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Spectral and Raman Analysis of Sm^{3+} ions doped Lead LithiumPotassiumniobateBorophosphate Glasses

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Abstract
Glass of the system: (40-x)P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:xSm_2O_3. (where x=1, 1.5,2 mol %) have been prepared by melt-quenching method (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,Excitation, fluorescence and Raman spectra have been recorded at roomtemperature for all glass samples. The various interaction parameters likeSlater-CondonparametersF$_k$ (k=2,4,6),Lande'parameters(ξ_{4f}),nephelauexetic ratio(β'),bondingparameters($b^{1/2}$)and RacahparametersE^k(k=1,2 3) have been computed.Judd-Ofelt intensity parameters and laser parameters have also been calculated.

Keywords:*LLPNBPGlasses,ThermalProperties,Opticalproperties,Raman analysis.*

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I. Introduction

Transparent glass–ceramic as host materials for active optical ions have attracted great interest recently due to their potential application such asoptical storage devices, amplifier, light-emitting devices, wave guide, laser andfrequency-conversion materials [1-6]. Phosphate glasses have been studied and commercialized for a variety of applications because of their useful physical properties including controllable chemical durability in aqueous environments, high thermal expansion coefficient, low phonon energies and high refractive index [7-12].Phosphate glasses have a best thermo-optical performance with considerable chemical durability, high gain as with low energy back transfer and weak up conversion [13, 14]. B_2O_3 is one of the best-known glass formers and it is present in varieties of commercial glasses.The HMO such as PbO in the glass composition increases the thermal stability and decreases the maximum phonon energy of the host in which it is present. Glasses having samarium oxide have attained great attention, since they are used in the wide area of applications such as spin glasses or optical isolators and optical switches [15-19].The low glass-transition temperature and the high thermal expansion coefficient of phosphate glasses make them the material of choice for glass-metal sealing applications.

The aim of the present study is to prepare theSm^{3+}dopedlead lithiumpotassiumniobateborophosphateglass with different Sm_2O_3concentrations.The Optical absorption,Excitation, fluorescence and Raman spectra have been recorded at roomtemperature for all glass samples. 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, branchingratio,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 lead lithiumpotassiumniobateborophosphateglass samples (40-x) P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:xSm_2O_3. (where*x*=1, 1.5, 2) have been prepared by melt-quenching method.Analytical reagent grade chemical used in the present study consist of P_2O_5,PbO,Li_2O,K_2O,Nb_2O_5,$B_2O_3$and Sm_2O_3. They were thoroughly mixed by using an agate pestle mortar. then melted at 1050^0C 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 %)
LLPNBP(UD)	40 P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3
LLPNBP(SM1)	39 P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:1Sm_2O_3
LLPNBP(SM 1.5)	38.5 P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:1.5Sm_2O_3
LLPNBP(SM 2)	38 P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:2Sm_2O_3

LLPNBP(UD) -Represents undopedLeadLithiumPotassiumniobateBorophosphate glass specimens.
LLPNBP(SM) -Represents Sm^{3+}dopedLead LithiumPotassiumniobateBorophosphate glass specimens.

III. Theory

3.1Oscillator Strength

The intensity of spectral lines are expressed in terms of oscillator strengths using the relation [20].

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

where, ε (*ν)* 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[21], using the modified relation:

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

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[22] and Ofelt[23] theory, independently derived expression for the oscillator strength of the induced forced electric dipole transitions between an initial Jmanifold $|4f^N$ (S, L) J> level and the terminal J' manifold $|4f^N$ (S', L') J'> is given by:

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

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

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

λ =2, 4, 6

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, 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:

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

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

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

where, the sum is over all terminal manifolds.

The radiative life time is given by

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

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

$$\sigma_p(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}}\right] \times A[(S', L')J'; (\bar{S}, \bar{L})\bar{J}] \qquad (7)$$

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 [24, 25]. The Nephelauxetic Ratio is given by

$$\beta' = \frac{\nu_g}{\nu_a} \qquad (8)$$

where, ν_a and ν_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

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

IV. Result and Discussion

4.1XRD 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.

