	1.41	1.49		
${}^{6}\text{H}_{9/2}$ + ${}^{6}\text{F}_{11/2}$	10.32	10.22	10.28	10.17	10.24	10.14		
<sup>6</sup> H <sub>7/2</sub> + <sup>6</sup> F <sub>9/2</sub>	5.60	5.29	5.55	5.24	5.51	5.22		
${}^{6}F_{7/2} + {}^{6}H_{5/2}$	4.76	3.81	4.72	3.75	4.67	4.10		
${}^{6}F_{5/2}$	1.36	1.72	1.32	1.69	1.27	1.95		
${}^{6}F_{3/2}$	0.35	0.32	0.33	0.32	0.30	0.37		
<sup>6</sup> F <sub>9/2</sub>	0.39	0.29	0.36	0.28	0.33	0.31		
${}^{4}I_{15/2}$	0.34	0.71	0.32	0.70	0.28	0.79		
${}^{4}G_{11/2}$	0.28	0.17	0.26	0.17	0.23	0.14		
${}^{6}\mathrm{F}_{7/2} + {}^{4}\mathrm{I}_{13/2}$	3.55	3.71	3.51	3.67	3.46	3.84		
<sup>6</sup> M <sub>19/2</sub> +4(P,D)3/2	7.99	10.01	7.93	9.97	7.86	9.16		
${}^{4}G_{9/2} + {}^{6}P_{3/2}$	1.70	2.10	1.66	2.08	1.62	2.24		
r.m.s. deviation	0.6791		0.6847		0.5253			

Table 4: Judd-Ofelt intensity parameters for Dy<sup>3+</sup> doped ZLLTAP glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(\mathrm{pm}^2)$	$\Omega_6(\mathrm{pm}^2)$	$\Omega_4/\Omega_6$		
ZLLTAP (DY01)	2.947	1.723	1.484	1.161		
ZLLTAP (DY1.5)	2.933	1.723	1.457	1.183		
ZLLTAP (DY02)	3.060	1.416	1.677	0.844		

## 4.4 Excitation Spectrum

The Excitation spectra of Dy<sup>3+</sup>doped ZLLTAP 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  ${}^{6}P_{7/2}$ ,  ${}^{4}P_{3/2}$ ,  ${}^{4}I_{13/2}$ ,  ${}^{4}G_{11/2}$ ,  ${}^{6}G_{11/2}$ ,  ${}^{6}G_{11/$ 



Fig. (4): Excitation spectrum of ZLLTAP DY (01) glass.

#### 4.5 Fluorescence Spectrum

The Fluorescence spectrum of  $Dy^{3+}$ doped in zinc lithium lead tungsten aluminophosphate glass is shown in Figure 5. There are four broad bands observed in the Fluorescence spectrum of  $Dy^{3+}$ doped zinc lithium lead tungsten



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Volume 13, Issue 1, January 2024

DOI:10.15680/IJIRSET.2024.1301032

aluminophosphate 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 and 752 nm corresponds to the  $({}^{4}F_{9/2} \rightarrow {}^{6}H_{15/2})$ ,  $({}^{4}F_{9/2} \rightarrow {}^{6}H_{13/2})$ ,  $({}^{4}F_{9/2} \rightarrow {}^{6}H_{11/2})$  and  $({}^{4}F_{9/2} \rightarrow {}^{6}H_{9/2})$  transition.



Fig. (5): Fluorescence spectrum of ZLLTAP DY (01) glass.

Table5: Emission peak wave lengths  $(\lambda_p)$ , radiative transition probability  $(A_{rad})$ , branching ratio ( $\beta$ ), stimulated emission cross-section ( $\sigma_p$ ) and radiative life time ( $\tau_R$ ) for various transitions in Dy<sup>3+</sup> doped ZLLTAP glasses.

Transition		ZLLTAP DY 01			ZLLTAP DY 1.5			ZLLTAP DY 02					
	$\lambda_{max}$	A <sub>rad</sub> (s	β	$\sigma_p$	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p$		A <sub>rad</sub> (s	β	$\sigma_{p_o}$	$\tau_{\rm R}$
	(nm)	1)		$(10^{-20})$				$(10^{-20})$	$\tau_{R}$ (µs)	1)		$(10^{-20})$	$(10^{-20})$
				$cm^2$ )				$cm^2$ )				$cm^2$ )	$cm^2$ )
${}^{4}F_{9/2} \rightarrow {}^{6}H_{1}$	485	79.13	0.1988	0.168		78.02	0.1975	0.162		85.99	0.2042	0.173	
5/2					2512.9				2531.6				2374.75
${}^{4}F_{9/2} \rightarrow {}^{6}H_{1}$	575	268.29	0.6742	1.219	3	266.68	0.6751	1.170	2	283.11	0.6723	1.202	
3/2													
${}^{4}F_{9/2} \rightarrow {}^{6}H_{1}$	665	282.71	0.0710	0.145		28.15	0.0712	0.141		29.56	0.0702	0.145	
1/2													
${}^{4}F_{9/2} \rightarrow {}^{6}H_{9/2}$	752	22.25	0.0559	0.138	1	22.15	0.0561	0.136		22.43	0.0532	0.135	
2													

### V. CONCLUSION

In the present study, the glass samples of composition (40-x)P2O5:10ZnO:10Li2O:10PbO:10WO3:20Al2O3:xDy2O3 (where x =1, 1.5and 2mol %) have been prepared by melt-quenching method. The value of stimulated emission cross-section ( $\sigma$ p) is found to be maximum for the transition ( $4F9/2 \rightarrow 6H13/2$ ) for all glass specimens. This shows that ( $4F9/2 \rightarrow 6H13/2$ ) transition is most probable transition. ). The FTIR of glasses revealed the presence of characteristic bonding vibrations of different functional groups.

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|e-ISSN: 2319-8753, p-ISSN: 2347-6710| www.ijirset.com | Impact Factor: 8.423| A Monthly Peer Reviewed & Referred Journal |

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| DOI:10.15680/IJIRSET.2024.1301032 |

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