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Optical Analysis of Rare Earth Doped Sodium Bismuth Silicate Glass

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Abstract

Samarium doped sodium bismuth silicate glass was synthesized using the melt quenching method. Detailed optical spectroscopic studies of the glassy material were carried out in the UV-Vis-NIR spectral range. Our analysis revealed that the novel glassy system has the optimum values for the key parameters viz. spectroscopic quality factor, optical gain, stimulated emission cross section and quantum efficiency, which are required for a high performance optical amplifier. Calculated chromaticity co-ordinates (0.61, 0.38) also confirm its application potential in display devices.

Key words:- A.Glasses, D. optical properties

1. Introduction

Rare earth (RE) ion activated materials find wide applications in display devices, lighting technology, solar cell energy conversion, telecommunications, remote sensing etc[1]. Recent years have witnessed a tremendous increase in research activities related to glasses doped with rare earth ions in various forms such as network formers, modifiers or luminescent ions[2,3]. Most often, research efforts have concentrated on fluoride, tellurite and chalcogenide glass systems because of their lower phonon energies compared to oxide glasses. Nevertheless, oxide glasses have proven more suitable for practical applications due to their high chemical durability and thermal stability. Among oxide glasses, silicate glasses are the most popular glass hosts for making optical fiber lasers and amplifiers[4-7]. They are also known to be an excellent host matrix for rare earth oxides because of their good glass forming ability when compared to several other conventional systems such as borate, phosphate, germinate, vanadate and tellurite glass families. This makes it an interesting system for experimental and theoretical studies [8-12].

Heavy-metal silicate glasses possess lower phonon energies compared to other silicate glasses and display intense visible and near-infrared fluorescence of the rare-earth ions within the system [13-16]. The trivalent samarium ion (Sm^{3+}) is one of the most important active ions in the RE family due to its closely lying energy level structure[17]. The ${}^4G_{5/2}$ level of Sm^{3+}has a relatively high quantum efficiency which shows luminescence in the orange and red regions of the visible spectrum via transitions to the ground state ${}^6H_{5/2}$ or to the higher energy states ${}^6Hj$ [(j = 7/2, 9/2, and 11/2) [17]. Although there have been a large number reports on various rare earth doped glassy systems, the synthesis and optical analysis of samarium doped bismuth silicate glasses have seldom been studied. At relatively low Bi_2O_3 content (≤10 mol%), Bi_2O_3incorporates into the interstices of glass as network modifiers which doesn’t cause a large-scale structural rearrangement of the local glassy network. At higher concentrations of Bi_2O_3 (>10 mol%), Bi_2O_3 enters into glasses as a network former and a large-scale structural rearrangement of the local glass network takes place, which leads to significant variation of its optical properties[18-20].

In this context, an optical analysis of samarium doped bismuth (10mol%)sodium silicate glass is deserving of attention which to our knowledge have not been reported so far. Understanding the optical properties of samarium doped bismuth sodium silicate glass is a pre-requisite for considering optical applications. The purpose of the present study is to prepare samarium doped bismuth sodium silicate glass and to study various optical properties such as radiative transition probability, optical gain, stimulated emission cross section and chromaticity parameters. All derived or observed results are compared with similar systems found in the literature.

