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Research Article

# OSL Dosimetry Using $LiCaAlF_6$:Eu Phosphor

Aditya Sahare[1], Abhishek[2], Siddharth Sahare[3], Manveer Singh[4] and P.D. Sahare*[4]

[1] *Department of Chemical Engineering, National Tsing Hua University, Hsinchu 300044, Taiwan*
[2] *Institute of Technology and Management, Technische Universität Berlin, Berlin, 10623, Germany*
[3] *Department of Physics, Hansraj College (University of Delhi), Delhi 110007, India*
[4] *Department of Physics & Astrophysics, University of Delhi, Delhi 110007, India*
***Corresponding author:** pdsahare@physics.du.ac.in

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**Abstract.** $LiCaAlF_6$:Eu (0.1 mol%) OSLD phosphor material in microcrystalline powder form was prepared in a two-step process. First, through co-precipitation method and second by quickly melting it in a graphite crucible at around 900 °C. It is a very novel and economic method of preparing this phosphor. The formation of the material in a single phase was characterized by XRD. Optically stimulated luminescence (OSL) and photoluminescence studies were done for its application in radiation dosimetry of high-energy. The materials were irradiated to different doses of $\gamma$-rays using $^{60}$Co radiation source and optically stimulated luminescence (OSL) decay curves were recorded. The powder material was found to be at least four times more sensitive than $Al_2O_3$:C (Landauer USA) commercially available OSL chips. Considering these facts, the material could be considered as a highly sensitive and suitable OSL phosphor.

**Keywords.** $LiCaAlF_6$:Eu, Optically stimulated luminescence (OSL), High sensitivity, Dosimetry, OSLD phosphor

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## 1. Introduction

Thermoluminescence is a widely used technique for monitoring high-energy radiations like, UV, X-rays $\beta$-rays, $\gamma$-rays and neutrons, etc. as they are hazardous to living beings. In spite of this, we need to make use of such radiations for research, medical diagnostics, power generation using nuclear reactors and so on. This is a relatively simple technique where the phosphor material after getting exposed to radiation could simply be stimulated by heating (thermal energy). The dosimeters are thus passive devices and could be used as cards, rings, necklaces, etc. and hundreds of such devices could be coded and handled easily [2, 3, 5]. However, there are certain drawbacks of this technique. For example, there could be permanent damage on repeated irradiation and/or change in the sensitivity due to other processes, such as, phase change, redox reactions due to repeated heating/cooling during readouts [6, 7, 9]. To avoid heating/cooling during readouts, the material could be stimulated optically, i.e., by blue, green or even by infra-red light by using certain filters to block the stimulating light and pass only the signal to the optical detector. The technique is called as optically stimulated luminescence technique (OSL). Other advantages are technique could be used as continuous-wave optically stimulated luminescence (CW-OSL) or pulsed-OSL (POSL), linearly modulated-OSL (LMOSL). It could also be used as optical fiber aided OSL technique used for distributed radiation sensing applications or OSL imaging [4, 10]. Nevertheless, as this technique is relatively newer, there are not many OSLD phosphors available and only two phosphors, i.e., $Al_2O_3$:C (Luxel$^{TM}$, Landauer, USA) and BeO (Thermalox 995, Materion, USA) are available commercially and they are expensive due to need of sophisticated instrumentation for producing. $LiCaAlF_6$ is a versatile and excellent host material for various applications, such as, windows material in vacuum ultraviolet (VUV) region due to its large band gap, active media for lasers, scintillators and radiation dosimetry after doping with suitable impurities [1, 3, 4, 6–10]. In the present paper, we have successfully synthesized a new OSLD material $LiCaAlF_6$:Eu (0.1 mol%), characterized and studies its dosimetry properties.

### Highlights

- OSLD phosphor material was synthesized successfully by coprecipitation/melt method.
- The novelty of the method of preparation is that it was prepared in a single phase which is otherwise difficult.
- The material was found to be in a single phase XRD studies.
- The material was found to be four times more sensitive than the standard $Al_2O_3$:C OSLD chips.
- The material is found to be a highly sensitive OSLD material.