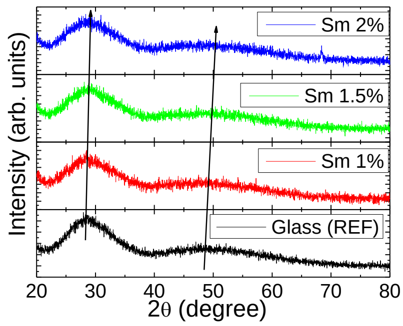

Fig. 1: X-ray diffraction pattern of P_2O_5:PbO:Li_2O:K_2O:Nb_2O_5:B_2O_3:Sm_2O_3

4.2 Raman spectra

The Raman spectrum ofLead LithiumPotassiumniobateBorophosphate(LLPNBP) glass specimens is recorded and is shown in Fig. 2. The spectrum peaks located at 395 and 775 cm^{-1}. The band at 395 $cm^{-1}$is is related to the bending motion of phosphate polyhedral PO_4 units with cation like Li_2O as the modifier.The broad band at 775 cm^{-1} is due to symmetric stretching of (P–O–P) bridging oxygen bonds in $(P_2O_7)_4$ units.

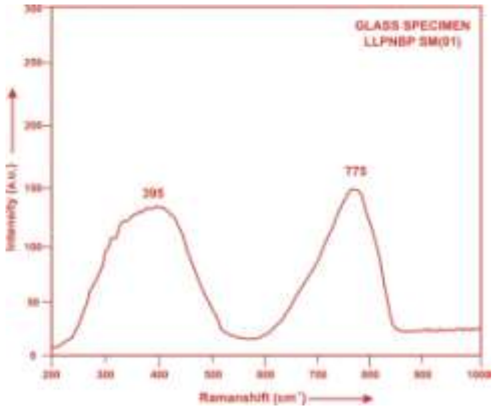

Fig.2 Raman spectrum of LLPNBP SM (01) glass.

4.3 Absorption Spectrum

The absorption spectra of Sm^{3+} doped LLPNBP(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 $^6H_{5/2}$to excited states $^6F_{1/2}$,$^6F_{7/2}$,$^6F_{9/2}$,$^4G_{7/2}$,$^4I_{9/2}$,$^4M_{7/2}$,$(^6P,^4P)_{5/2}$,$^4F_{7/2}$, $^4D_{1/2}$, and $(^4D,^6P)_{5/2}$for Sm^{3+}dopedLLPNBP glasses.

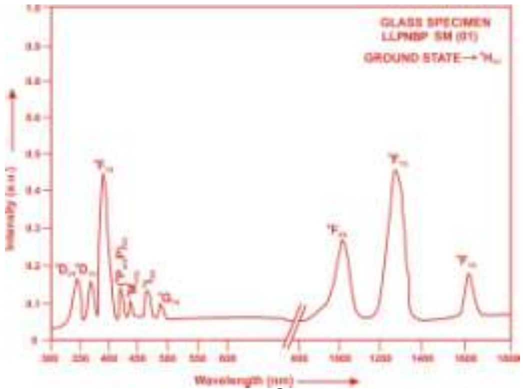

Fig.3: Absorption spectrum of Sm^{3+}doped LLPNBPSM (01)glass.

The experimental and calculated oscillator strengths for Sm^{3+} ions inlead lithiumpotassiumniobateborophosphateglasses are given in Table 2.

Table2: Measured and calculated oscillator strength (P_m×10^{+6}) of Sm^{3+}ions in LLPNBP glasses.

Energy level from $^6H_{5/2}$	**Glass LLPNBP (SM01)**		**Glass LLPNBP (SM1.5)**		**Glass LLPNBP (SM02)**	
	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$	$P_{exp.}$	$P_{cal.}$
$^6F_{1/2}$	1.68	1.72	1.64	1.68	1.60	1.66
$^6F_{7/2}$	5.51	5.56	5.47	5.53	5.42	5.49
$^6F_{9/2}$	3.86	3.88	3.82	3.86	3.76	3.82
$^4G_{7/2}$	0.19	0.12	0.17	0.12	0.14	0.12
$^4I_{9/2}$,$^4M_{15/2}$,$^4I_{11/2}$	1.20	1.90	1.16	1.89	1.12	1.87
$^4M_{17/2}$,$^4G_{9/2}$,$^4I_{15/2}$	0.30	0.25	0.28	0.25	0.25	0.24
$(^6P,^4P)_{5/2}$,$^4L_{13/2}$	1.38	1.30	1.32	1.30	1.27	1.30
$^4F_{7/2}$,$^6P_{3/2}$,$^4K_{11/2}$	5.57	5.60	5.53	5.60	5.48	5.59
$^4D_{1/2}$,$^6P_{7/2}$,$^4L_{17/2}$	2.50	2.46	2.45	2.45	2.41	2.42
$^4D_{3/2}$, $(^4D,^6P)_{5/2}$	2.64	3.48	2.60	3.46	2.56	3.45
r.m.s. deviation	0.3487		0.3599		0.3708	

