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# Instantaneous surface temperature measurements in combustion by an adapted processing of the Laser Induced Phosphorescence signal

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## Abstract

This paper proposes a new approach of the Laser Induced Phosphorescence (LIP) signal for instantaneous surface temperature measurements: the “full spectrum fitting” method. LIP is a semi-invasive optical diagnostic based on coated thermographic phosphor surfaces, emitting a temperature-dependent signal under laser irradiation. Thousands of  $\text{Mg}_{3.5}\text{FGeO}_5:\text{Mn}$  phosphorescence spectra are acquired to build a database at each Kelvin in a temperature range from 100 K to 900 K with a unique set of experimental acquisition parameters. The constituted database is used to retrieve a single-shot temperature measurement using a least mean square method. A statistical study on 70,000 single-shots acquisitions shows good precision and low uncertainties with this method.

## Introduction

Surface temperature knowledge is essential in energy converting systems and transfer processes such as turbines, rocket engines, nuclear reactors, or even glass melting furnaces. Their efficiency and lifetime are strongly dependent on wall temperature, which requires accurate and resolved information. In combustors, transitions between different flame topologies may be triggered by wall temperature [1]. More precise thermal boundary conditions improve numerical modeling, providing better prediction of these transitions [2], [3].

In the combustion research field, numerous experiments are performed in the transient thermal regime and in a short time to protect the setup. These experiments demand to track the temporal resolution of the surface temperature, which can reach various levels. Thus, diagnostics exploiting single-shot measurements are required to quantify wall heat fluxes and heat transfer modes in a wide range of temperatures.

Among experimental techniques to measure the temperature, thermocouple measurements require little effort. They are sensitive to a wide range of temperatures, and can capture fast temperature changes [4], [5]. However, they are invasive probes, and their implementation need to be planned at the design level of the investigated system. Pyrometry is another possibility, that requires the exact knowledge of the surface emissivity, which is difficult to assess and may change during operation. Moreover, radiations from flames or other bodies may affect the measurement [6]. Heat sensitive paints allow an estimation of the peak temperature through a change of color of the paint [7]. They are however inappropriate for time-resolved measurements in high temperature combustion environments.

In combustion diagnostics, Laser Induced Phosphorescence (LIP) thermometry is an established method to measure surface temperature [8]–[12]. Surfaces of interest are coated with a rare-earth-doped phosphor, which emits a phosphorescence signal under

laser irradiation. The temporal and spectral properties of this signal are temperature-dependent. These properties are used to determine an unknown temperature through an appropriate calibration. Two approaches are mainly used for the determination of the temperature. The “lifetime” method takes advantage of the temperature dependence of the exponential decrease of the phosphorescence signal after laser excitation [13]. The function of the decay time with the temperature,  $\tau(T)$ , is a calibration curve. Depending on the phosphor used, cryogenic [14], [15] to high temperatures [16] can be measured, but the decay time can vary in few decades, from milliseconds to microseconds. A high to very high detector sampling rate is then needed to resolve the temporal decay. The “spectral” or “intensity ratio” method takes advantage of the emission spectrum temperature dependence [17]. The calibration curve is built by computing the ratio of intensity,  $R(T)$ , from two spectral bands of the spectrum. Detectors with proper filters are used to collect the light from these two regions of the spectrum. This method can be used for 2D [18], [19] or even fluid temperature measurements with dispersed phosphor particles [20], [21].

Both methods use only a small portion of the phosphorescence spectrum, which may restrict the sensitive range. The equipment capacity limits the decay detection, even if automatic routines for calibration have been imagined to extend the dynamic range [22]–[24]. The exploitation of single-shot measurements is challenging for both methods as the signal-to-noise ratio evolves with temperature. Precision and uncertainty thus depend on the temperature but also on the experimental setup sensitivity (optical alignment, laser energy level, fluorescence interferences) [20], [21], [23]–[29], [30].

Adapted processing of the laser induced phosphorescence signal is needed to exploit single-shot measurements with a good signal-to-noise ratio in a wide and accurate temperature range. A unique set of experimental parameters (or fast-changing parameters) are required to work in short time operating systems

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without operator adjustments. The method must be easy to implement and also to extrapolate to other phosphors to extend measurement possibilities.

