

Communication

Green- and Blue-Emitting Tb³⁺-Activated Linde Type A Zeolite-Derived Boro-Aluminosilicate Glass for Deep UV Detection/Imaging

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Abstract: Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples with a composition of xTb₂O₃-68(Na₂O-Al₂O₃-SiO₂)–32B₂O₃ (x = 0.2, 1.0 and 10 extra wt%) were prepared using the melt-quenching method. The emission spectra recorded upon ultraviolet (UV) excitation with two different wavelengths of 193 and 378 nm showed blue light (⁵D₃ to ⁷F_{J=6,5,4} and ⁵D₄ to ⁷F₆ transitions of Tb³⁺) and green light (⁵D₄ to ⁷F₅ transition of Tb³⁺) emissions with comparable intensities up to a Tb³⁺ concentration of 10 extra wt%. Of note, the mean decay times of the green luminescence of the glass samples were relatively fast (<20 µs). The synthesized glass has potential in applications concerning UV imaging, UV detection, and plasma display panels.

Keywords: boro-aluminosilicate glasses; luminescence; Tb³⁺; phosphor; UV detection and imaging



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1. Introduction

There is ongoing research on the luminescence properties of rare earth-doped glasses and ceramics [1,2] potentially suitable for applications concerning lighting [3], displays [4,5], X-ray scintillation [6], increasing the efficiency of solar cells [7,8], and UV detection and imaging [9]. Emission in such materials is the result of optical transitions attributed to energy levels of 4f-electrons of rare earth ions (e.g., 4f⁸ (for Tb³⁺), 4f⁷ (for Gd³⁺), 4f⁶ (for Eu^{3+}), and $4f^{12}$ (for Tm^{3+})) [10]. Thus, for the case of Tb^{3+} -doped materials, the emission is due to transitions from ⁵D₃ (violet-blue light) and ⁵D₄ (green-orange light) levels to integer multiplets of ⁷F_I of Tb³⁺ ions [11]. Additionally, Tb³⁺ is an important dopant in commercial phosphor LaPO₄:Ce³⁺, Tb³⁺ [12,13]. Nevertheless, in practice, the emission spectra of Tb³⁺-doped materials are often dominated by a characteristic green light (543 nm; ${}^{5}D_{4}$ to $^{7}F_{5}$ transition) due to a non-radiative relaxation from the $^{5}D_{3}$ level to the $^{5}D_{4}$ level via crossrelaxation to a neighbor Tb^{3+} [11,14–16]. In fact, examples of Tb^{3+} -doped glasses showing ${}^{5}D_{3}$ -originated blue emission with intensities comparable to those of the green emission are limited to glasses with low contents of Tb^{3+} (typically no more than 1 mol%) [14,15,17–19]. The green luminescence decay time of Tb³⁺-doped glasses is typically on the order of ms [16,20–26], which normally does not satisfy the fast response time required for some applications, e.g., scintillation detectors [27] and plasma display panels [28,29]. In fact, examples of Tb^{3+} -doped glasses with decay times on the order of μs are rare [29].

Different host lattices have been used for Tb³⁺-activated glasses, e.g., aluminates [14,19,21,26,30], phosphates [16,18,22,23], and germanates [24,25,31]. Linde Type A (LTA) zeolites are synthetic aluminosilicates with a porous and periodic structure with the composition of Na₁₂(AlSi)₁₂O₄₈·27H₂O, which can adopt an amorphous structure via thermal treatment at high temperatures [32]. To our knowledge, only a few studies have examined the luminescence properties of rare earth-activated LTA zeolite-derived ceramics/glasses, which



have included ions of Tb^{3+} [20], Dy^{3+} (with or without Ag^+) [33], and Eu^{2+} [20,34]. In the study that used Tb^{3+} as the dopant [20], the sample was prepared by ion exchanging Tb^{3+} for Na^+ in the LTA zeolite in deionized water, followed by applying the melt quenching method. The sample was green-emitting and showed a green luminescence decay time of 2.398 ms. Thus, in the present paper, we report on the preparation, physical characterization, and luminescence study of green- and blue-emitting Tb^{3+} -doped LTA zeolite-derived boro-aluminosilicate glasses that show fast green luminescence decay times on the order of μ s.

