Chapter 6: Surface Thermometry – Thermographic Phosphors

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Outline phosphor thermometry

• Introduction & Motivation
• “Decay-Time Method” versus “Ratio Method”: comparing precision and accuracy
• Error treatment for decay-time method
• Application examples
Surface temperature: diagnostics

Thermocouples
- Invasive, limited spatial and temporal resolution, only point measurements

Pyrometry (Infrared thermometry)
- Emission coefficient generally unknown
- Sensitive against chemiluminescence and stray light

Temperature sensitive paints (TSP - coated), thermoliquid crystals (TLC)
- Temperature range < 380 K

Heat sensitive paints (HSP - coated)
- No temporal resolution

Thermographic phosphors (TGP – coated)
+ Broad temperature sensitive range (7 K -1870 K)
+ Insensitive against blackbody radiation, stray light and chemiluminescence
+ High spatial and temporal resolution, 2D diagnostics
Thermographic phosphors:

- Rare-earth or transition metal doped ceramic materials
- Exploit temperature dependent spectroscopic properties following electronic excitation
Principle of Phosphor Thermometry:

- Coat surface with thermographic phosphor
- Excite coating with UV-light source
- Detect emission with appropriate device either
  - time-resolved (fixed spectral range) or
  - Time-integrated and spectrally resolved (fixed temporal range)
Introduction & Motivation II

Approaches of Phosphor Thermometry

Phosphor Thermometry

Time-resolved
- Time-domain
  - Decay-time
  - Rise-time
- Frequency-domain

Time-integrated
- Single spectral band
  - Integrated intensity
  - Line shift
- Two spectral bands

\[ R = \frac{I(\lambda_1)}{I(\lambda_2)} \]
Introduction & Motivation III

- Comparison of the two most popular approaches:
  - Luminescence Lifetime (Decay-time)
  - Intensity Ratio (Two-line)
- Motivation:
  - Decision support required for users
  - Comparison to highlight pros vs cons
  - Investigation of sensitivities, precision, accuracy and application
- Procedure
  - Use two CMOS high-speed cameras (same data-set)
  - Exemplified for phosphor which exhibits sensitivities in both approaches: Mg$_4$FGeO$_6$: Mn
Experimental Setup

- HSS6
- Interference filter
- Homogenizer
- Nd:YAG laser
- Glan polariser
- Halfwave plate
Data evaluation

- Nonlinearity intensity-correction of the CMOS Chip
- Image matching by pinhole model (Software: Davis 7)

**Lifetime**
- Pixelwise fitting of waveforms
- Brübach Algorithm + Linear Regression of the Sum (LRS)

**Intensity ratio**
- Pixelwise temporal integration:

\[
R = \frac{\int_{t_1}^{t_2} I_{633\text{ nm}}(t) \, dt}{\int_{t_1}^{t_2} I_{660\text{ nm}}(t) \, dt}
\]

- Two ratios R1 and R2 used:
  - R1:
    \[
    t_1 = t_0 + 100 \, \mu s
    \]
    \[
    t_2 = t_0 + 120 \, \mu s
    \]
    (corresponds to 1 image @50kHz)
  - R2:
    \[
    t_1 = t_0 + 100 \, \mu s
    \]
    \[
    t_2 = t_0 + 1100 \, \mu s
    \]
    (corresponds to 50 images @50kHz)

---

Results: Temperature dependent characteristics

• Temperature-Lifetime / Temperature-Ratio characteristics

• Temperature-Lifetime characteristics of both spectral bands similar

• Temperature-Ratio characteristics for both ratios similar as well

• Very different sensitivities for lifetime approach and only slightly differing sensitivities for ratio approach
Results: Precision I

- Shot-to-Shot (temporal) standard deviations

- Comparable precision of both techniques at lower temperatures

- Lifetime method ~2 orders of magnitude better at higher temperatures
Results: Precision II

- Pixel-to-pixel (spatial) standard deviations

- Huge difference in spatial precision between the two techniques

- Spatial precision of lifetime method superior over the entire temperature range
Results: Accuracy I

