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White Light Generation in Dy^{3+}-Doped Sodium Lead Alumino Borosilicate Glasses for W-LED Applications

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Abstract: Excitation and emission spectra of sodium lead alumino borosilicate glass doped with different concentrations of Dy^{3+} have been reported. The concentration of Dy^{3+} were varied from 0.5 to 1.5 mol%. As a result of 385 nm excitation wavelength, the luminescence spectra showed three characteristic bands at 482 nm and 575 nm and 665 nm. These absorption bands were attributed to $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transitions respectively of Dy^{3+} ions. $^4F_{9/2} \rightarrow {}^6H_{15/2}$, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ and $^4F_{9/2} \rightarrow {}^6H_{11/2}$ transitions respectively of Dy^{3+} ions. The yellow to blue intensity ratio increases (1.063 to 1.093) with increase in Dy^{3+} ion concentration. The decay rates exhibit single exponential for lower concentrations and turns into non exponential for higher concentrations. The non-exponential nature of the decay rates is well-fitted to the Inokuti-Hirayama model for S = 6, which indicates that the nature of the energy transfer between donor and acceptor ions is of dipole-dipole type. The lifetime for the $^4F_{9/2}$ level of Dy^{3+} ion decreases (0.634 to 0.580 ms). The chromaticity coordinates have been calculated from the emission spectra and analyzed with Commission International de I'Eclairage diagram. The chromaticity coordinates visible in the white light region for all concentrations of Dy^{3+} ions in the present glasses. The correlated color temperature value varies from 5107 to 5362 K (closer to the day light value of 5500 K). These results indicate that Dy^{3+}-doped sodium lead alumino borosilicate glasses can be development of white light emitting diodes and suitability for solid state lasers applications.

Keywords: Sodium Lead Alumino Borosilicate Glasses, Excitation, Emission, Fluorescence, Decay Analysis

Introduction

In the recent years White Light Emitting Diodes (W-LEDs, next generation of Solid-State Lighting (SSL) technology has been the subject of increasing interest due to their potential applications in lasers, automobile headlights and light indicators etc., (Nakamura and Fasol, 1997). The wide spread of solid-state lighting is of great importance to reduce significantly the global electricity consumption and the use of fossil fuels (OIDA, 2002). These W-LEDs have the advantages of high luminous efficiency, long operation lifetime reliability, safety and environmental friendly characteristics (Lin *et al*., 2008). At present W-LEDs are usually fabricated by the combination of blue Light Emitting Diode (LED) chip with fluorescence phosphors (Park *et al*., 2003) However, the individual degradation rates of the blue LED chip and the phosphors coated on the chip would cause chromatic aberration and poor white light performance. To solve this problem, it is essential to develop novel materials that could emit bright white light by Ultraviolet (UV) excitation (Liang *et al*., 2008; Chang and Chen, 2007). In the scheme of current constructing W-LEDs is used to mixed phosphor powder to enable its coating on the chip, which may shorten the lifetime of the LED owing to unavailable aging of the coating layer under long-term UV irradiation. To overcome this problem, recently, the

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trivalent lanthanide (Ln^{3+}) ions doped luminescent materials like glasses and glass-ceramics are reported as a good alternative for the phosphor powders, since they can be formed in a plate like shape to ensure the direct encasement of the UV chip (Tanabe *et al*., 2005). In addition to that, they have some other advantages such as homogenous light emission, simpler manufacture procedure, lower production cost and excellent thermal and mechanical stabilities (Xu *et al*., 2003). Borosilicate glasses possess high mechanical, chemical, thermal stability and low phonon energy, which indicates that these are excellent materials for optoelectronic applications (Fujita *et al*., 2005). Among the Ln^{3+} ions, dysprosium has special interest due to the existence of three luminescence bands in the blue ($^4F_{9/2} \rightarrow {}^6H_{15/2}$), yellow ($^4F_{9/2} \rightarrow {}^6H_{13/2}$) and weak red ($^4F_{9/2} \rightarrow {}^6H_{11/2}$) regions. An appropriate combination of these blue and yellow luminescence leads to generation of white light that can be tuned in glasses and glass ceramics. Moreover, dysprosium doped solid state systems can be quite easily excited by the commercial UV or blue LEDs because their excitation spectrum exhibits several 4f-4f electronic transitions in the 320-500 nm spectral range.