Computed values of F_2, Lande′ parameter (ξ_{4f}), Nephlauxeticratio(β') and bonding parameter($b^{1/2}$) for Sm^{3+} doped LLPNBP glass specimen are given in Table3.

Table 3. F_2,ξ_{4f},β' 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 $\Omega_\lambda$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 1.0765 and 1.0941 in the present glasses.

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

Table4: Judd-Ofelt intensity parameters for Sm^{3+} doped LLPNBPglass specimens.

Glass Specimen	**Ω_2(pm^2)**	**Ω_4(pm^2)**	**Ω_6(pm^2)**	**Ω_4/Ω_6**	**Ref.**
LLPNBP (SM01)	5.254	4.561	4.237	1.0765	[P.W.]
LLPNBP (SM1.5)	5.151	4.553	4.203	1.0833	[P.W.]
LLPNBP (SM02)	5.051	4.546	4.155	1.0941	[P.W.]
GPBS (SM)	8.56	3.02	2.37	1.274	[26]

4.4 Excitation Spectrum

Excitation spectra of LLPNBP 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 $^6H_{5/2}$ to the various excited states $^4H_{9/2}$, $^4D_{3/2}$, $^6P_{7/2}$, $^4F_{7/2}$, $^4M_{19/2}$, $^4G_{9/2}$ and $^4I_{11/2}+^4I_{13/2}$ at the wavelengths of 337, 361, 375,401, 412, 436 and 478 nm respectively. Among these, a prominent excitation band at 401 nm has been selected for the measurement of emission spectrum of Sm^{3+} glass.

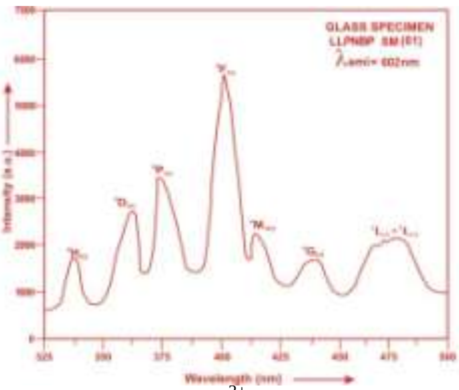

Fig.4: Excitationspectrum of Sm^{3+}doped LLPNBPSM (01) glass**.**

4.5. Fluorescence Spectrum

The fluorescence spectrum of Sm^{3+}doped in lead lithiumpotassiumniobateborophosphateglass is shown in Figure 5. There are nine broad bands observed in the Fluorescence spectrum ofSm^{3+}dopedlead lithiumpotassiumniobateborophosphateglass. The wavelengths of these bands along with their assignments are given in Table 5. Fig. (5).Shows the fluorescence spectrum with nine peaks ($^4G_{5/2}\rightarrow{}^6H_{5/2}$), ($^4G_{5/2}\rightarrow{}^6H_{7/2}$), ($^4G_{5/2}\rightarrow{}^6H_{9/2}$),($^4G_{5/2}\rightarrow{}^6H_{11/2}$),($^4G_{5/2}\rightarrow{}^6H_{11/2}$),($^4G_{5/2}\rightarrow{}^6F_{3/2}$),($^4G_{5/2}\rightarrow{}^6F_{5/2}$),($^4G_{5/2}\rightarrow{}^6F_{7/2}$) and ($^4G_{5/2}\rightarrow{}^6F_{9/2}$)respectively for glass specimens.