In this study, a new approach based on the dependence of the entire shape of the phosphorescence emission spectrum with the temperature is proposed to retrieve a single-shot measurement temperature: the “full spectrum fitting” method. First, existing methods and the phosphor choice for this study are presented. Next, the experimental setup used for the calibration procedure is described. Then, the method for the determination of a single-shot temperature measurement is presented. Precision and uncertainties are determined using a statistical analysis. Finally, a quantified comparison with the “intensity ratio” method is performed.

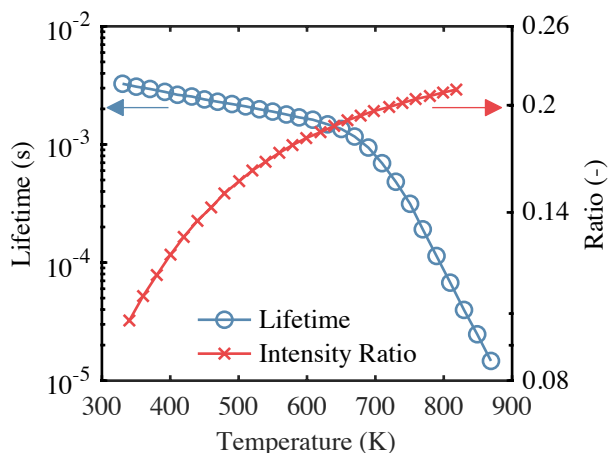
### LIP methods and choice of the phosphor

The “lifetime” method relies on the measurement of the phosphorescence signal as a function of time. If  $t = 0$  corresponds to the end of the laser excitation, the intensity  $I(t)$  of the phosphorescence signal can be described with a mono-exponential decrease:

$$I(t) = I_0 \exp\left(-\frac{t}{\tau}\right)$$

$I_0$  denotes the intensity at  $t = 0$ , whereas  $\tau$  the lifetime (or decay-time) is temperature-dependent. The function  $\tau(T)$  is used as a calibration curve in the “lifetime” method, as seen in figure 1. An unknown temperature is determined by measuring the decay-time of the phosphorescence signal and reversing this function.

The “intensity ratio” method takes advantage of the temperature-dependent changes of the emission spectrum (see figure 2). A ratio  $R(T)$  is computed at each temperature by dividing intensities from two spectral regions acquired with two adequate filters. An example of the calibration curve  $R(T)$  is plotted in figure 1. An unknown temperature is determined by computing the intensity ratio of the phosphorescence signal and reversing this function.

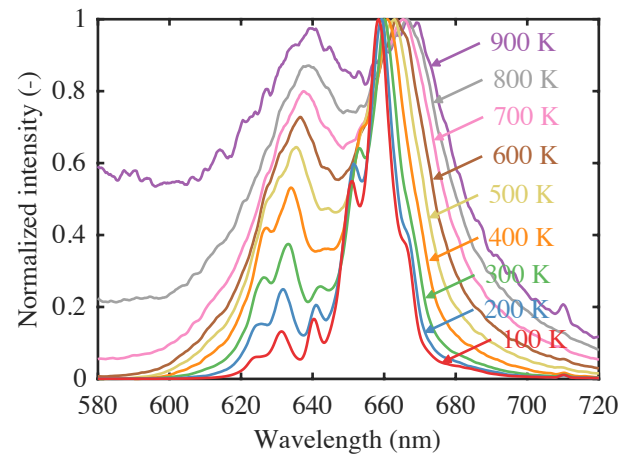


**Figure 1** – Lifetime and intensity ratio for  $Mg_{3.5}FGeO_5:Mn$  phosphor as a function of temperature [26].

For both methods, the calibration curve is determined experimentally and depends on the phosphor temperature sensitivity and the equipment capacities [8]–[12].

The “full spectrum fitting” method proposed in this paper uses the dependence of the entire phosphorescence spectrum instead of narrow bands. The calibration curve is replaced by a database of spectra at known temperatures. An unknown temperature is determined by finding the best agreement between the measurement spectrum and a spectrum in the database thanks to a fitting algorithm.