2. Experimental

Samarium doped sodium bismuth silicate glasses (83.5 SiO_2-5Na_2CO_3-10Bi_2O_3-1.5Sm_2O_3) were prepared by the well-known melt quenching method. Appropriate amounts of Bi_2O_3 (99.99% purity, Sigma Aldrich),Na_2CO_3 and SiO_2(99.99% purity, Sigma Aldrich) were mixed and ground continuously using an agate mortar. The powder mixture was placed in proclaim crucible and melted in a box furnace at a temperature of 1200^0C for 3 hours before the molten mixture was poured into a stainless steel mold heated to 100^0C. The sample was then annealed at a temperature of 200^0Cfor 1hour. The density of the sample was measured by Archimedes' principle. To avoid any undesired reaction with the synthesized glass, xylene was taken as the immersion liquid. Refractive index of the glasses were measured using Abbe refractometer (accuracy 0.001). Concentration of rare earth ion in the glasses was calculated from the starting batch composition and density of the glass samples. U-V Visible-NIR absorption spectra of the sample were measured using a UV-Visible-NIR spectrophotometer Varian Cary 5000 in the wavelength range form250nm to 2000nm. Fluorescence spectra of the sample were recorded using spectrofluro-photometer Shimadzu RFPC 5301 in the range from 200 to 900nm. All the measurements were done at room temperature.

3. Results and Discussions

Figure 1 shows the optical absorption spectra of samarium in bismuth silicate glass in the NIR region. All absorption bands are due to transitions between the multiplets of $4f^5$ configurations of Sm^{3+} with the ground state $^6H_{5/2}$. The typical absorption peaks of Sm^{3+}resultfrom transitions from the ground state $^6H_{5/2}$ to the excited states $^4H_{9/2}$,$^4D_{3/2}$,$^6P_{7/2}$,$^6F_{7/2}$,$^4I_{13/2}$,$^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^6F_{1/2}$ and $^6H_{13/2}$[21].

Indirect but convincing information regarding the RE-ligand bond strength can be obtained from the nephelauxatic ratio(β). The nephelauxatic ratio is given by:

$$\beta = {}^{\nu_m}/{\nu_a}$$

where ν_m and ν_a are the wave numbers(cm^{-1}) of the particular transitions in the host matrix and aqua, respectively. Larger nephelauxatic ratios indicate a reduction in the strength of the covalent bond between a RE ion and a ligand. The nephelauxatic parameter is directly related to the bonding parameter (δ) by:

$$\delta = {}^{(1-\bar{\beta})}/{}_{\bar{\beta}}$$

where $\bar{\beta}$ is the average value of β for the observed transitions. The positive or negative sign of (δ) indicates the covalent or ionic bonding nature of the RE –ligand bond. The small positive value of δ (0.002) measured in the sodium bismuth silicate glass indicates a decreased strength of the RE–O covalent bond in bismuth sodium silicate glass compared to other oxide systems[22].

The intensities of all the absorption bands were evaluated by measuring their oscillator strengths (f), which are proportional to the area under the absorption line shapes. For single peaks the absorption band was integrated directly after subtracting the background loss. The oscillator strengths can be expressed in terms of the molar extinction coefficient (ε) as [23]:

$$f = 4.32 \times 10^{-9} \int \varepsilon(\nu) d\nu \quad (1)$$

where $d\nu$ is the full width at half height for a given energy ν.

According to the Judd-Ofelt [24,25] (JO) theory, the oscillator strengths of an intra configurational electric dipole transition within the 4f shell of a trivalent rare earth ion can be obtained from the relationship:

$$f_{ed} = \frac{\nu}{(2J+1)} \left[\frac{8\pi^2 mc}{3h} \frac{(n^2+2)^2}{9n} \right] \sum_{\lambda=2,4,6} \Omega_\lambda \left\langle \psi J \| U^\lambda \| \psi' J' \right\rangle^2 \quad (2)$$

where (2J+1) is the degeneracy of the ground state, ν is the mean energy of the $|\psi J\rangle \rightarrow |\psi' J'\rangle$ transition, For the calculation of JO intensity parameters (Ω_λ) of Sm^{3+}, the levels of this ion are divided into a low set of energies (<10000cm^{-1}) and a high lying set of levels (>17000cm^{-1}). Only the low set is compatible with JO theory[26]. U^λ's are the doubly reduced unit tensor operators of rank λ calculated in the intermediate coupling approximations[Table 1]. U^λ are known to be almost insensitive to the ion's environment [27] . Using the matrix elements and experimental oscillator strengths, Ω_λ 's have been evaluated by least square programming. The experimentally obtained oscillator strengths of various absorption transitions and the evaluated JO parameters of Sm^{3+} ions in the present glassy matrix are found to be in good agreement with those of other similar matrices[see Table 2 and 3].