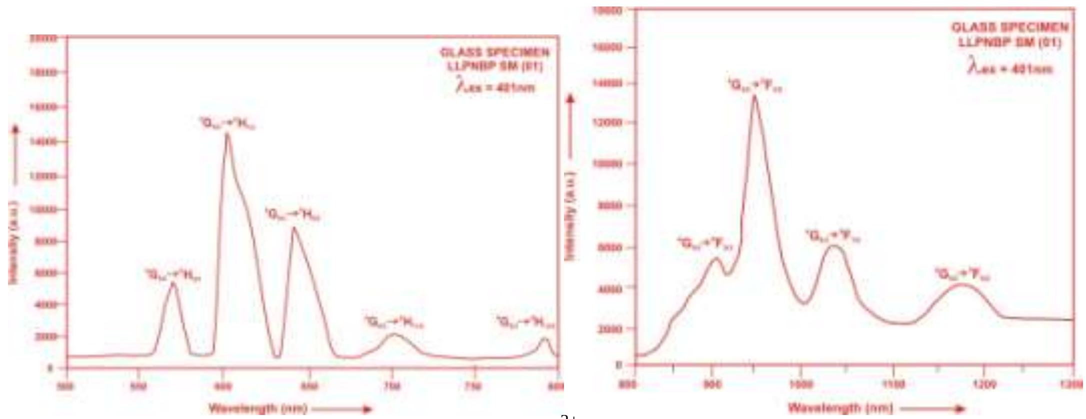

Fig.5:fluorescence spectrum of Sm^{3+}doped LLPNBPSM (01) glass.

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 Sm^{3+} doped LLPNBPglasses.

Transition		LLPNBP SM 01				LLPNBP SM 1.5				LLPNBPSM 02			
	λ_{max} (nm)	$A_{rad}(s^{-1})$	β	σ_p ($10^{-20}cm^2$)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	σ ($10^{-20}cm^2$)	$\tau_R(\mu s)$	$A_{rad}(s^{-1})$	β	$\sigma_p(10^{-20}$ $cm^2)$	$\tau_R(\mu s)$
$^4G_{5/2}\rightarrow{}^6H_{5/2}$	562	10.79	0.0366	0.0037		10.74	0.0367	0.0041		10.69	0.0368	0.0045	
$^4G_{5/2}\rightarrow{}^6H_{7/2}$	602	112.82	0.3826	0.0453		112.47	0.3841	0.0492		111.88	0.3853	0.053	
$^4G_{5/2}\rightarrow{}^6H_{9/2}$	645	111.06	0.3766	0.0433		109.87	0.3752	0.0447		108.59	0.3740	0.047	
$^4G_{5/2}\rightarrow{}^6H_{11/2}$	705	27.60	0.0936	0.0129		27.56	0.0941	0.0134		27.47	0.0946	0.014	
$^4G_{5/2}\rightarrow{}^6H_{13/2}$	786	2.65	0.0089	0.0017	3391.34	2.63	0.0089	0.00176	3415.28	2.60	0.0090	0.0018	3444.17
$^4G_{5/2}\rightarrow{}^6F_{3/2}$	915	4.68	0.0159	0.0071		4.60	0.0157	0.00726		4.52	0.0155	0.0075	
$^4G_{5/2}\rightarrow{}^6F_{5/2}$	955	20.91	0.0709	0.0312		20.60	0.0703	0.0320		2.03	0.0699	0.0328	
$^4G_{5/2}\rightarrow{}^6F_{7/2}$	1036	2.48	0.0084	0.0044		2,48	0.0084	0.0045		2.48	0.0085	0.0047	
$^4G_{5/2}\rightarrow{}^6F_{9/2}$	1180	1.88	0.0064	0.0048		1.85	0.0063	0.0049		1.82	0.0063	0.0049	

V. Conclusion

In the present study, the glass samples of composition (40-x) P_2O_5:10PbO:10Li_2O:10K_2O:10Nb_2O_5:20B_2O_3:xSm_2O_3. (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 Ω_λ(λ=2, 4, 6). The radiative transition probability, branching ratio are highest for ($^4G_{5/2}\rightarrow{}^6H_{7/2}$) transition and hence it is useful for laser action. The stimulated emission cross section (σ_p) has highest value for the transition ($^4G_{5/2}\rightarrow{}^6H_{7/2}$) in all the glass specimens doped with Sm^{3+} ion. This shows that ($^4G_{5/2}\rightarrow{}^6H_{7/2}$) transition is most probable transition. The obtained results indicated that LLPNBP (SM) glass should be suitable for laser and optoelectronic applications.

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