Green fluorescence of terbium ions in lithium fluoroborate glasses for fibre lasers and display devices

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Abstract. In this paper, for the first time, the visible fluorescence properties, resonance energy transfer mechanism responsible for non-radiative decay rates of $^5D_4 \rightarrow ^7F_5$ transition and also quenching of fluorescence intensity of the $^5D_3 \rightarrow ^7F_5$ transition of various concentrations of Tb^{3+} ions in LBZLFB glasses are reported. Optical absorption, fluorescence spectra and quantum efficiencies are measured and analysed. Green fluorescence related to $^5D_4 \rightarrow ^7F_5$ (548 nm) transition is registered under excitation of 378 nm of Tb^{3+} ions. Based on excitation and fluorescence measurements, several spectroscopic parameters for Tb^{3+} ions are examined as a function of concentration by Judd–Ofelt theory to judge the suitability of studied glasses for display devices and fibre lasers.

Keywords. Glasses; terbium ion; oscillator strengths; fluorescence; lifetimes; fibre lasers.

1. Introduction

Today glasses are most favourable engineering materials for abundant applications due to the wide ability of property altering by compositional modifications. The considerable examination of glass science to achieve required properties is due to innovative role of glasses in optical communication and solid-state lasers attracted remarkable attention in the last few decades. Host phonon energy is one of such intrinsic property of glass which plays key role in favouring the luminescence performance of active ions. Low phonon energy glasses doped with rare earth (RE) ions are attractive materials for photonic devices. A great attention of research has been focused on RE ions-doped glasses owing to their wide ranging applications in laser and optical communication devices. Fluoride glasses in comparison with typical silica glasses used in fibre optic telecommunication are characterized by low attenuation of radiation significantly wider range of wavelength (2000–7000 nm) [1]. The oxyfluoroborate glasses are promising candidates for laser host materials due to their high transparency, chemical durability and thermal stability and large RE ion solubility. The intensities of the transitions for RE ions are evaluated using the procedure based on the Judd–Ofelt (J–O) theory [2,3]. A comparable

knowledge on the fundamental data that includes optical efficiency, transition positions, transition probabilities, radiative and non-radiative decay rates, branching ratios, etc. to optimize the best configuration of the ion-host to improve the laser efficiency of a specific electronic transition. The host-dependent J–O parameters have been used to obtain above-mentioned radiative properties for the excited states and are very essential to estimate/design optical devices such as lasers, colour displays, upconverters, optical fibres and optical amplifiers and so on.

Glasses as laser hosts have advantages to form fibres more easily than single crystals and RE ions-doped glass fibre lasers have been applied in optical communication, optical fibre sensing, displays, medical treatment, etc. However, in the last decades, green fibre lasers was mainly achieved by using Er^{3+} -doped glass fibres by upconversion luminescence process [4,5]. The green-pulsed laser operation based on $^5D_4 \rightarrow ^7F_5$ transition of Tb^{3+} was first reported in 1967 [6]. Recently, Yamashita and Ohisi [8] reported green laser from Tb^{3+} ions-doped fluoride glass fibres pumped by 488 nm laser diode [7,8]. In this investigation, we select borate glass as the host for Tb^{3+} ions for display devices and to realize green fibre lasers. Absorption spectra and concentration dependent emission spectra, quantum efficiencies of the Tb^{3+} ions-doped borate glasses were measured. The spectroscopic properties of borate glasses containing high Tb^{3+} ion concentration may make them promising as materials for green fibre laser and solid-state materials.

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2. Experimental

The LBZLFB glasses used in this work were prepared with the composition in mol% 20PbO + 5BaO + 5ZnO + 10LiF + (60 - x) B₂O₃ + x Tb₂O₃ (where x = 0.1, 0.5, 1.0, 1.5 and 2.0 mol%). Each 10 g mixtures of reagent grade (99.9%), Pb₃O₄, BaCO₃, ZnO, LiF, H₃BO₃ and Tb₂O₃ chemicals were heated at 950°C for 1 h in an electric furnace using porcelain crucible. The melt was poured in a pre-heated brass cast and pressed with another brass plate to get a flat glass of 0.3 cm thick and then annealed at 300°C for 5 h to remove thermal strains. The obtained glasses were polished for measuring their physical and optical properties. Densities of glass samples were measured at room temperature (RT) with distilled water as the immersion liquid by the conventional Archimedes's principle. Abbe's refractometer was used to obtain the refractive indices for all the glass samples at sodium wavelength using 1-bromonaphthalene as the contact liquid. The various measured and calculated physical properties of the 1.0 mol% Tb³⁺-doped LBZLFB glass are presented in table 1. The absorption spectra were recorded using a double beam Perkin Elmer Lambda 950 spectrophotometer in the wavelength range of 250–2500 nm. The excitation, emission and lifetime measurements were carried out using Jobin Yvon Fluorolog-3 spectrofluorometer with xenon flash lamp as source. All the measurements were carried out at RT.