2. Experimental Processes

2.1. Sample Preparation

The Tb³⁺-doped glass samples were prepared via the melt quenching method using LTA zeolites (4A, A. R, Tosoh, Tokyo, Japan; Na₁₂(AlSi)₁₂O₄₈·27H₂O), boron oxide (B₂O₃, 98%, Shanghai Aladdin Biochemical Technology Co. Ltd., Shanghai, China), and Tb₄O₇ (Shanghai Diyang Chemicals Co., Ltd., Shanghai, China). The amounts of LTA zeolite and boron oxide were fixed at 34 g (68 wt%) and 16 g (32 wt%), respectively. Thus, to 50 g mixtures of LTA zeolite and boron oxide, Tb₄O₇ was added in amounts of 0.1 g (extra 0.2 wt%; sample #1), 0.5 g (extra 1.0 wt%; sample #2), and 5 g (extra 10 wt%; sample #3). The powder mixtures were placed in an agate mortar and mixed homogeneously. Heating the mixture to 1200 °C using a high-temperature muffle furnace [35], the Tb³⁺ ions diffuse and evenly distribute within the LTA molecular sieve. Subsequently, the thoroughly melted liquid is quenched by pouring it into water, thus obtaining colorless transparent glass with the nominal composition xTb₂O₃-68(Na₂O-Al₂O₃-SiO₂)-32B₂O₃ (x = 0.2, 1.0 and 10 extra wt%). We have also illustrated a brief description of the preparation process [36–39] of LTA:Tb³⁺, as shown in Figure 1.



Figure 1. Schematic diagram of the preparation process of LTA:Tb³⁺ glass samples.

2.2. Characterization

The structures of all of the obtained samples were analyzed with X-ray diffraction (XRD) (Rigaku, Model Mini Flex 600, Tokyo, Japan), using Cu K_{α} irradiation (λ = 1.5418 Å) operated at 40 kV, 15 mA. The morphology of the fracture surfaces of the samples was observed using a scanning electron microscope (SEM), while energy dispersive X-ray spectroscopy (EDX) was performed in the SEM (Verios 5 UC, Eindhoven, The Netherlands). The optical transmission spectra were recorded with an ultraviolet–visible–NIR spectrophotometer (PerkinElmer, LAMBDA 1050, Waltham, MA, USA). The steady photoluminescence (PL) and photoluminescence excitation (PLE) spectra and the dynamic emission decay curves were recorded using a fluorescence spectrophotometer (Edinburgh Instruments, FLS-1000, Livingston, UK).

3. Results and Discussion

The SEM images did not show pores in the samples. The results of the experimental determination of the wt% of the elements in the samples using EDX are presented in Table 1. The values in Table 1 are the average values of two measurements. The inconsistencies between the nominal and experimental compositions can be explained by (i) the uncertainty in the determination of wt% of the elements (18–25%) dictated by the instrumentation used, and (ii) the fact that the glass matrix consisted of light elements such as B, Al, Si, Na, and

O, which could be evaporated from the surface, e.g., B could have been undetected due to being very light.

	Nominal (wt%)						Experimental (wt%)						
	Na	Al	Si	0	В	Tb	Na	Al	Si	0	В	Tb	Y
#1	8.5	10.0	10.4	58.7	9.8	0.2	6.2	15.1	25.3	47.9	0	2.4	3.2
#2	8.5	10.0	10.3	58.3	9.8	0.9	6.8	16.95	21.2	45.1	0	3.5	6.5
#3	7.8	9.1	9.5	54.8	9.0	7.7	10.1	12.65	13.8	45.1	0	6.7	11.8

Table 1. Comparison of nominal and experimental compositions obtained from EDX analysis.

