

# Chapter 6: Surface Thermometry – Thermographic Phosphors

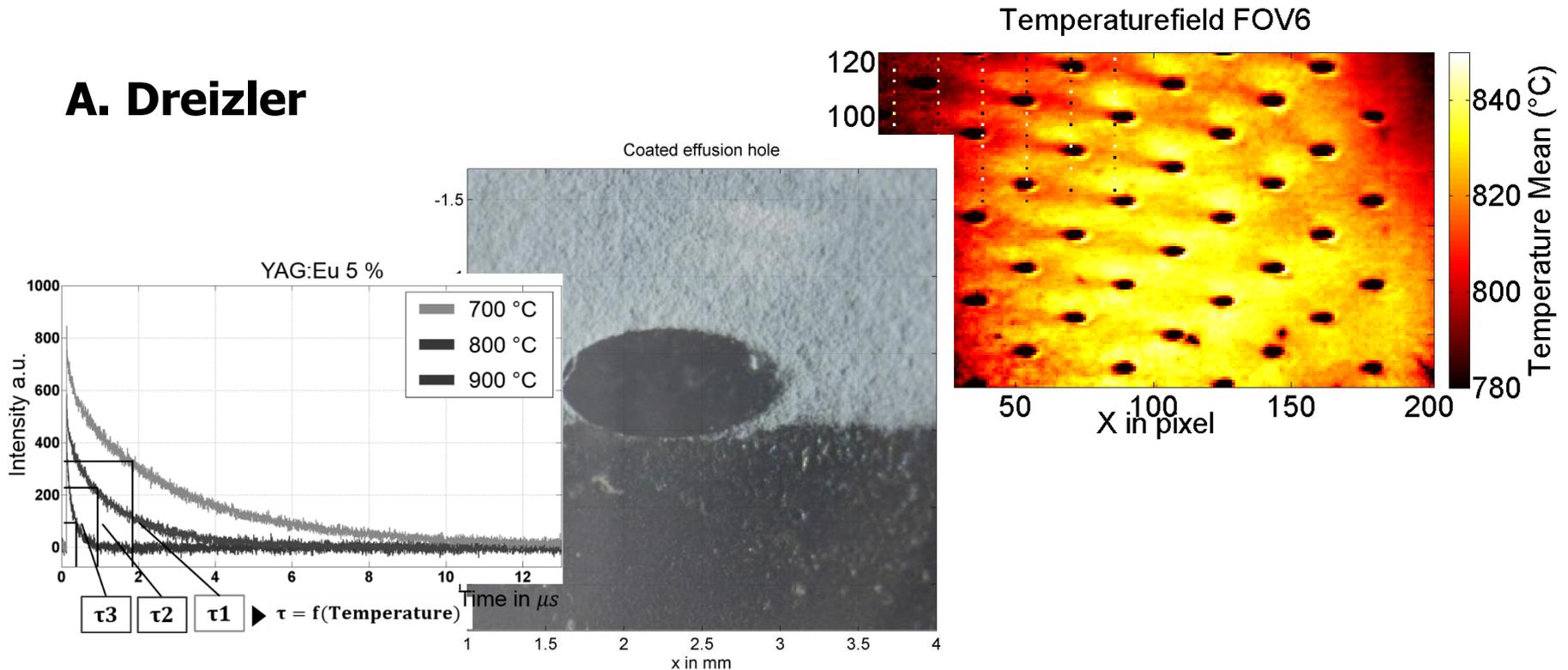
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TECHNISCHE  
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DARMSTADT



