# Automated phosphor thermometry lifetime calibration of multiple phosphors and emission lines to above 1900 K

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**Technical Note**

# Automated phosphor thermometry lifetime calibration of multiple phosphors and emission lines to above 1900 K

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**Abstract**

A method is specified which enables lifetime calibration of multiple phosphors and emission lines at the same time to temperatures above 1900 K. The experimental setup and algorithm used for data collection and experimental equipment control are described. The phosphors were coated on an alumina oxide disc and the reference temperature was measured using three type B thermocouples. The algorithm automates the data collection process such that no input from an operator is required during operation. The potential systematic error in calibration temperature was evaluated and was less than 1% around 1400 K.

Keywords: thermographic phosphors, calibration, laser induced phosphorescence, thermometry, high temperature

(Some figures may appear in colour only in the online journal)

## 1. Introduction

Phosphor thermometry is a remote technique which uses the luminescence of phosphors to measure temperature. The most frequently used methods include the lifetime method and the intensity ratio [1, 2]. Although the uncertainty of intensity ratio measurements has reduced in recent studies [3, 4], the lifetime method is used here due to its superior ability to measure higher temperatures [5, 6]. In lifetime phosphor thermometry the decay time of the phosphor luminescence is used to measure temperature using a calibration where a decay time is uniquely correlated with a temperature.

Measuring the phosphorescence behaviour of phosphors is necessary because it is difficult to adequately predict the behaviour of thermographic phosphors [7]. Investigating potential thermographic phosphor candidates and different emission lines for lifetime phosphor thermometry measurements can become a time-consuming task. In particular using the conventional method where the calibration is recorded when the temperature of the phosphor sample is in thermal equilibrium [8–11]. The automatic calibration routine developed by Abou Nada *et al* [12] went a long way to improve the time efficiency of phosphor calibrations and also to reduce errors from interpolating between calibration points due to increased data density. This automatic routine could shorten the experimental

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duration by a factor of four. However, with this method only one phosphor at a time could be calibrated to a maximum temperature of 1450 K.

Having a phosphor with bright luminesce and the capability of measuring high temperatures is desirable in several applications including in gas turbines, particularly on the surface of thermal barrier coatings which can reach very high temperatures [5, 13]. With the practise of co-doping, the potential candidates for phosphor thermometry testing increases significantly [14]. The potential time saving with the calibration method described in the current study is significant especially as the process requires no input from the operator other than during the starting and ending process. For the first time, to the authors' knowledge, the calibration of surface coated phosphors to temperatures above 1900 K is demonstrated. The method allows for four phosphors and multiple emission lines of each phosphor to be studied at a time. The main benefit of the multi-phosphor calibration method is that it guarantees the same heating history and conditions of all phosphors. Therefore, one can guarantee that comparison of phosphors is performed at the same conditions and the same heating history. The lifetime of phosphors has been calibrated up to almost 2000 K previously with doped crystals [5] and phosphors in powder form [11], but the maximum temperature in a surface coating is 1800 K [13]. The novelty here is that multiple surface coated phosphors and emission lines can be calibrated at the same time with an automatic process to temperatures above 1900 K.

## 2. Experimental setup and calibration program

The third harmonic from a 10 Hz Nd:YAG laser with a pulse energy of 11.5 mJ resulting in a fluence of 14.6 mJ $cm^{-2}$ was used to excite the phosphors. This laser fluence is in the typical range used for high temperature YAG:Dy lifetime thermometry [5, 14]. The laser pulse energy could be varied using a motorized rotatable half-wave plate, controllable by the program and the laser pulse energy was monitored using an Ophir PE50-DIF-C energy meter. Three type B thermocouples were attached into indentation holes on the back of the alumina oxide (DEGUSSIT AL23) disc (see the schematic in figure 1) which were coated with the phosphors on the front. The thermocouples were attached using a high temperature ceramic binder. The reference temperature for calibration is measured in the same substrate on which the phosphor coatings are applied to increase the confidence in the accuracy of the reference temperature compared with reference temperature methods where the thermocouple is not in contact with the sample. Multiple thermocouples are used to further increase the confidence in the reference temperature. An alumina oxide substrate was used because it can handle higher temperatures than metal-based substrates. When using the Hastelloy C disc, four type K thermocouples were used which were compression fitted into holes of the disc the same way as in [12] and the locations can be seen by the schematic in figure 1. Both phosphor coated discs are 6 mm thick and were heated by the furnace in figure 1.