The XRD analysis of samples #1, #2, and #3 did not show crystalline peaks, thus confirming the glass nature of the samples (Figure 2). The transmission spectra of samples #1, #2, and #3, presented in Figure 3, showed that the visible light transmittance was beyond 85%. Also, photos of these samples were colorless and transparent (Figure 4). The transmittance of a standard sample showed absorption peaks at 378 nm and 485 nm, which were attributed to ${}^{7}F_{6}$ to ${}^{5}D_{3}$ and ${}^{7}F_{6}$ to ${}^{5}D_{4}$ transitions of Tb³⁺, respectively [30,31]. The high transparency of the fabricated LTA zeolite glass samples in the visible light range and the low doping concentration of Tb³⁺ could have resulted in overshadowing or overlapping of the absorption peak of Tb³⁺ by those of the LTA base material in the absorption spectra. Therefore, the transmittance spectra of the Tb³⁺:LTA material did not show a distinct absorption peak for Tb³⁺, and there was no significant correlation with the doping amount of Tb³⁺. The peaks observed and the corresponding transitions in the PLE spectra ($\lambda_{em} = 543$ nm) were 304 nm (${}^{7}F_{6}$ to ${}^{5}H_{6}$), 318 nm (${}^{7}F_{6}$ to ${}^{5}D_{4}$), 369 nm (${}^{7}F_{6}$ to ${}^{5}D_{2}$), 378 nm (${}^{7}F_{6}$ to ${}^{5}D_{3}$), and 485 nm (${}^{7}F_{6}$ to ${}^{5}D_{4}$) (Figure 5) [30,31].



Figure 2. XRD analysis of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples.



Figure 3. Transmission spectra of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples.



Figure 4. Photos of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples (The bottom font is the name of our laboratory).

The PL of the samples was examined by measuring their emission spectra upon excitation with two ultraviolet (UV) light excitations, one with a wavelength of $\lambda_{exc} = 378$ nm, which lay in the ultraviolet A (UV-A)/near-ultraviolet (N-UV) regions, and another with a wavelength of λ_{exc} = 193 nm (a laser light), which lay in the ultraviolet C (UV-C)/farultraviolet (F-UV) regions. Peaks of both ${}^{5}D_{3}$ and ${}^{5}D_{4}$ transitions were present in the emission spectra upon excitation with UV-A/N-UV light, which were located at 418 nm $({}^{5}D_{3} \text{ to } {}^{7}F_{5})$, 440 nm $({}^{5}D_{3} \text{ to } {}^{7}F_{4})$, 487 nm $({}^{5}D_{4} \text{ to } {}^{7}F_{6})$, 543 nm $({}^{5}D_{4} \text{ to } {}^{7}F_{5})$, 586 nm $({}^{5}D_{4} \text{ to }$ $^{7}F_{4}$), and 621 nm ($^{5}D_{4}$ to $^{7}F_{3}$) [16,30,36] (Figure 6). Upon excitation with UV-C/F-UV light, the same peaks from the ${}^{5}D_{4}$ to ${}^{7}F_{I}$ transitions were present, but emission from ${}^{5}D_{4}$ to ${}^{7}F_{I}$ was limited to one peak at 386 nm (${}^{5}D_{4}$ to ${}^{7}F_{6}$) [14] (Figure 7). In all of these transitions, ${}^{5}D_{4}-{}^{7}F_{6}$ (487 nm) and ${}^{5}D_{4}-{}^{7}F_{5}$ (543 nm) are magnetically dipole and parity-forbidden transitions, respectively [20]. Therefore, LTA:Tb³⁺ exhibits a strong emission intensity at 543 nm. In this context, we should point out that a non-radiative relaxation from the ${}^{5}D_{3}$ to the ${}^{5}D_{4}$ levels via cross-relaxation to a neighbor Tb³⁺ is a well-known phenomenon in Tb³⁺ systems [11,14-16]. This process occurs because due to the closely matched energy difference between the ${}^{5}D_{4}$ and ${}^{5}D_{3}$ levels (5800 cm⁻¹) and the ${}^{7}F_{6}$ and ${}^{7}F_{0}$ levels (5700 cm⁻¹), excitation from ⁷F₆ to ⁷F₀ promotes the non-radiative drain from ⁵D₃ to ⁵D₄ of a nearby ion ((${}^{5}D_{3}, {}^{7}F_{6}$) \rightarrow (${}^{5}D_{4}, {}^{7}F_{0}$)) [11,14,15]. Thus, if the dispersion of Tb³⁺ in the matrix is ideal, the ratio of the intensity of the green light to the intensity of the blue light (I_G/I_B) is expected to increase when Tb³⁺ concentration is increased [15]. In our experiments, while I_G/I_B increased when the Tb³⁺ concentration was increased from 1 to 10 extra wt%, it decreased when the Tb^{3+} concentration was increased from 0.1 to 1 extra wt% (Figure 8), possibly because Tb³⁺ pair formation, clustering, and phase separation played a role in our