## A. Dreizler



# Outline phosphor thermometry



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



## 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



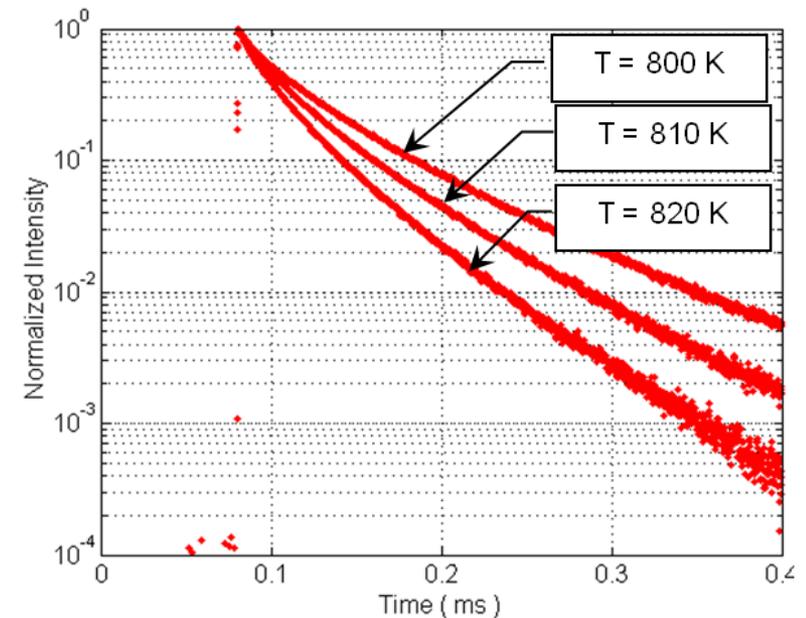
## Thermographic phosphors:

- Rare-earth or transition metal doped ceramic materials
- Exploit temperature dependent spectroscopic properties following electronic excitation

# Introduction & Motivation I

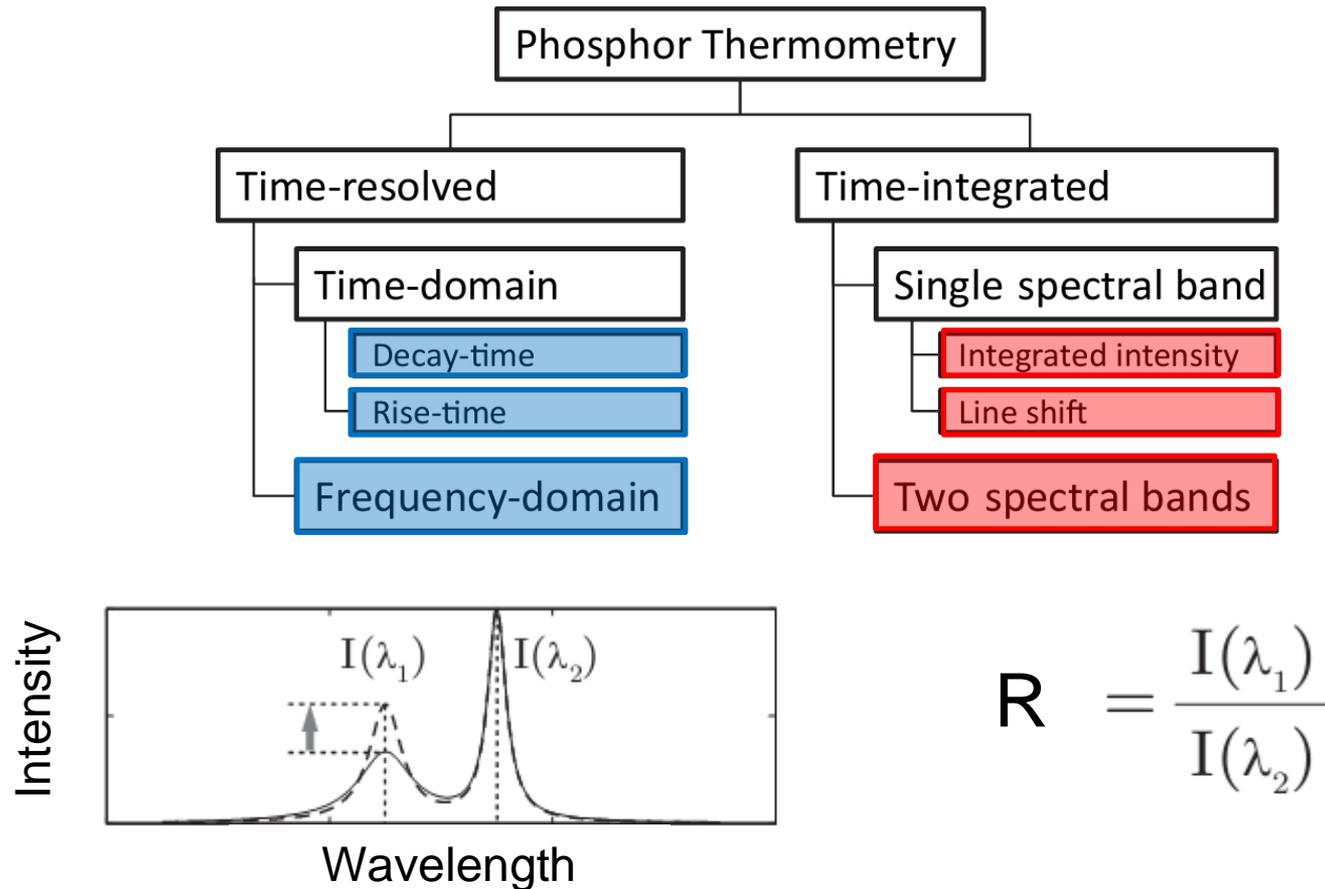
## Principle of Phosphor Thermometry:

- 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)



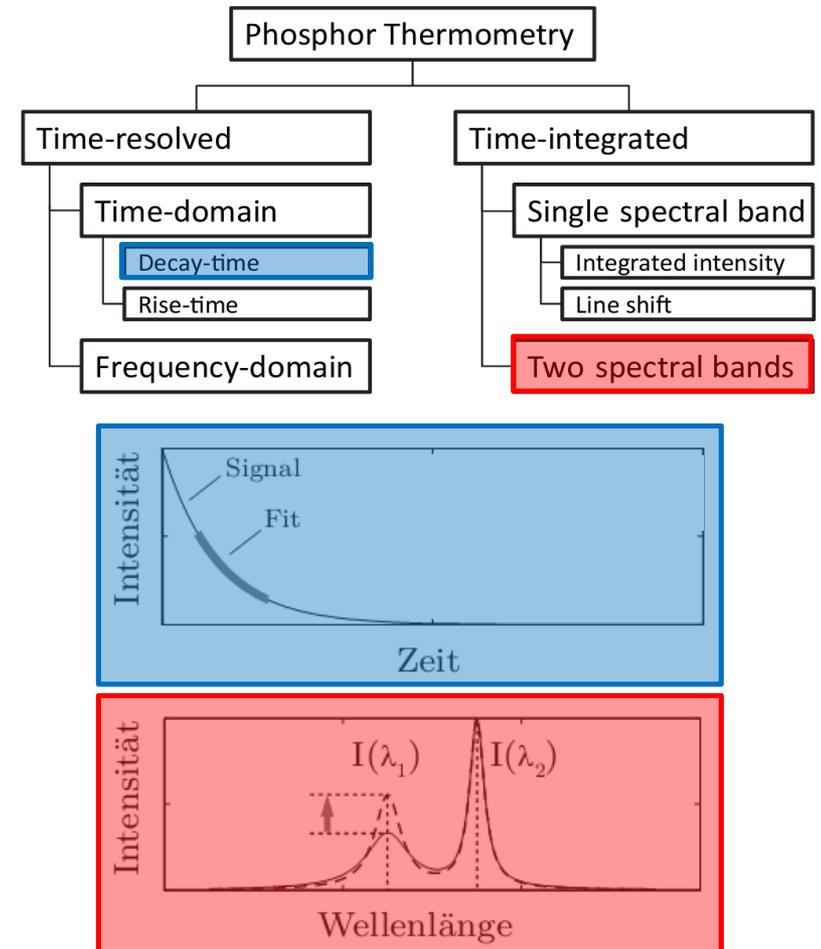
# Introduction & Motivation