Four phosphors were coated in each quadrant of the disc: YAG:Dy, YAG:Dy,Ce, BSAS ($(Ba_{0.75}Sr_{0.25})Al_2Si_2O_8$):Tb, and $Y_2Si_2O_7$:Dy. Only data from YAG:Dy and YAG:Dy,Ce is shown in this study because they were the only ones which reliable temperature measurement could be performed with mono-exponential fitting. The phosphor powders were mixed with a YAG binder (ZYP Coatings) and ethanol and applied to the disc surface. There may be interactions between the YAG binder and phosphors with hosts other than YAG. This may have led to the difficulties of using BSAS:Tb and $Y_2Si_2O_7$:Dy for temperature measurements. The phosphor application was standardized with an average coating thickness of 40 μm. No significant temperature gradient in the coating is expected due to the uniform temperature distribution inside the oven and small temporal temperature gradients of 4 K $min^{-1}$. A two axis Galvo system (Thorlabs QS15XY-Y3), controllable by the program, was used to aim the laser at one of the four phosphors at a time as seen in figure 1.

A PMT was used to measure the phosphor luminescence and a motorized wheel with bandpass filters, controlled by the program, was used to change what emission line studied. For Dy doped phosphors, three different emission lines were investigated using 458 ± 10 nm (OD 4), 483 ± 31 nm (OD 6) and 586 ± 20 nm (OD 6) bandpass filters. The collection lens needed to be carefully managed such that the phosphorescence from all quadrants of the disc was imaged on the photocathode equally well.

The function of the program, which was written in MATLAB is described in the flowchart in figure 2. The program continuously acquires calibration data until operator is satisfied. During one iteration of the program, all phosphors and emission lines are investigated, where a unique combination of a phosphor and bandpass filter is called a configuration. The three emission lines from the Dy doped phosphors as well as the single emission line investigated for BSAS:Tb, lead to a total of ten configurations investigated per iteration. The order of the phosphors and their emission lines in each iteration was randomized to avoid systematic errors caused by the order of the measurement.

For each configuration, first the correct galvo position, filter wheel position and oscilloscope settings are set. Then a user-defined number of waveforms are collected by the oscilloscope. Either a sequence of waveforms is collected or the average signal of the specified number of waveforms. The mean pulse laser energy and mean temperature of the three thermocouples attached to the alumina oxide disc is also recorded during the measurement.

The program performs real time fitting of a mono-exponential decay curve to the data using a trust-region-reflective least-squares algorithm in MATLAB. The oscilloscope settings are optimized by the program based on the decay time and the intensity of the phosphorescence. The oscilloscope settings are then saved and used the next time the same configuration is used.

