




Enhanced thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor

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ABSTRACT

The application of thermoluminescence technique in radiation dosimetry spans field of health physics, biological and geological sciences and personnel monitoring; this led to the search for new compositions with desirable dosimetric properties. In the present work, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors were prepared by solid-state reaction method and their TL properties were studied in detail. The comparison of their TL results showed that co-doping of Ce^{3+} ions enhanced TL response of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphors; this was also verified from measurement of TL emission spectra of the samples. Optimized glow curves were analysed and TL parameters were extracted from Chen's method. Co-doped $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor is found to be useful in dosimetric application.

1 Introduction

When an insulating or a superconducting material is exposed to any kind of ionizing radiation, deposited energy is stored in the defect sites and colour centres of the crystal lattice. Due to action of heat energy, a fraction of this stored energy released and emitted as visible light which is called thermoluminescence [1, 2]. Nowadays application of various radiations such as ultraviolet, X-rays, β -rays, γ -rays in the different fields like medical, industrial, agriculture, etc., is increasing [3]. Thermoluminescence (TL) is one of

the techniques used in radiation dosimetry [4]. Thermoluminescent materials are more investigated in on-going researches, because they found to have increasing application in thermoluminescence dosimeters. Thermoluminescent dosimeters necessarily have linearity of TL response with exposed radiation dose.

$\text{CaSrAl}_2\text{SiO}_7$ is one of the members of melilite group; these melilites that are basically silicate-based materials showed their use in TL dosimetry. In this work, thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor are investigated and also

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observed that co-doping of Ce^{3+} ions enhanced TL of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor. The effect of different Tb^{3+} concentration in both single and co-doped samples on the TL glow curves has been recorded and glow curve is analysed by computerized glow curve deconvolution method. To the best of our knowledge, there is no earlier report on thermoluminescence investigation on $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors.

2 Synthesis and experimental

Phosphors for the study were prepared by solid-state reaction method at high temperature using analytical reagent (AR) grade raw chemicals with more than 99% accuracy. Calcium carbonate, strontium carbonate, aluminium oxide, silicon di-oxide, ceric oxide and terbium oxide were the starting materials. The amounts of the raw chemicals were calculated according to stoichiometric ratio in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (y mol%), where $y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%. Required quantities of raw materials were mixed homogeneously using agate mortar and pestle for 3 h. The resultant mixtures were fired at high temperature 1300 °C for 5 h. It was then slowly cooled to room temperature inside the closed furnace. The powders so formed were collected by crushing the prepared samples. Now the samples were ready for all other characterization studies.

Phase of synthesized phosphor was confirmed by X-ray diffraction (XRD) technique using $\text{Cu-K}\alpha$ radiation with the help of D2 phaser Bruker diffractometer. Thermoluminescence investigation of phosphor was performed by TLD reader, Nucleonix TL 1009I with constant heating rate of 5 °C/s. As sample must be exposed to any kind of radiation before measurement of TL, UV cabinet is used, where the samples were exposed to UV radiation of 254 nm. Band pass filters of different wavelengths have been used to record TL emission spectrum. To record TL emission spectrum, the processes used in recording TL glow curves were repeated; here the sample that is placed in canthal strip was covered with band pass filter. The band pass filter allows the signals of selected range of wavelengths; hence in TL emission spectrum, characteristic emission peaks were observed.

3 Results and discussion

3.1 XRD investigation

To verify the preparation of phosphors, they were analysed by XRD tool that reveals the phase composition. Figure 1 shows the XRD patterns of Tb^{3+} singly doped and $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors along with JCPDS file. It is clear that phosphors have good crystallization with no any impurity phase, as experimental diffraction patterns well matched with JCPDS file 26-0327 of $\text{CaSrAl}_2\text{SiO}_7$. Hence, the as-prepared phosphors have tetragonal crystallographic structure whose space group is $P4_21m$ [5].

