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Spectroscopic properties of Nd^{3+}-doped barium phosphate glasses for Laser application

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Abstract—Barium phosphate glasses doped with varying Nd^{3+} ions concentration were prepared by melt quenching method and characterized through absorption and luminescence measurements. Judd-Ofelt (JO) intensity parameters were derived from the absorption spectrum of 1.0 mol % Nd_2O_3 -doped glass. Nd^{3+} ions exhibit strong near infrared luminescence at 1.06 μm ($^4F_{3/2} \rightarrow {}^4I_{11/2}$) has been obtained for all the glasses upon 808 nm diode laser excitation. Obtained JO parameters and refractive index are used to calculate the radiative properties, such as effective bandwidth ($\Delta\lambda_{eff}$, nm), emission cross-section (σ_{emi}, cm^2), radiative transition probabilities (A_R, s^{-1}) and branching ratio (β_R) for the $^4F_{3/2} \rightarrow {}^4I_{J/2}$ (where J = 9, 11 and 13) transitions of Nd^{3+} ions. Among three transitions $^4F_{3/2} \rightarrow {}^4I_{11/2}$ shows higher value of stimulated emission cross-section and branching ratio indicates that the present glasses could be useful for the development of NIR laser at around 1.06 µm.

Keywords—*Glasses, Nd^{3+} ions, Judd–Ofelt analysis, Radiative properties, emission.*

I. Introduction

In recent years rare earth ions (RE^{3+}) doped glasses have been investigated because of their potential applications in the field of laser technology and optical communication systems [1,2]. Oxide glasses have much technological importance because of their unique properties like simple preparation, easy shaping, good strength, and can be doped at very high concentrations of activator ions with excellent homogeneity [3,4]. Among different oxide glasses, phosphate glasses are very interesting materials due to physical properties. These glasses are regarded as better hosts for RE^{3+} ions due to high transparency, low melting temperature, high thermal stability and low viscosity [5, 6], which render them useful for a wide range of real-world applications. But, these glasses show low chemical durability. However it can be improved by the addition of suitable modifiers like CaO and BaO [7,8]. BaO can also improve thermal properties of phosphate glasses [9].

Among various RE^{3+} ions, the Nd^{3+} is one of the most investigated ions for high-power laser applications, in which high gain, energy storage capacity and low optical losses are required [10]. Moreover, Nd^{3+} ions exhibit large cross-sections of optical absorption near 800 nm ($^4I_{9/2} \rightarrow {}^4F_{5/2}$). They can be easily excited by diode laser and shows emission near 1060 nm corresponding to $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition having high quantum efficiency.

The present paper explores the preparation of barium phosphate glasses doped with different concentration of Nd^{3+} ions and their characterization using optical absorption and near infrared (NIR) emission in order to test the suitability for laser applications in NIR region. Using Judd–Ofelt (J–O) theory [11,12] the intensity parameters and various radiative properties have been evaluated and the results are compared with existing Nd^{3+}: systems.

The paper contains four sections in which Section I provides the brief introduction and importance of Nd^{3+}-doped phosphate glasses. Earlier work related to Nd^{3+}-doped glasses is presented in Section II. Section III gives the experimental techniques used for the preparation and characterization of barium phosphate glasses. Section IV describes results and discussion and finally, Section V gives the conclusions drawn from the present investigations.

