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Influence of Coating Thickness on Thermographic Phosphorescence Diagnostics Developed for Optical Engine Applications

T.C. Chi[1], S. Kook[1,*], Q.N. Chan[1], and E.R. Hawkes[1]

[1]School of Mechanical and Manufacturing Engineering
The University of New South Wales, Sydney, NSW 2052, Australia

Abstract

Thermographic phosphorescence measurements are widely used as a non-invasive technique to investigate the changes in the wall temperature distribution of the pistons and engine cylinders. However, the performance of this technique is highly sensitive to the phosphor layer coating process and method of adhesion, which requires an extensive standardised approach for accurate and reliable results. This paper presents the optimisation of phosphor coating thickness for thermographic phosphorescence diagnostics developed for optical engine applications. Based on literature review, europium-doped lanthanum oxysulphide (La2O2S:Eu) was selected as a phosphor material due to its high temperature sensitivity at engine relevant conditions of 0~300°C, which could be excited by 355 nm light with strong phosphorescence emissions at 615~625 nm. Using a water-based magnesium aluminium silicate binder and a central pneumatic air brush, phosphor coating with various thicknesses were formed on glass plates, which were imaged in a scanning electron microscope (SEM) for thickness measurements before being tested for the phosphorescence signal delay in a purpose-built calibration rig. It is found that, for a given optical setup, the phosphorescence emission signal at fixed wall temperature increases with increasing coating thickness with 11.9 µm being the minimum layer thickness required for measurable signals. However, the thick 19.0-µm coating shows lower signal intensity than the 14.3-µm layer because of the increased distance between the high-temperature wall and phosphors. This optimised phosphor thickness will be applied to optical engines in future studies.

Introduction

Flame-wall interaction occurs in all engines, as the fuel jet impinges and then spreads out over the wall of the combustion chamber, which makes a significant impact on engine efficiency due to variations in heat transfer. For instance, the heat loss occurring through the combustion chamber wall accounts for about 20~30% of the total heat released from combustion [1]. Therefore, the need is clear for the development of wall temperature measurement techniques for engine applications. One of the simplest temperature measurement methods is to use fast-response surface thermocouples [9]. However, an individual thermocouple only measures temperature at a single point and is intrusive to install on moving engine parts such as the piston surface. The thermocouple-based temperature measurements are also rather complex since the electrical signal needs to be retrieved in the fast-moving parts.

Thermographic phosphorescence measurements have been proved to be a good alternative, with high spatial and temporal resolution while maintaining good accuracy and precision [1-5]. Exposing the phosphors to light sources such as high-pulse energy lasers can excite its electronic energy level to a higher state. Upon returning to the ground state level of electronic energy, it emits phosphorescence which corresponds to the difference in the energy level or by non-radiative relaxation [3]. Since the probabilities for the radiative and non-radiative transitions depend on the temperature of the phosphor, the phosphorescence spectrum, lifetime and intensity can be used in surface thermometry [1].

An important part of the phosphorescence diagnostics is forming a thin coating of phosphor materials. It is that the thinner the layer the closer in contact it is with the piston surface, the more accurate the coating is in reflecting the original surface temperature. Past studies used a phosphor coating with 20 µm layer thickness or lower for the temperature range to that of a typical engine (e.g. 0~200°C) because films thicker than 20 µm showed an intrusive effect on the surface temperatures [6, 8]. However, there is often a trade-off between layer thickness and durability of the coating to withstand the harsh environment of the engine such as thermal shocks and vibrations [7].

In this paper, the phosphor coating thickness and its effect on phosphorescence signal intensity is investigated on quartz glass surface in consideration of the future optical engine applications. The phosphor coating thickness was measured using a scanning electron microscope (SEM) and in-house developed image post-processing codes. The coatings with various thicknesses were then tested in a calibration rig to obtain phosphorescence signal intensity decay curves and calibration graphs.

Phosphor Coating Method

Selected Phosphor Material

Based on literature review [1-5], europium doped lanthanum oxysulphide (La2O2S:Eu) was selected due to the following reasons. Firstly, the temperature sensitivity of La2O2S:Eu is high in the 0~200°C range, typical cylinder wall temperature. Secondly, regarding the pressure effects, the decay time of La2O2S:Eu is only affected with the application of 3.5 GPa [3], which is an order of magnitude greater than our engine operation limits (<20 MPa). Thirdly, variations in oxygen, nitrogen, helium, carbon dioxide, water vapour, and methane do not affect the phosphor decay time [4]. In consideration of all the preceding factors, La2O2S:Eu was selected as a phosphor material of the present study.