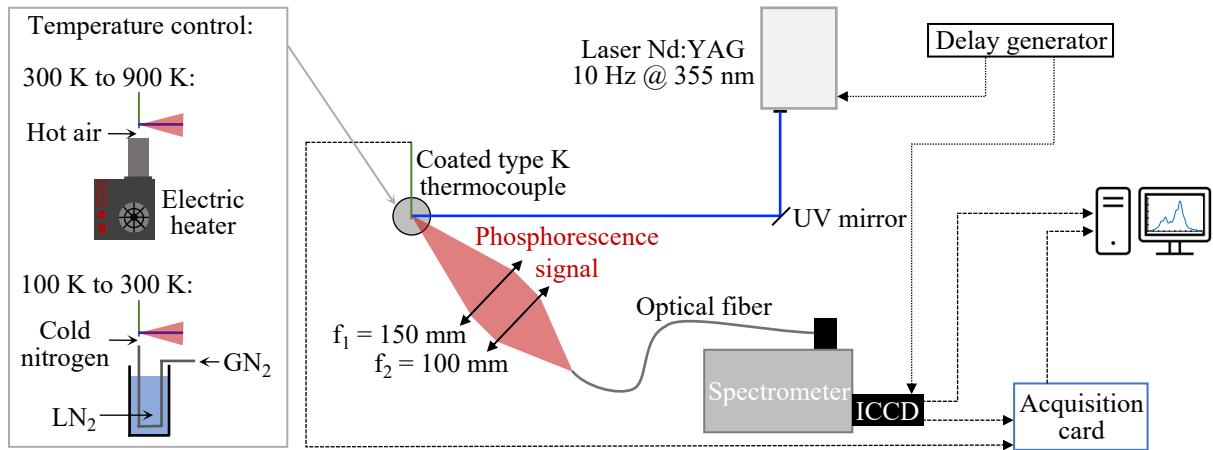
A phosphor with a wide range of temperature sensitivity and good spectral dynamics is required. The phosphor  $Mg_{3.5}FGeO_5:Mn$  is sensitive with the “lifetime” method from cryogenic to high temperatures (from to 8 K [15] to 1200 K [31]). The signal decay time is about few milliseconds at ambient temperature and hundreds of nanoseconds at high temperatures. The fully resolved phosphorescence spectrum is presented and analyzed in [19], [23], [27], [32] and [33]. Figure 2 shows average spectra from 100 K to 900 K every 100 K. The most intense peak, at 660 nm, is used for the “lifetime” method. The 630 nm peak increases in intensity and expands with temperature. This line is compared with the 660 nm line in the “intensity ratio” method. Starting from 700 K, another component of the spectrum appears under 580 nm and increases with temperature [33], but it is not exploited with the “lifetime” or “intensity ratio” method. As the entire shape of the spectrum is dependent on temperature, the phosphor  $Mg_{3.5}FGeO_5:Mn$  is a good candidate for this approach. It is then selected to test the method along a wide range of temperatures.



**Figure 2** – Normalized average  $Mg_{3.5}FGeO_5:Mn$  phosphorescence spectra at different temperatures after laser excitation at 355 nm with a fluence of  $106 \text{ mJ}\cdot\text{cm}^{-2}$ .

### Experimental setup

Spectra from 100 K to 900 K must be acquired to build the database experimentally. For this purpose, a thermographic painting is applied on a thermocouple and excited by a laser. Phosphorescence signals are acquired with a spectrometer at different temperatures. The spectrum is stored with its temperature at each laser



**Figure 3** - Experimental setup for phosphorescence spectra acquisition from 100 K to 900 K.

pulse. Figure 3 shows the experimental setup, which is described in the next paragraphs.

The thermographic phosphor powder  $\text{Mg}_{3.5}\text{FGeO}_5\text{:Mn}$  (Phosphor-Technology, EQD25/NU1) is mixed with an industrial binder (ZYP Coatings, HPC Binder). A dilution water volume corresponding to 10% of the binder volume is added. This mixture is then coated with an airbrush (AEROGRAFHE Services, Utopia C05) with ten layers. Each layer is dried at ambient air conditions for a few minutes. The thickness has been estimated not to exceed  $100\ \mu\text{m}$  in a previous study in the laboratory [34]. The coating is applied on a type K sheathed thermocouple with a diameter of  $1.5\ \text{mm}$ .

