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Absorption and emission spectral studies of Sm^{3+}-doped lead tungstate tellurite glasses

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ABSTRACT

The present work reports the effect of concentration on photoluminescence properties of Sm^{3+} ions doped lead tungstate tellurite (LTTSm) glasses by using the absorption, emission and decay measurements. The Judd–Ofelt theory has been used to evaluate the three Judd–Ofelt intensity parameters ($\Omega_{2,4,6}$) and calculated oscillator strengths (f_c). LTTSm glasses exhibited intense reddish–orange emission when excited with 477 nm wavelength. Concentration quenching has been noticed beyond 1.0 mol% of Sm^{3+} ion concentration. The decay curves of $^4G_{5/2}$ level exhibited single exponential behavior for all the concentrations and the measured lifetimes are found to depend strongly on Sm^{3+} concentration. From the emission characteristic parameters of $^4G_{5/2}$ level, it is concluded that the LTTSm glasses could be useful for photonic devices like visible lasers, fluorescent display devices and optical amplifiers.

1. Introduction

In the search of new materials for optical devices such as lasers, fiber amplifiers, upconvertors, light emitting diodes (LED's) and memory devices, several rare-earth doped inorganic glasses with suitable formers and modifiers were prepared by different workers [1–10]. Host glass composition plays very important role on the spectral characteristics of rare-earth ions. The characteristic features of tellurite based glasses like low-melting temperature (~800 °C), slow crystallization rate, excellent transparency in a wide spectral region (0.3–6.0 μm), good mechanical stability, maximum chemical durability, low cut-off phonon energy and high refractive indices make them as the best host materials for obtaining efficient luminescence from trivalent rare-earth ions [8,9]. The presence of PbO, PbF_2 and Bi_2O_3 etc., compounds in conventional glasses such as silicates and borates enhances their luminescence properties [11–16]. Schoonover et al. [17] investigated the emission properties of Sm^{3+} ions in lead borate glasses and found that heavy metal lattices enhance the fluorescence yield of rare-earth ions due to their low phonon energies.

The optical properties of Sm^{3+}-doped glasses have attracted much attention because of their technological applications. Additionally, the Sm^{3+} ions exhibit broad emission bands due to $^4G_{5/2} \rightarrow {}^6H_J$ (J = 5/2, 7/2, 9/2, 11/2) transitions in any host matrix. Moreover, the phonon energies of hosts are not so critical to the reddish–orange emission at ~600 nm, because of the large energy difference of $^4G_{5/2}$ metastable level to its next lower lying level [18,19]. It is also well known that the intensities of emission bands of Sm^{3+} ion in glasses depend on its concentration and glass composition [20]. In order to obtain optimum emission characteristics for device applications, the characteristic features of host as well as concentration dependent studies of Sm^{3+} are essential.

In the present study the spectroscopic and stimulated emission characteristics of Sm^{3+} ions in lead tungstate tellurite (LTT) glasses were investigated by using the absorption, photoluminescence and decay measurements. Judd–Ofelt (J–O) [21,22] intensity parameters Ω_λ (λ = 2, 4, 6) were determined from the experimental oscillator strengths and the spontaneous transition probabilities (A_R) for the observed emission transitions were predicted. The laser characteristic parameters such as the measured branching ratios (β_m), stimulated emission cross-sections (σ_e), gain bandwidths ($\sigma_e \times \Delta\lambda_P$) and optical gain ($\sigma_e \times \tau_R$) parameters were evaluated.

