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Spectral and Thermal Properties of Tm^{3+} Doped in Zinc Lithium Tungsten Antimony Borophosphate Glasses

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Abstract
Glass sample of zinc lithium tungsten antimony borophosphate: (45-x) P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3:xTm_2O_3. (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. DTA curve was analysed to evaluate the glass transition temperature (T_g), onset crystallization temperature (T_c),melting temperature(T_m) and hence study the thermal properties. Optical absorption and fluorescence spectra were recorded at room temperature for all glass samples. Judd-Ofelt intensity parameters Ω_λ (λ=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: *ZLTABP Glasses, Optical Properties, Judd-Ofelt Theory, Rare earth ions.*

I. Introduction

Glass materials doped with rare earth ions are widely used mainly for optical devices, wave guide laser, sensors, up-conversion lasers, white light emitting diodes [1-5]. Glasses based on heavy metal oxide have received increased attention due to their manifold possible applications in the field of glass ceramics, layers for optoelectronics devices, thermal and mechanical sensors [6-10]. Phosphate glasses possess easier preparation, large transparency window, high refractive index, low phonon energy, better thermal stability, high density, good mechanical and chemical durability [11-14]. Phosphate glasses are promising laser hosts because they are able to accommodate higher content of rare earth ions and still remain amorphous in comparison with other glass systems. Due to their excellent thermal, physical and optical properties, they are used in fiber lasers, spectral conversion, photo-voltaic solar cells, temperature sensors and optical coherence tomography [15-17]. The addition of network modifier (NWF) Li_2O is to improve both electrical and mechanical properties of such glasses [18]. Phosphate glasses containing Tm^{3+} ions good for candidates because of their strong absorption and emission in the UV and visible spectral regions [19-21].Recently Tm^{3+} ions doped glasses found important in the area of wave guide laser, laser action, solar cells and optical fibers [22-24].

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 absorption ,emission and thermal properties of Tm^{3+} doped zinc lithium tungsten antimony 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 Tm^{3+}doped borophosphate glass samples (45-x) P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3: xTm_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,WO_3, Sb_2O_3, B_2O_3 and Tm_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1070^0C 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 250^0C 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 %)
ZLTABP (UD)	45 P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3
ZLTABP (TM 1)	44 P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3:1Tm_2O_3
ZLTABP (TM 1.5)	43.5 P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3:1.5 Tm_2O_3
ZLTABP (TM 2)	43 P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3:2 Tm_2O_3

ZLTABP (UD) -Represents undoped Zinc Lithium Tungsten Antimony Borophosphate glass specimen.
ZLTABP (TM) -Represents Tm^{3+} doped Zinc Lithium Tungsten Antimony Borophosphate glass specimens.

III. Theory

3.1 Oscillator Strength

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

$$f_{expt.} = 4.318 \times 10^{-9} \int \varepsilon(\nu)\, d\nu \qquad (1)$$

where, $\varepsilon(\nu)$ 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 [26], using the modified relation:

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

where c is the molar concentration of the absorbing ion per unit volume, I is the optical path length, $\log I_0/I$ is optical density and $\Delta\upsilon_{1/2}$ 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:

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

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

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

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 (λ=2,4and 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:

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

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

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

S L J

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

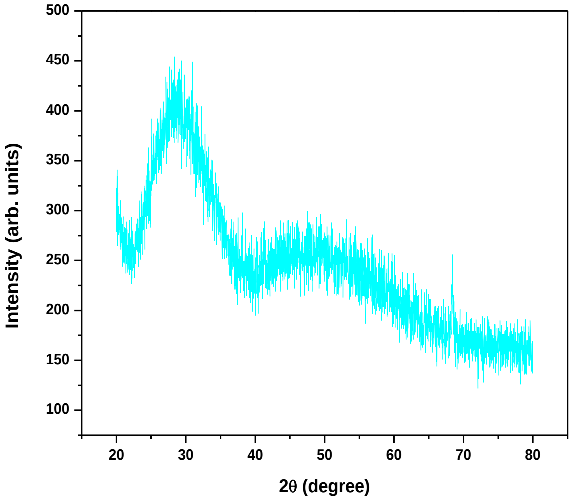

Fig. 1 X-ray diffraction pattern of ZLTABP TM (01) glass.

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 ZLTABP sample. The glass transition temperature (T_g), onset crystallization temperature (T_c), crystallization temperature (T_p), melting temperature(T_m), thermal stability (T_s), thermal stability parameter(S),Hurbe's criterion (H_r) and reduced glass transition temperature(T_{rg}) were calculated. All the determined thermal parameters are given in table 2.