II

## Approaches of Phosphor Thermometry

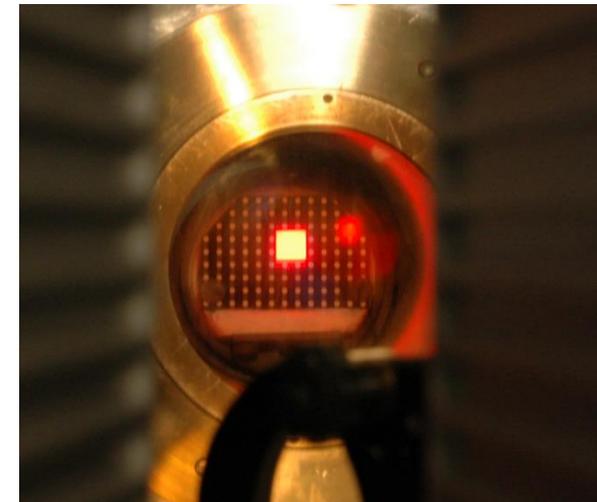
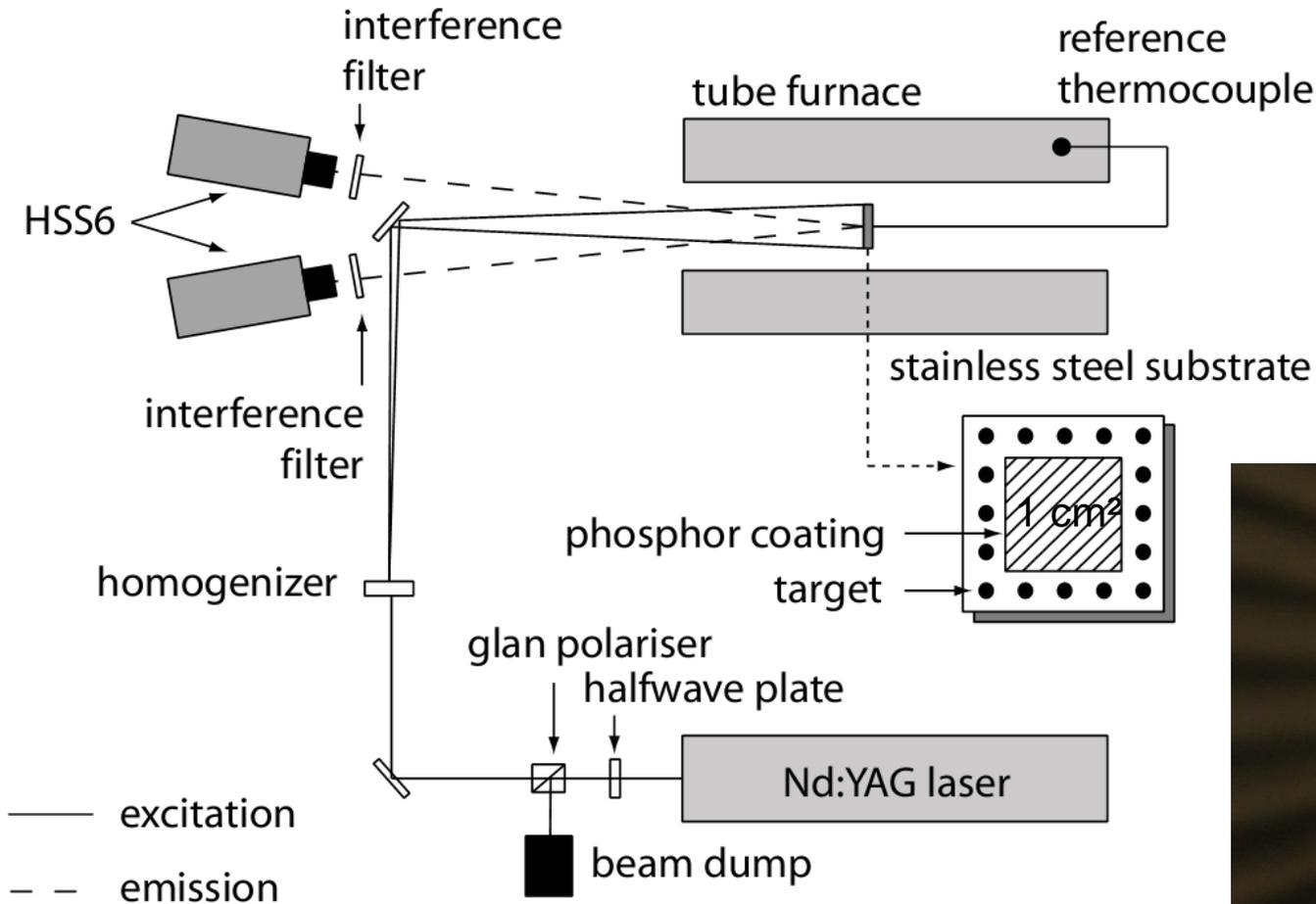