## 2. Experimental

The detailed method of preparation could be found elsewhere [1]. It is also given here in short. The materials $LiCaAlF_6$ doped with rare earth impurity Eu (0.05-2.0 mol%) were prepared by two step method, in the first step through wet chemical method, where analytical reagent grade chloride compounds of constituent elements (i.e., LiCl, $CaCl_2$ and $AlCl_3$) were dissolved in their stoichiometric ratios. Then the stoichiometric amount of ammonium fluoride ($NH_4F$) was added slowly dropwise (at the rate of 5.0 ml/min) while stirring rigorously. All the utensils (beaker, burette, etc.) used were made of Polytetrafluoroethylene (Teflon) as the reaction involves very reactive ions like Li and F. The precipitate was washed several times with distilled water/ethanol and dried in a vacuum oven at ~70 °C overnight. However, material prepared by this method was not found to be in a single phase, therefore, in a second step, it was then rapidly heated in a graphite crucible in air till the powder completely melted (at ~900 °C). The melt was then quickly quenched by pouring it into another graphite crucible to get it in as a single phase.

The characterization of the material was done by PXRD. The PXRD patterns were recorded using a high-resolution X-ray diffractometer (model D8 Discover, Bruker, Germany) equipped with a point X-ray detector (i.e., scintillation counter). Cu-K$_\alpha$ radiation line ($\lambda$ = 1.54056 Å) was used to obtain the PXRD patterns. The PXRD patterns were recorded at the scan rate of 1.0 s/step and the step size of 0.013 s. Luminescence studies, i.e., Optically stimulated luminescence (OSL) were done on a TL/OSL Reader (Nucleonix Systems, Hyderabad, India, Model TL/OSL-1008). Optical annealing was done in a light-tight cabinet (home-made) consisting of 15 blue LEDs and having ~300 mW power.

## 3. Results and Discussion

### 3.1 X-Ray Diffraction (XRD)

The PXRD patterns for the LiCaAlF$_6$:Eu phosphor material are the same as given in another paper [1]. It could be seen there that PXRD pattern matches well with the JCPDF data found in the literature (JCPDF file # 73-2441). This also shows that the material prepared by two step method of preparation is in a single phase.

### 3.2 Decay Curves and Dose Response

Figure 1 shows the dose response of the material annealed at 200 °C. The dose response is very much linear the dose range of 5-50 Gy. The CW-OSL decay curves of the material irradiated for different doses are also shown in the figure. It could be seen in the inset of the figure that the dose response is quite linear in this dose range.

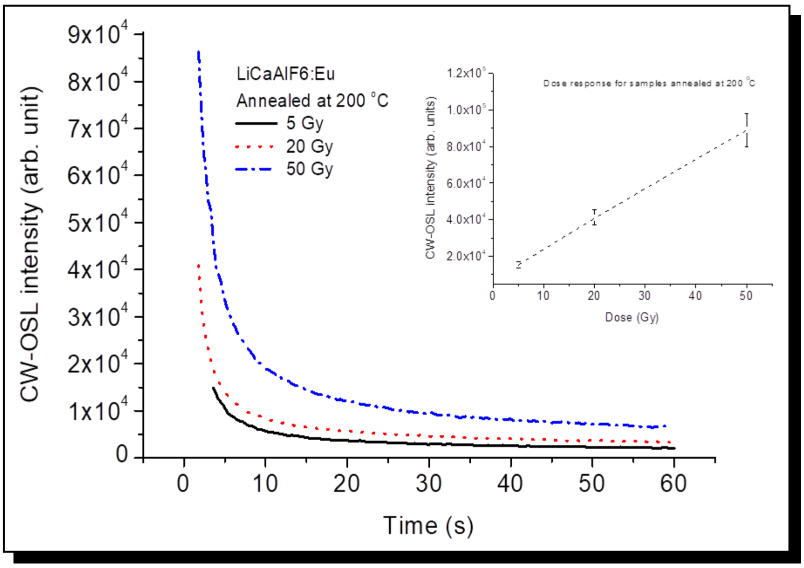


**Figure 1.** Dose response of LiCaAlF$_6$:Eu OSLD phosphor material annealed at 200 °C. The particle size of the material was in the range of 63-90 $\mu$m

### 3.3 Comparison of OSL Response of the LiCaAlF$_6$:Eu With Standard Al$_2$O$_3$:C Chips

The powder LiCaAlF$_6$:Eu phosphor material was irradiated to 5.0 Gy dose of $\gamma$ ray dose from $^{60}$Co with standard Al$_2$O$_3$:C chips The OSL decay curves are as shown in Figure 2. It could be seen in the figure that the LiCaAlF$_6$:Eu phosphor material under investigation is at least four times more sensitive than Al$_2$O$_3$:C (Landauer USA) commercially available OSL chips.