3.2 Thermoluminescence investigation

Thermoluminescence (TL) is light emission due to moderate heating of a solid, previously exposed to ionizing radiation. The absorption of the ionizing radiation (UV, X-, β - or γ -rays, high-energy particles) creates traps, and fills them (or those already exist) with electrons and/or holes even at low temperatures. The subsequent heating migrates the electrons

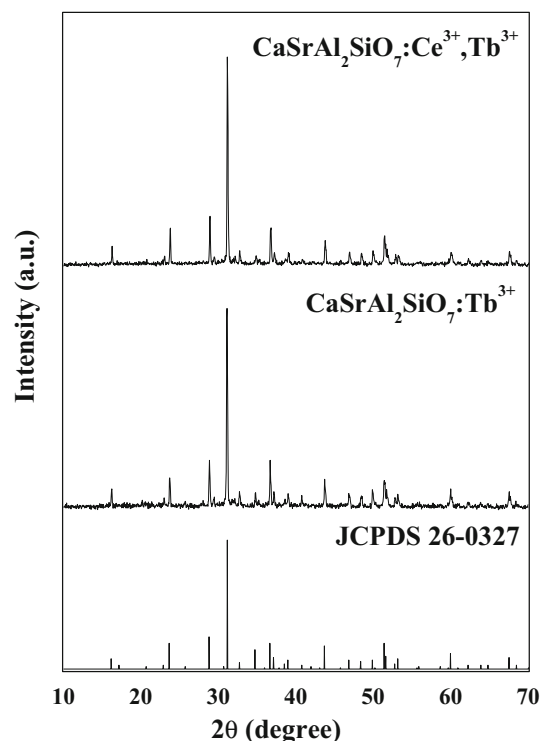


Fig. 1 Diffraction patterns of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

and/or holes in the lattice, until they fall into other traps (or recombine) with a consequent photon emission [6]. Thermoluminescent emission from TL materials is very sensitive to the amount and nature of dopant element and radiation effect. So in the present research work, we used Ce^{3+} and Tb^{3+} as single and co-dopant ions with their various concentrations; after that TL measurement was performed for each sample with optimized dopant and co-dopant concentration with various ultraviolet (UV) exposure time and then TL kinetic parameters were extracted from the Chen's peak shape method.

3.2.1 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$

Let us first focus on the investigated thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor; this was already discussed in our previous work in which Ce^{3+} concentration was varied as 0.1, 0.3, 0.5, 1.0, 2.0, 3.0 and 4.0 mol% [7]. The optimized glow curve was obtained for 0.5 mol% Ce^{3+} -doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor. This optimized phosphor showed an increment in TL intensity up to 35 min of UV irradiation time. As we further increased UV exposure time, saturation in TL intensity was found.

3.2.2 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$

At the second attempt, TL properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor was seen and this report is dealing here for the first time. Figure 2 represents a series of Tb^{3+} concentration (1.0, 3.0, 5.0, 7.0, 10.0 mol%)-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ samples. TL peak position is independent of the concentration of Tb^{3+} element and exhibits no shift with change in doping concentration. The only variation obtained in the TL peak intensity while increasing Tb^{3+} concentration. It can be understood from the inset of Fig. 2 that TL intensity of sample first increased with rise in Tb^{3+} concentration, attained a highest value at 5.0 mol% of Tb^{3+} ions and then decreased with further increase in Tb^{3+} concentration; this behaviour arises from concentration quenching process after a particular Tb^{3+} concentration. As we increase impurity concentration, there is an increase in the number of defects/traps which in turn implies a growth in the density of charge carriers being trapped upon irradiation. Therefore the initial rise in the TL peak intensity or area of the glow curves. Furthermore, on being thermally stimulated,

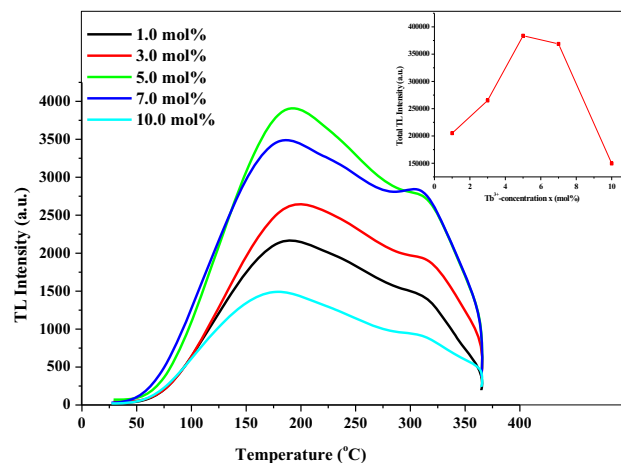


Fig. 2 Concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphors with 10-min UV exposure time

these charge carriers release from traps which in turn recombine with their counterparts at the recombination centre and yield diverse TL glow peaks with elevated height [8].