The present work reports, preparation and fluorescence study of Dy^{3+}-doped borosilicate glasses and the white luminescence with the combination of yellow and blue emissions as a function of Dy^{3+} ion concentration through Commission International de I'Eclairage (CIE 1931) chromaticity diagram (Fred Schubert, 2006) to develop W-LEDs by combining with GaN-based blue LEDs.

Experimental Details

The new glasses were prepared by using the conventional melt quenched technique. The current composition was prepared based on the proposed ratio: $20Na_2O$-$10PbO$-$(5-x)Al_2O_3$-$40B_2O_3$-$25SiO_2$-xDy_2O_3, x various from 0.5 to 1.5 mol% respectively. The mixed batches about 10 g were well grounded in an agate mortar and transferred to a silica crucible and then kept in an electric furnace for melting around 1200°C to 1250°C for 30 min. until the homogeneous melt is obtained. The melt was then poured onto a preheated brass plate and annealed at 450°C for 8 h and then cooled to room temperature to remove thermal stress and strains. Finally, the glasses were thoroughly polished to achieve good optical surfaces. The excitation, emission and decay rates were recorded by using JOBIN YVON HORIBA Fluorolog-3 spectrofluorometer at room temperature using xenon arc lamp (450 W) as an excitation source at 385 nm.

Theory

White Light Simulation

The degree of stimulation required to match the color of spectral power density $P(\lambda)$ is expressed as (Shanmugavelu and Kanth Kumar, 2014):

$$X = \int \bar{x}(\lambda) P(\lambda) d\lambda \tag{1}$$

$$Y = \int \bar{y}(\lambda) P(\lambda) d\lambda \tag{2}$$

$$Z = \int \bar{z}(\lambda) P(\lambda) d\lambda \tag{3}$$

where, X, Y and Z are the tri stimulus values which gives the power for each of the three primary colors to match with the color of $P(\lambda)$ and from the tri stimulus values the color chromaticity coordinates x and y can be determined from the following expression (Shanmugavelu and Kanth Kumar, 2014):

$$X = \frac{x}{x + y + z} \tag{4}$$

$$Y = \frac{y}{x + y + z} \tag{5}$$

The standard (x, y) coordinates (where x = 0.33 and y = 0.33) corresponding to the location of the white light emission is always situated at the center of the CIE 1931 chromaticity diagram.

Generally white light deals with CCT from the Planck Ian's locus approximation, the range of CCT can be derived from CIE coordinates using the third power polynomial which is applied between 2,222 K and 13,000 K and is given by the McCamy's approximate formula (Kaushal Jha, 2016; Som *et al*., 2013; Lokesh *et al*., 2016):

$$CCT(x, y) = -44n^3 + 3525n^2 6823.3n + 5520.33 \tag{6}$$

where,

$$n = \frac{(x - x_e)}{(y - y_e)} \tag{7}$$

(x_e = 0.3320 and y_e = 0.1858) (Som *et al*., 2013).

Decay Analysis

The effective decay time is evaluated using the following expression:

$$\tau_{\exp} = \tau_{eff} = \frac{\int tI(t)dt}{\int I(t)dt} \tag{8}$$

where, $I(t)$ is the emissive intensity at time 't'. For the Rare Earth (RE) doped glasses, the experimental lifetime ($\tau_{\exp}$) is expressed using the below given expression:

$$\frac{1}{\tau_{\exp}} = \frac{1}{\tau_{rad}} + W_{MPR} + W_{ET} + W_{OH} + \cdots \tag{9}$$

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where, τ_{rad} is the radiative lifetime determined through J-O theory, W_{MPR} is the multi phonon relaxation rate, W_{ET} is the rate of energy transfer and W_{OH} is the energy transfer rate between RE^{3+} ion and OH groups.