The magnitude of the spectroscopic quality factor($\chi = \Omega_4 / \Omega_6$)is used to characterize stimulated emission in the host matrix and the quality factor (1.65) for the present system promises efficient stimulated emission[28].

Various radiative parameters, viz radiative transition probability (A), radiative life time(τ_{rad}), fluorescence branching ratio (β),the integrated absorption crossection or effective cros-section (σ_a) for a stimulated emission of the active ion are directly evaluated using the expressions:

$$A^{ed}{}_{JJ} = \frac{64\pi^2 e^2 \upsilon^3}{3h(2J+1)}\left[\frac{n(n^2+2)^2}{9}\right]S_{ed} \qquad (3)$$

where $S_{ed} = \sum_{\lambda=2,4,6} \Omega_\lambda \left\langle \psi J \left\| U^\lambda \right\| \psi' J' \right\rangle^2$ (4)

$$\beta_{JJ'} = \frac{A_{JJ'}}{\sum_{J'} AJJ'} \qquad (5)$$

$$\tau_{rad} = \frac{1}{\sum_{J'} A_{JJ'}} \qquad (6)$$

and $\sigma_a = \frac{1}{\nu^2}\frac{A}{8\pi cn^2}$ (7)

The electric dipole line strength (S_{ed}), radiative transition probability (A) , total radiative transition probability (A_R), radiative life time (τ), branching ratio(β) and stimulated absorption crossection (σ_a) for all $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$ and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ transitions and are presented in Table4. The trend in the branching ratio (β_R) is $^4G_{5/2} \rightarrow {}^6H_{7/2} > {}^4G_{5/2} \rightarrow {}^6H_{5/2} > {}^4G_{5/2} \rightarrow {}^6H_{9/2} > {}^4G_{5/2} \rightarrow {}^6H_{11/2}$. The branching ratio for the $^4G_{5/2}$-$^6H_{7/2}$ transition in the present glass is 49 %, exhibiting a maximum in emission spectra at 598nm.

Figure 2 shows the fluorescence spectra of Sm^{3+} ions in sodium bismuth silicate glass and the corresponding excitation spectrum for 600nm emission is given in Figure 3. Emission bands of Sm^{3+} , $^4G_{5/2} \rightarrow {}^6H_{5/2}$(562 nm), $^4G_{5/2} \rightarrow {}^6H_{7/2}$(598 nm), $^4G_{5/2} \rightarrow {}^6H_{9/2}$(644 nm) and $^4G_{5/2} \rightarrow {}^6H_{11/2}$(694 nm) are observed(Figure 4). To minimize the possibility of reabsorption, concentration of Sm^{3+} ion was kept low. Emission transitions $^4G_{5/2} \rightarrow {}^6H_{5/2}$ and $^4G_{5/2} \rightarrow {}^6H_{7/2}$ are formally magnetic dipole (MD) dominated and the magnetic dipole strengths are much larger than for the transitions $^4G_{5/2} \rightarrow {}^6H_{9/2}$ and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ with little electric dipole (ED) contribution which is less sensitive to the site symmetry. The transitions $^4G_{5/2} \rightarrow {}^6H_{9/2}$ and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ on the other hand are electric dipole (ED) in nature and magnetic dipole line strengths are considerably smaller than for the transitions $^4G_{5/2} \rightarrow {}^6H_{5/2}$ and $^4G_{5/2} \rightarrow {}^6H_{7/2}$. The intensity ratio (ED/MD) is influenced by the site symmetry, electronegativity and covalency of the ligand atoms. Larger the inequality of emission intensity larger is the asymmetry and covalency effects. A larger ratio corresponds to a more distorted or asymmetric local cation environment. In the present case a numerical value of 0.44obtained for the ratio ${}^4G_{5/2}\rightarrow{}^6H_{9/2}$ / ${}^4G_{5/2}\rightarrow{}^6H_{5/2}$ suggests less asymmetry and covalency[29]. The stimulated emission crossection ($\sigma_p(\lambda_p)$) and quantum efficiency of ${}^4G_{5/2}$ fluorescence upon excitation to a higher level (at 402 nm which is the most intense band) can be calculated by use of the expressions [30]:

$$\sigma(\lambda_p) = \left[\frac{\lambda_p^4}{8\pi c n^2 \Delta\lambda_{eff}}\right] A_R \qquad (8)$$

and Quantum efficiency (QE)

$$QE_u = QE_s \frac{F_u A_s (\lambda_{ex})_s}{F_s A_u (\lambda_{ex})_u} \qquad (9)$$

where F is the total area under the emission peak, A the absorbance, λ_{ex} the excitation wavelength and the subscripts u and s denote unknown and standard respectively. The calculated QE of the 1.5 mol% Sm^{3+} doped glass sample was 5%, 20% and 0.7% for emissions at 562, 598and 643 nm which is comparable with the values obtained for laser glasses and reported found in the literature[31].

Radiative life time, effective emission bandwidth, stimulated emission cross-section, gain bandwidth ($\sigma_{(e)}$FWHM),optical gain ($\sigma_{(e)}\tau_r$) and quantum efficiency are key parameters if a material is to be used as an optical amplifier. The higher values obtained for these parameters suggest better amplifier performance. Table 5 reports the calculated values of these parameters for the${}^4G_{5/2}\rightarrow{}^6H_{5/2}$, ${}^6H_{7/2}$ and ${}^6H_{9/2}$ transitions. The calculated values of gain bandwidth and optical gain shows the a trend ${}^6H_{7/2} > {}^6H_{5/2} > {}^6H_{9/2}$. A comparison of these values with other best performing glassy systems like zinc bismuth borate glasses [32], sodium borate and sodium fluoro-borate glasses[33],L5FBS glasses [34], sodium tellurate glass[28], lead tungstate glasses [35] and cadmium silicate [8] leads to the conclusion that the ${}^4G_{5/2}$-${}^6H_{5/2}$ and${}^4G_{5/2}$- ${}^6H_{7/2}$ transition(at 562nm and 598nmrespectively) provide favorable lasing action, high gain bandwidth and optical gain for amplifier applications. Hence, Sm^{3+}-doped bismuth sodium silicate glass is a promising material for photonic applications.

For device applications, in addition to the quantum efficiency and stimulated emission cross section, the colour chromaticity of the samples is also a key parameter for performance evaluation. Colour chromaticity of a sample is estimated using the Commission Internationale de L'Eclairage (CIE) in1931system [36, 37]. The chromaticity specifies the hue and saturation of a color and hence chromaticity coordinates are useful in determining the exact emission color and color purity of a system [38]. Using spectral power distribution of the glassy sample and the CIE colour matching functions, the tristimulus values X (λ), Y(λ) and Z(λ) are calculated. Chromaticity co-ordinates (x,y) are estimated from the tristimulus values as x= X/(X+Y+Z) and y= Y/(X+Y+Z).The (x,y) coordinates of the present glassy system are found to be (0.61, 0.38) and the corresponding color of the sample is marked in the CIE chromaticity diagram in Fig.5. The CIE diagram confirms that our novel sodium bismuth silicate glassy material produces emissions in the orange region, which increases its potential for use in display applications.