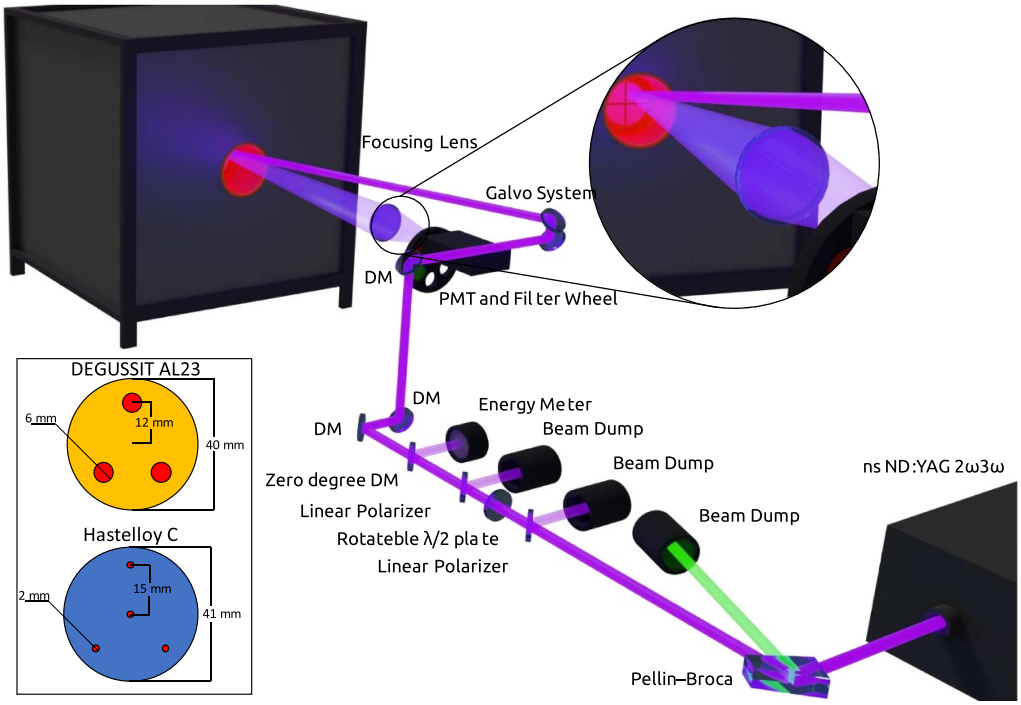


**Figure 1.** Experimental setup. DM—dichroic mirror. The zoomed view shows how the galvo system is set to excite the phosphor in the upper quadrant to the right. The schematic drawings in the box in the bottom left shows where the thermocouples are attached on the two discs. The thermocouples are shown as red dots.

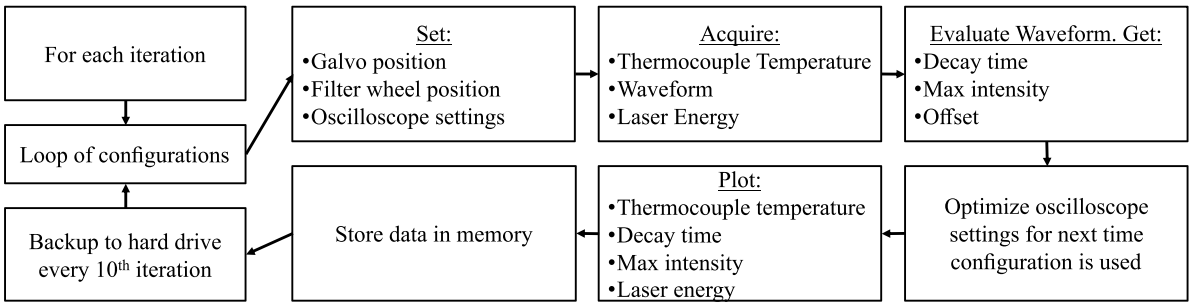


**Figure 2.** Flowchart of the algorithm used to collect decay curves from multiple phosphors and multiple emission lines. In each iteration, the order of the phosphors and emission line configurations are randomized.

Sources of error due to PMT nonlinearity were considered and minimized [15]. In situations where the luminescence of the configurations differs significantly, the laser pulse energy can be customized for each configuration to improve detector linearity. This can be done using the motorized rotatable halfwave plate.