3. Results and discussion

3.1 Optical absorption spectra

Figures 1 and 2 represent the RT absorption spectra of 1.0 mol% Tb³⁺ ions-doped LBZLFB glass in the visible and near-infrared region, respectively. The absorption spectra consist a total of four bands at 486, 1891, 1954 and 2177 nm corresponding to the ⁷F₆ → ⁵D₄, ⁷F_J = 1–3 transitions, respectively, and are assigned according to the energy level

positions of Tb³⁺ ions in the literature [9]. The spectral intensities for the observed bands in the absorption spectra are expressed in terms of oscillator strengths and are analysed with the help of Judd–Ofelt (J–O) theory using the procedure available in the literature [10–13]. The experimental oscillator strength (f_{exp}) and calculated oscillator strength (f_{cal}) values along with assignment of absorption bands are tabulated in table 2. The goodness of the fit is obtained in terms of root mean square deviation (δ_{rms}). As shown in the table, the obtained δ_{rms} deviation of $\pm 0.16 \times 10^{-6}$ is well consistent with the f_{exp} and f_{cal} values and also the validity of J–O intensity parameters.

In the 1.0 mol% LBZLFB glass, the evaluated set of J–O intensity parameters: $\Omega_2 = 16.34 \times 10^{-20} \text{ cm}^2$, $\Omega_4 = 0.25 \times 10^{-20} \text{ cm}^2$ and $\Omega_6 = 5.74 \times 10^{-20} \text{ cm}^2$ follow the trend $\Omega_2 > \Omega_6 > \Omega_4$. These parameters play an important role

Figure 1. Vis absorption spectrum of 1.0 mol% Tb³⁺-doped LBZLFB glass.

Figure 2. NIR absorption spectrum of 1.0 mol% Tb³⁺-doped LBZLFB glass.

Table 1. Measured and calculated physical properties for 1.0 mol% Tb³⁺-doped LBZLFB glass.

Physical quantities	LBZLFB
Sample thickness (cm)	0.300
Refractive index (n)	1.580
Density (g cm ⁻³)	4.960
Concentration (mol l ⁻¹)	0.256
Concentration (ions cm ⁻³ × 10 ²⁰)	1.542
Average molecular weight (g)	193.770
Dielectric constant (ϵ)	2.496
Molar volume, V_m (cm ³ mol ⁻¹)	39.060
Glass molar refractivity (cm ⁻³)	12.990
Electronic polarizability, α_e (× 10 ⁻²⁴ cm ³)	5.150
Reflection losses, R (%)	5.053
Polaron radius, r_p (Å)	7.514
Inter ionic distance, r_i (Å)	18.648
Field strength, F (× 10 ¹⁴ cm ⁻²)	5.300

to investigate the coordination environment as well as bonding in the vicinity of RE ions. Ω_2 parameter is sensitive to the local environment and often attributed to the degree of covalence between the dopant ion and surrounding ligand, also it depends mainly on the asymmetry of the coordination structure and bonding nature [14,15]. The higher magnitude of Ω_2 parameter is an indication of higher covalency of the dopant ion–ligand bonds and lower symmetry around RE ion. Moreover, the Ω_4 and Ω_6 parameters are less sensitive to the medium and related to the bulk properties such as viscosity and rigidity. The J–O intensity parameters of Tb^{3+} ions doped different hosts are listed in table 3. From the table, it is clear that the Ω_2 parameter varies significantly in different hosts. The Ω_2 parameter of 1.0 mol% Tb^{3+} ions-doped LBZLFB glass is found to be higher to those reported in the literature. When compared to the other hosts [13,16–20], the sites occupied by Tb^{3+} ions in LBZLFB glasses possess lower symmetry and greater degree of covalence for Tb–O bond. On the observation of intensity parameters, better luminescence properties may be expected in the present glasses than the reported glasses [13,16–20].