Glass samples	T_g(°C)	T_c(°C)	T_p(°C)	T_m(°C)	T_s(°C)	H_r(°C)	S(°C)	T_{rg}(°C)
ZLTABP HO(01)	374	505	546	682	131	0.232	14.36	0.548
ZLTABP HO(01)	376	506	548	685	130	0.235	14.52	0.549
ZLTABP HO(01)	377	508	550	688	131	0.244	13.90	0.548

The thermal stability of the glass samples can be calculated by difference between onset crystallization temperature and transition temperature [29].

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

Hruby's criterion is calculated using the Hurby's relation [30].

Hruby's criterion (H_r) = $[(T_p-T_c)/(T_m-T_c)$ (10)

Reduced glass transition temperature is given as [31].

Reduced glass transition temperature (T_{rg}) $=T_g/T_m$ (11)

Thermal stability parameter can be calculated using [32].

Thermal stability parameter (S) = $[(T_p-T_c) \times (T_c-T_g)]/ T_g$ (12)

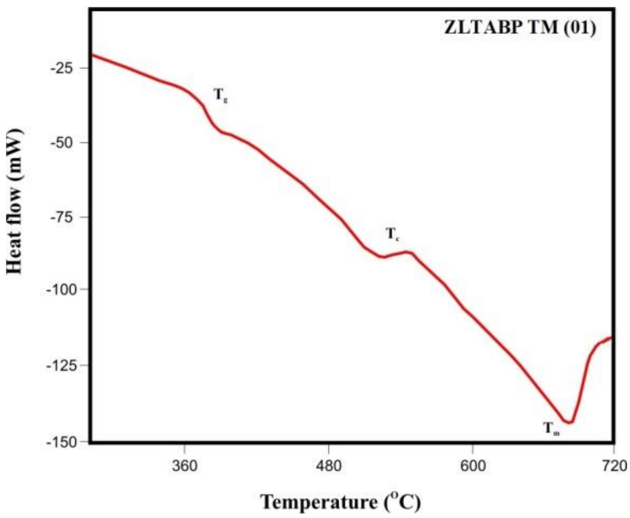

Fig.2: DTA curve of ZLTABP TM (01) glass.

4.3 Absorption Spectrum

The absorption spectra of Tm^{3+}doped ZLTABP glass specimens have been presented in Figure 3 in terms of optical density versus wavelength. Five absorption bands have been observed from the ground state $^3H_6$ to excited states $^3F_4$, $^3H_5$, $^3H_4$, $^3F_3$ and $^1G_4$ for ZLTABP TM(01) glass.

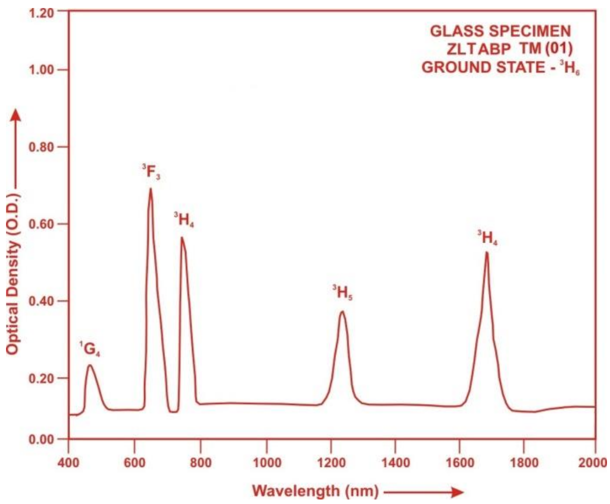

Fig. (3) Absorption spectrum of ZLTABP **TM (01) glass.**

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

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

Energy level from $^3H_6$	**Glass ZLTABP (TM01)**		**Glass ZLTABP (TM1.5)**		**Glass ZLTABP (TM02)**	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^3F_4$	1.89	1.91	1.88	1.91	1.85	1.88
$^3H_5$	1.50	1.50	1.48	1.49	1.46	1.49
$^3H_4$	2.07	2.13	2.06	2.13	2.04	2.12
$^3F_3$	3.06	3.13	3.04	3.12	3.02	3.11
$^1G_4$	0.84	0.92	0.82	0.92	0.80	0.92
r.m.s. deviation	0.0565		0.0654		0.0770	

In the zinc lithium tungsten antimony borophosphate 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 $\Omega_4>\Omega_2>\Omega_6$ 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 1.436 and 1.444 in the present glasses.
The values of Judd-Ofelt intensity parameters are given in **Table 4.**

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

Glass Specimen	**Ω_2(pm^2)**	**Ω_4(pm^2)**	**Ω_6(pm^2)**	**Ω_4 /Ω_6**
ZLTABP (TM 01)	7.170	8.771	6.076	1.444
ZLTABP TM 1.5)	7.203	8.699	6.056	1.436
ZLTABP (TM 02)	7.083	8.566	6.070	1.411

4.4. Fluorescence Spectrum

The fluorescence spectrum of ZLTABP TM (01) doped in zinc lithium tungsten antimony borophosphate glass is shown in Figure 4. There are nine broad bands observed in the Fluorescence spectrum of Tm^{3+}doped zinc lithium tungsten antimony borophosphate glass. The wavelengths of these bands along with their assignments are given in Table 5. The peak with maximum emission intensity appears at 1810 nm and corresponds to the ($^3F_4 \rightarrow ^3H_6$) transition.