Phosphor Binder and Coating Method

Preliminary testing was first performed using the ceramic adhesive (Aron Ceramic Type C) as the phosphor binder which was applied onto glass plates, as shown in figure 1 (left). This technique is advantageous because the coating is opaque enough to block the background flame luminescence as well as durable enough to withstand repeated engine cycles without being burnt off or compromised when removing soot deposition after each firing

Figure 1. Phosphor ceramic adhesive coating cracking after 150°C (left) and water-based silicate paint binder coating with 10 airbrush-sprayed layers cured at 200°C (right).

cycle. Similar to previous studies (e.g. [2]), a phosphor by mass of 5% was mixed with the ceramic adhesive (95%) for the clearest emission resolution at high temperatures where the phosphorescence signal is weakest. This ratio was kept constant throughout the experiment.

However, the ceramic adhesive coating technique showed limited temperature ranges; the prolonged exposure of the coating to temperatures of 100°C detached the layer from the glass plate and the crack occurred at 150°C as shown in figure 1 (left). This is mainly due to the thermal expansion coefficient difference between the ceramic adhesive and the glass plate. That is, the coating expanded at a greater rate than the glass causing subsequent detaching and cracking of the test sample when subjected to increasing temperature. The ceramic adhesive, together with a high-precision milling technique, could be very effective to form thin coating on a metal surface. However, for optical window applications, the limited temperature range was unavoidable.

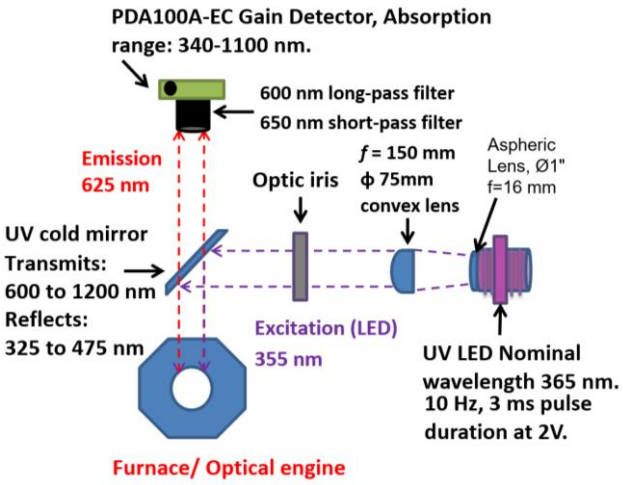
Alternatively, a water-based paint binder containing magnesium aluminium silicate (ZYP Coatings HPC binder) was tested considering its high durability that can withstand temperatures of up to 1700 K, low thermal expansion coefficient similar to quartz glass, and capability of forming homogenous coating at thicknesses as thin as 5 µm [2]. The coating was applied using an airbrush. The phosphor is first mixed with ethanol just enough to ensure homogenous dispersion of the phosphor when mixed with the binding agent. Ethanol also helped avoid clogging the airbrush nozzle during application. Each layer is cured individually at fixed temperature up to 200°C such that any residual ethanol or water in the layer vaporised and layer cracks did not occur at target temperature ranges.

Phosphor Layer Thickness Determination

Due to the use of a simple airbrushing method, it was necessary to check the uniformity of the phosphor distributions and measure the coasting thickness. As mentioned previously, an SEM was used for this purpose. The SEM focuses a high-energy electron beam onto the surface of the cross-section of phosphorus paint samples to enable the determination of their crystalline structure and hence allows sample thickness to be measured precisely. The coating thickness of the La2O2S:Eu and paint binder mixture was varied by adjusting the number of airbrushed layers. After a few preliminary tests, the coating of 6, 10, and 15 layers were selected for the tests of the present study.