system [15]. Interestingly, for both excitation wavelengths (λ_{exc} = 193 and 378 nm), I_B and I_G were comparable for the samples studied (Figure 8).

Figure 5. PLE spectra of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples ($\lambda_{em} = 543 \text{ nm}$).



Figure 6. PL spectra of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples ($\lambda_{exc} = 378 \text{ nm}$).



Figure 7. PL spectra of the Tb³⁺-activated LTA zeolite-derived boro-aluminosilicate glass samples ($\lambda_{exc} = 193 \text{ nm}$).



Figure 8. Variation in I_G/I_B with varying concentrations of Tb^{3+} (I_G and I_B were determined by integrating the intensities of the green and blue peaks in the emission spectra, respectively).

A graph of the green PL decay curves is shown in Figure 9 ($\lambda_{exc} = 378 \text{ nm}$; $\lambda_{em} = 543 \text{ nm}$). The curves did not follow single exponential decays, indicating the presence of a radiationless process due to the energy transfer among active Tb³⁺ ions, caused by either cross-relaxation or a cooperative energy transfer to upper levels [21]. We employed two methods for obtaining the luminescence decay times. In the first method, the following theoretical intensity curve with two exponential decay terms was fitted to the experimental data (Figure 9):



$$I = I_0 + A1 \times exp\left(\frac{-t}{\tau_1}\right) + A2 \times exp\left(\frac{-t}{\tau_2}\right)$$
(1)

Figure 9. Green luminescence decay curves of the Tb³⁺-activated LTA zeolite-derived boroaluminosilicate glass samples (each curve was normalized according to the intensity of sample #1). The solid lines represent the results of fitting Equation (1) to the experimental data.

The values for the luminescence decay times (τ_1 and τ_2) obtained using this method were similar for the samples and included τ_1 = 1.21, 1.21, and 1.20 µs and τ_2 = 11.80, 12.28, and 12.00 µs for samples #1, #2, and #3, respectively. In the second method, the mean luminescence decay time (τ_m) was calculated from the following equation [17,22,30,36]

$$\tau_m = \frac{\int I(t)tdt}{\int I(t)dt}$$
(2)

The values obtained for τ_m from this method were 18, 14, and 17 µs for samples #1, #2, and #3, respectively. A decay time on the order of µs is potentially suitable for applications concerning static imaging, UV detection [27], and plasma display panels [28–30]. Importantly, these decay times are substantially shorter than those typically obtained for Tb³⁺-doped glass materials, which are on the order of ms (Table 2). The Tb³⁺-doped glass materials listed in Table 2 were based upon calcium aluminosilicate [14,19,21], fluorophosphate [16], fluoroborate [17], zinc phosphate [16,20], LTA zeolite-derived aluminosilicate [20], zinc phosphate [20], zinc fluorophosphate [26], and strontium fluoroaluminate [30] glasses. Of note, in the only other example of LTA zeolite-derived Tb³⁺-doped glass we are aware of [20], the sample was green-emitting and had a decay time of 2.398 ms.