2. Experimental

2.1. Glass preparation

LTT glasses with different concentrations of Sm^{3+} ion were prepared by conventional melt quenching technique using the chemicals PbF_2 (99.9%, Aldrich), WO_3 (99.9% Fulka), TeO_2 (99.9%, Aldrich) and Sm_2O_3 (99.9%, Indian Rare-Earths) with the following molar percentages:

LTTSm01 : $15PbF_2 + 25WO_3 + 59.9TeO_2 + 0.1Sm_2O_3$

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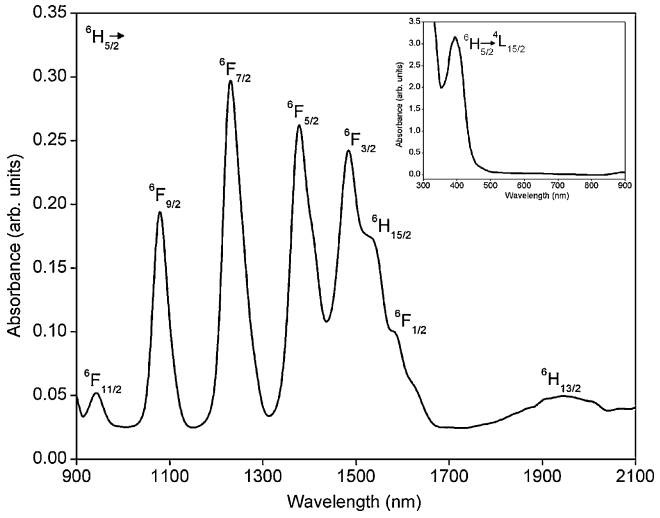

Fig. 1. Optical absorption spectrum recorded for LTTSm10 glass.

$$\text{LTTSm05} : 15PbF_2 + 25WO_3 + 59.5TeO_2 + 0.5Sm_2O_3$$

$$\text{LTTSm10} : 15PbF_2 + 25WO_3 + 59.0TeO_2 + 1.0Sm_2O_3$$

$$\text{LTTSm20} : 15PbF_2 + 25WO_3 + 58.0TeO_2 + 2.0Sm_2O_3$$

About 10 g batches of pre-weighed quantities of chemicals were thoroughly grinded in an agate mortar to homogenize the chemical constituents. The mixture was taken in a platinum crucible and heated at 700–750 °C for 40 min. The melts were poured on a preheated brass moulds and annealed at 300 °C for 12 h to remove thermal strains occurred during sudden quenching. In order to carry out the physical and optical measurements, the glasses were cut into 15 × 10 × 2 mm^3 shape and polished well.

2.2. Physical and optical measurements

The amorphous nature of the glasses was identified by the Siefert x-ray diffractometer using CuKα as the radiation source at 40 kV and 30 mA. The absence of XRD peaks (not shown) indicates the amorphous nature of title glasses. For all the glasses, the densities (d) of ~6.605 g/cm^3 with an estimated error of ±0.001 g/cm^3 were found by the Archimedean's principle using water as immersion liquid. The refractive index (n) of 2.2352 for LTTSm10 glass was determined by Brewster's angle method at He–Ne polarized laser (632.9 nm) with 2 mW power and no considerable variation has been noticed for other glasses. The concentrations (C) of Sm^{3+} ions (in ions/cm^3) were calculated as 0.42, 2.11, 4.09 and 8.19 (×10^{20} ions/cm^3) for LTTSm01, LTTSm05, LTTSm10 and LTTSm20 glasses, respectively. The optical absorption spectrum of Sm^{3+} ions in LTTSm10 glass in the spectral region 300–2100 nm was recorded at room temperature by using Varian Cary 5E UV–vis-NIR double beam spectrophotometer. Excitation, visible photoluminescence and decay measurements were performed by using JOBIN YVON Fluorolog-3 spectrofluorimeter with xenon flash lamp as excitation source.