Calibration Rig and Optical Setup

Figure 2 shows the electric furnace and optical setup used to obtain calibration graphs for the phosphorescence measurements. The furnace (Tetlow Multi-Sided Octagonal Kiln) provides an enclosed environment for fixed surface temperature on quartz windows. The furnace temperature was constantly monitored by a



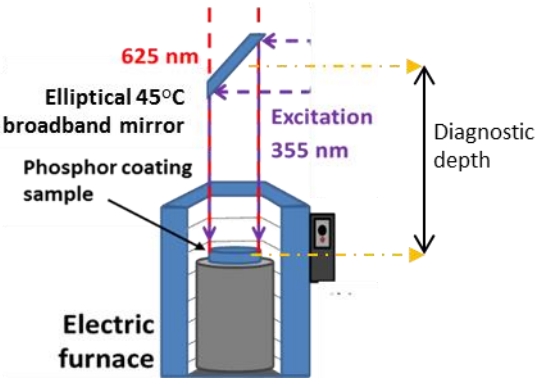


Figure 2. Top and side views of a calibration rig using an electric furnace and optical setup for phosphorescence measurements.

type S thermocouple (specified error: ΔT=±1°C). The phosphorescence signal decay was measured for a number of temperatures in the expected temperature range, i.e. 21~200°C. The surface temperature of the coating was confirmed using an infrared thermometer again.

To induce phosphorescence signals of La2O2S:Eu, a 365-nm, high-power LED (Thorlabs M365L2) was used as the excitation light source. An LED driver (Thorlabs DC2100) was used to generate 3-ms pulse at the input of 700 mA and repetition rate of 10 Hz. As shown in figure 2 (top), a set of optical lenses were used to generate an LED beam of 47 mm in diameter, which fits to the size of the quartz window placed within the furnace. The emitted phosphorescence signal was detected using a gain photodetector (Thorlabs PDA100A). A set of filters comprising 600-nm long-pass filter and a 650-nm short-pass filter were placed in front of the photodetector to isolate the resulting signal at 625 nm. The total distance from the phosphor coating to the photodetector (including 45° reflex mirror) was identically set to that of the optical engine [10] for future applications.

Results and Discussion

Figure 3 shows example SEM images of a 10-layer phosphor coating, which suggests spatial variations (i.e. non-uniform distributions) in the layer thickness along just one sample of coating. However, the close evaluation of the SEM images suggested that the variations could be due to off-focused crystalline structures signals. When a section of the fully focused signals is looked at, the coating thickness increases with the increasing number of phosphor layers, as shown in figure 4. Therefore, it was necessary to obtain many SEM images across the

coated surface and perform statistical analysis of the thickness variations.

The coating thickness was determined by post-processing three separate cross-sectional areas of 10 SEM images (i.e. a total of 30 images) for each sample of 6, 10, and 15 layers. The post processing of SEM images was automated with an in-house-developed Matlab code, as shown in figure 5. The code converts each pixel of the raw images into a grey scale and then into a binary

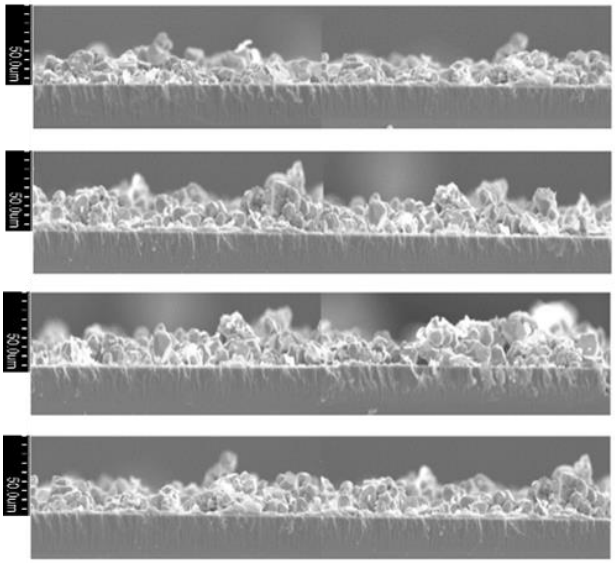


Figure 3. Four raw SEM images of 10-layer phosphor coating.

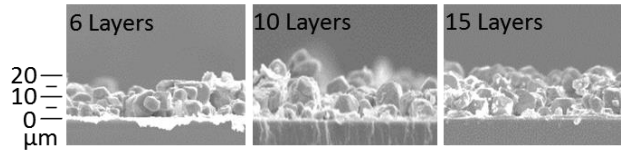


Figure 4. SEM images for various numbers of phosphor layers.

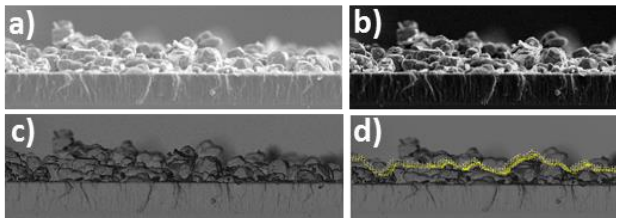


Figure 5. Post-processing of SEM images for the phosphor layer detection: a) original SEM image, b) adjustment of image sharping, contrast and brightness, c) self-subtraction from the original image, and d) the phosphor layer detection.