In the calibration performed, the average of 100 decay curves were recorded for each of the ten configurations in each iteration. This resulted in one iteration taking approximately 200 s. The temperature increased by 4 K $\text{min}^{-1}$; therefore, the temperature increased approximately 0.7 K during averaging of 100 decay curves for each configuration acquisition. The mean temperature change between each iteration was approximately 13 K. The program stops using emission lines of phosphors if the background from the blackbody radiation becomes too strong. This way, emission lines that are less affected by background radiation are calibrated to higher temperatures.

## 3. Results and discussion

The decay time of the three different emission lines for YAG:Dy in figure 3(a) were the same for any given temperature in the temperature interval investigated. This is also true for the YAG:Dy,Ce phosphor in figure 3(b). The decay times were found by fitting a mono-exponential decay curve fit using a well-established method based on adaptive fitting window with $c1 = 0.5\tau$ and $c2 = 4\tau$ [16]. The ensures that the strong and fast components of the phosphor decay is excluded from decay time fitting and that only longer decay time components are used for lifetime thermometry purposes.

The 458 nm emission was calibrated until 1920 K, at the limit of the maximum feasible temperature of the tube furnace used. The 458 nm emission is less luminous at lower temperatures relative to the 483 and 586 nm emissions so these longer emission wavelengths are preferred in situations