II. RELATED WORK

N. Chanthima et al [13] have reported Luminescence study and Judd-Ofelt analysis of CaO-BaO-P_2O_5 glass doped with Nd^{3+} ions and from J-O analysis concluded that calcium barium phosphate glass with 1.0 mol% Nd_2O_3 can be useful for laser applications. K.Linganna et al [14] have reported Thermal and optical properties of Nd^{3+} ions in K-Ca-Al fluorophosphates glasses, their research Decay analysis has been carried out and found that their lifetime for the $^4F_{3/2}$ level of Nd^{3+} ion was found to be higher compared to the other Nd^{3+} -doped glass host matrices. R.Narro-García et al 15] have observed Spectroscopic properties of Eu^{3+} / Nd^{3+} co-doped phosphate glasses and opaque glass-ceramics. In their work observed that the lifetimes of the $^4F_{3/2}$ level of Nd^{3+} of both glasses. V.M. Martins and group [16] have studied Thermo-optical properties of Nd^{3+} doped phosphate glass determined by thermal lens and lifetime measurements and their study they synthesized the thermal and optical properties of a set of Nd^{3+}: PAN phosphate glass samples were obtained using the Normalized lifetime thermal lens technique. Kirti Nanda et al [17] have investigated Effect of doping of Nd^{3+} ions in BaO-TeO_2-B_2O_3 glasses: A vibrational and optical study. In their study the values of optical band gap decrease with increase in concentration of Nd_2O_3. C.Pérez-Rodríguez et al [18] have examined relevance of radiative transfer processes on Nd^{3+} doped phosphate glasses for temperature sensing by means of the fluorescence intensity ratio technique and they investigated that Nd^{3+} show a luminescence spectrum due to transitions from thermally coupled levels; $^4F_{3/2}$ and $^4F_{5/2}$ that exhibit a change in emission band shape with temperature. Ying Tian and group [19] have reported optical absorption and near infrared emissions of Nd^{3+} doped fluorophosphates glass and they were studied that Nd^{3+} doped fluorophosphates glass with desirable low OH^- concentration and optical properties may become an appropriate material for developing solid state lasers around infrared region.

III. EXPERIMENTAL PROCEDURE

Nd^{3+}-doped barium phosphate (PBaNd) glasses with the molar composition of (50-x/2) P_2O_5 – (50-x/2) BaO - x Nd_2O_3, where x = 0.01, 0.05, 0.1, 0.5, 1.0, 2.0, 3.0 and 4.0 mol % referred as PBaNd001, PBaNd005, PBaNd01, PBaNd05, PBaNd10, PBaNd20, PBaNd30 and PBaNd40, respectively, were prepared by the conventional melt quenching technique. The stoichiometric compositions of the batch at batch materials (~25g) were melted in a platinum crucible at 1200 °C for 1 hour 30 min. The melt was stirred with a platinum rod and then poured onto a preheated brass plate, annealed at 400 °C for 12 h to relieve thermal stresses and strains and then slowly cooled to room temperature. Finally, the glass samples were cut and polished for optical measurements.

For PBaNd10 glass, the optical path length (l = 0.527 cm) and refractive index (n = 1.598) were measured by digital Vernier calipers and Abbe refractometer at wavelength of 589.3 nm using 1-bromonapthalene as contact liquid, respectively. The densities of all the glasses were determined by Archimedes' method using distilled water as an immersion liquid. The optical absorption spectrum was measured using a Perkin Elmer Lambda-950 UV-visible-NIR spectrophotometer in the range of 300-950 nm with a spectral resolution of 1 nm. The luminescence spectra of Nd^{3+} ions in PBaNd glasses were measured by using diode laser at 808 nm. All these measurements were carried out at room temperature only.

IV. RESULTS AND DISCUSSION

Absorption Spectrum and Judd-Ofelt analysis

The absorption spectrum of 1.0 mol % of Nd_2O_3 doped barium phosphate glasses in the wavelength range 300-950 nm is shown in Figure 1. The spectrum consists of 11 inhomogeneously broadened spectral profiles due to poor resolution of individual stark components due to disordered environment of glass matrix [20]. The absorption peaks centered at around 356 nm, 429 nm, 476 nm, 512 nm, 524 nm, 582 nm, 627 nm, 678 nm, 745 nm, 802 nm and 871 nm are assigned to transitions from ground state $^4I_{9/2}$ to the higher excited states $^4D_{3/2}$+$^4D_{5/2}$, $^2P_{1/2}$, $^4G_{11/2}$ + $^2D_{3/2}$ + $^2G_{9/2}$ + $^2K_{15/2}$, $^4G_{9/2}$, $^4G_{7/2}$, $^4G_{5/2}$ + $^2G_{7/2}$, $^2H_{11/2}$, $^4F_{9/2}$, $^4F_{7/2}$ + $^4S_{3/2}$, $^4F_{5/2}$+$^2H_{9/2}$ and $^4F_{3/2}$ respectively in $4f^3$ electronic configuration of Nd^{3+} ion. The assignment of the observed absorption bands has been made according to the earlier reports on Nd^{3+}-doped glasses [21-23].

