Optically stimulated luminescence and thermoluminescence in newly developed LiMgPO4:Gd

Authors: Tang, Kaiyong, Tang, Kaiyong

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Abstract

Five samples of LiMgPO4:Gd were prepared via five different production processes using a solid-state reaction method. The effects of the preparation process on optically stimulated luminescence (OSL) and thermoluminescence (TL) were investigated. Considering its high sensitivity, low fading, and minimum detectable dose (MDD), the LiMgPO4:Gd phosphor heated to 900°C for 15h is concluded to be optimal. The effects of annealing on the OSL sensitivity, relative residual OSL signals measured after 24h of irradiation, and MDD of LiMgPO4:Gd phosphors heated to 900°C for 15h were also investigated. Considering its high sensitivity, low fading, and MDD, annealing at 350°C for 1h is concluded to be optimal. The OSL signal of LiMgPO4:Gd was derived from the principal TL glow peak. For a maximum integration time of 5s, the OSL signal was stable, with no fading 30 days after irradiation. LiMgPO4:Gd eliminated approximately 2.2% of the OSL signal at each readout for a readout time of 0.1s, which is sufficient for fast and multiple OSL readout. The sensitivity of LiMgPO4:Gd phosphor, annealed for 1h at 350°C with a reading time of 0.1s, was found to be approximately 98% of that observed for α-Al2O3:C(TLD-500k), which should be sufficient for low-dose measurements in personal, workplace, and environmental dosimetry.

Full Text

Preamble

Optically stimulated luminescence (OSL) and thermoluminescence (TL) in newly developed LiMgPO4:Gd phosphors were investigated. Five samples were prepared via solid-state reaction using five different production processes. Based on sensitivity, fading characteristics, and minimum detectable dose (MDD), the LiMgPO4:Gd phosphor heated to 900 °C for 15 h was determined to be optimal. The effects of annealing on OSL sensitivity, relative residual OSL signals measured 24 h post-irradiation, and MDD were also examined

for this optimal phosphor. Annealing at 350 °C for 1 h yielded the best performance. The OSL signal originated from the principal TL glow peak. For integration times up to 5 s, the OSL signal remained stable with no fading observed 30 days after irradiation. At a readout time of 0.1 s, LiMgPO4:Gd eliminated approximately 2.2% of the OSL signal per readout, enabling fast and multiple OSL measurements. The sensitivity of LiMgPO4:Gd phosphor annealed at 350 °C for 1 h (with 0.1 s readout time) was approximately 98% of that observed for α-Al2O3:C (TLD-500k), sufficient for low-dose measurements in personal, workplace, and environmental dosimetry.

Keywords: Fading; LiMgPO4:Gd; Optically stimulated luminescence; Phosphors; Thermoluminescence.

Introduction

Optically stimulated luminescence (OSL) and thermoluminescence (TL) describe phenomena where irradiated materials emit luminescent signals upon exposure to appropriate optical or thermal stimuli [1–4]. TL is a well-established technology widely employed in various dosimetric applications, including personal, workplace, and environmental monitoring; medical dosimetry; ionizing radiation imaging; archaeological and geological dating; and radiation accident assessment [1, 5]. Currently, thermoluminescent dosimeters (TLDs) predominate in individual radiation exposure assessment, with LiF:Mg,Cu,P (GR-200A) being the most prominent example [9–13].

The fundamental principles of TL and OSL are identical, differing only in their stimulation method: TL uses heat, while OSL uses light [14]. This distinction confers several advantages to OSL [2, 15]. A major benefit is the ability to read signals at room temperature without heating the detector, enabling compatibility with plastic adhesives. In OSL mode, excitation light intensity and duration can be controlled to read only a fraction of the signal at a time [14], unlike TL where all signals are read simultaneously. This allows for multiple readout sessions and rapid measurements by discharging only a small percentage of the OSL signal per reading. In the InLight reader, each detector is stimulated for 1 s, with weak stimulation removing only 0.07% of the signal and strong stimulation removing 0.25% [14]. Consequently, several Individual Monitoring Service (IMS) laboratories have transitioned from TLD to OSL dosimeters [16].