The non-radiative decay rate is determined using the following expression:

$$W_{NR} + \frac{1}{\tau_{\exp}} - \frac{1}{\tau_{rad}} \tag{10}$$

The quantum efficiency (η) is defined as the ratio of the number of photons emitted to the number of photons absorbed. In the case of RE^{3+} ion incorporated systems, it is equal to the ratio of the measured lifetime to the predicted lifetime for the corresponding levels and is given by:

$$\eta = \frac{\tau_{\exp}}{\tau_{rad}} \times 100 \tag{11}$$

Results

Luminescence Spectra

The PL excitation spectrum of sodium lead alumino borosilicate glass doped with 1.0 mol% of Dy^{3+} is as shown in Fig. 1. The spectrum is measured while keeping the monitoring wavelength that λ_{emi} = 575 nm, as it corresponds to the most intense peak in emission spectra (Fig. 1). In the excitation spectra, sharp peaks are observed due to the 4f-4f transitions of Dy^{3+} ions, which are forbidden by the Laporte parity selection rule. They grow from the ground level ($^6H_{15/2}$) to higher excited levels. From the figure it is observed that the positions of the excitation bands of NPABSDy10 glasses. Firstly, the screening effect of the electron clouds of the 5s and 5p orbital shields the intra-4f transitions from any variation in the local crystal-field environment. This renders the electrons in the 4f orbital relatively insensitive to the glassy matrix, hence the positions of the bands (or their energies, which is strongly correlated with the configuration of electrons in the 4f orbital) does not change with changing Dy^{3+} ion concentration. Secondly, as the concentration of Dy^{3+} ions is increased, the average Dy^{3+}- Dy^{3+} distance decrease which in turn increases the probability of non-radiative energy transfers between the activator ions through cross relaxation beyond the critical distance. The excitation spectra exhibit several major bands centered at 336 nm ($^6H_{15/2} \rightarrow {}^4I_{9/2}$), 348 nm ($^6H_{15/2} \rightarrow {}^6P_{7/2}$), 363 nm ($^6H_{15/2} \rightarrow {}^6P_{5/2}$), 385 nm ($^6H_{15/2} \rightarrow {}^4F_{7/2}$), 424 nm ($^6H_{15/2} \rightarrow {}^4G_{11/2}$), 451 nm ($^6H_{15/2} \rightarrow 4I_{15/2}$) and 472 nm $^6H_{15/2} \rightarrow {}^4F_{9/2}$) (Mahamuda *et al.*, 2014; Kaushal Jha, 2016; Selvi *et al*., 2014; Klosowicz *et al*., 2012; Wanyi *et al*., 2014). The result confirms that the sample can be effectively excited from Ultra Violet light (UV), Near Ultra Violet light (N-UV) and blue light. It is well known that an ideal phosphor should show strong absorption band in the wavelength range from 338 to 410 nm, which matches with the emission of commercially available N-UV and blue LED chips. Thus, the above results establish that these glasses could be efficiently excited by using commercially available GaN based light sources, providing an additional support in favor of the titled glasses.

In order to analyze luminescence properties for the NPABSDy10 glasses, it is essential to know the suitable excitation wavelengths of Dy^{3+} ions. The luminescence behavior of Dy^{3+}-doped NPABS glasses have been studied throughout the emission spectral measurement.

White Light Simulation

The assessment and quantification of color is referred as colorimetric and the CIE system is the commonly used method to describe the composition of any color. The color of the luminescent source can be described through the color matching function values $\bar{x}(\lambda)$, $\bar{y}(\lambda)$, $\bar{z}(\lambda)$ and they are dimensionless quantities.

All the multi chromatic wavelengths are expected to lie within the area of chromaticity diagram. The luminescent intensity of the emission spectral measurements has been characterized using the CIE 1931 chromaticity diagram. Figure 4 shows the CIE chromaticity diagram for the prepared glasses (λ_{ext} = 385 nm) and the values of the *x*, *y* color coordinates are found to be (0.34, 0.38), (0.34, 0.39) and (0.34, 0.39) corresponding to the prepared NPABSD5, NPABSD10 and NPABSD15 glasses respectively.