- Evaluation of systematic errors

- Evaluated at two different temperatures corresponding to two different sensitivities in the temperature lifetime characteristic

- Calibration at fixed conditions and then parametric variation:
  - Energy variation
  - Changing settings in optical setup
Results: Accuracy II

- Dependency on excitation energy at $T = 515K$

- Inaccuracy for too low excitation energies of lifetime approach well known (i.e. Brübach et al. 2008)

- Ratio 2 rather robust against energy variations, but inaccuracy over whole energy range
Results: Accuracy III

- Dependency on excitation energy at $T = 780$K

![Graph showing temperature and excitation energy relationship]

- Lifetime approach very accurate due to high sensitivity

- Ratio 1 & 2 show overestimation of more than 100 K at lower energies
Results: Accuracy IV

- Accuracy due to changes of settings in the optical setup

**T = 515 K**

- Lifetime (660 nm)
- Ratio 1
- Ratio 2

**T = 780 K**

Index
1 – Filter rotation
2 – Increase distance (1 cm)
3 – Increase distance (2 cm)
4 – Increase distance (4 cm)
5 – Change camera angle

Temperature difference (K)

Index

Temperature difference (K)
Results: Cooling device I

- Stainless steel block with cooling channel placed inside oven
- When block is at designated temperature, cold water is pumped through cooling channel
- For image mapping purposes target dots have been transferred to device

Results: Cooling device II

- Transient Experiment
- Same set of data for all three methods
Results: Cooling device III

- Stationary T-distribution over last 16 frames
- Statistics for these temperature maps:
Results: Cooling device III

• Stationary T-distribution over last 16 frames
• Statistics for these temperature maps:

Temperature in K

Vertically averaged temperature (K)

Normalized temporal standard deviation (-)

Normalized spatial standard deviation (-)

Temperature difference (K)

Index

Lifetime (660 nm)

Ratio 1

Ratio 2

Vertical binning

Andreas Dreizler | 20
Results: Cooling device III

• Stationary T-distribution over last 16 frames
• Statistics for these temperature maps:

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

Normalized temporal standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature difference (K)

Index

300 400 500

0.02

0.04

0.06

0.08

0

5

-5

-10

-15

-20

-25

-30

-30

-25

-20

-15

-10

-5

0

5

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

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Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)

Temperature in K

300 400 500

Vertically averaged temperature (K)

Normalized spatial standard deviation (-)

x (mm)
y (mm)

Lifetime (660 nm)
Summary & Conclusions

Comparison of Mg$_4$FGeO$_6$: Mn for the lifetime and the intensity ratio method in terms of
- Precision
- Accuracy
- Application (cooling device)

Main results
- High temperature range (>650K): Lifetime method is superior in all aspects
- Low temperature range: Lifetime method superior for spatial standard deviation and for accuracy, similar temporal standard deviations
- Only valid for the phosphor under consideration
- For other phosphors similar investigations required

Fuhrmann et al. PCI 34, 2013
Error treatment for the decay-time method

1. Introduction
2. Measurement chain / Error sources
3. Applications
4. Summary

1. Introduction

2. Measurement chain / Error sources

3. Applications

4. Summary
Combustion applications: mostly used approaches

- Most often used approaches are **decay-time** and two-spectral-band-method
- As discussed: Decay-time method does have the potential for superior precision and accuracy (especially in 2D application); see Fuhrmann, Brübach and Dreizler, PCI 33, 2013
- Decay-time varies with temperature due to varying energy transfer processes
Thermographic Phosphors: materials showing temperature dependent decay times

Approx. 100 different phosphors documented in literature
See Brübach et al. PECS 2013