Despite extensive research on new OSL materials, only two commercial OSL dosimeters based on Al2O3:C and BeO are available, in contrast to the significantly larger number of TL dosimeters [1]. α-Al2O3:C is an ideal OSL material with high stability and sensitivity. While many preparation methods exist, only crystal growth technology yields the highest OSL sensitivity [17–22]. However, carbon-doped alumina crystal growth is susceptible to condition fluctuations, causing considerable property variations between samples and batches [14]. To enhance uniformity, commercial Luxel+ and InLight dosimeters blend α-Al2O3:C powders with polyester on transparent film, though this

compromises the initial dose response linearity compared to the pure material [14]. BeO exhibits pronounced exponential sensitivity decline upon initial use [23] and post-irradiation signal fading [24].

Many countries are developing improved OSL materials, with numerous compounds reported [25–29], though most suffer from high fading—an unacceptable limitation for personal dosimetry and environmental monitoring as it causes substantial inaccuracies. For instance, Li2B4O7:Ag shows a minimum detectable dose of 15 µGy but experiences ~30% CW-OSL signal decay within 24 h post-irradiation [30]. CaSO4:Dy and CaSO4:Eu exhibit ~50% and ~10% decay after one day, respectively [31, 32]. Mg4SiO4:Tb and LiAl2O4:Tb show 30–40% intensity decline over ~20 h [33]. NaMgF3 doped with Ce, Tm, and Tb showed 20%, 13%, and 35% OSL response decreases after 1 day, 60 h, and 5 days, respectively [34, 35]. MgO:Li,Ce,Sm achieves an MDD as low as 0.2 µGy but shows 15% fading within the first hour [36].

This study aims to improve the temporal stability of LiMgPO4 OSL signals for fast, multiple readouts. Five LiMgPO4:Gd phosphor samples were prepared using five different methods. We investigated preparation effects on OSL decay curves, TL glow curves, sensitivity, MDD, and 24-h post-irradiation fading. The impact of annealing on OSL sensitivity, residual signals, and MDD was also examined for phosphors heated to 900 °C for 15 h. The fundamental characteristics of LiMgPO4:Gd annealed at 350 °C for 1 h were evaluated.

LiMgPO4 represents one of many promising OSL materials investigated globally [26, 37–50], but suffers from high fading. Previous studies attempted to minimize fading through dopant variation, impurity concentration adjustment, process modification, and prereadouts to eliminate low-temperature peaks [51]. Preheating or optical treatment can reduce fading but significantly decreases OSL intensity by 60–70% [51]. For LiMgPO4:Tb,Sm,B, preheating at 160 °C for 30 s reduced fading from 30% to ~7% after 30 days [52]. LiMgPO4:Tb,B showed 15–30% fading reduction, though the low-temperature peak remained problematic for personal dosimetry [53]. Varying preparation temperature significantly reduced fading in LiMgPO4:Tb,Sm,B, achieving 92.3% residual signal after 24 h [47]. Improved preparation processes also reduced fading in LiMgPO4:Er, with samples heated to 900 °C for 10 h showing <5% loss over 30 days when integration time $ $20 s, but >5% loss when integration time <20 s [41]. A study of LiMgPO4:RE (RE = Nd, Sm, Gd, Tb, Dy, Ho, Er, Tm) revealed that LiMgPO4:Er and LiMgPO4:Gd both exhibit broad 360 nm emission bands in TL spectra, with Gd showing higher intensity [39], though OSL characteristics of LiMgPO4:Gd remained unexamined.

Significant efforts have focused on developing OSL materials with Al2O3:C-like properties, including simple synthesis, readily available raw materials, low cost, and mass production suitability. The main challenge lies in achieving stable signals for rapid, multiple readouts.

Materials and Methods

A. Material Synthesis

LiMgPO4:Gd materials were synthesized using a simple solid-state reaction method. Reagents were weighed according to stoichiometric ratios: Li2CO3 (lithium carbonate), 3MgCO3·Mg(OH)2·3H2O (magnesium carbonate, 99.9%), and NH4(H2)PO4 (ammonium dihydrogen phosphate, 99.99%). These were thoroughly mixed, and Gd2O3 (0.25 mol%) [39] was added before final mixing. Five distinct processes were employed, divided into three stages where only the third stage varied.