The coated thermocouple is placed above two complementary devices to access a temperature range from 100 K to 900 K. A home-made heat exchanger is used for cryogenic temperatures from 100 K to 300 K. The exchanger is fed by gaseous nitrogen at ambient pressure and immersed in a Dewar filled with liquid nitrogen. The coated thermocouple is positioned in the cold flow of gaseous nitrogen at the exit of the exchanger. The temperature is decreased from ambient to 100 K by adjusting the mass flow controller. The exchanger is replaced by an electric heater (LEISTER, CH-6056, 2850W) to access the temperature range from ambient up to 900 K.

The phosphor coating is excited at 355 nm using the 3<sup>rd</sup> harmonic of an Nd:YAG laser (Continuum, Minilite ML II) triggered at 10 Hz. The pulse energy is adjusted by the included dielectric polarizer attenuator and recorded with a pyroelectric energy detector (Gentech-EO, QE25-SP-S-MB). The energy is set at 5 mJ to ensure no dependence on the spectrum shape and a high signal-to-noise ratio.

A telescope composed of two-inch achromatic cylindrical lenses is used to collect the phosphorescence signal ( $f_1 = 150\ \text{mm}$  and  $f_2 = 100\ \text{mm}$ , Thorlabs, AC508-150-A and AC508-100-A). The light is then focused in a  $200\ \mu\text{m}$  multimode optical fiber leading to the entrance slit of a spectrometer. With a 1.5 magnification, the phosphorescence signal is collected from a  $300\ \mu\text{m}$  diameter spot on the coated thermocouple, smaller than the 3 mm diameter laser beam. Records of

the laser beam (Gentech BEAMAGE-4M beam profiler) show that the energy distribution follows a Gaussian profile and is homogeneously distributed in the  $300\ \mu\text{m}$  spot. The laser energy changes neither the shape of the beam, nor the fluence. An average fluence can then be determined from the distribution at  $106\ \text{mJ}\cdot\text{cm}^{-2}$ .

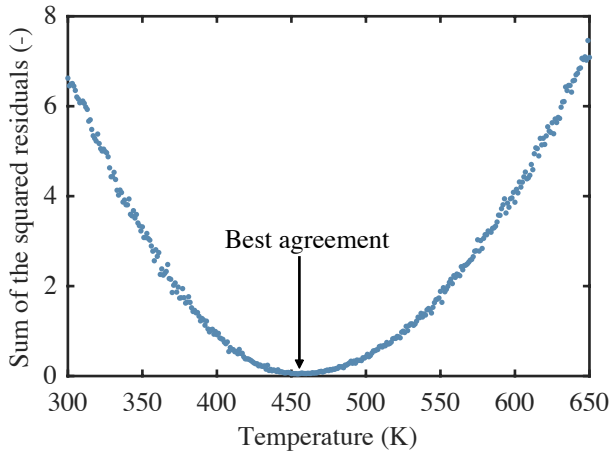
The phosphorescence signal is imaged at 10 Hz with a spectrometer (Princeton Instrument, Spectra Pro 500i,  $f = 500\ \text{mm}$ ,  $150\ \text{grooves}\ \text{mm}^{-1}$ ) coupled with an intensified CCD camera (Princeton Instrument, Pi-Max,  $512 \times 512$  pixels) covering a spectral range of 160 nm centered at  $\lambda = 650\ \text{nm}$  with a resolution of  $0.3\ \text{nm}/\text{pixel}$ . The chosen wavelength resolution allows capturing enough details in the spectrum to distinguish them at each temperature (see figure 2). A wavelength calibration was carried out (but no intensity calibration yet). The laser and the spectrometer are synchronized with an external delay generator (BNC, Model 577 Pulse Generator). A 50 ns delay between the laser pulse and the camera exposition is set to avoid any laser contribution and prevent potential interferences from fluorescence sources [30]. The exposure time is set to  $30\ \mu\text{s}$ . These parameters are fixed for temperatures from 100 K to 900 K.

The construction of the database requires the acquisition of thousands of single-shot spectra at known temperatures. For this purpose, single-shot spectra are acquired at 10 Hz during a slow evolution of the temperature ( $\sim 0.2\ \text{K}\cdot\text{s}^{-1}$ ). The heat exchanger mass flow is increased to change the temperature from ambient to 100 K. The procedure is the same with the electric heater: the current is adjusted to change the temperature from ambient to 900 K. In both situations, the temperature is acquired with an acquisition card (National Instruments, PCI-6111). The camera exposition signal is recorded as well to match the single-shot spectra with the corresponding temperature.