Magnesium-Fluorogermanate doped with manganese:

\[ \text{Mg}_4\text{FGeO}_6: \text{Mn} \]
Spectra

Mg₄FGeO₆:Mn

Excitation
Possible by standard laser wavelength

Emission
may interfere with luminous combustion
Experimental Setup – 0D

A - Aperture
BD - Beam dump
D - Dichroitic mirror
He-Ne - Helium neon laser
M - Mirror
P - Polarizator
PB - Pellin-Brocca prism
$\lambda/2$ - Halfwave plate
Experimental Setup – 2D

CMOS Kamera LaVision HSS6:

• Dynamic Range: 12 bit
• max. frame rate of 675 kHz at a resolution of 64 x 16 Pixels
Calibration in well-controlled environments (oven)

- Phosphor: Mg$_4$FGeO$_6$:Mn
- Substrate: Stainless Steel (1.4301)
- Gas phase pressure: 1 bar (air)
- Detection: PMT at 500 Ω
1. Introduction

2. Measurement chain / Error sources

3. Applications

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Measurement chain

Actual Measurement

- Measurand
  - Heat transfer
    - Temperature of the phosphor
  - Phosphor / Photophysics
    - Luminescence properties
  - Signal detection
    - Signal response
  - Algorithm
    - Scalar value

Comparison

- Measured value

Calibration

- Temperature of the calibration environment
  - Heat transfer
    - Temperature of the phosphor
  - Phosphor / Photophysics
    - Luminescence properties
  - Signal detection
    - Signal response
  - Algorithm
    - Scalar value

Thermometer

Brübach, J.; ISA 55th Int. Instrumentation Symposium, 2009
Measurement chain

Actual Measurement:
- Measurand
  - Heat transfer
    - Temperature of the phosphor
  - Phosphor / Photophysics
    - Luminescence properties
  - Signal detection
    - Signal response
  - Algorithm
    - Scalar value

Comparison:
- Measured value

Calibration:
- Heat transfer
  - Temperature of the calibration environment
  - Phosphor / Photophysics
    - Luminescence properties
  - Signal detection
    - Signal response
  - Algorithm
    - Scalar value

Temperature reading of the thermometer
Thermometer

Brübach, J.; ISA 55th Int. Instrumentation Symposium, 2009
Error class 1: Thermal interactions

1. Heat transfer
   - Excitation
   - Temperature of the phosphor
   - Temperature of the calibration environment

2. Phosphor / Photophysics
   - Environment
   - Heat treatment
   - Luminescence properties

3. Signal detection
   - Interferences
   - Signal response

4. Algorithm
   - Scalar value

5. Comparison
   - Temperature reading of the thermometer
   - Measured value

6. Thermometer
Error class 1: Thermal interactions

- Device under test (DUT) and phosphor or phosphor and calibration thermometer are not in thermal equilibrium

- Impact of the presence of the phosphor on the thermal state of the DUT (e.g. heat insulation) → “semi-invasive” method

- Excitation induced heating of the phosphor → check by power scan
Error class 2: Photophysical properties of the phosphor
Error class 2: Photophysical properties of the phosphor

- Impact of the transfer function of the phosphor
  - Temperature sensitivity, varies with temperature
  - Temporal low pass character due to finite decay time (thermal inertia versus decay time)

- Parameters that manipulate the transfer function of the phosphor
  - Diffusion processes between the phosphor material and the substrate due to heat treatments
  - Chemical and physical environment (oxygen quenching)
  - Laser excitation (variations with intensity)
Error class 2: Excitation irradiance

- Decay behaviour “more multi-exponential” at higher laser intensities
- Significant influence of the position of the fitting window

Error class 2:
Decay time @ 294 K after heat treatment

![Graphs showing decay time vs. max. temperature for stainless steel and powder samples.]