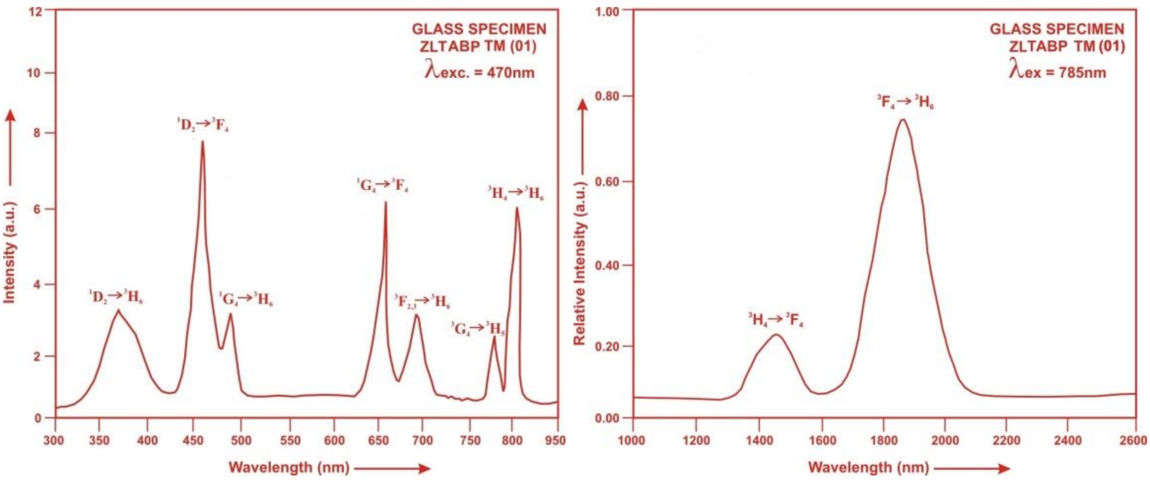

Fig. (4). **Fluorescence spectrum of ZLTABP TM (01) glass.**

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 Tm^{3+} doped ZLTABP glasses

Transition		ZLTABP (TM 01)				ZLTABP (TM 1.5)				ZLTABP (TM 02)			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20} cm^2$)	τ_R(μs)	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20} cm^2$)	τ_R (μs)	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20} cm^2$)	τ_R ($10^{-20} cm^2$)
$^1D_2 \rightarrow {}^3H_6$	365	54163.00	0.6284	1.517		53909.90	0.6270	1.538		53491.70	0.6274	1.555	
$^1D_2 \rightarrow {}^3F_4$	455	17730.30	0.2057	2.353		17794.20	0.2070	2.454		17545.60	0.2058	2.483	
$^1G_4 \rightarrow {}^3H_6$	480	2235.42	0.0259	0.555		2232.85	0.0260	0.587		2205.01	0.0259	0.617	
$^1G_4 \rightarrow {}^3F_4$	651	734.31	0.0085	1.425	11.6027	732.33	0.0085	1.558	11.6311	731.81	0.0086	1.677	11.7293
$^3F_{2,3} \rightarrow {}^3H_6$	689	5552.59	0.0644	2.358		5535.85	0.0644	2.433		5525.78	0.0648	2.495	
$^1G_4 \rightarrow {}^3H_5$	785	2137.96	0.0248	2.482		2137.38	0.0249	2.593		2139.77	0.0251	2.646	
$^3H_4 \rightarrow {}^3H_6$	798	2821.69	0.0327	4.350		2823.03	0.0328	4.558		2814.10	0.0330	4.690	
$^3H_4 \rightarrow {}^3F_4$	1450	359.63	0.0042	3.826		359.35	0.0042	3.915		356.82	0.0042	4.005	
$^3F_4 \rightarrow {}^3H_6$	1810	451.84	0.0052	7.299		451.22	0.0052	7.394		446.01	0.0052	7.513	

V. Conclusion

In the present study, the glass samples of composition (45-x) P_2O_5:10ZnO:10Li_2O:10WO_3:10Sb_2O_3:15B_2O_3:xTm_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 ($^3F_4 \rightarrow {}^3H_6$) for glass ZLTABP (TM 02), suggesting that glass ZLTABP (TM 02) is better compared to the other two glass systems ZLTABP (TM 01 and ZLTABP (TM 1.5).The prepared glass samples have good thermal stability as specified by calculated glass stability factors and therefore, these samples can be good materials for fiber fabrication.

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