



## A Study on Thermoluminescence Characteristics of Rare-Earth Doped Phosphors

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### Abstract

Thermoluminescence (TL) is a thermally stimulated light-emission process in which charge carriers created by prior irradiation are stored at metastable defects and released during controlled heating. Rare-earth ions are particularly useful in phosphor hosts because they can create or modify trapping states and can also act as efficient radiative recombination centers. This methodology paper proposes a systematic framework for studying the TL characteristics of rare-earth-doped phosphors, using alkaline-earth aluminate phosphors as the principal model system and published work up to 2018 as the evidence base. The study design integrates controlled synthesis, X-ray diffraction, morphology analysis, photoluminescence, ultraviolet and gamma irradiation, glow-curve acquisition, dose-response measurements, fading tests, and kinetic analysis. Eu<sup>2+</sup> is treated primarily as an emission center, while Dy<sup>3+</sup> and related rare-earth co-dopants are evaluated for their influence on trap density and trap depth. Peak-shape analysis and computerized glow-curve deconvolution are proposed to estimate activation energy, frequency factor, and kinetic order. An illustrative simulation is included to show how increased rare-earth-assisted trapping can enhance and reshape a TL glow curve; the simulated values are not presented as experimental measurements. The methodology is intended to support reproducible comparison of phosphor composition, irradiation dose, heating rate, and thermal treatment, and to identify compositions with strong, stable, and approximately linear TL responses suitable for dosimetry, persistent-luminescence studies, and radiation-sensing applications.

### Introduction

Thermoluminescence occurs when an insulating or semiconducting material is first exposed to ionizing or optical radiation and then heated. Irradiation produces mobile electrons and holes; a fraction becomes trapped at lattice defects or impurity-related states. During subsequent heating, trapped carriers acquire sufficient thermal energy to escape and recombine, producing photons whose intensity is recorded as a function of temperature. The resulting glow curve contains information about trap concentration, trap depth, recombination

probability, and thermal stability. Because the integrated TL signal can be related to absorbed radiation dose, thermoluminescent materials are widely investigated for radiation monitoring and dosimetry. The same trap physics also underpins persistent luminescence, making TL spectroscopy a useful diagnostic tool for understanding long-afterglow phosphors (Van den Eeckhout, Smet, & Poelman, 2010).

Rare-earth activators such as Eu, Dy, Tb, Ce, Nd, and Sm are attractive because their electronic configurations generate characteristic

luminescent states while simultaneously perturbing the defect chemistry of a host lattice. In  $\text{Eu}^{2+}$ -activated aluminates, broad 5d-4f emission is strongly influenced by the local crystal field, whereas trivalent co-dopants may introduce or stabilize charge-compensating defects that act as trapping centers. For example,  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+},\text{Dy}^{3+}$  is a benchmark persistent phosphor in which thermoluminescence has been repeatedly used to probe trap distributions and long-afterglow mechanisms (Mothudi et al., 2012; Zeng et al., 2018). Similar TL behavior has been reported for  $\text{Dy}^{3+}$ -activated calcium aluminates,  $\text{Dy}^{3+}$ -doped haloborates, mixed Eu/Dy silicates, and other rare-earth-containing hosts (Oza et al., 2015; Ziyauddin et al., 2016; Jadhaw et al., 2017).

The objective of the proposed study is therefore to establish a reproducible methodology for determining how rare-earth type and concentration influence TL glow-peak temperature, intensity, dose linearity, fading, and trapping parameters. The study emphasizes a controlled comparison rather than a single isolated glow curve. Structural characterization is linked to TL measurements so that phase formation, crystallite size, thermal treatment, and dopant concentration can be interpreted together. All literature used in the design was published in or before 2018, consistent with the requested reference cutoff.

## Literature Review

Foundational TL analysis relies on describing individual glow peaks through kinetic models. Kitis, Gómez-Ros, and Tuyn (1998) introduced widely used analytical deconvolution functions for first-, second-, and general-order thermoluminescence kinetics, allowing overlapping glow curves to be separated into physically interpretable components. This approach is especially important for rare-earth-doped hosts because multiple defects may contribute simultaneously to a broad experimental curve.