The average spectrum of the background noise is subtracted for each of the 70,000 phosphorescence spectra acquired. An average spectrum is computed with dozens of single-shot spectra at each Kelvin to build the final database. A 3-order Savitzky-Golay smoothing filter with a moving window of 13 points is applied.

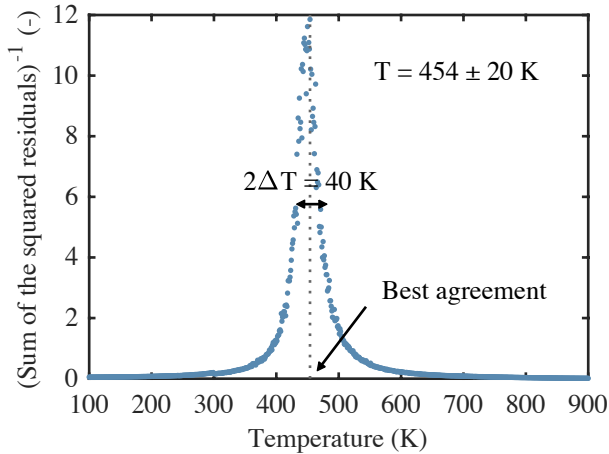
### Data evaluation

The constituted database is used to determine the temperature of a single-shot spectrum. The spectrum from the database, which corresponds best to the single-shot measurement, gives the temperature. As a first step, the classical least-mean-square (LMS) method is chosen to fulfill this task. The algorithm computes the sum of the squares of the residuals between the single-shot spectrum and all the average spectra in the database. This sum describes a parabolic curve as a function of the temperature, as illustrated in figure 4. The target temperature corresponds to the minimum of this function, which tends toward zero. Before computation, spectra from the database and the single-shot spectrum are normalized by the maximum intensity, around 660 nm. The same Savitzky-Golay filter is applied on the single-shot spectrum.



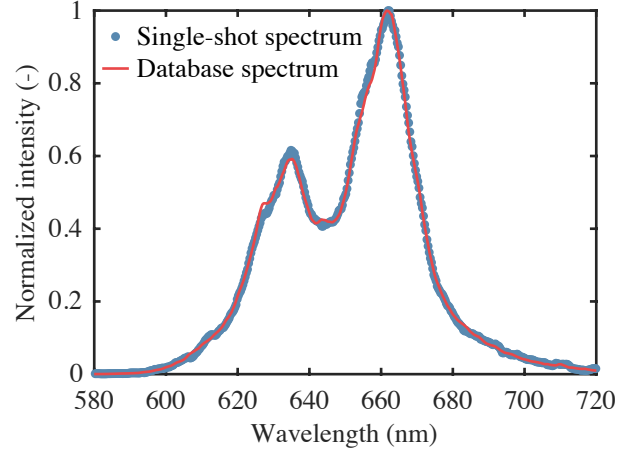
**Figure 4** - Example of the sum of the squared residuals between a single-shot spectrum and average spectra from the database as a function of temperature

An example of this procedure is shown in figures 4 to 6. A single-shot spectrum is chosen among the 70,000. The sum of the squared residuals between the single-shot spectrum and averaged spectra from the database is computed at each temperature. The inverse of this function is shown in figure 5.



**Figure 5** - Inverse of the sum of the squared residuals between a single-shot spectrum and average spectra from the database as a function of temperature.

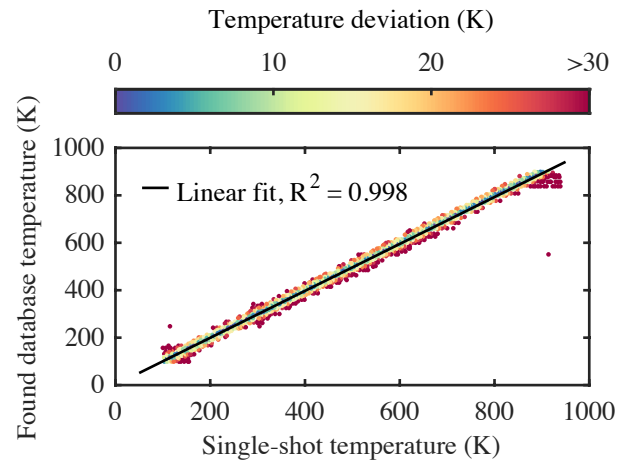
The best temperature corresponds to the maximum of this curve, 454 K, for this example. The uncertainty  $\Delta T$  can be taken at the FWHM leading to  $\Delta T = 20$  K. The corresponding spectrum from the database and the single-shot one are plotted in figure 6 showing a very good agreement.