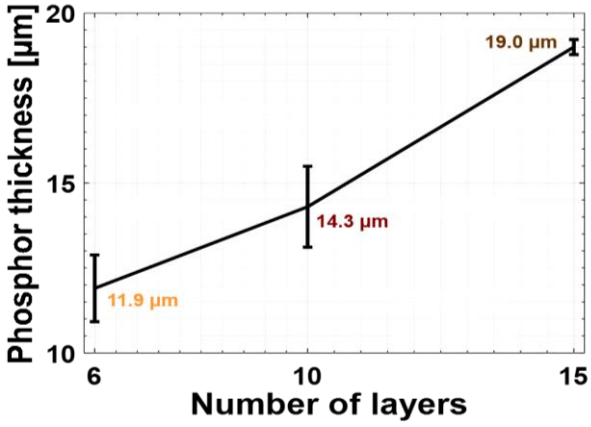


Figure 6. Phosphor coating thickness for various numbers of air-brushing layers.

matrix, the thickness is then determined by the length between the lowest to the highest number. To eliminate the off-focused crystalline structures, the images were enhanced by applying the image sharping and contrast/brightness adjustment, as well as the image self-subtraction with a weighting factor. The detected phosphor layer boundary excluding the off-focused region is illustrated using a yellow line in figure 5d.

The resulting layer thickness for 6, 10, 15-layer samples is shown in figure 6. The error bar (95% confidence) is also shown for each case. It is observed that the phosphor coating of 6, 10, and 15 airbrushed layers correspond to 11.9, 14.3 and 19.0 μm in average thickness. The results show that the thickness increase with the increasing number of layers exceeds the error margin, suggesting despite the spatial variations, the overall coating thickness increase is real.

The phosphor coatings with three different thicknesses were tested in the calibration rig for their phosphorescence signal delay curve. The results are plotted in figure 7. For all of the three coating thicknesses, the trend is clear that the phosphorescence signal delays and returns to the initial point in less than 1 ms, which suggests that the timescale of this calibration test will be applicable to diesel combustion occurring in a few milliseconds [10]. A noticeable trend is that, as the coating thickness increases from 11.9 to 14.3 μm, the signal intensity increases at any fixed time after the start of emission decay. This was simply due to the increased phosphorescence signal with increasing amounts of phosphor materials. However, as the coating thickness increases further to 19 μm, the phosphorescence signal intensity becomes lower than that of the 14.3-μm coating. The decreasing phosphorescence emission trend with the increasing layer

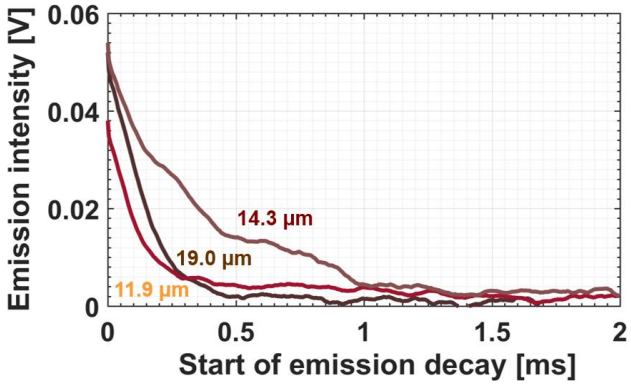


Figure 7. Effect of coating thickness on the phosphorescence signal at fixed wall temperature of 200°C.

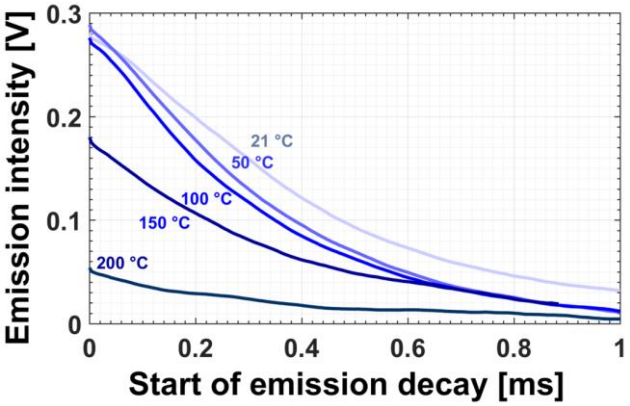


Figure 8. Effect of wall temperature on phosphorescence emission signals for temperatures ranging from 21~200°C. The phosphor coating thickness of 14.3 μm was used for the test.

thickness was also reported in previous studies [7,8] noting that the phosphor coating thicknesses higher than 20 µm has an intrusive effect on correctly reading the desired optical quartz surface temperature. This is primarily due to the increased mismatch between the quartz surface temperature and the phosphor layer temperature as the thickness increases. Therefore, for a tested condition of the present study, the highest signal was achieved with the 14.3-µm coating.