From figure 1, it is noticed that $^4I_{9/2} \rightarrow {}^4G_{5/2}$+$^2G_{7/2}$, transition is more intense than any other transitions in present PBaNd10 glass. This transition is known as the hypersensitive transition, for which the selection rules $\Delta J \leq 2$; $\Delta L \leq 2$ and $\Delta S = 0$ holds good. The absorption intensity

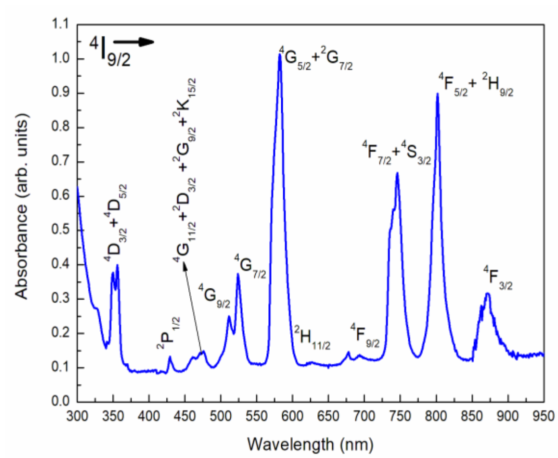

Figure.1 Absorption spectrum of Nd^{3+} ions in PBaNd10 glass.

of the hypersensitive transition is found to be more sensitive to the environment of Nd^{3+} ion. Also the band at 801 nm is more intense in the NIR region and this wavelength is most commonly used for optical pumping of Nd^{3+} based lasers, either by flash lamps or by semiconductor GaAs laser diodes [24]

The J-O theory [11, 12] provides reasonably good description for the spectral transition intensities of Nd^{3+} ions in glass matrices, since the theory assumes that all crystal field levels of the ground state are equally populated at room temperature [25]. By measuring the area under the absorption bands the experimental oscillator strengths have been evaluated using Ref. [26]. Table 1 presents the experimental (f_{exp}) and calculated (f_{cal}) oscillator strengths for various absorption transitions of Nd^{3+} ions in PBaNd10 glass. The value of f_{exp} is found to be high for $^4I_{9/2}\rightarrow{}^4G_{5/2} + {}^2G_{7/2}$ transition indicates that the intensity of absorption band depends on the neighboring ligands and it's hypersensitive transition. The f_{exp} values are in good agreement with the f_{cal} yielding small rms deviation of $\pm0.48\times10^{-6}$. Using least squares fit method the phenomenological J-O intensity parameters Ω_λ, (λ=2, 4, 6) are determined and presented in Table 2 along with some other reoprted Nd^{3+}: systems [14,16,27-29]. These are host dependent parameters and evaluated intensity parameters follow the trend as $\Omega_4>\Omega_6>\Omega_2$. Similar trend has been observed in PKCFAN10 [14], PAN [16], and ZBSN5[28] glasses.

Table 1. Experimental energies (E_{exp}, cm^{-1}), experimental (f_{exp}) and calculated (f_{cal}) oscillator strengths ($\times 10^{-6}$) of PBaNd10 glass.

[a]σ indicates root mean square deviation between experimental and calculated energy values and N represents the number of energy levels used in the fitting.

