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Spectral and Thermal Properties of Sm³⁺Doped in Zinc Lithium Alumino Antimony Borogerminate Glasses

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ABSTRACT:Glass of the system: (35-x) GeO₂:10ZnO:10Li₂O:10Al₂O₃:10Sb₂O₃:25B₂O₃:xSm₂O₃. (where x=1,1.5,2 mol%) have been prepared by melt-quenching technique.(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. The absorption spectra of three Sm³⁺ dopedzinc lithium alumino antimony borogerminateglasses have been recorded at room temperature. The various interaction parameters likeSlater-Condon parameters F_k (k=2,4,6),Lande parameters (ξ_{4f}),nephelauexetic ratio (β '),bonding parameters (b^{1/2}) and Racah parametersE^k(k=1,2 3) have been computed.Judd-Ofelt intensity parameters and laser parameters have also been calculated.

KEYWORDS:ZLAABG Glasses, Thermal Properties, Optical properties, Judd-Ofelt analysis.

I. INTRODUCTION

Rare- earth ion doped glasses have attracted much attention as potential materials for various optical devices such as lasers, up-converters and optical fibers[1-5]. Transparent glass-ceramic as host materials for active optical ions have attracted great interest recently due to their potential application in optical devices such as low non-linear refractive index, good physical and chemical stability and frequency-conversion materials [6-8]. Germinate based glasses have been recently the subject of many types of research according to their promising mechanical, optical and magnetic properties as well their chemical durability, large refractive indices, excellent optical properties and good optical transmission [9-12]. Germinate glass exhibit very important physical properties such as low melting temperature, low glass transition temperature and low softening temperature. Due to their good chemical durability, samarium-doped ceramic glasses are attractive materials for the fabrication of low-cost integrated optical amplifiers by using the ion-exchange technique [13-15].

The aim of the present study is to prepare the Sm^{3+} dopedzinc lithium alumino antimony borogerminateglass with different Sm_2O_3 concentrations. The absorption spectra, fluorescence spectra of Sm^{3+} of the glasses were investigated. The Judd-Ofelt theory has been applied to compute the intensity parameters Ω_{λ} (λ =2, 4,6). These intensity parameter have been used to evaluate optical optical properties such as spontaneous emission probability, branching ratio, radiative life time and stimulated emission cross section. Large stimulated emission cross section is one of the most important parameters required for the design of high peak power solid state lasers.

II. EXPERIMENTAL TECHNIQUES

Preparation of glasses

The following Sm^{3+} doped zinc lithium alumino antimony borogerminateglass samples (35-x) GeO₂:10ZnO:10Li₂O:10Al₂O₃:10Sb₂O₃:25B₂O₃:xSm₂O₃. (where *x* =1, 1.5, 2) have been prepared by melt-quenching method. Analytical reagent grade chemical used in the present study consist of GeO₂,ZnO, Li₂O, Al₂O₃, Sb₂O₃, B₂O₃ and Sm₂O₃. They were thoroughly mixed by using an agate pestle mortar. then melted at 1065^oC 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^oC 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.



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Table 1

Chemical composition of the glasses								
Sample	Glass composition (mol %)							
ZLAABG (UD)	35 GeO ₂ :10ZnO:10Li ₂ O:10Al ₂ O ₃ :10Sb ₂ O ₃ :25B ₂ O ₃							
ZLAABG(SM1)	34 GeO ₂ :10ZnO:10Li ₂ O:10Al ₂ O ₃ :10Sb ₂ O ₃ :25B ₂ O ₃ :1 Sm ₂ O ₃							
ZLAABG(SM 1.5)	33.5 GeO ₂ :10ZnO:10Li ₂ O:10Al ₂ O ₃ :10Sb ₂ O ₃ :25B ₂ O ₃ : 1.5 Sm ₂ O ₃							
ZLAABG(SM 2)	33 GeO ₂ :10ZnO:10Li ₂ O:10Al ₂ O ₃ :10Sb ₂ O ₃ :25B ₂ O ₃ : 2 Sm ₂ O ₃							

ZLAABG(UD) -Represents undopedZinc Lithium Alumino Antimony Borogerminate Glass specimens. ZLAABG(SM) -Represents Sm³⁺ dopedZinc Lithium Alumino Antimony Borogerminate Glass specimens.

III. THEORY

(1)

(3)

3.1Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [16].

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

where, ε (*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 [17], using the modified relation:

$$P_{\rm m} = 4.6 \times 10^{-9} \times \frac{1}{cl} \log \frac{I_0}{I} \times \Delta \upsilon_{1/2}$$
⁽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 $\Delta v_{1/2}$ is half band width.