- **Dependency on:**
  - Substrate material
  - Duration of the heat treatment
  - Heat treatment temperature

Diffusion process between phosphor and substrate

Error class 2: 
Surrounding gas phase

\[ \text{Mg}_4\text{FGeO}_6:\text{Mn} \]

No significant dependency on the composition and pressure of the gas phase
Error class 2: 
Surrounding gas phase

\[ Y_2O_3:Eu \]
Dependency on the oxygen concentration

Error class 3: Signal detection

- **Measurand**
  - Excitation
  - Temperature of the calibration environment

- **Heat transfer**
  - Excitation
  - Temperature of the phosphor

- **Phosphor / Photophysics**
  - Excitation
  - Environment
  - Heat treatment
  - Luminescence properties

- **Signal detection**
  - Interferences
  - Signal response

- **Algorithm**
  - Scalar value

- **Comparison**
  - Temperature reading of the thermometer
  - Measured value

- **Phosphor / Photophysics**
  - Excitation
  - Environment
  - Heat treatment
  - Luminescence properties

- **Signal detection**
  - Interferences
  - Signal response

- **Algorithm**
  - Scalar value

- **Thermometer**
Error class 3: Signal detection – overview

- Impact of the transfer function of the detection system
  - limited spatial, temporal and spectral resolution
  - nonlinear behaviour of the detector (CMOS camera)

- Parameters that manipulate the transfer function of the detection system
  - small changes in the alignment (intensity ratio, see PCI 33-paper 2013)
  - terminating resistor, BNC length, amplifier (PMT, decay time)

- Optical and electrical interferences
  - optical interf. (e.g. background radiation, CL, fluorescence of substrate,…) most often temporally not correlated (worse precision)
  - electrical interf. (e.g. high voltage Q-switch electronics) might be temporally correlated (worse accuracy)

- Dynamic DUTs (e.g. moving surfaces)
  - signal decay might be superimposed by spatial variations of the absolute luminescence intensity; depending on the homogeneity of the excitation irradiance and the phosphor coating as well as on spatial temperature variation
Error class 3:
Signal detection – PMT versus CMOS

- Photomultiplier unproblematic
- CMOS camera shows problems regarding
  → Linearity
  → Pixel-to-pixel homogeneity
  → Offset stability

Error class 3: Non-linear detector behaviour

Correction function for each pixel fixes the problem

Error class 3:
Coating thickness and CMOS frame rate

Variation of coating thickness and luminescence intensity

Increasing Thickness

Variation of frame rate:

~70 values

~7 values probing the decay

Resulting Temperature:

299.4 K ±3.0 K
298.0 K ±3.3 K
297.4 K ±5.7 K

Minimum of standard deviation at high temporal discretisation.

No significant influence!
Error Class 3: Parameters impacting the transfer function

- Modified low pass character of the detection system (PMT) due to terminating resistor
- Might strongly change the calibration curve, especially at low decay times
Error class 4:
Algorithm for the data reduction

Andreas Dreizler / TU Darmstadt
Problem: Decay characteristics are not single-exp.

Given the decay function $I(t) = I_0 \exp \left( -\frac{t}{\tau} \right)$, the issue at hand is to accurately fit the data within a predefined or iterative fitting window.

**Predefined fitting window:**
- $t_1 = t_0 + c_1 \cdot \Delta t$
- $t_2 = t_0 + c_2 \cdot \Delta t$

**Iterative fitting window:**
- $t_1 = t_0 + c_1 \cdot \tau$
- $t_2 = t_0 + c_2 \cdot \tau$

Error class 5: Comparison of the scalar values
Error class 5: Comparison of the scalar values

- Error depending on the calibration temperature intervals and the quality of the interpolation in between the calibration points.

- High error potential in strongly curved regimes of the calibration curve.

- Calibration intervals of at least $\Delta T = 20K$ or even $\Delta T = 10K$ are recommended.
Error class 6:
Uncertainty of the calibration thermometer
Error class 6: Uncertainty of the calibration thermometer

- Most often, thermocouples are employed

- High quality thermocouples offer an accuracy of better than 0.4 % of the absolute temperature.
Error Review