3. Results and discussion

3.1. Analysis of absorption spectrum – intensity parameters

The optical absorption spectrum of LTTSm10 glass in the spectral region 900–2100 nm is shown in Fig. 1. The inset figure represents the absorption spectrum in the region of 300–900 nm. The absorption bands identified at 394, 942, 1080, 1230, 1377, 1484, 1542, 1583 and 1944 nm are assigned to $^6H_{5/2} \rightarrow {}^4L_{15/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^6H_{15/2}$, $^6F_{1/2}$ and $^6H_{13/2}$ transitions, respectively. According to Boehm et al. [23], the absorption bands of Sm^{3+} ion can be classified into two groups: the first is the low-energy group which contains the transitions upto 10,700 cm^{-1} (≈935 nm) and the other is high-energy group containing the transitions in the range 17,600–32,000 cm^{-1} (≈570–313 nm). The presence of Pb^{2+} ions in LTTSm10 glass may be responsible for the disappearance of some of the absorption transitions of Sm^{3+} ion at higher-energy range [14]. The oscillator strengths of the absorption bands are determined using our previous work [24]. The experimental oscillator strengths of the absorption bands of LTTSm10 glass are relatively much higher than those reported for Sm^{3+} ion in $CdO–Al_2O_3$ [15], LBTAF [20], LiLTB [25] and N4BS [26] glasses as presented in Table 1. The reasonably small δ_{rms} of ±0.30 × 10^{-6} between the measured and calculated oscillator strengths indicates that the intensity parameters obtained in the present work are more reliable.

Table 1
Assignment of absorption bands, measured (f_m) and calculated (f_c) oscillator strengths (×10^{-6}) and comparison of intensity parameters ($\Omega_\lambda \times 10^{-20}$ cm^2) of Sm^{3+} ion.

Transition	λ (nm)	LTTSm10	
		f_c	f_m
$^6H_{5/2} \rightarrow {}^6H_{13/2}$	1944	0.23	0.13
$^6H_{5/2} \rightarrow {}^6F_{1/2}$	1583	0.70	0.37
$^6H_{5/2} \rightarrow {}^6H_{15/2}$	1542	0.02	0.56
$^6H_{5/2} \rightarrow {}^6F_{3/2}$	1484	1.80	2.32
$^6H_{5/2} \rightarrow {}^6F_{5/2}$	1377	2.93	2.66
$^6H_{5/2} \rightarrow {}^6F_{7/2}$	1230	3.96	3.95
$^6H_{5/2} \rightarrow {}^6F_{9/2}$	1080	2.41	2.45
$^6H_{5/2} \rightarrow {}^6F_{11/2}$	942	0.38	0.35

Glass host	Ω_2	Ω_4	Ω_6
LTTSm10 [This work]	1.30	3.08	1.54
LBTAF [20]	0.27	2.52	2.47
$PbO–PbF_2–B_2O_3$ [27]	1.28	2.78	1.97
$PbO–PbF_2$ [28]	1.16	2.60	1.40
ZnBS [29]	0.29	3.82	3.65

The intensity parameters evaluated for LTTSm10 glass are comparable to those reported for different Sm^{3+}-doped host matrices [20,27–29] as given in Table 1. In order to minimize the δ_{rms}, the oscillator strength corresponding to the $^6H_{5/2} \rightarrow {}^4L_{15/2}$ (394 nm) transition is not considered in determining the intensity parameters. The intensity parameters follow the trend, $\Omega_2 < \Omega_6 < \Omega_4$ and they play very important role for the investigation of local structure as well as bonding in the vicinity of RE ions. The higher magnitude of Ω_2 indicates that the asymmetry sites occupied by Sm^{3+} ions are higher and the higher mixing of the opposite parity electronic configurations are responsible for the large spectral intensities. Also, the higher value of Ω_2 parameter indicates the higher degree of covalency of Ln–O bond. When compared to other glasses, the sites occupied by Sm^{3+} ions in LTT glasses posses higher asymmetry and greater degree of covalency for the Sm–O bond.