In the first stage, the mixture was placed in an alumina crucible, heated in a tube furnace from room temperature to 300 °C over 30 min, held at 300 °C for 2 h, then rapidly cooled to room temperature. Samples were manually pulverized and sieved. In the second stage, the material was heated from 300 to 650 °C over 30 min and held at 650 °C for 2 h. For preparations 1–3, the third stage involved heating from 650 to 900 °C over 30 min, followed by holding at 900 °C for 10, 15, or 20 h, respectively. For preparations 4 and 5, the material was heated from 650 to 950 °C or 1000 °C over 30 min, then held at 950 °C or 1000 °C for 2 h, respectively.

For comparison, an α-Al2O3:C detector (TLD-500K) with 5 mm diameter and 0.9 mm thickness was used.

B. Characterizations

Powder structure was characterized by X-ray diffraction (XRD; Bruker D8 Advance). Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) using a Hitachi SU8600 confirmed Gd incorporation.

OSL decay curves, TL glow curves, and β-source irradiation were measured using a Risø TL/OSL reader (TL/OSL-DA-20), extensively documented elsewhere [41, 52]. Continuous-wave OSL (CW-OSL) measurements used 90% stimulus power for 88 s. TL glow curves were recorded from room temperature to 450 °C at 5 °C s^{-1}.

To evaluate preparation effects, five sample types were fully bleached via TL readout (pretreatment at 5 °C s^{-1} ramp to 450 °C), then their OSL background signals measured. Samples were irradiated for 2 s using a 90Sr/90Y β source (50 µGy s^{-1} nominal dose rate) to measure OSL decay curves, TL glow curves, and 24-h fading signals.

To assess annealing effects on OSL sensitivity, MDD, and 24-h fading, LiMgPO4:Gd powders synthesized at 900 °C for 15 h were annealed at 350–500 °C (50 °C intervals) for 1 h. Background signals were quantified post-annealing, followed by 2 s irradiation at 50 µGy s^{-1} and OSL measurement. Samples were then fully bleached (TL readout to 450 °C at 5 °C s^{-1}) and re-irradiated for 24-h post-irradiation OSL measurement.

Long-term fading was assessed using LiMgPO4:Gd synthesized at 900 °C for 15 h and annealed at 350 °C for 1 h. For kinetic order investigation, samples were fully bleached (5 °C s^{-1} to 450 °C), irradiated with 0.1 Gy using an in-situ β irradiator, and TL glow curves recorded at 450 °C. This was repeated at doses of 0.2, 0.5, 1, 2, 5, and 10 Gy (heating rate: 5 °C s^{-1}).

For experimental long-term fading assessment, samples were irradiated with 50 µGy from a 60Co gamma source, with OSL signals read at 0 days (~40 min) and 1, 2, 5, 10, 20, and 30 days post-irradiation.

Reusability was evaluated using five 10 mg LiMgPO4:Gd samples placed at different positions in the TL/OSL reader. After full bleaching (TL readout to 450 °C at 5 °C s^{-1}), samples were irradiated for 2 s at 50 µGy s^{-1} and OSL signals recorded; this cycle was repeated ten times.

Results

A. X-ray Diffraction (XRD)

Figure 1 [Figure 1: see original paper] shows XRD patterns of LiMgPO4:Gd samples from five preparation processes, compared with ICSD Card No. 201138. Measured diffraction patterns matched the reference for most reflection positions, though sensitivity differences may arise from preparation variations.

B. Elemental Analysis

Figure 2 [Figure 2: see original paper] presents map sum spectra confirming gadolinium presence in all five LiMgPO4:Gd samples.

C. Effect of Preparation Processes

Figure 3 [Figure 3: see original paper] illustrates preparation effects on LiMgPO4:Gd phosphors. Normalized OSL decay curves (Fig. 3a) show the 900 °C/15 h sample decayed slowest, while the 1000 °C/2 h sample decayed fastest; the remaining three showed similar profiles. Figure 3b compares normalized OSL signals (averaged over five readings, standard deviations <10%) across integration times, all lower than TLD-500k, with 900 °C/15 h showing highest sensitivity.

Relative residual OSL signals after 24 h (Fig. 3c) were stable across integration times for four samples, while the 1000 °C/2 h sample showed values of 0.701–0.880. MDD results (Fig. 3d) demonstrate significant preparation process impact, with relatively low MDD observed in the 950 °C/15 h sample.

TL glow curves and integrated signals (Figs. 3e and 3f) show the 900 °C/15 h sample exhibited highest TL sensitivity, while the 1000 °C/2 h sample showed lowest. Considering high sensitivity, low fading, and MDD, the 900 °C/15 h LiMgPO4:Gd phosphor is optimal; subsequent studies focused on this material.