As thermoluminescence properties are greatly affected by exposure time, TL property of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) phosphor was measured with different UV exposure time. For variable UV exposure time, some of the selected TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) are represented in Fig. 3. Spectral behaviour was same with increase in length of UV exposure time but peak TL intensity or glow curve area altered with increase in exposure time. The growth in TL response was observed as exposure time was boosted and after 60 min of UV exposure TL response diminished with more added exposure time. With increasing irradiation dose more and more trapping centres or luminescent centres responsible for the TL glow peaks are getting filled. Upon thermal stimulation, these traps liberate their charge carriers and they get recombine with their counterparts, giving rise to different glow peaks. When all the trapping centres that are subjected to desired TL emission get filled, the saturation or decrease in the TL intensity starts appearing [8–10].

3.2.3 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$

Thermoluminescence experiments were performed on the series of $\text{CaSrAl}_2\text{SiO}_7:x\text{Ce}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:y\text{Tb}^{3+}$ phosphors that the results point out optimal concentration of Ce^{3+} as $x = 0.5$ mol% and of Tb^{3+} as $y = 5.0$ mol%. In the present work, co-doping

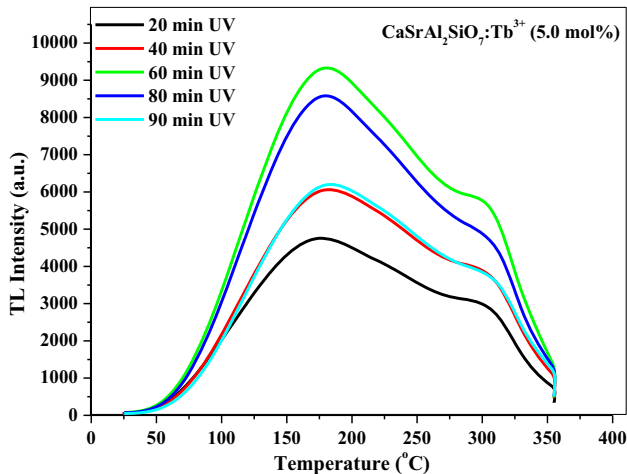


Fig. 3 Effect of UV exposure time on the TL glow curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) phosphor

of Ce^{3+} and Tb^{3+} was also performed in $\text{CaSrAl}_2\text{SiO}_7$ host and TL properties of this co-doped sample were also investigated and compared with TL of singly doped samples. Co-doping was performed with fixed concentration of Ce^{3+} ($x = 0.5$ mol%) and variable concentration of Tb^{3+} ($y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%).

Figure 4 shows TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ ($x = 0.5$ mol%), Tb^{3+} ($y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%) phosphors with fixed UV irradiation time. Glow curves for all samples had similar shape but with change in glow curve area. Concentration quenching occurred at 5.0 mol% of Tb^{3+} ions (see inset of Fig. 4). Like Ce^{3+} and Tb^{3+} singly doped samples, impact of various UV irradiation time on the TL glow curve of optimized co-doped $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%) sample was seen and this result is depicted in Fig. 5. It can be clearly seen from Fig. 5 that TL intensity of optimized co-doped phosphor increased up to 100 min of UV exposure and then decreased.

3.2.4 Evaluation of TL parameters

To determine TL parameters, broad glow curves of 60-min UV exposed $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) and 100-min UV exposed $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%) samples were first applied to computerized glow curve deconvolution (CGCD) method. Both samples were found to have four overlapping peaks in their parent glow curves. The position of constituent peaks is shown in Fig. 6. TL parameters like geometrical shape factor (μ_g),

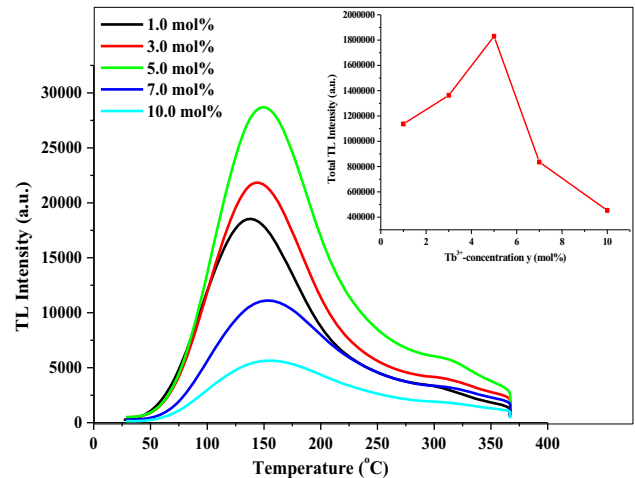


Fig. 4 Tb^{3+} concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor with 30-min UV exposure time