3.2. Emission spectra and radiative parameters

The excitation wavelength plays an important role in recording the emission spectra of rare-earth ions doped luminescent materials. The excitation spectrum of LTTSm10 glass by monitoring the emission at 600 nm is shown in Fig. 2. It contains nine excitation bands centered at 376, 404, 420, 441, 448, 477, 501, 528 and 561 nm corresponding to the $^6H_{5/2} \rightarrow {}^6P_{7/2}$, $^4F_{7/2}$, $^4M_{19/2}$, $^4G_{9/2}$, $^4M_{17/2}$, $^4I_{11/2}$, $^4G_{7/2}$, $^4F_{3/2}$ and $^4G_{5/2}$ transitions, respectively. The excitation band due to the $^6H_{5/2} \rightarrow {}^4I_{11/2}$ (477 nm) transition is found to be more intense and hence the emission measurements were carried out by exciting the samples with this wavelength. The room temperature fluorescence spectra recorded for LTTSm01, LTTSm05, LTTSm10 and LTTSm20 glasses in the spectral region 500–750 nm are shown in Fig. 3. For all the concentrations, the fluorescence spectra exhibit four emission transitions from $^4G_{5/2}$ excited level to its lower lying levels ^6H_J (J = 5/2, 7/2, 9/2 and 11/2) at 563, 600, 646 and 707 nm, respectively. Among these, the emission band corresponding to $^4G_{5/2} \rightarrow {}^6H_{7/2}$ (600 nm) transition possesses highest intensity. The $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^6H_{7/2}$ transitions contain both electric

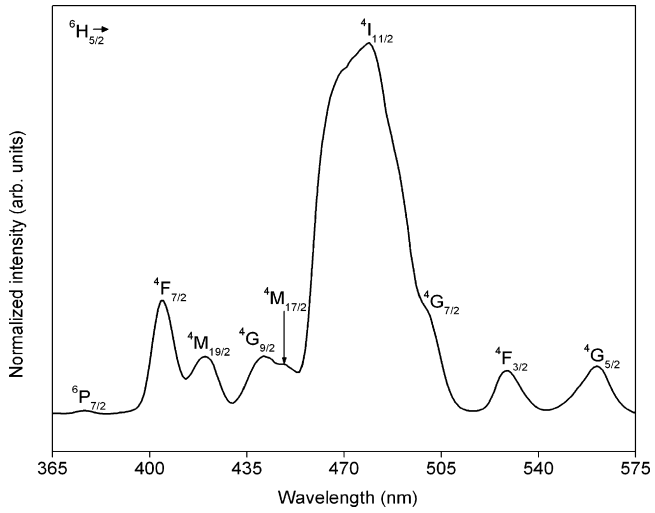

Fig. 2. Excitation spectrum of LTTSm10 glass monitoring the emission at 600 nm.

and magnetic dipole contributions obeying the selection rules $\Delta J = 0, \pm 1$ [30], while the other two transitions $^4G_{5/2} \rightarrow {}^6H_{9/2}, {}^6H_{11/2}$ are purely electric dipole interaction. The partial energy level diagram shown in Fig. 4 describes the emission mechanism of Sm^{3+} ions in LTT glasses. The closely spaced higher energy levels causes fast non-radiative (NR) decay from $^4I_{11/2}$ excited state to $^4G_{5/2}$ metastable state. Due to the large energy gap of ~7150 cm^{-1} between $^4G_{5/2}$ state and its next lower state $^6F_{11/2}$, the radiative decay through intense reddish–orange emission occurs from all the samples.

The J–O intensity parameters Ω_λ are used to obtain the radiative parameters such as spontaneous emission probabilities (A_R), luminescence branching ratios (β_R) and radiative lifetimes (τ_R) of $^4G_{5/2}$ excited state which are mainly depend on the network former and modifier of the glass composition as presented in Table 2. The magnitude of branching ratios characterizes the lasing power of a transition and it is well established that an emission transition with the measured branching ratio greater than 0.50 is more potential for laser emission [31]. From the predicted branching ratios of Sm^{3+} ions in LTTSm10 glass (see Table 2), it is clear that the $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition is found to have the highest β_R value compared to other transitions. The measured branching ratios (β_m) determined from the relative areas of the emission bands of LTTSm10 glass are 0.20,