Tb ³⁺ -Doped Glass Material	Composition	Decay Time (ms)	Refs.
LTA zeolite-derived boro-aluminosilicate	xTb ₂ O ₃ -68(Na ₂ O-Al ₂ O ₃ -SiO ₂)-32B ₂ O ₃ (x = 0.2, 1.0 and 10 extra wt%)	$τ_1 \sim 1.2$ μs, $τ_2 \sim 12$ μs $τ_m$ = 18, 14, 17 μs	Present study
calcium aluminosilicate	$\begin{array}{l} 47.2 CaO-41.3 Al_{2}O-4.1 MgO-7.0 SiO_{2}\text{-}x Tb_{4}O_{7} \\ (x=0.0415) \text{ (in wt\%) and} \\ 33.2 CaO-27.7 Al_{2}O_{3}4.1 MgO-34 SiO_{2}0.5 Tb_{4}O_{7} \\ (\text{in wt\%)} \end{array}$	1.9, 2.3	[14,19]
fluorophosphate	$\begin{array}{l} 44P_2O_5\text{-}17K_2O\text{-}(29-x)\ SrF_2\text{-}9Al_2O_3\text{-}x\ Tb_4O_7\\ (x=0.1\text{-}4)\ (\text{in mol}\%) \end{array}$	2.65–2.94	[16]
fluoroborate	$(50 - x)B_2O_3-20ZnF_2-30BaF_2-xTbF_3$ (x = 0.1-4.0) (in mol%)	3.33–4.57	[17]
zinc phosphate	60P ₂ O ₅ -15ZnO-5Al ₂ O ₃ -10BaO-10PbO-xTb ₂ O ₃ (in mol%) (x = 1.0–5.0) (in wt%)	2.62–2.94	[18]
LTA zeolite-derived aluminosilicate	Na^{+} was ion-exchanged with Tb^{3+} in $Na_{12}Al_{12}Si_{12}O_{48}$	2.398	[19]
calcium aluminosilicate	(100 - x)(58SiO ₂ -23CaO-5Al ₂ O ₃ -4MgO-0NaF in mol%)-xTb ₂ O ₃ (x = 0.25-40 in wt%)	2.32–3.38	[20]
zinc phosphate	$(100.0 - x)Zn(PO_3)_2-xTb_2O_3 (x = 0.6-5.0)$ (in mol%)	2.76-2.97	[22]
zinc fluorophosphate	$\begin{array}{l} 44P_2O_5\text{-}17K_2O\text{-}9Al_2O_3\text{-}(29-x)ZnF_2\text{-}xTb_4O_7\\ (x=0.1\text{-}2.0) \text{ (in mol\%)} \end{array}$	3.12–3.78	[23]
borogermanate	$25B_2O_3-40GeO_2-(35 - x)Gd_2O_3-xTb_2O_3$ (x = 0.25-16) (in mol%)	1.0–1.8	[24]
lead germanate	45PbO-45GeO ₂ -9.5Ga ₂ O ₃ -0.5Tb ₂ O ₃ (in mol%)	1.34	[25]
strontium aluminoborate	$\frac{50B_2O_3-15Al_2O_3-35-xSrO-xTb_4O_7 (x = 0.1-5.0)}{(in mol\%)}$	2.2–2.6	[26]
strontium fluoroaluminate	$70SiO_2$ - $7Al_2O_3$ - $16SrF_2$ - $7GdF_3$ - $4TbF_3$ (in mol%)	~3.1	[30]
borogermanate	$50GeO_2-25B_2O_3-(25 - x)La_2O_3/Gd_2O_3-xTb_2O_3 (x = 1-4)$	1.87–1.97	[31]

Table 2. Green luminescence decay times of various Tb^{3+} -doped glasses under excitation with UV light.

4. Conclusions

Colorless, transparent, Tb³⁺-doped, LTA zeolite-derived boro-aluminosilicate glass samples were prepared using the melt quenching method. The emission spectra obtained using two different excitation wavelengths (λ_{exc} = 193 and 378 nm) showed blue and green light emissions with comparable intensities. The green luminescence decay curves were not single-exponential. The mean decay times were 18, 14, and 17 µs for 0.2, 1.0, and 10 extra wt% of Tb³⁺, respectively, and the computed decay times from fitting a theoretical curve with two decay terms were ~1.2 and ~12 µs (irrespective of the Tb³⁺ concentration). Given their relatively fast green luminescence decay times, these synthesized glass materials have potential for applications concerning static imaging, UV detection, and plasma display panels.

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