The color temperature of a light source is the temperature of the Planck Ian's black body radiator, whose radiation is the same chromaticity as the light source. The chromaticity of nature and artificial light sources, including day light are not on this locus, hence "Correlated Color Temperature" (CCT) is used to indicate the temperature of the black body whose chromaticity is nearest to that of the light source. The CCT is defined as “the color temperature corresponding to the point on the planckian locus which is nearest to the point representing the chromaticity of the illuminant considered on an agreed uniform-chromaticity-scale diagram” by CIE.

Decay Analysis

Figure 5 shows the decay profiles of the $^4F_{9/2}$ energy level of the Dy^{3+} ions monitoring an excitation 385 nm and emission at 575 nm. The decay curves exhibited single exponential nature at lower concentrations and turned into the non-exponential nature at a higher concentration of Dy^{3+} ions (Madhukar Reddy *et al*., 2011).

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Discussion

Luminescence Spectra

The luminescence spectra have been recorded by monitoring an excitation at 385 nm as a function of various Dy^{3+} ion concentrations and are shown in Fig. 2. The spectrum contains three intense luminescence bands corresponding to $^4F_{9/2} \rightarrow {}^6H_J$ (J = 15/2, 13/2 and 11/2) transitions located nearly at the same wavelengths. In the blue region at 482 nm ($^4F_{9/2} \rightarrow {}^6H_{15/2}$), yellow region at 575 nm ($^4F_{9/2} \rightarrow {}^6H_{13/2}$) and a feeble band is in red region at 665 nm ($^4F_{9/2} \rightarrow {}^6H_{11/2}$) are observed. From Fig. 2, it also indicates that the luminescence intensity increases along with increase in Dy^{3+} ion concentration (Balakrishna *et al*., 2012; Wanyi *et al*., 2014; Joanna *et al*., 2010; Manpreet *et al*., 2016; Yuhua *et al*., 2010; Jia-Li *et al*., 2012). Among the three emission bands, the band at $^4F_{9/2} \rightarrow {}^6H_{15/2}$ is Magnetic Dipole (MD) transition possessing the most intense, $^4F_{9/2} \rightarrow {}^6H_{13/2}$ transition possessing moderate intensity, which is related to hypersensitive Electric Dipole (ED) transition with $\Delta J = \pm 2$ and $\Delta L = \pm 2$ which has been strongly influenced by the coordination environment [65], on the other hand, the $^4F_{9/2} \rightarrow {}^6H_{15/2}$ transition is a Magnetic Dipole (MD) transition ($\Delta J = 0, \pm 1$ but $0 \rightarrow 0$ forbidden) and less sensitive environment on this yellow emission band is much more pronounced (Dwivedi and Rai, 2009). As shown in energy level diagram of Fig. 3, when the Dy^{3+} ions are excited with 385 nm to the $^4I_{15/2}$ level, due to the small energy gap between the $^4I_{15/2}$ and lower $^4F_{9/2}$ level, the meta-stable $^4F_{9/2}$ level is populated through rapid non-radiative process. Since this level has sufficient energy gap of ≈ 8000 cm^{-1} with respect to the next lower level of $^6F_{3/2}$, it emits blue, yellow and red luminescence as shown in Fig. 2. As can be seen from the inset of Fig. 2, it is clear that the intensity of emission bands increases with increase in concentration of Dy^{3+} ion.

White Light Simulation

The luminescence spectra of the present glasses recorded monitoring an excitation at 385 nm is shown in Fig. 4 and the inset shows the CIE chromaticity diagram for the same. It is observed from figure that, the *x*, *y* color coordinate values are found to lie in the white light region.