4. Conclusions

The synthesis and optical characterization of samarium doped bismuth sodium silicate glass is reported. A detailed spectroscopic investigation of the glassy material has been carried out using the Judd-Ofelt analysis of the UV-Vis-NIR absorption spectra and photoluminescence emission spectra. Stimulated emission cross-section, optical gain and quantum efficiency of the transitions have been evaluated. The investigation reveals that the transition from the energy level $^4G_{5/2}$ to $^6H_{7/2}$ of a Sm^{3+} ion has the optimum values of full-width half maximum, branching ratio, stimulated emission cross section, quantum efficiency, optical gain and gain bandwidth required for optical amplifiers. Calculated chromaticity co-ordinates (0.61, 0.38) confirm the potential of this new material for use in display devices.

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Table 1. Doubly reduced unit tensor operators $\langle \psi J \| U^{\lambda} \| \psi' J' \rangle$ for the transitions of Sm^{3+} used for the calculation of JO parameters[26].

Transitions between		$(U^2)^2$	$(U^4)^2$	$(U^6)^2$
$^6H_{5/2}$ →	$^6F_{1/2}$	0.1947	0.0	0.0
	$^6F_{3/2}$	0.1389	0.1329	0.0
	$^6F_{5/2}$	0.0346	0.2594	0.0
	$^6F_{7/2}$	0.0042	0.1099	0.3939
	$^6F_{9/2}$	0.0001	0.0183	0.3526
	$^6F_{11/2}$	0.0	0.0006	0.0527
$^4G_{5/2}$ →	$^6H_{5/2}$	0.0002	0.0006	0.0
	$^6H_{7/2}$	0.0	0.0067	0.0081
	$^6H_{9/2}$	0.0112	0.0066	0.0021
	$^6H_{11/2}$	0.0	0.0056	0.0030
	$^6H_{13/2}$	0.0	0.0	0.0022
	$^6F_{1/2}$	0.0008	0.0	0.0
	$^6F_{3/2}$	0.0013	0.0001	0.0

Table 2 Experimentally observed energy, oscillator strength and electric dipole line strength of low energy lying set Sm^{3+} in sodium bismuth silicate glass.

Transition from $^6H_{5/2}$ to	Energy (cm^{-1})	F_{meas}(10^{-5})cm^2	S_{ed}(10^{-22})
$^6F_{9/2}$	9259	0.12	4.93
$^6F_{7/2}$	8170	0.14	6.52
$^6F_{5/2}$	7289	0.67	35.1
$^6F_{3/2}$	6786	0.51	28.6
$^6F_{1/2}$	6477	0.39	22.9
$^6H_{13/2}$	6329	0.078	4.21

Table 3. JO parameters of Sm3+ in sodium bismuth silicate glass and similar glassy systems.

Matrix	Ω_2 (10^{-20}) (cm^2)	Ω_4 (10^{-20}) (cm^2)	Ω_6 (10^{-20}) (cm^2)	Reference
Phosphate	1.65	2.66	2.65	30
Tellurite	1.49	5.68	3.33	17
borate	1.28	2.78	1.97	39
Fluoride	1.16	2.60	1.40	32
Silicate	6.56	5.14	4.08	3
ZBLAN	2.15	3.05	1.56	40
Bismuth silicate	2.26	3.46	2.09	Present work

Table 4.Calculated values of electric dipole line strength (Sed), radiative transition probability(A), total radiative transition probability(A_T), radiative life time (τ), branching ratio(β) and stimulated absorption crossection (σ_a) of Sm^{3+}in sodium bismuth silicate glass.

Level from $^4G_{5/2}$	Energy cm^{-1}	S_{ed} x10^{22}	A (s^{-1})	A_T (s^{-1})	τ (ms)	β	σ_a ($10^{-18)}$ cm^2
$^6H_{5/2}$	17857	4.12	105	278	3.5	0.38	9.9
$^6H_{7/2}$	16780	7.47	138			0.49	14.7
$^6H_{9/2}$	15550	2.7	27			0.1	33.7
$^6H_{11/2}$	14224	1.02	8			0.03	1.6

Table 5.Emission crossection, the product of emission cross-section and FWHM and the product of radiative life time and stimulated emission cross-section for the observed fluorescence transitions.