3.2 Analysis of emission intensity

The excitation spectrum shown in figure 3 was measured by monitoring the green emission (548 nm) of Tb^{3+} ion. The observed sharp bands at 353, 370, 378 and 486 nm corresponding to the $^7\text{F}_6 \rightarrow ^5\text{L}_9$, $^5\text{L}_{10}$, $^5\text{G}_6$ and $^5\text{D}_4$ transitions, respectively. These bands are due to the $4f-4f$ transitions of Tb^{3+} ions [21]. To produce stronger emission and to study

Table 2. Experimental and calculated oscillator strengths ($\times 10^{-6}$) for 1.0 mol% Tb^{3+} -doped LBZLFB glass.

Transition ($^7\text{F}_6 \rightarrow$)	Energy (cm^{-1})	f_{exp}	f_{cal}
$^5\text{D}_4$	20661	0.10	0.10
$^7\text{F}_1$	5291	1.59	1.35
$^7\text{F}_2$	5118	1.42	1.64
$^7\text{F}_3$	4545	1.35	1.31
$\delta_{\text{rms}} = \pm 0.16 \times 10^{-6}$			

Table 3. Comparison of J–O intensity parameters ($\times 10^{-20} \text{ cm}^2$) and their trends for Tb^{3+} ions in LBZLFB glass with different glass hosts.

Glass system	Ω_2	Ω_4	Ω_6	Trend
LBZLFB (present work)	16.34	0.25	5.74	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{LiNO}_3\text{--KNO}_3$ [20]	15.00	2.86	5.24	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{PbO--CaO--ZnO--NaF--B}_2\text{O}_3$ [13]	14.54	0.17	4.09	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{BaF}_2\text{--ThF}_4\text{--ZnF}_2\text{--LiF}$ [18]	13.40	2.15	4.90	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{KF--B}_2\text{O}_3$ [19]	11.92	0.26	2.16	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{PbF}_2\text{--B}_2\text{O}_3\text{--Al}_2\text{O}_3$ [16]	8.20	1.95	3.31	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{CaO--B}_2\text{O}_3$ [17]	6.95	1.28	2.91	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{Na}_2\text{O--P}_2\text{O}_5\text{--Al}_2\text{O}_3$ [17]	6.80	1.11	2.27	$\Omega_2 > \Omega_6 > \Omega_4$
$\text{Li}_2\text{O--B}_2\text{O}_3$ [17]	5.56	1.33	3.22	$\Omega_2 > \Omega_6 > \Omega_4$

the luminescence behaviour of Tb^{3+} ions in the present glass host, the excitation wavelength of 378 nm is used to record emission spectra. The emission spectra recorded for different concentrations of Tb^{3+} ions-doped glasses have similar profiles except in intensity, shown in figure 4. The emission spectra comprise of several peaks both from $^5\text{D}_3$ and $^5\text{D}_4$ emission states. The luminescence below 480 nm originates from $^5\text{D}_3$ excited state, and above 480 nm originates from $^5\text{D}_4$ excited state. The peaks originating from the $^5\text{D}_3$ state are located at 418, 441 and 458 nm assigned to the $^5\text{D}_3 \rightarrow ^7\text{F}_5$, $^7\text{F}_4$ and $^7\text{F}_3$ transitions, respectively, and while the peaks originating from the $^5\text{D}_4$ metastable state are located at 491, 548, 589 and 624 nm assigned to the $^5\text{D}_4 \rightarrow ^7\text{F}_6$, $^7\text{F}_5$, $^7\text{F}_4$ and $^7\text{F}_3$ transitions, respectively. Among these transitions, the bands corresponding to the $^5\text{D}_3 \rightarrow ^7\text{F}_5$, $^7\text{F}_4$ and $^7\text{F}_3$ and $^5\text{D}_4 \rightarrow ^7\text{F}_4$ and $^7\text{F}_3$ transitions are found to be very weak. The transitions related to the $^5\text{D}_4 \rightarrow ^7\text{F}_6$ and $^7\text{F}_5$ transitions

Figure 3. Excitation spectrum of 1.0 mol% Tb^{3+} -doped LBZLFB glass.