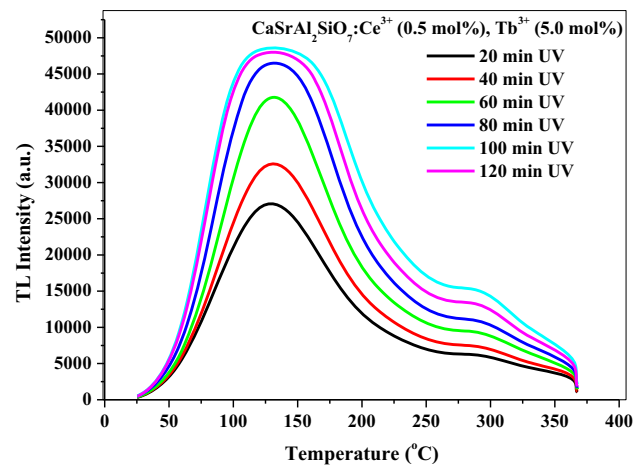
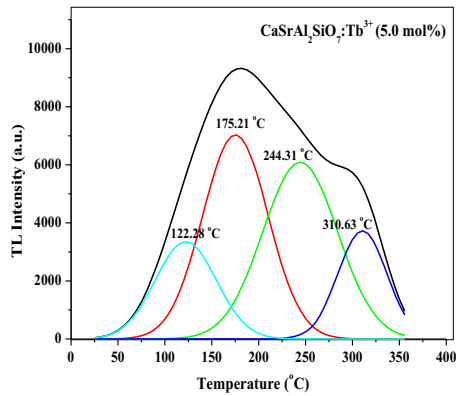


Fig. 5 Effect of UV exposure time on the TL glow curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor

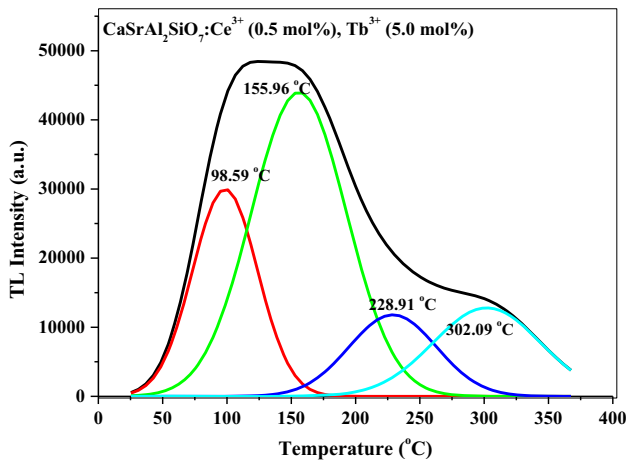
order of kinetics (b), frequency factor (S) and activation energy (E) were extracted using Chen's peak shape method. The calculated TL parameters of Tb^{3+} -doped and $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors are listed in Table 1.

3.2.5 TL emission spectra

Figure 7 represents comparison of TL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphors. Emission spectra were recorded using interference band pass filters of various wavelengths from 400 to 700 nm. Emission spectra of Ce^{3+} -doped sample was already discussed in our previous article [7], here it is shown for comparison only. The three samples have characteristic



(a)



(b)

Fig. 6 Representation of peak deconvolution on parent glow curve of **a** $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and **b** $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

emission peaks of respective dopant and co-dopant ions. $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ sample has emission peaks at around 480, 545 and 590 nm these are due to characteristic transition $^5\text{D}_4 \rightarrow ^7\text{F}_6$, $^5\text{D}_4 \rightarrow ^7\text{F}_5$ and $^5\text{D}_4 \rightarrow ^7\text{F}_4$ of Tb^{3+} ions [11]. $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ sample has characteristic peak of Ce^{3+} ions at around 420 nm, due to $5d (^5\text{D}_{3/2}) \rightarrow 4f (^2\text{F}_{5/2})$ [12, 13] and