Level from $^4G_{5/2}$	Energy (cm$^{-1)}$	Δλ (nm)	$\sigma_p(\lambda_p)$ (10^{-22}) cm^2	σ_eX FWHM(10^{-29}) cm	σ_e x τ (10^{-25}) cm^{2}s	QE (%)
$^6H_{5/2}$	17857	16	2.41	38.56	8.44	5.1
$^6H_{7/2}$	16780	21	3.08	64.68	10.78	20
$^6H_{9/2}$	15550	13	0.64	17.28	2.24	0.7
$^6H_{11/2}$	14224	-	-	-	-	

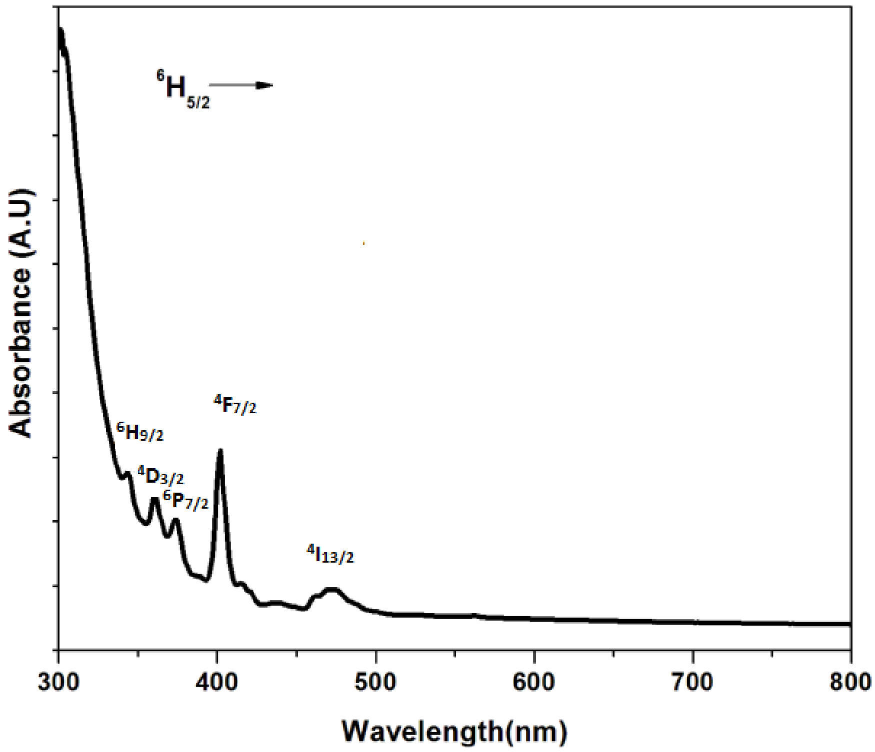

Figure 1(a) Uv-Vis Absorption spectrum of Sm^{3+} in sodium Bismuth silicate glass

