



# Synthesis and characterization of flexible thermographic phosphor temperature sensors



Katherine E. Mitchell <sup>a</sup>, Victor Gardner <sup>b</sup>, Stephen W. Allison <sup>c</sup>, Firouzeh Sabri <sup>a,\*</sup>

<sup>a</sup> Dept. of Physics and Materials Science, University of Memphis, Memphis, TN 38152, USA

<sup>b</sup> White Station High School, Memphis, TN 38117, USA

<sup>c</sup> Emerging Measurements, Collierville, TN 38017, USA

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## ABSTRACT

The temperature dependence of the emission characteristics of thermographic phosphors has been used extensively for surface temperature measurements of systems where thermal management is critical for the safe operation of the system. The instantaneous, remote, and highly accurate nature of this form of temperature measurement makes it a very attractive measurement technique. However, the destructive nature of depositing phosphors directly onto the surface of interest and the complications of working with fine powders has limited the use of this technique in all areas. This work focuses on the design and characterization of polymer-encapsulated thermographic phosphor flexible sensors for surface temperature assessment.  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  powder was embedded in an elastomeric sleeve at concentrations of 10%, 25%, and 50% wt. and fully characterized. The effect of spin-coating on emission characteristics of  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  was tested and the decay times were compared to results obtained from bulk-doped samples previously created by the authors.

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## 1. Introduction

Thermographic phosphors are ceramics consisting of rare earth oxides vanadates, phosphates, or oxysulfides which, when excited, emit a spectrum characterized by unique emission lines and both the intensity and fluorescence lifetime of these lines vary with temperature [1,2]. Thermographic Phosphor Thermometry (TPT) offers significant advantages over other temperature sensing means such as resistive heating and the Seebeck effect, as examples [3,4]. In the case of solid-state based thermometry, shot noise and tunnel junctions can be significant at the nanoscale and can create inaccuracies in temperature readings that are based on electrical resistance readings [5]. Advantages of phosphor thermometry include wide temperature range of operation-cryogenics [6] to 1800 °C [7], remotely accessible, up to 0.05 °C accuracy (for some compounds), low cost, and high versatility. However, in most cases, for the TPT method to be implemented in surface temperature reading effectively, the compound must be sprayed directly onto

the surface of interest as a thin uniform film or with a mixture of an adhesive agent [8–10]. This can create surface imperfections, on the surface that is being coated and in some cases applying a thin film coating can be extremely difficult. Finally, the phosphors that are used in this manner are no longer reusable. In an attempt to create a non-destructive means to assess surface temperature, recent publications [2,11,12] have demonstrated the feasibility of TPT with the phosphor particles encapsulated within an elastomeric polymer casing with no adverse effects on the emission characteristics and temperature response of the thermographic phosphors used. Our previously reported work concerned thick samples. A logical next step, reported here, was to proceed to thin samples. For thermometry purposes, thin samples will conform to curved surfaces. Moreover, the samples will equilibrate more quickly, resulting in more accurate temperature measurements.  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  was chosen for this work because of its high sensitivity, availability to the authors, and ease of excitation with inexpensive light sources. A further advantage is that the luminescence is comprised of both temperature-dependent bands and temperature-independent bands. In this work, the authors present the first thin-film  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  thermal sensors that are reusable, detachable, and geometrically conformable. Spin-coating techniques were implemented in order to create thin flexible film

\* Corresponding author.

E-mail addresses: [kmtchl12@memphis.edu](mailto:kmtchl12@memphis.edu) (K.E. Mitchell), [steve.allison@emergingmeasurements.com](mailto:steve.allison@emergingmeasurements.com) (S.W. Allison), [fsabri@memphis.edu](mailto:fsabri@memphis.edu) (F. Sabri).

sensors containing 10, 25, and 50% wt of  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$ .

This work was in part motivated by the possible application of this form of thermometry to cold chain shipping. The cold chain refers to the series of locations of an item while in the custody of the shipper as it is moved from its origin to its destination. For many vaccines and products the constraint is shipment within a range of 2–8 °C. This phosphor can indicate temperature over a wide range within, above, and below this region [2,11]. However, previous work with this phosphor involved either high power lasers and/or thick layers of phosphor. For some practical applications it may be desirable to use inexpensive but low power light emitting diodes or laser diodes. Thus, signal levels would be lower. In conjunction with this is the attractiveness and utility of removable and flexible phosphor-doped films of varying concentrations and thicknesses, also possibly degrading signal levels. However, for this phosphor, signal levels and decay times increase as temperature is lowered, hence the impetus for this investigation. The temperature measurements reported here in this rather narrow range are viewed as a stepping stone to applications that extend both below and above this stated range.