D. Effect of Annealing on LiMgPO4:Gd

Figure 4 [Figure 4: see original paper] shows annealing effects. OSL signals (Fig. 4a) of samples annealed at 350, 400, 450, and 500 °C for 1 h were normalized to unannealed TLD-500k (mean of five samples, standard deviations <10%). Annealing enhanced OSL sensitivity, with 350 °C/1 h showing highest sensitivity; sensitivity decreased with higher annealing temperatures. At 350 °C, initial and total OSL signals matched TLD-500k, with initial OSL signal at ~0.98 relative to TLD-500k.

Relative residual OSL signals after 24 h (Fig. 4b) were highest for unannealed phosphor. For annealed samples, signals were stable at integration times <10 s. Annealing significantly reduced MDD (Fig. 4c), achieving 2.2 µGy at 1 s integration and 16.2 µGy at 0.1 s for 350 °C/1 h annealing. Considering high sensitivity, low fading, and MDD, 350 °C/1 h annealing is optimal; subsequent studies used this condition.

E. Basic Properties of LiMgPO4:Gd

1. TL Dose Response and Kinetic Order Figure 5 [Figure 5: see original paper] shows dose, heating rate, and OSL readout effects. Glow curves (Fig. 5a) for 0.1–10 Gy irradiation (heated to 450 °C at 5 °C s^{-1}) show peak temperature consistently at 131.4 ± 1.1 °C across doses, indicating first-order kinetics [5]. Dose response (Fig. 5b) demonstrates excellent linearity from 0.1 to 10 Gy.

2. TL Kinetics Parameters Five glow curves recorded at 0.2–5 °C s^{-1} heating rates (Fig. 5c) show peak temperature increasing and peak/total integral signals decreasing with higher rates. The variable heating rate (VHR) method [5] was applied to obtain kinetic parameters. For first-order kinetics, the relationship between peak temperature (Tm) and heating rate (β) is:

$$\ln\left(\frac{T_m^2}{\beta}\right) = \frac{E}{kT_m} + \ln\left(\frac{E}{ks}\right)$$

where E is activation energy, k is Boltzmann constant, and s is pre-exponential factor. Plotting $\ln(\mathrm{Tm}^2/\beta)$ versus 1/Tm (Fig. 5d) yields E = 1.44 eV and s = 4.82×10^{17} s^{-1}.

3. TL after OSL Comparison of TL glow curves measured immediately after irradiation versus post-OSL readout (Fig. 5e) shows the immediate peak height was ~14× higher. The TL peak area after OSL was 8.5% of the immediate irradiation area (50–250 °C range), confirming OSL signal originates from the principal TL glow peak, with OSL readout eliminating most associated traps.

4. OSL Decay Curve Figure 5f compares mass-normalized decay curves for LiMgPO4:Gd and TLD-500k. LiMgPO4:Gd decayed faster initially, then

slower than TLD-500k, causing relative sensitivity to decrease then increase with measurement time. At 0.1 s readout, LiMgPO4:Gd and TLD-500k eliminated ~2.2% and ~2.9% of OSL signal per readout, respectively.

5. OSL Long-Term Fading Table 1 presents relative residual OSL signals over time and integration time. For integration times $ $5 s, signals remained stable with no fading 30 days post-irradiation. Data represent mean values from five aliquots.

6. Reusability Table 2 shows normalized OSL signal repeatability across ten cycles. Coefficient of variation was <0.8% at all integration times, demonstrating excellent reproducibility.

Discussion

A. Sensitivity

Radiation sensitivity is crucial for OSL materials in low-dose personal, workplace, and environmental dosimetry. While IEC 62387 does not specify sensitivity requirements for integrated passive detectors, only mandating dose linearity floor at 0.1 mSv for the complete system, novel material assessment typically involves comparison with established commercial materials under identical conditions [1].

OSL dosimetry offers two key advantages over TL: rapid readout and multiple readout capability. Rapid processing benefits high-throughput applications, while multiple readouts prevent data loss and preserve evidence. To enable these features, only minimal OSL signal portions should be read per measurement. The InLight OSL Reader achieves this by removing just 0.07% (weak stimulus) or 0.25% (strong stimulus) per reading [14].