The excitation spectra composing of several strong excitation bands have been observed from N-UV to blue region in Fig. 1, which demonstrates that the developed NPABS: xDy^{3+} glasses can be effectively excited by commercial N-UV (350-420 nm) and blue (440-470 nm) LED chips. Based on the emission spectra in Fig. 2, the chromaticity coordinates and the integral intensity ratios of yellow to blue bands (Y/B) are calculated and listed in Table 1, respectively. The corresponding Commission International de I'Eclairage (CIE) 1931 x-y chromaticity diagram are depicted in Fig. 4, where inset shows the enlarged spectrum for color coordinate (x) value from 0.338 to 0.342. For Dy^{3+} ions, the Y/B ratio is of importance because it will determine white light location in the CIE chromaticity diagram. It is interesting that all Y/B ratios retain in the vicinity of unity and vary little with the concentration and excitation wavelength of Dy^{3+} ions, which results in considerable stability of the CIE color coordinates against both concentration and excitation wavelength. The excellent chromaticity coordinates (0.34, 0.39) in the white region are obtained in light point (0.333, 0.333). The CCT values of the prepared glasses are found to be 5577, 5583 and 6420 K corresponding to the NPABSDy5, NPABSDy10 and NPABSDy15 respectively. Further, CCT values for all the prepared glasses are found to be higher than 3935 K of fluorescent tube and exhibit values between 5500 K of the day light 6400 K. It is observed from the tabulated results that the prepared Dy^{3+} doped NPABS glasses can act as excellent cool white light sources and the W-LEDs fabricated by these glasses can be commercially used for public luminescence devices, reading lamps and to illuminate the living rooms. Among the prepared glasses, NPABSDy10 glass possesses chromaticity coordinated nearest to the exact white light region and optimum CCT values and hence suitable for white light emitting sources for various photonic applications (Xin-Yuan *et al*., 2013; Mohan *et al*., 2011; Krishnaiah *et al*., 2013; McCamy, 1992; Ambast *et al*., 2014).

Decay Analysis

From the decay profiles, the experimental lifetime (τ_{exp}) of the $^4F_{9/2}$ transition are found to decrease with the increase of Dy^{3+} ions concentration and the obtained values are 634, 623 and 580 μs corresponding to the NPABSDy5, NPABSDy10 and NPABSDy15 glasses respectively (Zulfiqar Ali Ahamed *et al*., 2013).

This is because of the energy transfer between the acceptor ions (ground state Dy^{3+} ions) and the donor ions (excited state Dy^{3+} ions). The calculated lifetime (τ_{cal}) of these glasses are found to be 861, 813 and 745 μs. The luminescence quantum efficiency (η) is defined as "the ratio of number of photons emitted to the number of photons absorbed" and it is equal to the ratio of the experimental lifetime (τ_{exp}) to the calculated lifetime (τ_R). The percentage of η values are found to be 78, 81 and 68 corresponding to the NPABSDy5, NPABSDy10 and NPABSDy15 glasses

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respectively and the results are presented in Table 2. From the evaluated luminescence quantum efficiencies, it can be found that, the prepared glass host is more useful as laser material.