Provided a careful practice: → Systematic Error < 1 %

<table>
<thead>
<tr>
<th>Error class</th>
<th>max. error</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Heat transfer</td>
<td>?</td>
</tr>
<tr>
<td>2. Photophysics</td>
<td>( O (10^1 \text{ K}) )</td>
</tr>
<tr>
<td>Excitation</td>
<td>( O (10^2 \text{ K}) )</td>
</tr>
<tr>
<td>Dopant concentration</td>
<td>( O (10^1 \text{ K}) )</td>
</tr>
<tr>
<td>Heat treatments</td>
<td>( O (10^2 \text{ K}) )</td>
</tr>
<tr>
<td>Surrounding gas phase</td>
<td>( O (10^2 \text{ K}) )</td>
</tr>
<tr>
<td>3. Detection system</td>
<td>( O (10^2 \text{ K}) )</td>
</tr>
<tr>
<td>4. Algorithm for data reduction</td>
<td>( O (10^1 \text{ K}) )</td>
</tr>
<tr>
<td>5. Comparison of calibration and measurement</td>
<td>( O (10^1 \text{ K}) )</td>
</tr>
<tr>
<td>6. Accuracy of the calibration thermocouple</td>
<td>( O (10^0 \text{ K}) )</td>
</tr>
</tbody>
</table>

Statistical Error:

Shot-to-shot standard deviation: approx. 2 K

Brübach, J.; ISA 55th Int. Instrumentation Symposium, 2009
1. Introduction

2. Measurement chain / Error sources

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4. Summary
RSM Combustor

<table>
<thead>
<tr>
<th>Combustor operation Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_3$</td>
</tr>
<tr>
<td>$P_3$</td>
</tr>
<tr>
<td>$m_3$</td>
</tr>
<tr>
<td>$\varphi_{\text{LeanPremixed}}$</td>
</tr>
<tr>
<td>$\varphi_{\text{LeanPremixed+Pilot}}$</td>
</tr>
<tr>
<td>$S$</td>
</tr>
</tbody>
</table>

RSM Combustor

- **Standard operation point**

  **Operation conditions**

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_3 )</td>
<td>623K</td>
</tr>
<tr>
<td>( P_3 )</td>
<td>0.25 MPa</td>
</tr>
<tr>
<td>( \dot{m}_3 )</td>
<td>0.030kg/s</td>
</tr>
<tr>
<td>( \Phi_{\text{LeanPremixed}} )</td>
<td>0.65/0% Pilot</td>
</tr>
<tr>
<td>( \Phi_{\text{LeanPremixed}+\text{Pilot}} )</td>
<td>0.65/10% Pilot</td>
</tr>
<tr>
<td>( S )</td>
<td>0.7</td>
</tr>
<tr>
<td>( \dot{m}_5 )</td>
<td>0.0125 kg/s</td>
</tr>
<tr>
<td>( T_5 )</td>
<td>623 K</td>
</tr>
</tbody>
</table>

  ![Chemiluminescence images from piloted and premixed flames](image)

  Low-pass (490nm) filtered
  Chemiluminescence images from piloted and premixed flames
Selected results: wall thermometry

- 2d wall temperature fields

Magnified view of the coated liner. Coating thickness $t \ 10\text{um} < t < 50\text{um}$

Mean and standard deviation from 500 single shots at the liner surface

Optically accessible combustion engine

CMOS
Optically accessible combustion engine


Temperatur at exhaust valve:
- DI-ICE @ 2000 min⁻¹
- Coating: Gd₃Ga₅O₁₂·Cr + Ulfalux Ofenlack
- Spatial resolution: 188 µm/Pixel @ 64x64 Pixel (360 kHz framerate)
Optically accessible combustion engine

**Fired engine operation:**
- Temperature measurement at selected CAD

Extension to high speed phosphor thermometry in ICE

- Former measurements use 10Hz laser-systems for phosphor thermometry
- Temperatures originate from different cycles → averaging of uncorrelated single shots

High speed phosphor thermometry
Use laser at high repetition rate to resolve temperatures within cycles
Resolving singly cycles by high speed TGP

- **High speed phosphor thermometry**
  - Very fast decaying phosphors
  - Well below 167µs (1°C at 1000 rpm)

![Graph showing lifetime vs temperature](image)

*Mg₂TiO₄:Mn⁴⁺*

Steep increase due to detection system. Can be optimized.