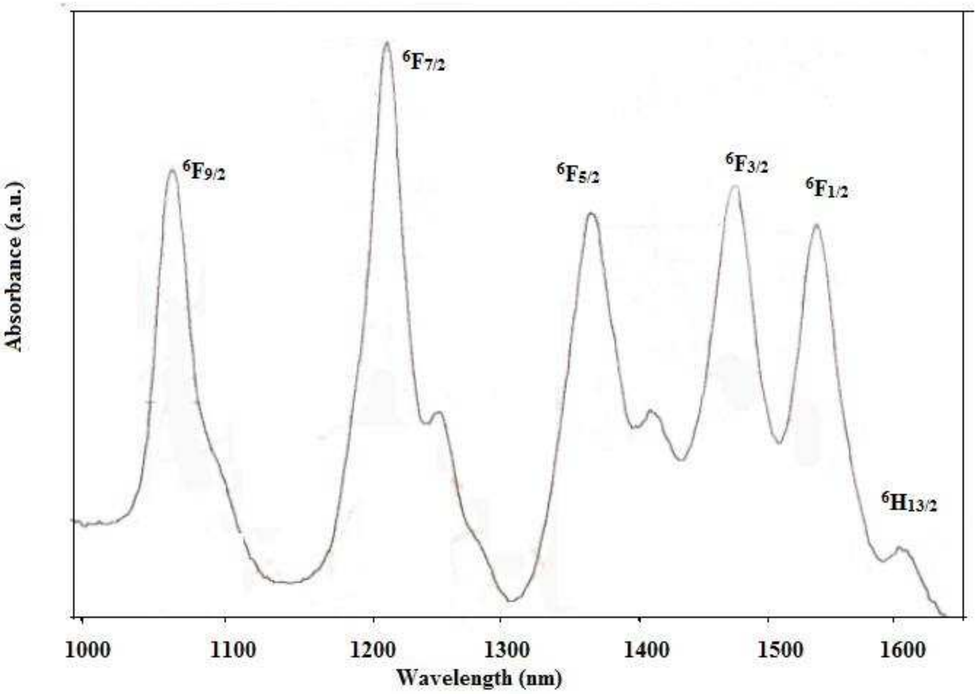

Figure 1(b) NIR Absorption spectrum of Sm^{3+} in sodium Bismuth silicate glass

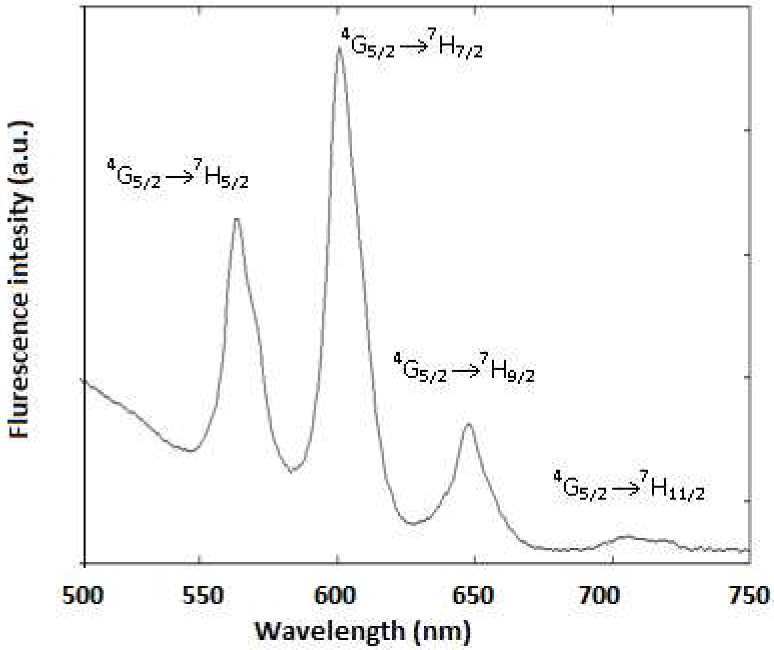

Figure 2. Fluorescence spectrum of Sm^{3+}in sodium bismuth silicate glass (λexc= 401 nm).