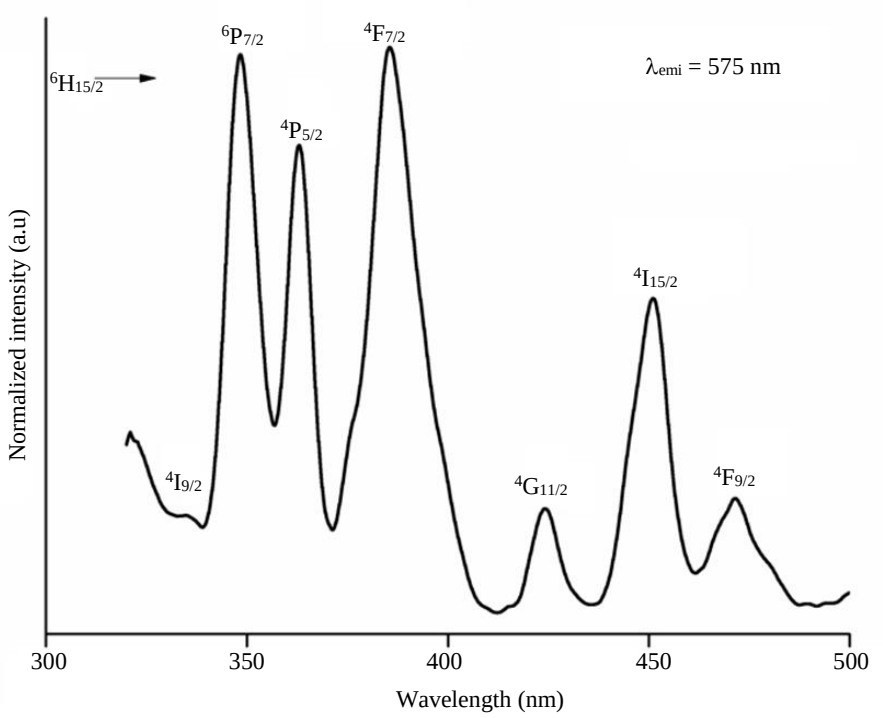

Fig. 1 Excitation spectrum of NPABSDy10 glass measured by monitoring emission at 575 nm

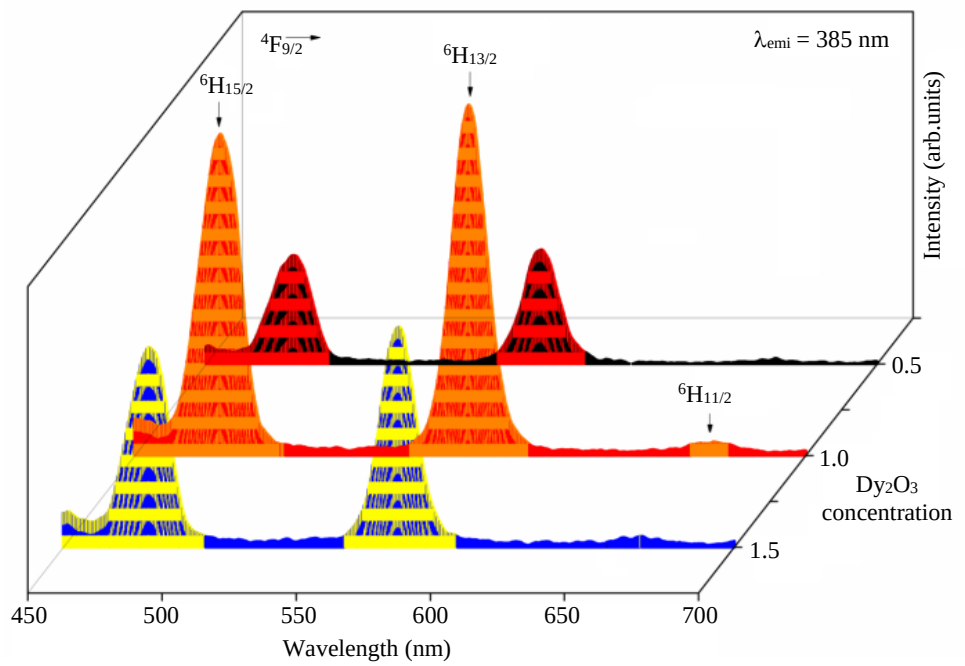

Fig. 2: Emission spectra of Dy^{3+} doped NPABS samples with different concentrations excited by 385 nm. Inset shows the variation of emission intensities for the respective transitions with concentration