[3]
Influence of laser-induced heating

- Challenges when using High Speed Phosphor Thermometry:
  - → Laser-induced heating effects

- Due to high power delivered by laser
- Quantify by energy-scan
- Trade-off between precision and accuracy

Optimum at $E = 13\mu$J:
Standard deviation below 1K
Systematic error of 2.5K
Set-up and realization

- **Experimental Setup**
  - Spark plug dummy
  - Inlet valves
  - Outlet valves
  - Quartz glass ring and piston disc
  - Broadband mirror
  - Photomultiplier-tube
  - High-speed UV-laser, 266nm, 6kHz

**Coating**
- Coated via airbrush
- Dispersion of binder and phosphor

**Engine**
- Motored at 1000rpm
- No injection and ignition
- Compression ratio 8.5:1
Results for motored engine (no combustion)

- Temperature progression of the spark plug dummy

- Mean values and temporal standard deviations of 100 cycles
- Resolution of 1 ° CA
- Precision of about 1-2 K
Wall temperature measurements during flame wall interactions in IC engine

flame position

OH-PLIF (3 pulse burst)

Temperature at piston surface

2D-phosphor thermometry (crank-angle resolved)

Phosphor thermometry

- Decay-time method
  - Ex-situ calibration

- Engine requirements
  - Fast temperature measurements (~ 10µs)
  - High sensitivity, high SNR (~ 5K)

- Suitable thermographic phosphor: Gd$_3$Ga$_5$O$_{12}$:Cr,Cer
Experimental setup for studying flame-induced heating

1: Fresnel rhomb
2: Pol. beam splitter
3: Zyl. lens, f=-400
4: Zyl. lens, f=+1000
5: Iris
6: Zyl. lens, f=+400
7: Pelin Broca
8: Rect. homogenizer
9: Zyl. lens, f=+1000
10: Sph. lens, f=500
11: Zyl. lens, f=+200
12: UV-Lenses (Halle)
13: Thermographic camera
F1: Bandpass filter

flame
OH-PLIF signal
OH-PLIF excitation
TPT excitation
TPT emission
piston glass
Engine operational conditions

Heating up the engine for several runs $\rightarrow$ multiple runs recorded

- 300 cycles motored
- 100 fired cycles

100 cycles, every second is recorded

HS-TPT
15 Images from -6.5 to 14.5 CAD aTDC

OH-PLIF
3 Images at -2, -0.5 and 1.0 CAD aTDC
### Engine operational conditions

<table>
<thead>
<tr>
<th>Operating Point</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>fuel CH₄</td>
<td></td>
</tr>
<tr>
<td>p&lt;sub&gt;in&lt;/sub&gt;</td>
<td>950 mbar</td>
</tr>
<tr>
<td>T&lt;sub&gt;in&lt;/sub&gt;</td>
<td>32°C</td>
</tr>
<tr>
<td>Speed</td>
<td>800 rpm</td>
</tr>
<tr>
<td>Imep</td>
<td>5.3 bar</td>
</tr>
<tr>
<td>lambda</td>
<td>1.0 and 1.3</td>
</tr>
<tr>
<td>t&lt;sub&gt;ignition&lt;/sub&gt;</td>
<td>-11 and -16 CAD aTDC</td>
</tr>
</tbody>
</table>

![Graph](image)
Flame position and wall temperature

- coherence of flame impingement and wall temperature increase
- reproducible temperature evolution
Temperature rise during flame-wall interaction

- Maximum heating rates of up to 20,000 K/s
1. Introduction

2. Measurement chain / Error sources

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Summary

- Systems for 0D und 2D phosphor thermometry
- Decay-time method superior compared to ratio method with respect to precision and accuracy
- Quantification of systematic and statistic error
- Application (relatively) straight forward, even to complex systems
- High potential for further applications