For persistent aluminate phosphors, Van den Eeckhout et al. (2010) reviewed  $\text{Eu}^{2+}$ -based systems and emphasized that thermally released trapped carriers are central to long-lasting emission. Mothudi et al. (2012) prepared

$\text{SrAl}_2\text{O}_4:\text{Eu}^{2+},\text{Dy}^{3+}$  by solid-state reaction and reported monoclinic phase formation, green  $\text{Eu}^{2+}$  emission near 497 nm, and broad TL glow curves centered near 120 °C for samples annealed at 1100-1200 °C. Their observations supported the role of  $\text{Dy}^{3+}$ -related trapping in prolonging luminescence. Later, Zeng et al. (2018) combined optically stimulated luminescence and thermoluminescence to examine  $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}/\text{Dy}^{3+}$ . Their results strengthened the interpretation that oxygen-vacancy-related electron traps and Dy-associated shallow traps contribute to persistent emission.

Other host lattices demonstrate that the effect is not limited to strontium aluminate. Oza et al. (2015) investigated  $\text{Sr}_2\text{B}_5\text{O}_9\text{Cl}:\text{Dy}^{3+}$  and observed a prominent high-temperature glow peak together with a lower-temperature component, indicating at least two classes of traps; the material also showed useful dose response and comparatively low fading. Ziyauddin et al. (2016) synthesized  $\text{CaAl}_2\text{O}_4:\text{Dy}^{3+}$  by combustion and found that TL intensity increased with Dy concentration up to an optimum before decreasing because of concentration quenching. Jadhaw et al. (2017) studied Eu-doped and Eu/Dy-codoped  $\text{Sr}_2\text{Al}_2\text{SiO}_7$  and evaluated the influence of heating rate, UV exposure, gamma irradiation, and peak-shape-derived trapping parameters. These studies collectively show why dopant concentration and readout conditions must be systematically controlled rather than treated as secondary variables.

By 2018, rare-earth TL research had expanded to nanophosphors and scintillator-like hosts. Samariha and Saraee (2018) showed that annealing temperature changed crystallite size and TL characteristics in Dy/Tb-doped  $\text{SrSO}_4$  nanophosphors, demonstrating a direct connection between thermal processing and trap structure. İlhan and Keskin (2018) reported concentration-dependent PL, radioluminescence, and TL in  $\text{Eu}^{3+}$ -doped  $\text{CdTa}_2\text{O}_6$ , with TL curves measured over 50-400 °C after UV irradiation. The literature therefore supports a methodology that integrates composition, phase/morphology, irradiation history, heating rate, and kinetic fitting in a single experimental plan.

**Table 1.** Selected literature informing the proposed methodology (references up to 2018)

| Study | Phosphor / system | Preparation or excitation | Key TL relevance | DOI |
|-------|-------------------|---------------------------|------------------|-----|
|-------|-------------------|---------------------------|------------------|-----|