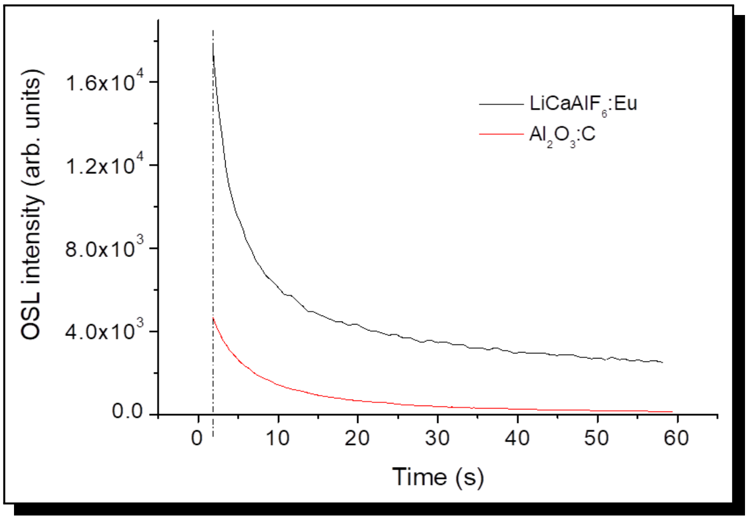


**Figure 2.** Comparison of OSL response of the LiCaAlF$_6$:Eu with standard Al$_2$O$_3$:C chips

### 3.4 Fading

The fading study of the material it was irradiated for nominal dose of 10 Gy and stored in a light tight container at room temperature. The OSL measurements were done at different intervals of time. The measured OSL intensity was plotted with time and is as shown in Figure 3. It could be observed from the figure that approximately 15% fading is observed in two weeks.

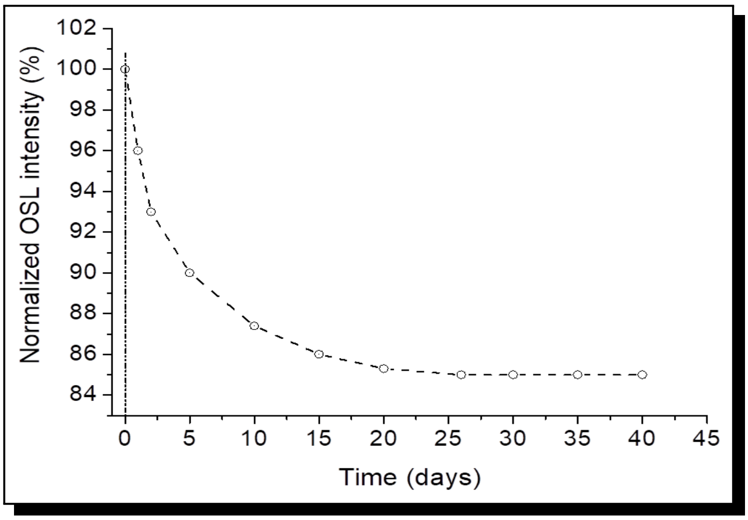


**Figure 3.** Fading for LiCaALF6:Eu OSL phosphor. The material was irradiated for 10 Gy dose and stored in dark at room temperature

### 3.5 Optical Annealing and Reusability

As mentioned earlier, OSL readouts as well as annealing (for making the detector ready for its reuse) is done optically. For optical annealing, a light-tight cabinet (home-made) consisting of 15 blue LEDs and having ~300 mW power at the sample was used. The material (after OSL readouts) was annealed for 2.0 h at room temperature. The readout was taken once again to ensure that all the optically active traps generated on irradiation are emptied. The material was reused several times and no appreciable change in the OSL intensity was observed after its reuse.

## 4. Conclusion

The material was synthesized successfully by a simple coprecipitation method and then melt-quenched in to form the material in a single phase with the desired dosimetric properties. The material was initially annealed thermally in an oven at around 200 °C for better sensitivity. It was characterized by XRD to confirm its formation and crystal structure and fond that it is in a single phase. Its OSL characteristics were studied and found that it is at least 4 times more sensitive than the commercially available $Al_2O_3$:C OSLD phosphor with around 15% fading in two weeks. It may be noted here that there are only two commercially available OSL detectors, i.e., $Al_2O_3$:C (Luxel$^{TM}$, Landauer, USA) and BeO (Thermalox 995, Materion, USA). So, in absence of many such commercially available OSLDs, the newly studied OSL phosphor could be a good candidate for the dosimetry of high-energy radiations like X-rays, $\gamma$-rays, $\beta$-rays, etc.

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### Competing Interests

The authors declare that they have no competing interests.

### Authors' Contributions

All the authors contributed significantly in writing this article. The authors read and approved the final manuscript.

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