In this study, LiMgPO4:Gd eliminated ~2.2% of OSL signal per 0.1 s readout, enabling multiple readouts. The initial OSL signal (sensitivity) of LiMgPO4:Gd annealed at 350 °C for 1 h was ~98% of TLD-500k, sufficient for low-dose measurements in personal, workplace, and environmental dosimetry.

B. Fading

OSL fading—the temporal response change post-irradiation—poses a greater challenge than TL fading. While TL fading can be addressed by spectral peak separation and unstable peak removal, OSL fading involves overlapping signals from multiple traps with different characteristics. Preheating can mitigate OSL fading but contradicts OSL' s advantage of rapid, room-temperature processing. Developing intrinsically stable OSL materials is the optimal solution.

Preparation process modifications significantly improve OSL fading. For MgB4O7:Ce,Li, fading varied from 10–15% over 72 h [55] to <1% over 40 days [56] depending on synthesis route. LiMgPO4:Tb,B powder showed ~20–25%

fading after three weeks, while crystalline samples exceeded 50% within 24 h [42]. LiMgPO4:Er samples heated to 900 °C for 10 h exhibited <5% loss over 30 days (integration time $ $20 s), but ~12% loss at 0.1 s integration [41].

For LiMgPO4:Gd, the 1000 °C/2 h sample showed 24-h residual signals of 0.701–0.880, while other samples were stable across integration times. The preparation process dependence was less pronounced than for LiMgPO4:Er. No fading was observed within 30 days for integration times <5 s, though ~18% total fading occurred by day 30. To prevent fading, LiMgPO4:Er requires $ $20 s integration time [41], which is impractical for rapid/multiple readouts. LiMgPO4:Gd achieved stable signals at 0.1 s integration, enabling fast, multiple OSL readouts.

C. Superiority of LiMgPO4:Gd

Table 3 compares properties and economics of α-Al2O3:C (TLD-500K) and LiMgPO4:Gd. Based on this study's results (Figs. 3b, 4a, and 5f), LiMgPO4:Gd offers comparable OSL sensitivity, effective atomic number, and fading characteristics suitable for fast/multiple measurements, with advantages in preparation simplicity and cost.

LiMgPO4:Gd is significantly easier to prepare than α-Al2O3:C. α-Al2O3:C' s high melting point (2050 °C) and challenging carbon doping conditions limit optimization scope and cause property variations within and between crystals. Commercial dosimeters blend α-Al2O3:C powders with polyester to improve homogeneity, but this reduces sensitivity compared to crystals and compromises initial dose response linearity. Using LiMgPO4:Gd in similar dosimeter configurations may offer sensitivity advantages. LiMgPO4:Gd matches Al2O3:C performance while being easy to synthesize, inexpensive, and suitable for large-scale production.

Conclusions

Five LiMgPO4:Gd phosphor powders were prepared via solid-state reaction using five production processes. XRD confirmed structures. Preparation effects on OSL decay curves, TL glow curves, sensitivity, MDD, and 24-h fading were investigated. The 900 °C/15 h phosphor showed highest OSL sensitivity. While the 1000 °C/2 h sample exhibited 24-h residual signals of 0.701–0.880, other samples were stable across integration times. Fading dependence on preparation was less pronounced for LiMgPO4:Gd than LiMgPO4:Er. Considering high sensitivity, low fading, and MDD, the 900 °C/15 h phosphor is optimal.

Annealing effects on OSL sensitivity, 24-h residual signals, and MDD were examined for the 900 °C/15 h phosphor. Annealing enhanced sensitivity, with 350 °C/1 h showing highest performance. Annealed samples were stable at integration times <10 s, and annealing significantly reduced MDD. Considering these factors, 350 °C/1 h annealing is optimal.

Basic properties of 350 °C/1 h annealed LiMgPO4:Gd were characterized. Dose

response was linear from 0.1–10 Gy. The TL glow peak exhibited first-order kinetics with E = 1.44 eV and s = 4.82×10^{17} s^{-1}. The OSL signal originated from the principal TL glow peak. At integration times \$ \$5 s, signals remained stable with no fading 30 days post-irradiation. At 0.1 s readout, ~2.2% of OSL signal was eliminated per readout, enabling fast/multiple measurements. Sensitivity was ~98% of TLD-500k, sufficient for low-dose personal, workplace, and environmental dosimetry.

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Note: Figure translations are in progress. See original paper for figures.