Transition	Energy	Oscillator strengths	
$^4I_{9/2}\rightarrow$	E_{exp}		
		f_{exp}	f_{cal}
$^4F_{3/2}$	11468	2.20	2.67
$^4F_{5/2}+{}^2H_{9/2}$	12469	7.34	6.79
$^4F_{7/2}+{}^4S_{3/2}$	13423	5.85	6.34
$^4F_{9/2}$	14749	0.43	0.51
$^2H_{11/2}$	15949	0.09	0.14
$^4G_{5/2}+{}^2G_{7/2}$	17182	16.30	16.34
$^4G_{7/2}$	19084	4.22	3.58
$^4G_{9/2}$	19531	2.58	1.57
$^4G_{11/2}+{}^2D_{3/2}+{}^2G_{9/2}+{}^2K_{15/2}$	21008	1.74	1.29
$^2P_{1/2}+{}^2D_{5/2}$	23310	0.74	0.72
$^4D_{1/2}+{}^4D_{3/2}+{}^4D_{5/2}$	28090	12.68	13.07
σ (N)[a]		±0.48 (11)	

Table 2. The Judd-Ofelt parameters (Ω_λ, $\times10^{-20}$ cm^2), their trend, spectroscopic quality factor (χ) and radiative lifetime (τ_{rad}, μs) of $^4F_{3/2}$ level in Nd^{3+}:glass systems.

Glass systems	Ω_2	Ω_4	Ω_6	Trend	χ	τ_{rad}
PBaNd10 [Present wor	2.62	5.47	4.24	$\Omega_4 > \Omega_6 > \Omega_2$	1.29	331
PKCFAN10 [14]	5.40	7.03	6.51	$\Omega_4 > \Omega_6 > \Omega_2$	1.08	254
PAN [16]	4.7	6.0	5.4	$\Omega_4 > \Omega_6 > \Omega_2$	1.11	-
NKZLSNd10 [27]	10.26	6.38	6.06	$\Omega_2 > \Omega_4 > \Omega_6$	1.05	173
ZBSN5 [28]	0.25	1.86	1.36	$\Omega_4 > \Omega_6 > \Omega_2$	1.37	--
Silica borate [29]	2.14	2.57	1.93	$\Omega_4 > \Omega_2 > \Omega_6$	1.33	-

The Ω_2 parameter is most sensitive to the local structural changes since it involves the long range terms in the crystal field potential [30]. The Ω_2 value in the present glass is higher than ZBSN5 [28] and Silica borate [29] but lower than that of other Nd^{3+}-doped PKCFAN10 [14], PKMAN [23], PAN [16] and NKZLSNd10 [27] glasses. Low value of Ω_2 parameter in the the present glass indicate the lower covalency between Nd-O bonds and asymmetry in the surrounding of Nd^{3+} ions. The parameters Ω_4 and Ω_6 depend on the mechanical rigidity of the host matrix [31]. The higher values of Ω_4 and Ω_6 for the present glass indicate its higher rigidity.

The spectroscopic quality factor (χ) is an important parameter in predicting the behaviour of various lasing transitions in a given matrix and generally, it is the ratio of Ω_4 and Ω_6 [32]. The χ-factor for the present PBaNd10 glass is presented in Table 2 along with reported glasses [14,16,27-29]. The obtained χ value for the PBaNd10 glass was more or less similar to the other host matrices [14,16,27-29] and indicating that the $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition possesses probability for laser action.

Emission Spectra and radiative properties

Figure 2 shows the concentration dependent NIR emission spectra for all the Nd^{3+} doped glasses under study which are recorded by exciting the samples at 808 nm using diode laser. The spectra exhibited the prominent emission bands at around 875 nm, 1056 nm and 1327 nm corresponding to the $^4F_{3/2}\rightarrow{}^4I_{9/2,\ 11/2,\ 13/2}$ transitions, respectively. From figure 2 it is observed that the intensity of emission bands increases with increase of Nd^{3+} ion concentration up to 1.0 mol % thereafter it decreases with concentration. This may be due to due to the enhanced interaction between excited state of Nd^{3+} ions, and/or ground state of Nd^{3+} ions and host defects leading to energy transfer process through cross-relaxation channels that could be attributed to $^4F_{3/2}+{}^4I_{9/2} \rightarrow {}^4I_{15/2} + {}^4I_{15/2}$ (resonant) or $^4F_{3/2} + {}^4I_{9/2} \rightarrow {}^4I_{13/2} + {}^4I_{15/2}$ (phonon assisted) energy transfer between the active ions [26].