|                          |                                                                      |                                            |                                                       |                              |
|--------------------------|----------------------------------------------------------------------|--------------------------------------------|-------------------------------------------------------|------------------------------|
| Mothudi et al. (2012)    | SrAl <sub>2</sub> O <sub>4</sub> :Eu <sup>2+</sup> ,Dy <sup>3+</sup> | Solid-state; 1100-1200 °C                  | Broad glow near 120 °C; trap-related afterglow        | 10.1016/j.physb.2011.10.033  |
| Oza et al. (2015)        | Sr <sub>2</sub> B <sub>5</sub> O <sub>9</sub> Cl:Dy <sup>3+</sup>    | Modified solid-state; $\gamma$ irradiation | Multiple trap populations; dosimetric response        | 10.1002/bio.2818             |
| Ziyauddi n et al. (2016) | CaAl <sub>2</sub> O <sub>4</sub> :Dy <sup>3+</sup>                   | Combustion; $\gamma$ irradiation           | Optimum dopant concentration and quenching            | 10.1002/bio.2926             |
| Jadhaw et al. (2017)     | Sr <sub>2</sub> Al <sub>2</sub> SiO <sub>7</sub> :Eu / Eu,Dy         | Solid-state; UV and 60Co $\gamma$          | Heating-rate and exposure effects; kinetic parameters | 10.1002/bio.3331             |
| Zeng et al. (2018)       | SrAl <sub>2</sub> O <sub>4</sub> :Eu <sup>2+</sup> /Dy <sup>3+</sup> | Optical stimulation + TL                   | Trap-depth interpretation of long afterglow           | 10.1016/j.jlumin.2018.03.088 |
| Samariha & Saraee (2018) | SrSO <sub>4</sub> :Dy,Tb                                             | Co-precipitation ; $\gamma$ irradiation    | Annealing/crystallite-size dependence of TL           | 10.1016/j.jlumin.2018.02.057 |
| İlhan & Keskin (2018)    | CdTa <sub>2</sub> O <sub>6</sub> :Eu <sup>3+</sup>                   | Solid-state; 254 nm UV                     | Concentration-dependent TL/PL/RL behavior             | 10.1039/C8DT02395F           |

## Methodology

*Step 1 - Selection of host and dopant series.* A controlled SrAl<sub>2</sub>O<sub>4</sub>-based series is proposed because its Eu/Dy trap chemistry is well documented. Five compositions should be prepared: undoped SrAl<sub>2</sub>O<sub>4</sub>, SrAl<sub>2</sub>O<sub>4</sub>:1 mol% Eu, and SrAl<sub>2</sub>O<sub>4</sub>:1 mol% Eu with 0.5, 1.0, and 2.0 mol% Dy. The fixed Eu concentration isolates the effect of Dy-related trap modification. A secondary Dy-only CaAl<sub>2</sub>O<sub>4</sub> sample may be used as an external comparison if resources allow, following the combustion-based concept used by Ziyauddin et al. (2016).

*Step 2 - Synthesis and thermal treatment.* High-purity SrCO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, Eu<sub>2</sub>O<sub>3</sub>, and Dy<sub>2</sub>O<sub>3</sub> are weighed stoichiometrically, mixed by wet milling, dried, and calcined. A small borate flux can be used when necessary to promote crystallization. Final firing is performed at approximately 1100-1200 °C under a reducing atmosphere suitable for converting Eu<sup>3+</sup> to Eu<sup>2+</sup>, followed by controlled cooling. The thermal schedule is kept identical for all compositions because annealing temperature can substantially alter trap structure and crystallite size (Mothudi et al., 2012; Samariha & Saraee, 2018).

*Step 3 - Structural and optical characterization.* Powder X-ray diffraction is used to verify host phase formation and identify impurity phases. Crystallite size is estimated from the principal diffraction peaks. SEM is used to compare morphology and agglomeration, while EDS verifies the presence of dopant elements.

Photoluminescence excitation and emission spectra are recorded to confirm the rare-earth emission center before TL testing. A concentration-dependent decrease in emission at high dopant loading should be interpreted carefully because it may indicate concentration quenching rather than loss of crystallinity (Ziyauddin et al., 2016; İlhan & Keskin, 2018).

*Step 4 - Annealing and irradiation protocol.* Before each TL experiment, samples are thermally erased using a repeatable annealing cycle, for example 400 °C for 30-60 min followed by cooling in darkness. Equal sample masses are then irradiated. Two excitation routes are proposed: 254 nm UV exposure for 1, 5, 10, and 20 min, and 60Co gamma irradiation at 0.1, 0.5, 1, 5, and 10 Gy. Irradiated samples are stored in light-tight containers for a fixed delay time before readout. Separating UV and gamma series allows comparison of trap filling by optical versus ionizing excitation, as previously examined in Eu/Dy phosphors (Jadhaw et al., 2017).