# Introduction & Motivation III

- 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:  $\text{Mg}_4\text{FGeO}_6:\text{Mn}$



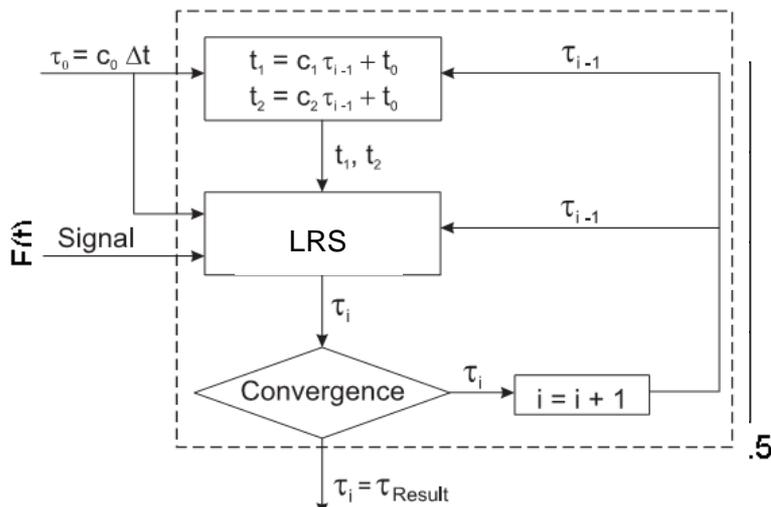
# Experimental Setup



- 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:

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

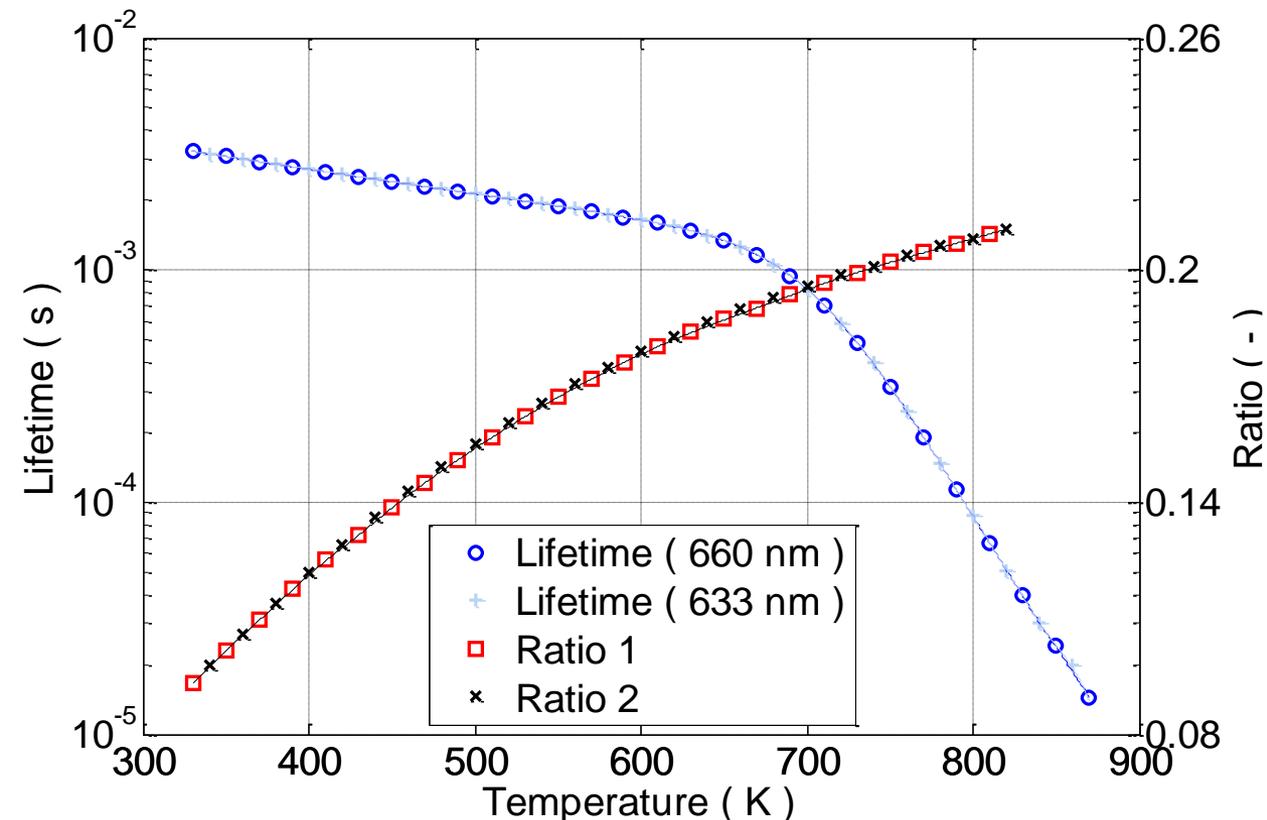
- Two ratios R1 and R2 used:

- R1:  $t_1 = t_0 + 100 \mu\text{s}$   
 $t_2 = t_0 + 120 \mu\text{s}$   
(corresponds to 1 image @50kHz)

- R2:  $t_1 = t_0 + 100 \mu\text{s}$   
 $t_2 = t_0 + 1100 \mu\text{s}$   
(corresponds to 50 images @50kHz)

# Results: Temperature dependent characteristics

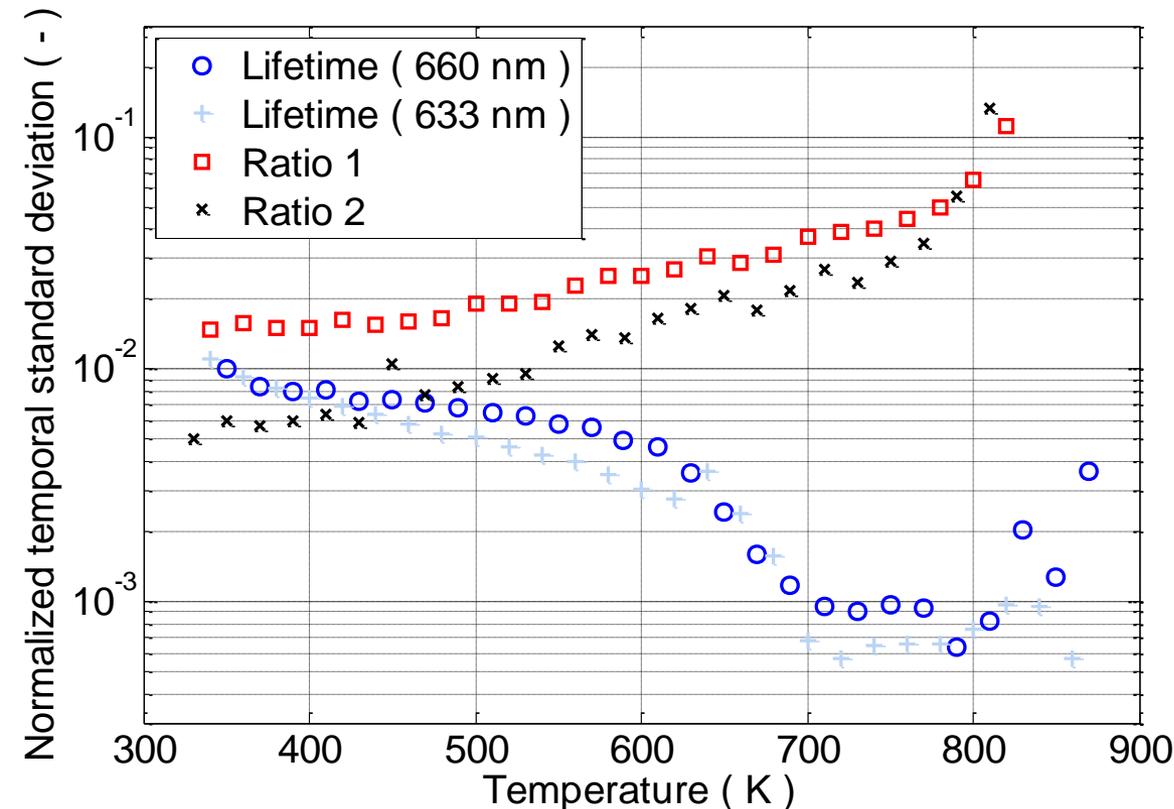
- Temperature-Lifetime / Temperature-Ratio characteristics