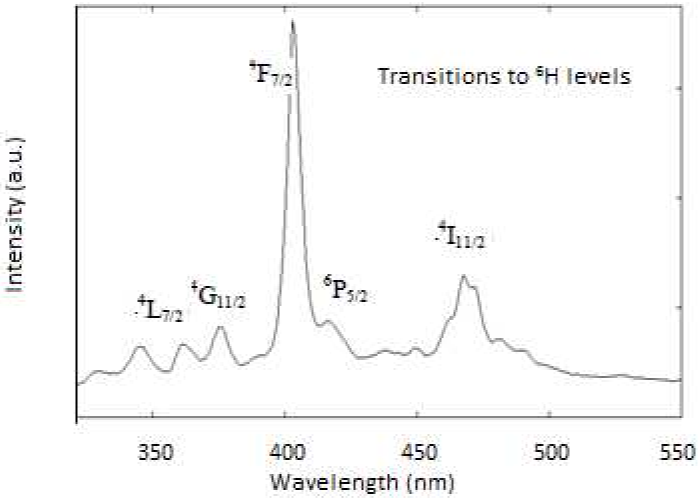

Figure. 3 Excitation spectrum of Sm^{3+}in sodium bismuth silicate glass(λem=600nm).

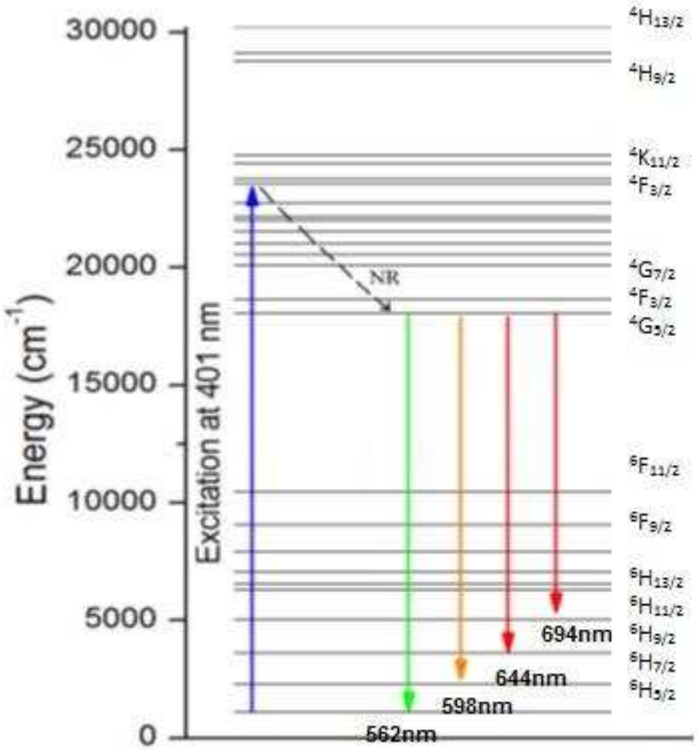

Figure 4 Optical transitions of Sm^{3+} in sodium bismuth silicate glass.

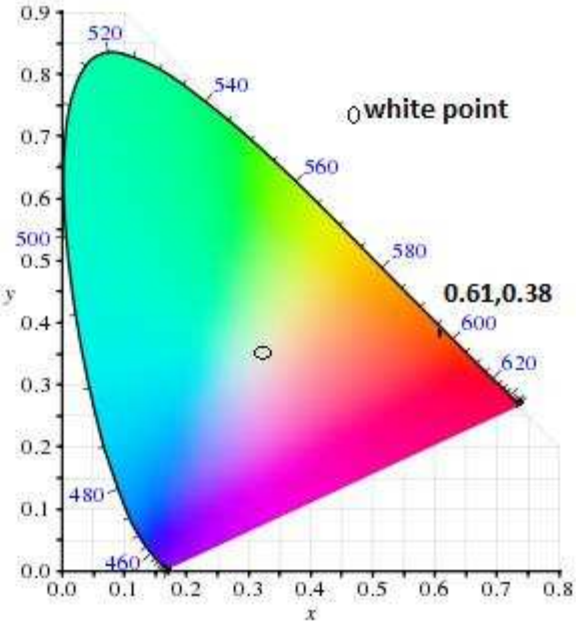

Figure 5. CIE chromaticity diagram of Sm^{3+} in sodium bismuth silicate glass.

Graphical abstract