3.2. Judd-Ofelt Intensity Parameters

According to Judd [18] and Ofelt [19] 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:

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

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

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

 $\lambda = 2, 4, 6$

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, Ω_{λ} ($\lambda = 2, 4, 6$) 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 $|4f^{N}(S', L') J'>$ to a final manifold $|4f^{N}(S,L) J >|$ is given by:

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A [(S', L') J'; (S,L)J] =
$$\frac{64 \pi^2 v^3}{3h(2J'+1)} \left[\frac{n(n^2+2)^2}{9} \right] \times S(J', J)$$
 (4)

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

$$\beta[(S', L') J'; (S, L) J] = \sum \frac{A[(S' L)]}{A[(S' L')J'(\bar{S} L)]}$$
(5)
S L J

where, the sum is over all terminal manifolds.

The radiative life time is given by

$$\tau_{\rm rad} = \sum A[(S', L') J'; (S,L)] = A_{\rm Total}^{-1}$$
(6)
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

(7)

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

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

3.4Nephelauxetic 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 [20, 21]. The Nephelauxetic Ratio is given by $\beta' = \frac{v_g}{v_a}$ (8)

where, v_a and v_g refer to the energies of the corresponding transition in the glass and free ion, respectively. The values of bonding parameter $b^{1/2}$ are given by

$$b^{1/2} = \left[\frac{1-\beta'}{2}\right]^{1/2} \tag{9}$$

IV. RESULT AND DISCUSSION

4.1XRD Measurement

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



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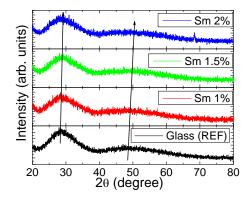


Fig. 1: X-ray diffraction pattern of GeO₂:ZnO:Li₂O:Al₂O₃:Sb₂O₃:B₂O₃

4.2 Thermal Properties

Fig. 2 depicts the DTA thermogram of powdered ZLAABG sample show an endothermic peak corresponding to glass transition event followed by an exothermic peak related to crystallization event. The glass transition temperature (T_g), onset crystallization temperature (T_c) were estimated to be 516^oC, 578^oC and 598^oC respectively. From the measured value of T_g , T_x and T_c , the glass stability factor ($\Delta T = T_x - T_g$) has been determined to be 62^oC indicating the good stability of the glass. Therefore, the present glass composition could also be used to draw fiber and used to determine the required heat temperatures applied to induce crystallization. Thermally stable glasses exhibit closed packed structure, while the unstable glasses exhibit loose packed structure.

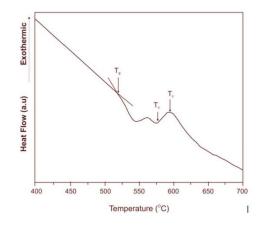


Fig. 2.DTA thermogram of powdered ZLAABG sample.

Obtained results indicate that by increasing the amount of mol% Sm_2O_3 , the T_g of the samples also increases, the small increase of T_g in these glasses shows that the structure is strongly and progressively modified. The thermal stabilities ΔT of the ZLAABG reference glass and Sm^{+3} doped ZLAABG glass has been evaluated from their T_g , T_c and T_c values, the results are listed out in Table 2.Hruby's parameter also calculated by using eq. (11), the greater values of the Hruby's parameter indicate higher glass forming tendency, the values of Hin our glasses increased with the addition of the Sm_2O_3 .Eqs. (12) and (13) present the GS parameter of Weinberg [22] and Lu and Liu [23], respectively.

$$H = \frac{T_X - T_g}{T_c - T_X} (11)$$

$$K_{W} = \frac{T_X - T_g}{T_c}$$
(12)
$$K_{LL} = \frac{T_X}{T_g + T_c} (13)$$



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Table 2: Thermal parameters determined from the DTA traces of ZLAABG (SM) glasses.