Table 1 Calculated TL parameters

Phosphor	T_m (°C)	μ_g	b	E (eV)	S (s^{-1})
$\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%)	122.28	0.48	1	0.46	8.07×10^6
	175.21	0.48	1	0.57	2.45×10^7
	244.31	0.49	1	0.75	1.82×10^8
	310.63	0.70	2	1.41	2.12×10^{13}
$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%)	98.59	0.49	1	0.54	2.35×10^8
	155.96	0.5	2	0.53	1.4×10^7
	228.91	0.49	1	0.78	7.45×10^8
	302.09	0.49	1	0.82	1.37×10^8

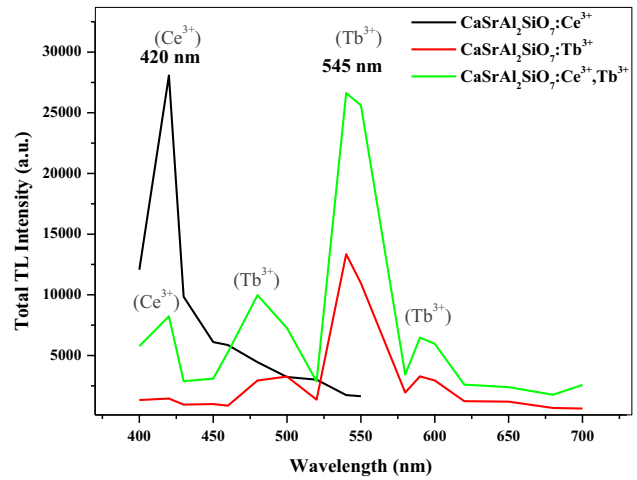


Fig. 7 TL Emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

characteristic peaks of Tb^{3+} ions situated at around 480, 545 and 600 nm; these are due to above-mentioned transitions of Tb^{3+} ions.

Figure 8 shows the comparative TL glow curves of the three optimized phosphors. $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped phosphor has most intense TL properties among all three. Also from Fig. 7 it is clearly visible that TL emission of cerium ions is decreased, while TL emission of terbium ions is increased in co-doped sample; hence co-doping of small amount of Ce^{3+} ions, i.e., 0.5 mol%, may enhance TL emission of Tb^{3+} ions in $\text{CaSrAl}_2\text{SiO}_7$ host.

Present study concludes that among the three, $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor has almost linear increment in total TL intensity with respect to UV exposure time up to 100 min, which is very large range as compared to other UV irradiated silicate, aluminate and aluminosilicate-based phosphors such as $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ [14]; $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Ce}^{3+}$ [15]; $\text{BaMgAl}_{10}\text{O}_{17}:\text{Ce}^{3+}$ [16]. Hence

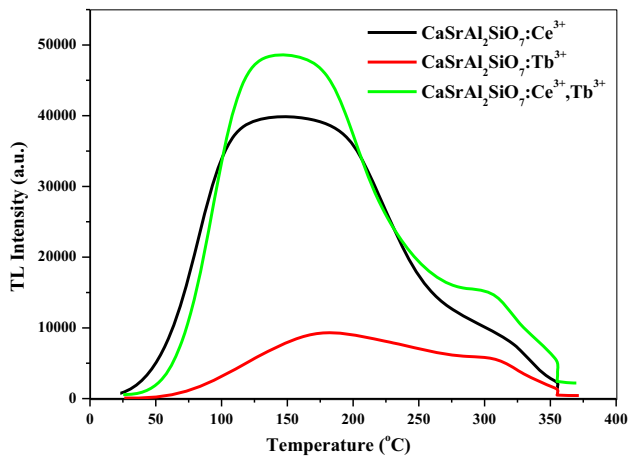


Fig. 8 Comparative TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor may be most appropriately useful for UV dosimeter application.

4 Conclusions

$\text{CaSrAl}_2\text{SiO}_7:\text{yTb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{yTb}^{3+}$ phosphors were prepared by solid-state reaction method. Detail comparative TL study of singly doped and co-doped samples was investigated. Concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{yTb}^{3+}$ phosphor and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{yTb}^{3+}$ phosphors were measured and found that 5.0 mol % is optimized concentration of Tb^{3+} ions in both samples. Tb^{3+} singly doped sample showed linear increment in TL intensity up to 60 min, while $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped sample showed this linearity up to 100 min, also TL properties of co-doped sample got enhanced with inclusion of 0.5 mol% of Ce^{3+} ions, this was also verified from TL emission spectra. Hence the optimized $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(0.5 \text{ mol}\%),\text{Tb}^{3+}(5.0 \text{ mol}\%)$ phosphor may be a potential candidate for UV dosimeter application.

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