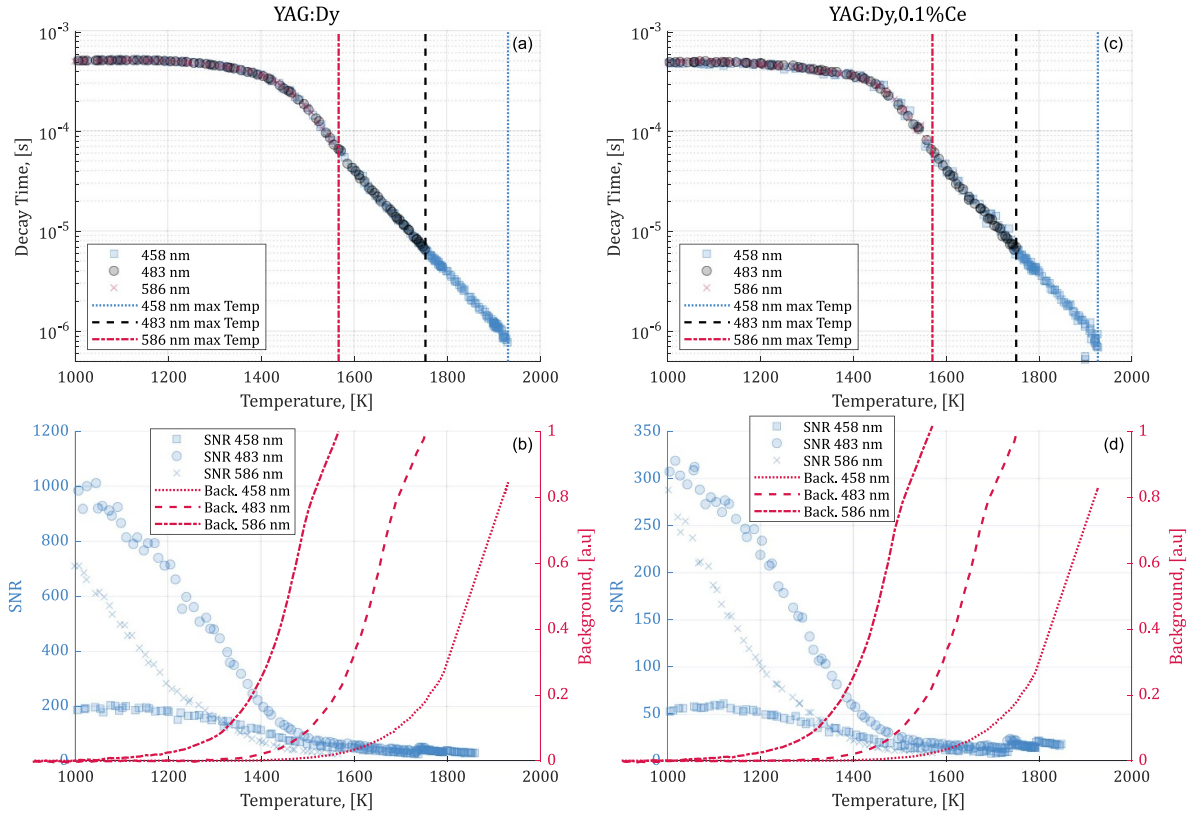


**Figure 3.** Calibration curves of YAG:Dy (a), (b) and YAG:Dy,0.1% Ce (c), (d). The legend in (a), (c) refers to the bandpass filter used. The vertical lines in (a), (c) show the maximum calibration temperature for the bandpass filter with the same colour. Shorter bandpass filters extend to higher temperatures due to lower blackbody radiation at those wavelengths as seen in (b), (d).

with lower temperature as seen by the signal-to-noise ratio (SNR) in figures 3(b) and (d). The SNR was calculated by dividing the peak phosphorescence signal of the lifetime component used for lifetime fitting with the standard deviation of the noise in the background. The strong dependence of the background blackbody radiation on the bandpass filter is evident in figures 3(b) and (d), highlighting the importance of short wavelength phosphor emissions for high temperature thermometry. The data collection methodology results in high data density of the calibration curves to resolve the temperature trends in the data. In figure 3(a), 120, 90, and 60 data points were collected for the 458, 483, 586 nm bandpass filters respectively.

A potential concern with using alumina oxide discs compared to using Hastelloy-C discs [12] is that the thermal conductivity of alumina oxide reduces significantly with temperature and is low at the high temperatures relevant for high temperature phosphor calibration. Therefore, the performance difference of the two substrates were compared at the limit of the maximum temperature of the Hastelloy C disc to give the best assessment of potential issues with the use of the alumina oxide substrate. At the maximum tested temperature of approximately 1400 K the DEGUSSIT AL23 substrates thermal conductivity is between 7 to 5 W $m^{-1}$ $K^{-1}$ and Hastelloy C's is approximately 28 W $m^{-1}$ $K^{-1}$. The concern with low thermal conductivity is that temperature gradients could occur through the disc and result in different temperatures for the phosphor coatings and the thermocouples.

To evaluate these potential temperature errors, YAG:Tm,Li was coated on both the DEGUSSIT AL23 substrate and a Hastelloy-C substrate. For each disc type, a calibration curve was generated when the temperature of the furnace increased by 4 K $min^{-1}$. Then the temperature in the furnace was varied as seen in figures 4(a) and (b) to look for temperature measurement errors with the ceramic disc (figures 4(a) and (c)) and Hastelloy C discs disc (figures 4(b) and (d)).

Due to the Hastelloy C disc's higher thermal conductivity and a previous investigation with the disc [12], it is assumed that the thermocouples and phosphor coating are at the same temperature during the constant 4 K $min^{-1}$ heating in the calibration processes. With this assumption one can investigate if there are temperature differences between the phosphor coating and the thermocouples for the ceramic disc by applying the calibration curve from the Hastelloy C disc to the decay times collected from the ceramic disc. This is investigated in figure 4(c). The temperature errors in figures 4(c) and (d) are the temperatures measured by the phosphor using the Hastelloy C calibration subtracted by the ceramic thermocouple temperature in figure 4(c) and Hastelloy C thermocouple temperature in figure 4(d). The maximum temperature measurement error with the ceramic puck due to temperature increases and decreases of the furnace was approximately 5 K (figure 4(c)).