Figure 4. Fluorescence spectra of $^5\text{D}_3$ and $^5\text{D}_4$ energy levels for different concentrations of Tb^{3+} -doped LBZLFB glasses.

Table 4. Radiative properties of $^5D_4 \rightarrow ^7F_5$ emission transition in 1.0 mol% Tb^{3+} -doped LBZLFB glass.

Radiative properties	$^5D_4 \rightarrow$			
	7F_6	7F_5	7F_4	7F_3
Peak emission wavelength (λ_p , nm)	491	548	589	624
Effective linewidth ($\Delta\lambda_p$, nm)	13.26	11.83	10.92	8.16
Radiative transition probability (A_R , s^{-1})	58	440	19	50
Radiative branching ratio (β_R)	0.1	0.78	0.03	0.09
Experimental branching ratio (β_m)	0.32	0.60	0.06	0.02
Stimulated emission cross-section ($\sigma_e \times 10^{-22}$ cm^2)	1.35	17.83	1.11	4.94
Gain bandwidth ($\sigma_e \times \Delta\lambda_p$, 10^{-28} cm^3)	1.79	21.1	1.2	4.03
Optical gain parameter ($\sigma_e \times \tau_m$, 10^{-25} cm^2 s)	2.4	31.4	1.95	8.7
Radiative lifetime (τ_R , μs)			1.76	

Table 5. Variation of lifetime (τ_m , ms), quantum efficiency (η , %) and energy transfer rate (W_{NR} , s^{-1}) with respect to concentration (mol%) of Tb^{3+} ions in LBZLFB glasses.

Concentration	Lifetime	η	W_{NR}
0.1	1.69	96	73
0.5	1.44	82	126
1.0	1.37	78	162
1.5	1.16	66	294
2.0	1.02	58	412

3.3 Radiative properties

The J–O intensity parameters (Ω_λ) have been used to compute the radiative properties of emission characteristics for $^5D_4 \rightarrow ^7F_5$ transition of the Tb^{3+} -doped LBZLFB glasses and presented in table 4. The radiative properties of Tb^{3+} ions depend on the number of factors such as property of network former and modifier of glass. The branching ratio of the fluorescent transitions characterizes the lasing power of the potential laser transition and it characterizes the possibility of attaining stimulated emission of any specific transition and is significant parameter for laser designer. An efficient laser transition has a branching ratio ≥ 0.50 . Among the observed emission transitions of 1.0 mol% Tb^{3+} -doped LBZLFB glass the experimental branching ratio of $^5D_4 \rightarrow ^7F_5$ transition is found to be 0.6 and this transition may therefore be considered as an effective laser transition suggesting its suitability for green colour display devices and to design a fibre laser. To investigate the possibility of laser emission through the $^5D_4 \rightarrow ^7F_5$ transition, the stimulated emission cross section (σ_e), gain band width parameter ($\sigma_e \times \Delta\lambda_p$) are obtained from the full-width at half maximum ($\Delta\lambda_p$) and optical gain parameter ($\sigma_e \times \tau_m$) are determined for all the intense transitions originating from 5D_4 metastable state and listed in table 5. From the table, it is observed that the values of σ_e , $\sigma_e \times \Delta\lambda_p$ are maximum for $^5D_4 \rightarrow ^7F_5$ transition. Based on the reasonably high branching ratio and laser characteristic parameters, when compared with the

Figure 7. Decay curves of $^5D_4 \rightarrow ^7F_5$ emission transition in Tb^{3+} -doped LBZLFB glasses.

other hosts [16,19,25,26], we suggest that 1.0 mol% Tb^{3+} -doped LBZLFB glass has the potential candidate to produce intense green laser at 548 nm through the $^5D_4 \rightarrow ^7F_5$ transition.