Using the 14.3-µm-thick phosphor coating, the signal decay profile was measured for various temperatures in the 21~200°C range. The results shown in figure 8 indicate that the signal intensity decreases with increasing surface temperature. For every 50°C, the signal reduction accelerates with the largest gap identified between 150 and 200°C. Using the measured phosphorescence decay curves, two calibration charts are formulated: one based on the signal intensity ratio with the reference temperature of 100°C (figure 9) and the other based on the inverse phosphorescence lifetime constant (figure 10). Both figures suggest that the intensity ratio or the lifetime constant has a temperature sensitivity adequate for thermometry between 21 and 200°C, well suited for our planned engine applications. Also, the overall profile with higher gradients in high and low temperature ranges is similar to those reported in previous studies [1, 3-6].

Conclusion

A ceramic adhesive and water-based paint binder were tested for the surface coating of La2O2S:Eu phosphor material to develop a thermographic phosphorescence diagnostic technique for future optical engine applications. The ceramic adhesive was proven to be unsuitable for the quartz windows due to its limited temperature range associated with largely different thermal expansion coefficients and thereby mechanical cracks. Alternatively, the water-based paint binder containing magnesium aluminium silicate was proven to be useful to form a thin layer on the quartz surface for the tested temperature range of 21~200°C. By varying the phosphor coating thickness and measuring the phosphorescence signal decay curves, it is found that, for a given setup of the present study, the measurable signal is detected when the coating thickness exceeds 11.9 µm. Importantly, the signal intensity shows the higher value for 14.3-µm coating than that of either 11.9 or 19.0-µm coating because the signal increases with increasing mass of the phosphor material while it decreases due to the increased distance from the quartz surface. Using this optimised coating thickness, two calibration charts are successfully developed for both the intensity ratio and inverse lifetime constant for future optical engine applications.

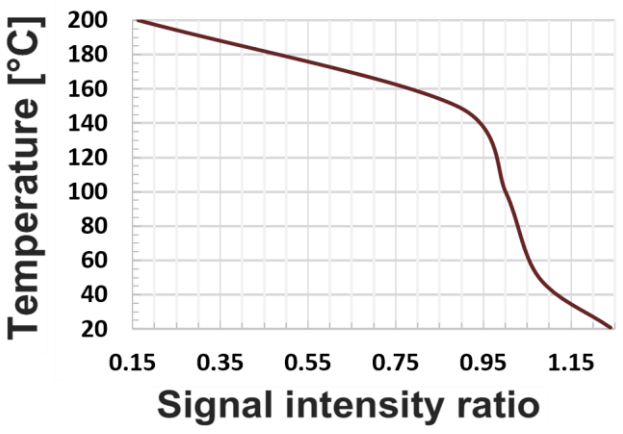


Figure 9. Signal intensity ratio based calibration curve for 14.3 µm coating.

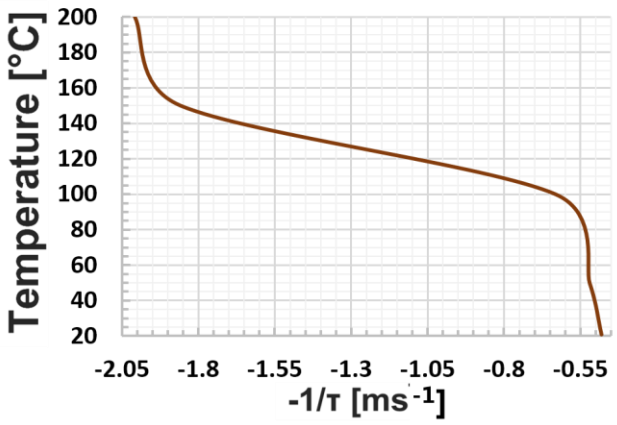


Figure 10. Inverse lifetime constant based calibration curve for 14.3 µm coating.

Acknowledgements

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