**Figure 6** – Normalized single-shot spectrum and average spectrum at 454 K extracted from the database using the LMS method.

### Uncertainty quantification and comparison

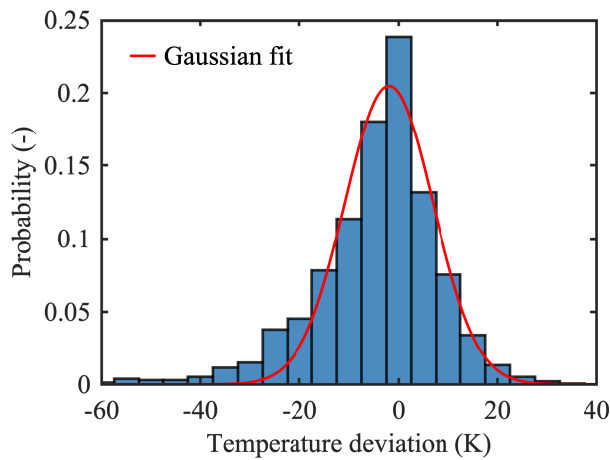
Precision and uncertainty of the “full spectrum fitting” method are determined with a statistical study carried out with the 70,000 spectra. The method is applied to determine the temperature for each single-shot measurement and then compared to its recorded value. Results are gathered in the scatter plot in figure 7.



**Figure 7** - Scatter plot of the temperature found with the LMS method and the recorded single-shot temperature. The color indicates the absolute temperature deviation.

Each of the 70,000 dots is colored depending on the absolute deviation with the single-shot temperature. Zero Kelvin indicates that the temperature found with the method is the same as the single-shot one. A linear fit is applied. The coefficient of determination  $R^2$  is 0.998, meaning minimal dispersion of the results around a nearly zero Kelvin temperature deviation. The error on the temperature determination is the equivalent for all

temperatures excepted for high temperatures (900 K), where the deviation is slightly higher. The phosphorescence signal intensity drastically decreases at these temperatures, resulting in a lower signal-to-noise ratio, making it more challenging to exploit single-shot spectra. The histogram in figure 8 shows the temperature deviation distribution for the 70,000 tests with the LMS algorithm. The deviation is smaller than 5 K for more than 40% of the single-shot spectra and between 5 K and 10 K for 25% of them. The maximum error is between 60 K and 65 K for only 0.2% of them. A Gaussian fit is applied. This fit is centered at an average deviation of -1.97 K and a standard deviation of 12.59 K, which confirms a limited dispersion of the results. This statistical study shows that the precision of the “full spectrum fitting” method is suitable for single-shot measurements.



**Figure 8** - Histogram of difference between the 70,000 single-shot temperature and the temperature found with the LMS method. Bar width is 5 K. A Gaussian fit is applied with 95% confidence bounds resulting in an average temperature deviation of  $\mu = -1.97$  K and a standard deviation of  $\sigma = 12.59$  K.

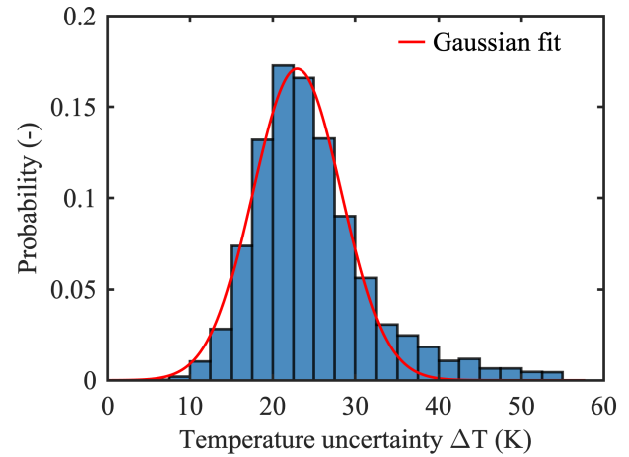
In figure 9, the uncertainty histogram for the 70,000 single-shot spectra is shown. A Gaussian fit gives an average temperature uncertainty of 23 K and a standard deviation of 15 K, which confirms the low dispersion of the results with this method again, even with single-shot measurements.