## 2. Materials and methods

### 2.1. Sample preparation

Composite polymer samples containing different concentrations of lanthanum oxysulfide  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$ , 0.1 mol% powder were prepared by combining Sylgard 184 (10:1 base to cross-linker ratio) with  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  powder acquired from phosphor technology SKL63, lot 15010. Appropriate amounts of  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  were added to the polymer mix gradually, prior to out gassing and curing to create 10%, 25%, and 50% wt.  $\text{La}_2\text{O}_2\text{S}:\text{Eu}$  concentration samples. The slurry was mixed vigorously and thoroughly for 2 min and then out-gassed in a precision 3-M vacuum oven at room temperature. No anti-clumping agents were used in this study. The slurry was dispensed onto a sacrificial layer (rimless aluminum pans) supported on the vacuum chuck of a Chemat Scientific spincoater and then spun at 1000, 1500, and 2000 rpm for each concentration, for 18 s (optimized experimentally). All samples were placed in the oven on a flat surface and cured at 100 °C for 1 h. After the samples were cooled down to room temperature, the cured samples were carefully peeled away from the sacrificial support layer. For control, 10%, 25%, and 50% samples were also prepared using the drop-casting method, whereby the mixture is allowed to disperse without the aid of spincoating. Sample thickness was determined by placing each film between two 1 mm thick microscope slides and measuring total thickness 3 times with a caliper. The film thickness was calculated by subtracting the thickness of the slides from the total value measured and taking the average of the three measurements.

### 2.2. UV–Vis characteristics

The transmission/absorbance behavior of the thin film composite samples prepared in the previous section was characterized by means of an Evolution 220 Thermoscientific spectrophotometer. Each polymer sample was placed on a quartz slide for support and scanned from 800 nm to 200 nm at a scan rate of 250 nm/min for each sample, at room temperature. The spectra were collected from different locations of each sample.

### 2.3. Luminescence characteristics

The arrangement for quantifying the fluorescence consisted of a 405 nm laser diode, gently focused to about 1 mm and aimed at the

target at about a 30° angle from the vertical. The photomultiplier detector viewed the target sample from directly above and at a distance of about 120 mm. An aluminum plate with an opening of about 25 mm<sup>2</sup> was attached to a table and protruded beyond the edge by about 40 mm. The spin-coated specimen was spread as evenly as possible over a metal washer with a 15 mm<sup>2</sup> hole with just enough tension such that the material laid flat and smooth. The laser was guided as close as possible to the center of the washer and aluminum opening. Each sample was mounted in this way in order to have as close to identical conditions as possible over the opening in order to minimize the possibility of reflection. The surface underneath the sample was 0.5 m below; therefore, back reflection from the surface was negligible. A Tektronix TDS 2012C digitized the detector output and communicated with a data acquisition laptop for data saving and analysis. Temperature dependence measurements were taken at the range of temperatures associated with cold-chain biologistics, 2 °C–8 °C, a current area of interest. A 50% doped Sylgard 184 sample spun at 1000 rpm was attached to the smooth surface of a glass pedestal placed in an insulated ceramic container. The container was filled with ice water to just above the sample surface. A thermocouple was placed within a few millimeters. Temperature increase during the course of a single measurement was negligible. For this temperature-dependence test, the excitation source was a 365 nm light emitting diode (LED) positioned at about a 30° angle as described above.

In the present work, a 405 nm laser diode was used to quantify the fluorescence of the samples, while a UV LED was used for temperature-dependence characterization. Each light source was selected for its efficacy in the specific measurement context: the laser diode produces a small, well-defined beam, while the UV LED produces a more efficient fluorescence.

The decay time of the luminescence is fit to a single exponential equation plus a constant (B):

$$I = I_0 e^{-t/\tau} + B$$

where  $I$  is the instantaneous luminescent intensity,  $I_0$  the initial luminescent intensity when the light source is terminated,  $t$  is time, and  $\tau$  is the characteristic decay, the time it takes for the luminescent intensity to decay to  $1/e$  of  $I_0$ .  $B$  is a constant offset which may be comprised of, for instance, background light, detector noise, and other phenomena. Decay times were obtained with a regression routine packaged with SigmaPlot software.

Ultraviolet excited fluorescence spectra were obtained using a StellarNet Blue-Wave microspectrometer, UVN-200, and accompanying SL3 Deuterium Lamp with a filter blocking light longer than 400 nm. The excitation is broad band from 250 to 400 nm with characteristic peaks at about 280 and 380 nm. An optical fiber delivered the excitation light to the sample that was placed at a distance of 1 cm. A receiving fiber collected the light. The lamp was on continuously and each spectrum was acquired for a total duration of 0.5 s.

### 2.4. Cluster size distribution

Images of the centers of the thin-film samples were taken under an Accu-Scope Unitron microscope using an AmScope MU1000 microscope digital camera. Using AmScope image software, the contrast on the images was increased until the surface phosphor was easily visible against the Sylgard 184 background. The images were then converted to binary format, with the phosphor particles as black and the Sylgard 184 as white. Data concerning the percent of each sample's surface covered in phosphor were gathered by determining the percent of the image's pixels that were black. Data concerning the average size of each phosphor particle were

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