Sample Name	% Sm ₂ O ₃	$T_g^0 C$	$T_X^0 C$	$T_{\rm C}^{0}$ C	ΔT	Н	K _W	K _{LL}
ZLAABG(SM 1.0)	1	516	578	598	62	3.10	0.1037	0.5189
ZLAABG (SM 1.5)	1.5	517	580	600	63	3.15	0.1050	0.5192
ZLAABG (SM 02)	2	520	584	603	64	3.37	0.1061	0.5200

4.3 Absorption Spectrum

The absorption spectra of Sm³⁺ doped ZLAABG(SM 01) glass specimen has been presented in Figure 3 in terms of optical density versus wavelength (nm). Ten absorption bands have been observed from the ground state ${}^{6}H_{5/2}$ to excited states ${}^{6}F_{1/2}$, ${}^{6}F_{7/2}$, ${}^{6}F_{9/2}$, ${}^{4}G_{7/2}$, ${}^{4}I_{9/2}$, ${}^{4}M_{7/2}$, ${}^{6}P$, ${}^{4}P$)_{5/2}, ${}^{4}F_{7/2}$, ${}^{4}D_{1/2}$, and $({}^{4}D$, ${}^{6}P$)_{5/2}for Sm³⁺dopedZLAABG glasses.

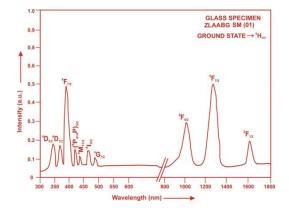


Fig.3: Absorption spectrum of Sm³⁺doped ZLAABG(01)glass.

The experimental and calculated oscillator strengths for Sm³⁺ ions in zinc lithium alumino antimony borogerminateglasses are given in Table 3.

Table3: Measured and calculated oscillator strength ($P_m \times 10^{+6}$) of Sm ³⁺ ions in ZLAABG	glasses.

Energy level from	Glass		Glass		Glass	
⁶ H _{5/2}	ZLAABG(SM01)		ZLAABG		ZLAABG(SM02)	
			(SM1.5)			
	P _{exp} .	P _{cal} .	P _{exp} .	P _{cal} .	P _{exp} .	P _{cal} .
⁶ F _{1/2}	1.62	1.66	1.58	1.63	1.52	1.58
${}^{6}F_{7/2}$	5.45	5.51	5.41	5.49	5.38	5.46
⁶ F _{9/2}	3.82	3.83	3.78	3.81	3.74	3.79
$^{4}G_{7/2}$	0.16	0.12	0.14	0.12	0.11	0.12
${}^{4}I_{9/2}, {}^{4}M_{15/2}, {}^{4}I_{11/2}$	1.16	1.88	1.12	1.87	1.09	1.85
${}^{4}M_{17/2}, {}^{4}G_{9/2}, {}^{4}I_{15/2}$	0.32	0.25	0.28	0.24	0.24	0.24
$({}^{6}P, {}^{4}P)_{5/2}, {}^{4}L_{13/2}$	1.33	1.31	1.29	1.31	1.25	1.30
${}^{4}\mathrm{F}_{7/2}, {}^{6}\mathrm{P}_{3/2}, {}^{4}\mathrm{K}_{11/2}$	5.62	5.62	5.58	5.64	5.54	5.62
${}^{4}D_{1/2}, {}^{6}P_{7/2}, {}^{4}L_{17/2}$	2.51	2.43	2.46	2.50	2.43	2.40
$^{4}D_{3/2}, (^{4}D, ^{6}P)_{5/2}$	2.62	3.47	2.58	3.46	2.54	3.45
r.m.s. deviation	0.3538		0.3674		0.3777	

Computed values of F₂, Lande' parameter (ξ_{4f}), Nephlauxetic ratio(β ') and bonding parameter($b^{1/2}$) for Sm³⁺ doped ZLAABG glass specimen are given in Table 4.



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Table 4. F₂, $\xi_{4f}\beta'$ and $b^{1/2}$ parameters for Samarium doped glass specimen.

Glass Specimen	F_2	ξ_{4f}	β'	b ^{1/2}
Sm ³⁺	358.82	1258.16	0.9337	0.1821

Judd-Ofelt intensity parameters Ω_{λ} (λ =2,4,6) were calculated by using the fitting approximation of the experimental oscillator strengths to the calculated oscillator strengths with respect to their electric dipole contributions. In the present case the three Ω_{λ} parameters follow the trend $\Omega_2 > \Omega_4 > \Omega_6$. The spectroscopic quality factor (Ω_4 / Ω_6) related with the rigidity of the glass system has been found to lie between 1.097 and 1.112 in the present glasses. The value of Judd-Ofelt intensity parameters are given in **Table 5**

 Table 5: Judd-Ofelt intensity parameters for Sm³⁺ doped ZLAABG glass specimens.