*Step 5 - Thermoluminescence readout.* Glow curves are recorded from approximately 30 to 350 °C using a linear heating ramp. The primary heating rate is 2 °C s<sup>-1</sup>, with additional measurements at 1, 5, and 10 °C s<sup>-1</sup> for selected samples to evaluate thermal-lag and kinetic effects. Each condition is measured in triplicate. Signal is normalized to phosphor mass and background-subtracted. Peak temperature (T<sub>m</sub>), maximum intensity (I<sub>m</sub>), integrated glow area, and full width at half maximum are extracted from each curve.

*Step 6 - Dose response, fading, and reproducibility.* For the gamma-irradiated series, integrated TL intensity is plotted against absorbed dose. Linear regression is applied over the region where proportionality is observed, with  $R^2$  and residuals reported. Fading is assessed by reading identically irradiated samples after 1 h, 24 h, 7 days, and 30 days of dark storage. Reusability is evaluated through at least five anneal-irradiate-read cycles. These measurements distinguish a bright phosphor from a practically stable dosimetric phosphor, consistent with the criteria emphasized in rare-earth TL dosimetry studies such as Oza et al. (2015).

*Step 7 - Trap-parameter analysis.* Complex curves are deconvoluted into elementary glow peaks. For each component, activation energy ( $E$ ), frequency factor ( $s$ ), and kinetic order ( $b$ ) are estimated using peak-shape analysis and computerized glow-curve deconvolution. The analytical framework of Kitis et al. (1998) is appropriate because it accommodates first-, second-, and general-order kinetics and is suited to overlapping TL peaks. Fits should be accepted only when both statistical residuals and physical parameter ranges are reasonable.

*Step 8 - Statistical comparison.* Mean  $\pm$  standard deviation is reported for peak intensity, integrated area, and  $T_m$ . One-way or two-way ANOVA can be used to test the effects of dopant concentration and dose, followed by post-hoc comparisons where appropriate. The optimum composition is defined by a combination of high signal, a thermally stable dosimetric peak, low fading, reproducible cycling, and a wide linear response region rather than by maximum intensity alone.

*Step 9 - Quality assurance and uncertainty control.* Instrumental and sample-related uncertainties should be evaluated explicitly. The TL reader

temperature calibration is checked using a reference material or manufacturer calibration procedure, and the photomultiplier or detector response is monitored with a stable light source. Sample masses are held within a narrow tolerance and the same planchet geometry is used for all readings. Background curves from unirradiated samples are subtracted, while empty-planchet readings are used to identify heater or detector background. Irradiation geometry, source-to-sample distance, dose rate, and exposure time are recorded for every batch. At least three independently prepared aliquots are used for each principal composition so that repeatability is not confused with variation between separate synthesis batches. Where a peak shift is smaller than the temperature calibration uncertainty, it should not be interpreted as a real trap-depth change.

*Step 10 - Data reduction and decision rules.* The primary response variable is the integrated glow-curve area after baseline correction because it is less sensitive than peak height to small temperature shifts. Relative sensitivity is calculated by dividing the area of a test composition by the area of the reference composition measured at the same dose. Percentage fading is calculated as  $100 \times (I_0 - I_t)/I_0$ , where  $I_0$  is the initial integrated signal and  $I_t$  is the signal after storage time  $t$ . Reproducibility is summarized using the coefficient of variation across repeated cycles. For kinetic analysis, individual deconvoluted peaks are retained only when they are stable against small changes in the starting fit parameters and when fitted peak temperatures remain within the experimentally observed glow region. These rules reduce the risk of obtaining mathematically good but physically meaningless multi-peak fits.