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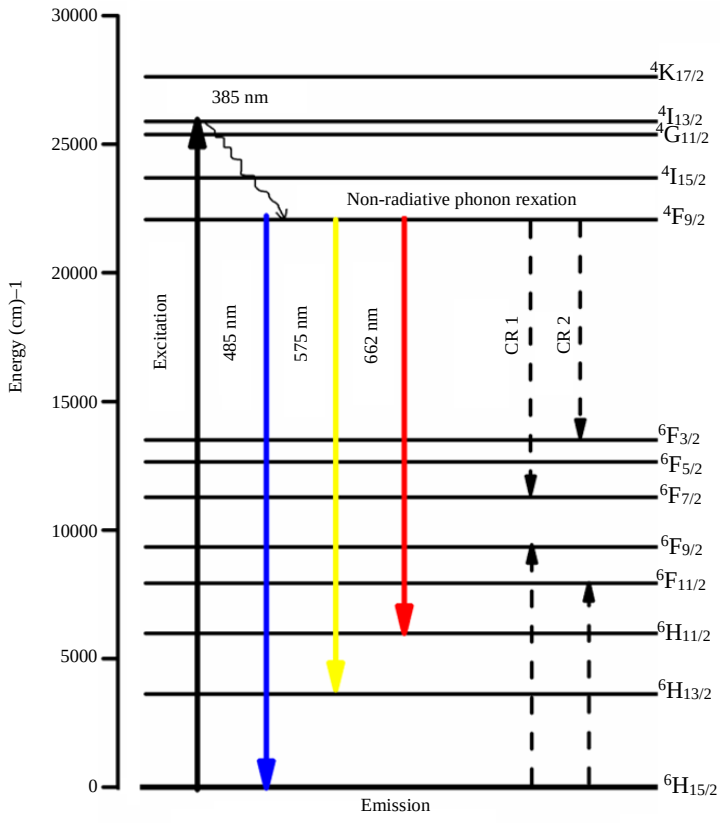

Fig. 3: Partial energy level diagram of Dy^{3+} ions showing possible excitation and emission transitions, Non-Radiative (NR) decays, Cross-Relaxation (CR) channels in the Dy^{3+} doped NPABS glasses

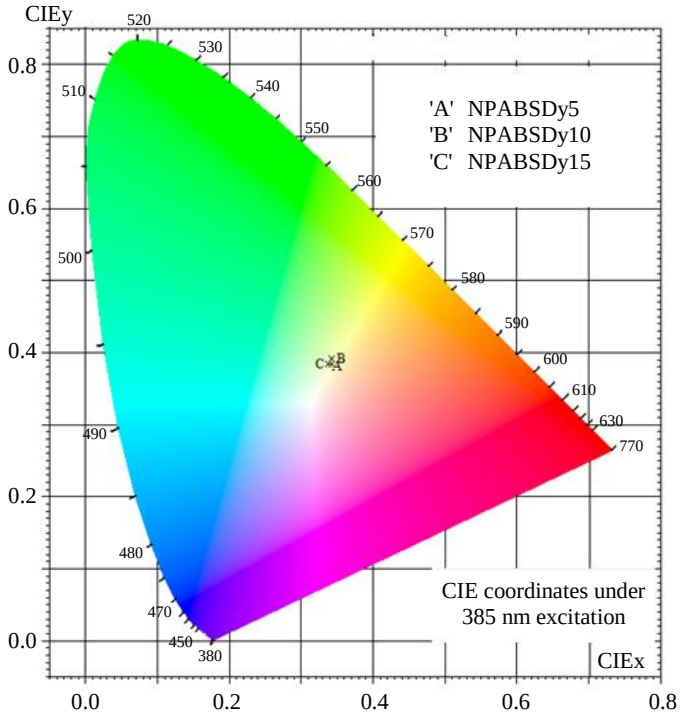

Fig. 4: Chromaticity diagram showing the chromaticity coordinates of Dy^{3+}: NPABS glasses

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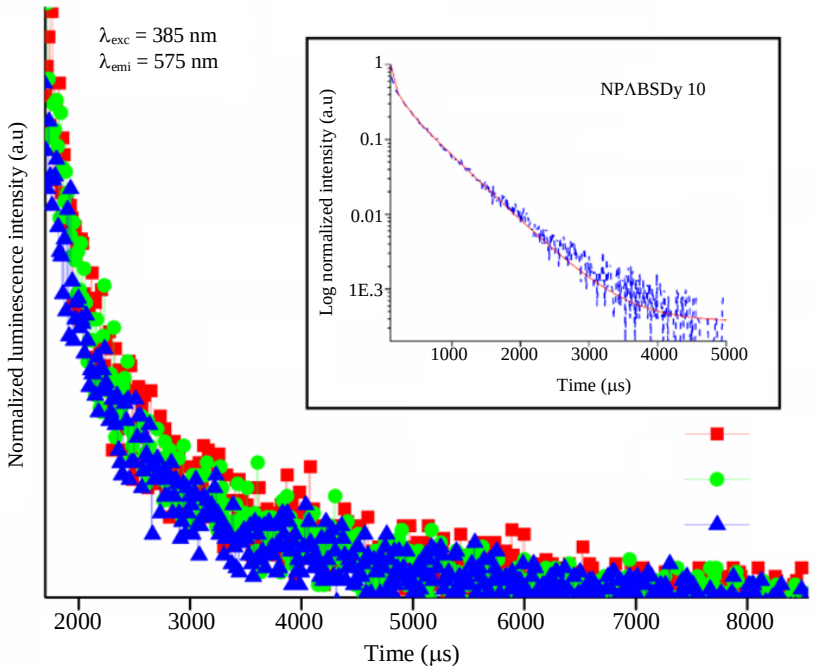