Glass Specimen	$\Omega_2(pm^2)$	$\Omega_4(pm^2)$	$\Omega_6(\mathrm{pm}^2)$	Ω_4/Ω_6	Ref.
ZLAABG (SM01)	5.527	4.993	4.551	1.0969	P.W.
ZLAABG (SM1.5)	5.427	5.005	4.515	1.1085	P.W.
ZLAABG (SM02)	5.241	4.987	4.486	1.1117	P.W.
LBGS (SM)	10.11	8.85	7.42	1.193	[24]

4.4 Excitation Spectrum

Excitation spectra of ZLAABG SM (01) glass recorded at the emission wavelength 602 nm is depicted as figure 4. The excitation spectra consists of seven peaks corresponding to the transitions from the ground state ${}^{6}H_{5/2}$ to the various excited states ${}^{4}H_{9/2}$, ${}^{4}D_{3/2}$, ${}^{6}P_{7/2}$, ${}^{4}F_{7/2}$, ${}^{4}M_{19/2}$, ${}^{4}G_{9/2}$ and ${}^{4}I_{11/2}$ + ${}^{4}I_{13/2}$ at the wavelengths of 337, 362, 375,401, 412, 435 and 478 nm respectively. Among these, a prominent excitation band at 401 nm has been selected for the measurement of emission spectrum of Sm³⁺ glass.

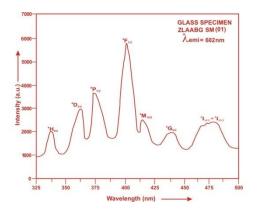


Fig.4: fluorescence spectrum of Sm³⁺doped ZLAABG (01) glass

4.4. Fluorescence Spectrum

The fluorescence spectrum of Sm³⁺doped in zinc lithium alumino antimony borogerminate glass is shown in Figure 4. There are four broad bands observed in the Fluorescence spectrum of Sm³⁺doped zinc lithium alumino antimony borogerminateglass. The wavelengths of these bands along with their assignments are given in Table 6.Fig. (4).Shows the fluorescence spectrum with four peaks(${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$), (${}^{4}G_{5/2} \rightarrow {}^{6}H_{9/2}$) and (${}^{4}G_{5/2} \rightarrow {}^{6}H_{11/2}$), respectively for glass specimens.



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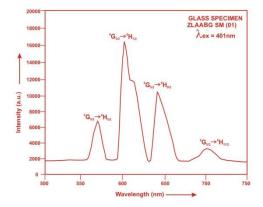


Fig.4: fluorescence spectrum of Sm³⁺doped ZLAABG(01) glass.

Table6. 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 Sm³⁺ doped ZLAABG glasses.

Transition		ZLAABG SM 01			ZLAABG SM 1.5				ZLAABG SM 02				
	λ _{max} (nm)	A _{rad} (s ⁻ ¹)	β	σ _p (10 ⁻²⁰ cm ²)	τ _R (μs)	A _{rad} (s ⁻ ¹)	β	σ (10 ⁻²⁰ cm ²)	τ _R (μs)	A _{rad} (s ⁻	β	σ _p (10 ⁻²⁰ cm ²)	τ _R (μs)
⁴ G _{5/2} → ⁶ H _{5/2}	562	9.349	0.041 3	0.0040		9.344	0.041 5	0.00440		9.253	0.041 5	0.0048	
⁴ G _{5/2} → ⁶ H _{7/2}	602	97.87	0.432 9	0.0462	4422.7 8	97.718	0.433 9	0.0485	4440.0 5	97.47	0.437 2	0.0526	4485.7 9
⁴ G _{5/2} → ⁶ H _{9/2}	645	94.84	0.419 4	0.0436		94.107	0.417 8	0.0460]	92.20	0.413 6	0.0476]
⁴ G _{5/2} → ⁶ H _{11/} 2	705	24.04	0.106 3	0.0134		24.055	0.106 8	0.0141	1	24.01	0.107 7	0.0146	1

V. CONCLUSION

In the present study, the glass samples of composition (35-x) GeO₂:10ZnO:10Li₂O:10Al₂O₃:10Sb₂O₃:25B₂O₃:xSm₂O₃. (where x=1, 1.5, 2mol %) have been prepared by melt-quenching method. The Judd-Ofelt theory has been applied to calculate the oscillator strength and intensity parameters $\Omega_{\lambda}(\lambda=2, 4, 6)$. The radiative transition probability, branching ratio are highest for (${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$) transition and hence it is useful for laser action. The stimulated emission cross section (σ_{p}) has highest value for the transition (${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$) in all the glass specimens doped with Sm³⁺ ion. This shows that (${}^{4}G_{5/2} \rightarrow {}^{6}H_{7/2}$) transition is most probable transition. The large temperature range (Δ T) indicates that the glass could be easily drawn into fiber.

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