**Table 2.** Proposed experimental factors and measurements

| Factor           | Proposed levels                               | Primary output                        | Purpose                     |
|------------------|-----------------------------------------------|---------------------------------------|-----------------------------|
| Dy concentration | 0, 0.5, 1.0, 2.0 mol%<br>(Eu fixed at 1 mol%) | $T_m$ , area, $I_m$ , $E$ , $s$ , $b$ | Locate optimum trap density |
| Gamma dose       | 0.1-10 Gy                                     | Integrated TL area                    | Dose-response linearity     |
| UV exposure      | 1-20 min                                      | Glow-curve intensity/shape            | Optical trap filling        |
| Heating rate     | 1, 2, 5, 10 °C s <sup>-1</sup>                | Peak shift and shape                  | Kinetic validation          |
| Storage time     | 1 h-30 d                                      | Residual TL signal                    | Fading stability            |

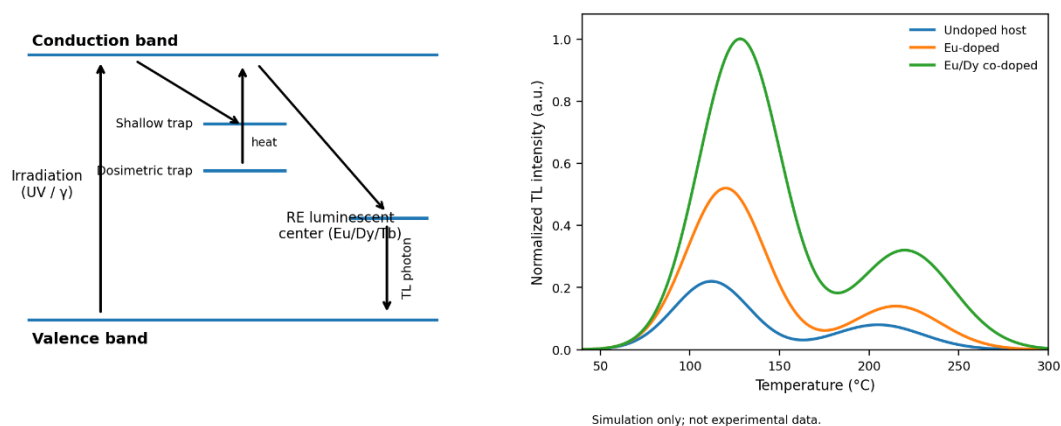


**Figure 1.** Conceptual thermoluminescence trapping/recombination process and illustrative simulated glow curves for a rare-earth-doped phosphor series. The plotted curves are schematic simulations, not experimental measurements.

## Results

Because this is a methodology paper rather than a report of newly performed laboratory measurements, the numerical glow curves in Figure 1(b) are illustrative simulations used to demonstrate the expected analysis workflow. The simulated sequence reproduces a commonly reported qualitative trend: an undoped host has relatively weak TL, addition of an emitting activator increases measurable recombination, and suitable co-doping can increase trap density and produce a stronger glow peak. This interpretation is consistent with the experimentally observed importance of Eu/Dy trapping in  $\text{SrAl}_2\text{O}_4$  systems (Mothudi et al., 2012; Zeng et al., 2018), but the plotted intensities should not be treated as literature-extracted values.

For the proposed experiment, the most informative result would be a composition-dependent maximum in integrated TL intensity rather than a monotonic increase with Dy content. At low dopant concentration, the number of useful traps is expected to increase; at excessive concentration, concentration quenching, competing defect complexes, or altered recombination pathways can reduce useful emission. Such an optimum has been reported in  $\text{Dy}^{3+}$ -activated calcium aluminate (Ziyouddin et al., 2016) and concentration-sensitive  $\text{Eu}^{3+}$  phosphors (İlhan & Keskin, 2018). Heating-rate measurements should shift  $T_m$  to higher temperatures as the ramp becomes faster, while deconvolution should reveal whether the apparent broad peak contains more than one elementary component. Multiple trap components are plausible from the behavior reported for Dy-doped haloborates and other rare-earth phosphors (Oza et al., 2015).