3.4 Decay curve analysis

The decay curves of the 5D_4 emission level of Tb^{3+} ions in LBZLFB glasses were recorded by monitoring the emission and excitation wavelength at 548 and 378 nm, respectively, and shown in figure 7. The analysis of fluorescence decay provides information regarding the energy transfer mechanism and fluorescence quenching of Tb^{3+} ions in LBZLFB glass host. At lower Tb^{3+} concentrations (≤ 0.5 mol%), the decay curves are well fitted to a single exponential function. However, at higher Tb^{3+} concentrations (≥ 1.0 mol%), the decay profiles exhibited non-exponential behaviour due to resonant energy transfer among the excited Tb^{3+} ions through [5D_4 , 7F_6] $\rightarrow$ [7F_6 , 5D_4] transitions (figure 5). The experimental lifetime (τ_m) of 5D_4 level determined by taking

Figure 8. CIE chromaticity diagram of 1.0 mol% Tb^{3+} -doped LBZLFB glass.

the first e-folding times of the decay curves of Tb^{3+} ions are 1.69, 1.44, 1.37, 1.16 and 1.02 ms for 0.1, 0.5, 1.0, 1.5 and 2.0 mol% of Tb^{3+} ion concentration, respectively. However, the radiative decay (τ_R) is calculated as 1.76 ms by applying the J-O theory. The difference between τ_m and τ_R may be due to the non-radiative contributions from the $^5\text{D}_4$ level of Tb^{3+} ions in LBZLFB glasses.

For photonic applications like fibre lasers and planar waveguides, the fluorescence quantum efficiency (η) is an important parameter controlling the performance of the material. For 1.0 mol% Tb^{3+} ions-doped LBZLFB glass, the value of quantum efficiency $\eta = \tau_m/\tau_R \times 100\%$ is found to be 78%. It is found that higher than that of 1.0 mol% Tb^{3+} -doped LCZSFB [13] glass. Further, the experimental decay time can be written as

$$\frac{1}{\tau_m} = \frac{1}{\tau_R} + W_{\text{MPR}} + W_{\text{ET}},$$

where W_{MPR} and W_{ET} represent the multi phonon relaxation rate and the rate of energy transfer, respectively. The energy gap of $\sim 14,750 \text{ cm}^{-1}$ between the $^5\text{D}_4$ emission state and the $^7\text{F}_0$ lower lying state, suppress the multi phonon relaxation rates in the LBZLFB glass system. Thus, the energy transfer is mainly due to resonant energy transfer and the rate of energy transfer

$$W_{\text{ET}} = \frac{1}{\tau_m} - \frac{1}{\tau_R} \text{ s}^{-1}.$$

For 1.0 mol% Tb^{3+} ions-doped LBZLFB glass, the energy transfer rate W_{ET} is found to be 162 s^{-1} and is found lower than those reported for LCZSFB glass [13].

3.5 Chromaticity coordinates

To recognize the emission colour of the prepared glasses, the CIE 1931 chromaticity coordinates were calculated by

using CIE calculation software. A typical CIE values of 1.0 mol% Tb^{3+} ions-doped LBZLFB glasses are shown in CIE diagram, figure 8. As indicated in the figure, the CIE values (0.303, 0.350) are located in near white light region. However, the emission of Tb^{3+} -doped LBZLFB glasses are showing predominant at 550 nm (green colour) as represented in figure 4. This suggests that the obtained values are slightly deviated from the pure white colour source chromaticity values (0.33, 0.33).

4. Conclusions

In summary, we reported transparent LBZLFB glasses with different Tb^{3+} concentrations, prepared by melt quenching method. A quenching in fluorescence intensity of $^5\text{D}_3$ state is observed beyond 1.0 mol% Tb^{3+} and is attributed to the energy transfer through cross-relaxation of $[^5\text{D}_3, ^7\text{F}_6] \rightarrow [^5\text{D}_4, ^7\text{F}_0]$ transitions. The fluorescence intensity of $^5\text{D}_4$ emission state is increased with increase of Tb^{3+} concentration due to fast non-radiative decay from $^5\text{D}_3$ to $^5\text{D}_4$ levels. The decrease of experimental decay times with increase of Tb^{3+} concentration is attributed to the resonant energy transfer through $[^5\text{D}_4, ^7\text{F}_6] \rightarrow [^7\text{F}_6, ^5\text{D}_4]$ emission channels. The radiative properties of 1.0 mol% Tb^{3+} ions-doped LBZLFB glasses strongly suggest their utility in the development of green colour display devices and fibre lasers.

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