This method is then compared to another spectral method, the “intensity ratio” method. A ratio is computed from 100 K to 900 K for each average spectrum in the database. The ratio  $R(T)$  is calculated to reproduce the usual experimental conditions [26]:

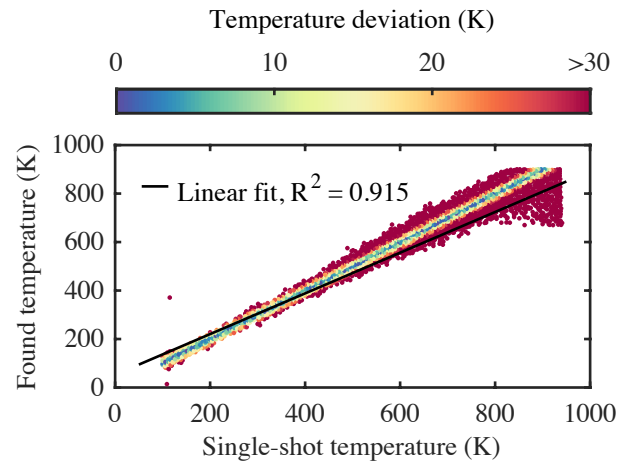
$$R(T) = \frac{\int_{628 \text{ nm}}^{638 \text{ nm}} I(\lambda, T) d\lambda}{\int_{655 \text{ nm}}^{665 \text{ nm}} I(\lambda, T) d\lambda}$$

The calibration curve is finally obtained by fitting a polynomial function on the ratio  $R(T)$ . A single-shot temperature is determined by computing its intensity ratio and reversing this function. This method is applied

to the 70,000 single-shot spectra. Results are gathered in the scatter plot in figure 10.



**Figure 9** - Histogram of the temperature uncertainty with the LMS method applied on the 70,000 single-shots spectra. Bar width is 5 K. A Gaussian fit is applied with 95% confidence bounds resulting in an average temperature uncertainty of  $\mu = 22.95$  K and a standard deviation of  $\sigma = 15.11$  K.



**Figure 10** - Scatter plot of the temperature found with the intensity ratio method and the single-shot temperature. The color indicates the absolute temperature deviation.

Even if the temperature is correctly determined most of the time, the dispersion of the results is more important than with the new methodology (see figure 7 for comparison). This is confirmed by the lower coefficient of determination  $R^2$ , which is now around 0.915. The temperature deviation increases with temperature, whereas it is practically independent with the LMS algorithm. In worst cases, an error of several dozens of Kelvin may occur with the “full spectrum fitting” method, while it may reach hundreds of Kelvin with the “intensity ratio” method, in particular when the temperature exceeds 800 K. The full exploitation of the phosphorescence spectrum and the use of a least mean square algorithm seems more robust and efficient for determining single-shot temperatures in a wide range of temperatures.

## Conclusions

A new methodology of post-processing of the Laser Induced Phosphorescence signal has been studied for instantaneous surface temperature measurements: the “full spectrum fitting” method. The dependence of the entire  $\text{Mg}_{3.5}\text{FGeO}_5\text{:Mn}$  phosphorescence spectrum is exploited for single-shot measurements. To this end, thousands of spectra have been acquired with a unique set of experimental parameters to build an average spectra database from 100 K to 900 K at each Kelvin. The least mean square method is used to retrieve the temperature from a single-shot spectrum, and it shows promising results. A statistical study on 70,000 single-shot spectra shows a very low dispersion of the results regardless of the temperature. The average temperature error is less than 2 K, and the average temperature uncertainty is 23 K. This method shows better results for single-shot measurements than the “intensity ratio” method. This method could easily be applied to other phosphors offering good spectral dynamics to reach different temperature ranges.

This methodology will be applied in future studies to determine the temperature of several surfaces inside the cryogenic testbench MASCOTTE [35] during an experimental campaign within the LabEM2C / ONERA / CNES / ArianeGroup R&D consortium. The structure of LOX/LCH<sub>4</sub> flames will be studied during this campaign. The harsh conditions reached in MASCOTTE – wide range of temperatures, pressure up to 60 bar, complex gas composition – and the short time of operation are perfect challenges for this technique.

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