A successful dosimetric composition should show four linked features: a reproducible glow peak in a temperature region sufficiently above ambient to resist rapid fading; an integrated signal that is approximately proportional to dose over a useful interval; low cycle-to-cycle variation after standard annealing; and physically consistent trap parameters obtained from curve fitting. Structural results should be interpreted alongside TL data. For example, if annealing increases crystallite size and changes peak intensity or peak temperature, the change may reflect modified defect concentrations rather than the rare-earth ion alone, an effect highlighted in  $\text{SrSO}_4\text{:Dy,Tb}$  nanophosphors by Samariha and Saraee (2018).

Another expected outcome is a separation between high TL intensity and high dosimetric quality. A sample can produce a strong low-temperature peak yet still be unsuitable for storage dosimetry because shallow traps empty rapidly at room temperature. Conversely, a somewhat lower-intensity peak at a higher temperature may provide superior temporal stability. The proposed fading series is therefore essential for assigning practical significance to the glow curves. Comparing UV-filled and gamma-filled traps can also reveal whether the same defect population is populated by both excitation routes. If peak temperatures are similar but relative peak areas differ, the difference would suggest selective trap filling rather than formation of an entirely new defect during irradiation.

The kinetic parameters should be interpreted as an internally consistent set rather than isolated numbers. Activation energy describes the thermal depth of the trap, the frequency factor represents an attempt-to-escape rate, and the kinetic order reflects the balance between direct

recombination and retrapping. Agreement between peak-shape estimates and deconvolution-derived values would increase confidence in the assignment. Large discrepancies would trigger re-examination of baseline selection, thermal lag, or the assumed

number of component peaks. This analysis strategy follows the rationale of glow-curve deconvolution established by Kitis et al. (1998) and is especially relevant for rare-earth phosphors whose broad glow curves can mask several overlapping trapping processes.

**Table 3.** Illustrative simulated trend for the proposed SrAl<sub>2</sub>O<sub>4</sub>:Eu,Dy series (not experimental data)

| Composition     | Main T <sub>m</sub> (°C) | Normalized area | Expected fading | Interpretation                            |
|-----------------|--------------------------|-----------------|-----------------|-------------------------------------------|
| Undoped host    | 112                      | 0.22            | High            | Few useful traps                          |
| 1% Eu           | 120                      | 0.52            | Moderate        | Stronger radiative recombination          |
| 1% Eu + 0.5% Dy | 125                      | 0.78            | Lower           | Additional suitable traps                 |
| 1% Eu + 1% Dy   | 128                      | 1.00            | Low             | Illustrative optimum                      |
| 1% Eu + 2% Dy   | 131                      | 0.83            | Low-moderate    | Possible concentration/defect competition |

### Conclusion and Discussion

The reviewed evidence up to 2018 demonstrates that rare-earth doping changes thermoluminescence through two coupled mechanisms: modification of defect-related trapping states and provision of efficient luminescent recombination centers. A rigorous study must therefore control both composition and processing. Eu<sup>2+</sup>/Dy<sup>3+</sup>-doped alkaline-earth aluminates provide a particularly useful model because they combine strong emission with well-documented trap-mediated persistent luminescence. However, results from Dy-doped haloborates, calcium aluminates, SrSO<sub>4</sub> nanophosphors, and Eu<sup>3+</sup>-doped tantalates show that the same experimental logic is transferable to other hosts. The proposed methodology combines synthesis, phase verification, morphology, PL, controlled irradiation, glow-curve measurement, dose response, fading, reproducibility, and kinetic deconvolution. Its major strength is that it avoids identifying the “best” phosphor from peak height alone. Instead, the optimum material is selected using trap stability, dose linearity, low fading, repeatability, and physically meaningful activation parameters. The principal limitation is that TL parameters can depend strongly on reader calibration, heating rate, atmosphere, sample mass, and thermal history; cross-study comparisons therefore require standardized conditions. Future laboratory implementation should report all readout settings and should distinguish measured data from modeled or fitted quantities. Under these conditions, rare-earth-doped phosphors can be evaluated systematically for radiation dosimetry, radiation

sensing, persistent-luminescence devices, and defect-spectroscopy applications.

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