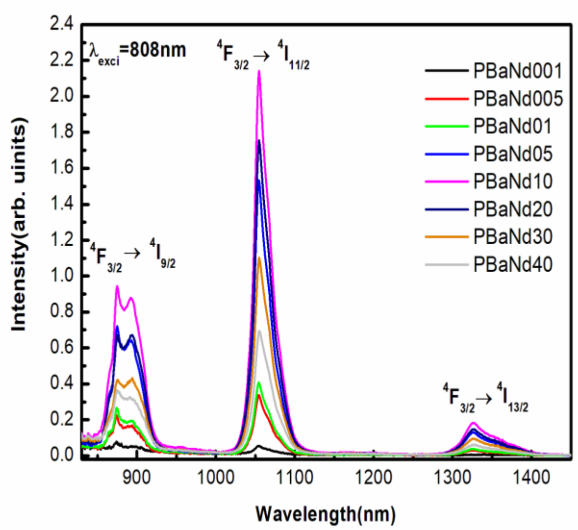

Figure 2. Near Infrared luminescence spectra Nd^{3+} ions in PBaNd glasses

The JO intensity parameters along with refractive index have been used to evaluate the various radiative properties using the procedure described in Refs. [33,34]. The total radiative transition probability ($\sum A_R$) is found to be 3193 s^{-1} and the τ_R ($1/\sum A_R$) is 313 μs. The radiative parameters such as effective bandwidths ($\Delta\lambda_{eff}$), radiative transition probabilities (A_R), experimental and calculated branching ratios (β_{exp}, β_R) and the stimulated emission cross-sections (σ_{emi}) have been estimated for the ${}^4F_{3/2}\rightarrow{}^4I_{9/2,11/2,13/2}$ transitions Nd^{3+} ion from the emission spectrum of PBaNd10 glass which are presented in Table 3.

Table 3. Emission band positions (λ_p, nm), effective bandwidths ($\Delta\lambda_{eff}$, nm), radiative transition probabilities (A_R, s^{-1}), peak stimulated emission cross-sections ($\sigma(\lambda_p)$, $\times10^{-20}$ cm^2), experimental and calculated branching ratios (β_R) for ${}^4F_{3/2} \rightarrow {}^4I_J$ (J = 9/2, 11/2, 13/2 and 11/2) transitions in PBaNd10 glass.

Transition ${}^4F_{3/2} \rightarrow$	λ_p	$\Delta\lambda_{eff}$	A_R	$\sigma(\lambda_p)$	β_R	
					Exp.	Cal.
${}^4I_{9/2}$	875	44	1469.5	1.03	0.38	0.46
${}^4I_{11/2}$	1056	24	1450.66	2.79	0.54	0.46
${}^4I_{13/2}$	1327	47	260.26	0.64	0.08	0.08

The luminescence branching ratio is a critical parameter for the laser designer, because it characterizes the possibility of attaining stimulated emission from any specific transition. In the present study, it is inferred that, the values of the branching ratio for ${}^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition is found to be maximum and also reasonable agreement is noted between measured and predicted β_R values. Moreover the higher value of A_R and σ_{emi} suggests that the present glass could be potentially useful for getting laser emission at around 1.06 μm [35].

V. Conclusions

Barium phosphate glasses doped with varying Nd^{3+} ions concentration were prepared by melt quenching method and characterized through absorption and luminescence measurements. Judd–Ofelt theory has been applied to evaluate the best fit intensity parameters, and used to calculate the radiative lifetime and stimulated emission cross section of the lasing transitions**.** The present glasses show an intense and sharp laser emission band ${}^4F_{3/2} \rightarrow {}^4I_{11/2}$ at 1056 nm and it has high stimulated emission cross-section and branching ratio compared to other transitions. Therefore, barium phosphate host glass doped with neodymium oxide is a good candidate for laser applications at 1.06 μm.

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