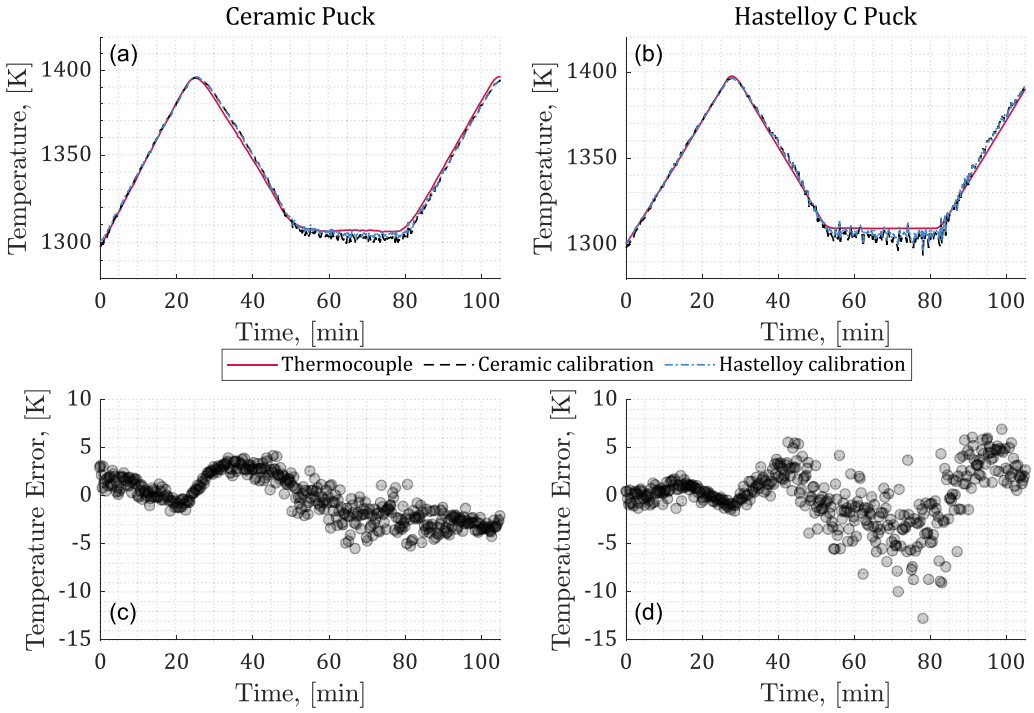


**Figure 4.** Thermocouple and measured phosphor temperature using either the ceramic or Hastelloy C calibration using decay time measured from the ceramic disc (a) or decay times measured with the Hastelloy C disc (b). Temperature error using the ceramic disc (c) and Hastelloy disc (d) using the calibration from the Hastelloy C disc.

The Hastelloy C temperature error in figure 4(d) gradually becomes noisier due to the oxidation rate of the disc negatively impacting the phosphorescence luminosity at temperatures above 1300 K.

The maximum achievable accuracy of a phosphor thermometry temperature calibration is limited by the uncertainty of the reference temperature, in this case the thermocouples. The rated uncertainties of the thermocouples are 0.75% and 0.5% for the type K (Hastelloy C disc) and type B (Ceramic disc) thermocouples respectively. The uncertainty reduces by using multiple thermocouples, resulting in an uncertainty of 0.38% and 0.29% for the four type K thermocouples and three type B thermocouples respectively. The mean standard deviation between the thermocouples attached to the same disc as a percentage of measured temperature for the data presented in figure 4 is 0.05% and 0.06% for the type K and type B thermocouples respectively. This is within the temperature measurement uncertainty of the thermocouples therefore no statistically significant spatial temperature variation on either of the discs can be measured. Additionally, the maximum 2 K difference between the ceramic calibration and Hastelloy C calibration in figure 4(a) for decay times measured from the ceramic disc is within the measurement uncertainty of the thermocouples.

## 4. Conclusion

The described experimental setup allows for calibration of multiple phosphors and emission lines to above 1900 K. The program allows for real time evaluation of decay time and automatic optimization of oscilloscope settings. The system significantly simplifies the calibration process for novel phosphors and can also be used to test hysteresis effects in phosphors. The systematic error in calibration temperature was evaluated and was less than 1% around 1400 K.

## Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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