Fig. 5: Decay profile corresponding to the 4F9/2 energy level of the Dy^{3+} doped NPABS glasses monitoring an emission at 575 nm

Table 1: Glass label, yellow to blue intensity (Y/B) ratio, chromaticity coordinates (*x*, *y*) and Correlated Color Temperature (CCT) for various Dy^{3+} glass systems.

		Chromaticity Coordinates			
Sample code	Y/B ratio	x	y	CCT (K)	References
NPABSDy5	1.063	0.34	0.38	5107	[Present]
NPABSDy10	1.092	0.34	0.39	5271	[Present]
NPABSDy15	1.088	0.34	0.39	5362	[Present]
ZnAlBiB	1.18	0.31	0.31	-	Arunkumar *et al*. (2015)
NAPDy	2.9	0.44	0.44	-	Amarnath Reddy *et al*. (2011)
LBODy1.0	1.019	0.35	0.38	-	Dillip *et al*. (2014)

Table 2: The calculated (τ_{cal}, μs) and experimental (τ_{exp}) life time, quantum efficiency (%η) And non-radiative transition rate (W_{NR}, s^{-1}) of the Dy^{3+}doped NPABS glasses

Sample code	τ_R	τ_{exp}	%η	W_{NR}	References
NPABSDy5	814	634	77.88	349	[Present]
NPABSDy10	765	623	81.38	298	[Present]
NPABSDy15	861	580	67.36	563	[Present]

Conclusion

Optical quality Dy_2O_3-doped borosilicate glasses have been prepared by conventional melt quenching technique and characterized through emission and decay rate analysis. The glass samples emit strong blue and yellow lights as well as a weaker red emission by 385 nm excitation. The Y/B ratio strongly depends on the Dy^{3+} ion concentration. Y/B intensity ratios of visible emissions vary over a wide range indicating the suitability of the material for color display devices. The decay rates exhibited single exponential for lower concentrations and turns into non exponential for higher concentrations. The non-exponential decay rates are well-fitted to I-H model for S = 6 and analyzed the interaction among the Dy^{3+} ions in the present glasses. The lifetime of the $^4F_{9/2}$ level decreased with increase of Dy^{3+} ion concentration. The CIE color chromaticity coordinates (0.34, 0.39) of the prepared glasses have been tuned for white light emission by varying the Dy^{3+} ions to be close to the standard values (0.33, 0.33). The CIE chromaticity coordinates lie in the white light region for all the concentrations. Among all the chromaticity coordinates 0.5, 1.0 and 1.5 mol % Dy_2O_3-doped glasses appeared very nearer and comparable to commercial pc-LED suggesting their suitability for WLED applications as well its CCT value of 5597 K closer to the day light, which can be a promising host for blue GaN-based W-LED applications. Among the prepared glasses, NPABSDy10 glass possess higher η value for the $^6H_{13/2}$ emission transition suggesting its suitability for lasers and fabrication of optical amplifiers.

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Author's Contributions

All authors equally contributed in this work.

Ethics

No Sir, it's my own work. I assure no ethical problems are arising after the publication of this manuscript (Corresponding author: Dr. Karumuri Vijaya Babu)

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