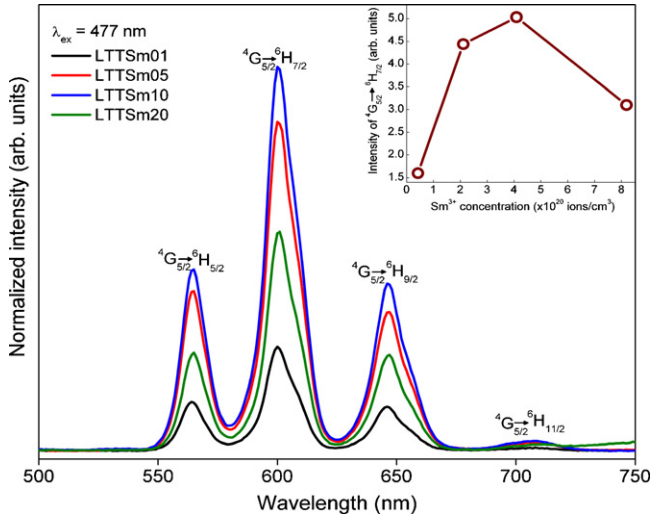

Fig. 3. Fluorescence spectra for different concentrations of Sm^{3+} ions in LTT glass system (λ_{ex} = 477 nm). Inset figure represents fluorescence intensity as a function of Sm^{3+} concentration.

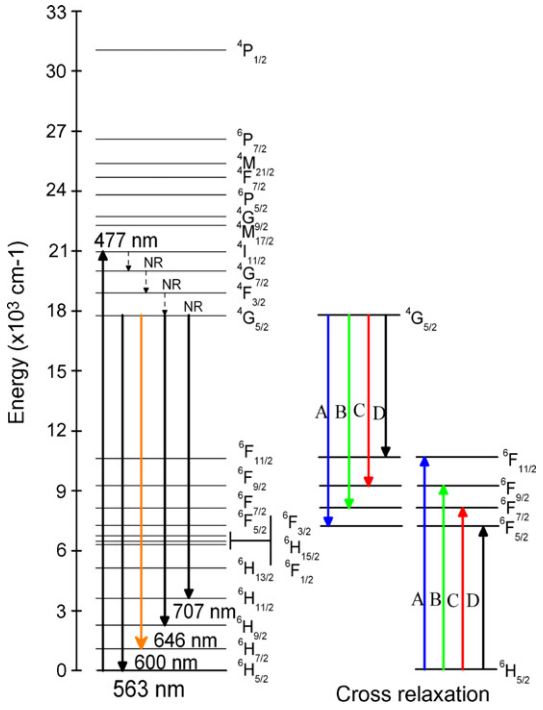

Fig. 4. Partial energy level diagram showing emission mechanism and cross relaxation channels in Sm^{3+} ions doped LTT glass system.

Table 2
Electric A_{ed} and magnetic A_{md} probabilities, spontaneous emission probabilities (A_R in s^{-1}), luminescence branching ratios β_R and radiative lifetime (τ_R) of $^4G_{5/2}$ state of Sm^{3+} ion in LTTSm10 glass.

Transition	A_{ed}	A_{md}	A_R	β_R
$^4G_{5/2} \rightarrow {}^6F_{11/2}$	0.50	0.00	~1	~0
$^4G_{5/2} \rightarrow {}^6F_{9/2}$	2.96	0.00	3	~0
$^4G_{5/2} \rightarrow {}^6F_{7/2}$	7.32	0.27	10	0.01
$^4G_{5/2} \rightarrow {}^6F_{5/2}$	24.69	0.66	35	0.05
$^4G_{5/2} \rightarrow {}^6F_{3/2}$	3.28	0.85	18	0.03
$^4G_{5/2} \rightarrow {}^6H_{15/2}$	0.53	0.00	~1	~0
$^4G_{5/2} \rightarrow {}^6F_{1/2}$	2.74	0.00	3	~0
$^4G_{5/2} \rightarrow {}^6H_{13/2}$	9.57	0.00	10	0.01
$^4G_{5/2} \rightarrow {}^6H_{11/2}$	78.98	0.00	79	0.11
$^4G_{5/2} \rightarrow {}^6H_{9/2}$	205.48	0.00	205	0.28
$^4G_{5/2} \rightarrow {}^6H_{7/2}$	272.73	0.58	309	0.42
$^4G_{5/2} \rightarrow {}^6H_{5/2}$	17.53	0.58	61	0.08
$A_T = 734\ s^{-1}$; $\tau_R = 1.36$ ms				

0.54, 0.24 and 0.02 for $^4G_{5/2} \rightarrow {}^6H_{5/2}, {}^6H_{7/2}, {}^6H_{9/2}$, and $^6H_{11/2}$ transitions, respectively. Amongst these β_m values the branching ratios of $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition in LTT glasses is found comparable to various glass hosts [20,23,25,32–36] as shown in Table 3.

In addition to the emission measurements, the spontaneous emission probabilities and radiative lifetimes obtained from the

Table 3
Measured (β_m) and predicted (β_R) branching ratios for $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition of Sm^{3+} ion in various glass compositions.

Glass composition	β_m	β_R
LTTSm10 [This work]	0.54	0.42
LBTAF [20]	0.55	0.50
P_2O_5 [23]	0.35	0.37
N4BS [26]	0.54	0.43
PKFBASm [32]	0.56	0.42
L5FBS [33]	0.55	0.47
Lithiumfluoroborate [31]	0.46	0.47
ZnCdF [35]	0.38	0.47
ZBLALi [36]	0.37	0.42

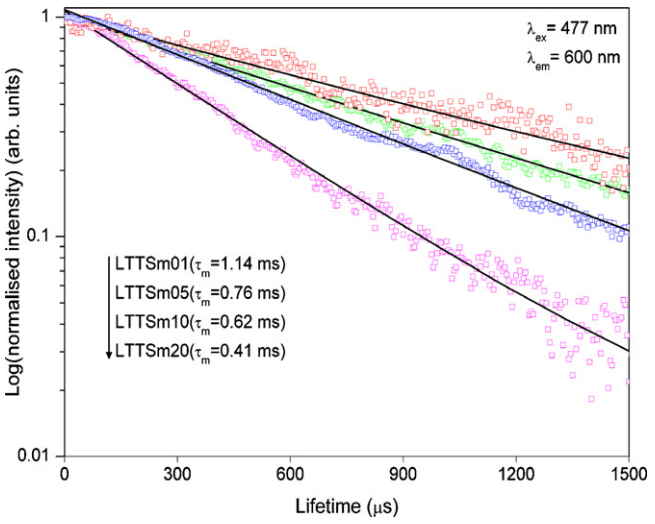

Fig. 5. Decay profiles of $^4G_{5/2}$ level for different concentrations of Sm^{3+} ion in LTT glasses.

J–O theory [21,22] have been used to evaluate the stimulated emission cross-sections (σ_e), gain bandwidths ($\sigma_e \times \Delta\lambda_P$) and optical gain ($\sigma_e \times \tau_R$) parameters for the $^4G_{5/2} \rightarrow {}^6H_J$ (J = 5/2, 7/2, 9/2 and 11/2) transition of LTTSm10 glasses. The stimulated emission cross-section is one of the important parameters used to identify the potential laser transitions in a laser active medium. The emission cross-section of 6.26×10^{-22} cm^2 obtained for $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition in LTTSm10 glass is found to be very close to the values of 6.92×10^{-22} cm^2 for calibo [14]; 6.30×10^{-22} cm^2 for lead fluoroborate [16]; 5.74×10^{-22} cm^2 for L5FBS [33] and 5.73×10^{-22} cm^2 for Mo–Sb_2O_3–B_2O_3 [37] glasses. The high gain bandwidths of 10.63×10^{-28} cm^3 and optical gain of 8.51×10^{-25} cm^2 s^{-1} obtained for $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition indicate that the LTTSm10 glass could be useful for the development of optical devices.

3.3. Luminescence quenching and decay characteristics

Fig. 5 shows the decay profiles of $^4G_{5/2}$ emission level of Sm^{3+} ions in LTT glasses upon 477 nm excitation by monitoring the emission at 600 nm. The decay curves exhibited single-exponential behavior according to equation: $I_t = I_o e^{-t/\tau}$, where I_o is the fluorescence intensity when $t = 0$ and τ represents the lifetime of the excited state. The predicted radiative lifetime (τ_R) of $^4G_{5/2}$ is 1.36 ms. The measured lifetimes (τ_m) obtained by taking the first e-folding times of the maximum intensities are 1140, 760, 620 and 410 μs for LTTSm01, LTTSm05, LTTSm10 and LTTSm20 glasses, respectively. From these measurements, it is clear that the measured lifetime decreases with increase of Sm^{3+} concentration.

The photoluminescence spectra shown in Fig. 3 also reveal the decrease of emission peak intensities for 2.0 mol% of Sm^{3+} ion concentration. The inset shows the variation of intensity of $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition with Sm^{3+} ions concentration. The main reason for the quenching of emission intensities and lifetimes may be due to the energy transfer through the cross-relaxation as displayed in Fig. 4, where the energies of emission transitions, A: $^4G_{5/2} \rightarrow {}^6F_{5/2}$ (10,500 cm^{-1}), B: $^4G_{5/2} \rightarrow {}^6F_{7/2}$ (9632 cm^{-1}), C: $^4G_{5/2} \rightarrow {}^6F_{9/2}$ (8503 cm^{-1}) and D: $^4G_{5/2} \rightarrow {}^6F_{11/2}$ (7146 cm^{-1}) closely matches with the energies of absorption transitions, A: $^6H_{5/2} \rightarrow {}^6F_{11/2}$ (10,616 cm^{-1}), B: $^6H_{5/2} \rightarrow {}^6F_{9/2}$ (9259 cm^{-1}), C: $^6H_{5/2} \rightarrow {}^6F_{7/2}$ (8130 cm^{-1}) and D: $^6H_{5/2} \rightarrow {}^6F_{5/2}$ (7262 cm^{-1}) respectively.

4. Conclusions

LTT glasses doped with different concentrations of Sm^{3+} ion have been investigated through absorption, photoluminescence and decay measurements. The visible photoluminescence spectra revealed four emission bands at 563, 600, 646 and 707 nm corresponding to the $^4G_{5/2} \rightarrow {}^6H_J$ (J = 5/2, 7/2, 9/2 and 11/2) transitions, respectively. From the magnitude of emission intensities it is concluded that the LTTSm01, LTTSm05 and LTTSm20 glass exhibit reddish–orange emission with 477 nm excitation, while the LTTSm10 glass emit deep-red luminescence. Among the calculated spontaneous probabilities and luminescence branching ratios of the $^4G_{5/2} \rightarrow {}^6H_J$ states, the $^4G_{5/2} \rightarrow {}^6H_{7/2}$ transition posses highest values. The decay curves of $^4G_{5/2}$ metastable state upon 477 nm excitation revealed single-exponential for all the concentrations. The decrease of lifetimes with an increase in Sm^{3+} concentration has been attributed to concentration quenching among the Sm^{3+} ions at higher concentrations. The results of the present investigation indicate that LTTSm glasses are promising materials for the development of visible lasers, fluorescent display devices and optical amplifiers.

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