- 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

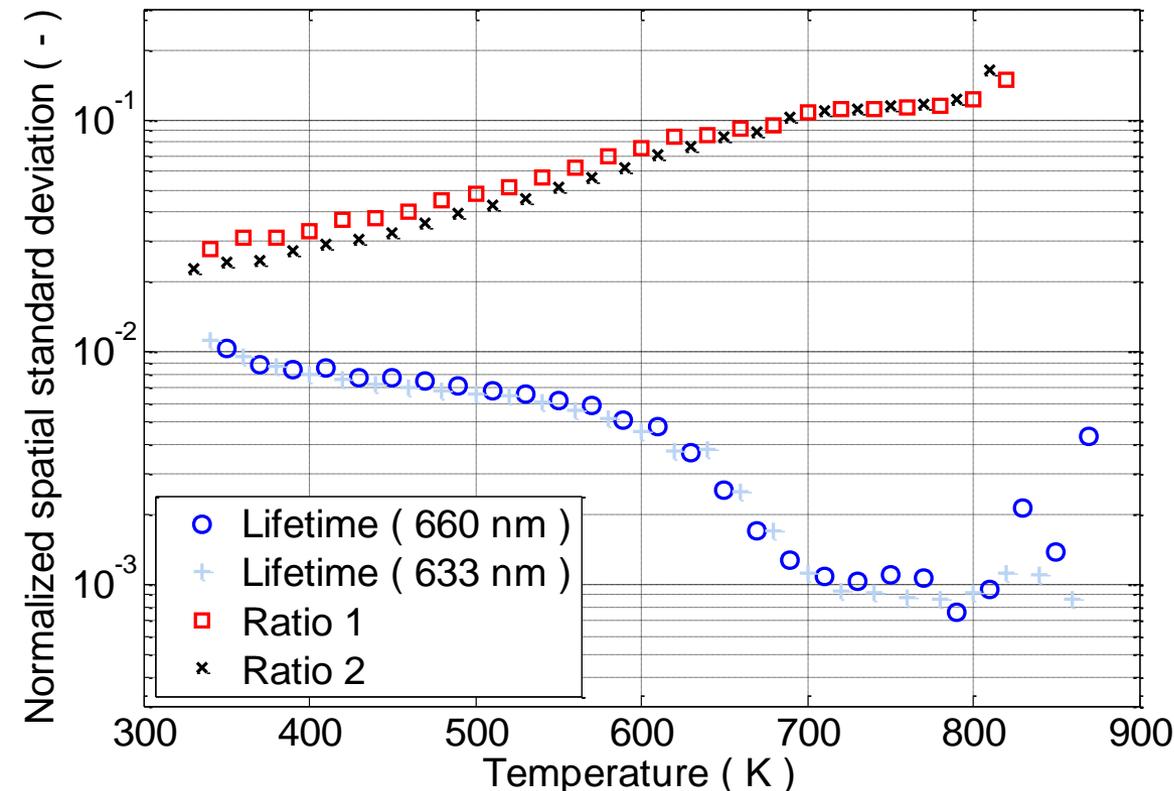
- Shot-to-Shot (**temporal**) standard deviations



- Comparable precision of both techniques at lower temperatures
- Lifetime method  $\sim 2$  orders of magnitude better at higher temperatures

# Results: Precision II

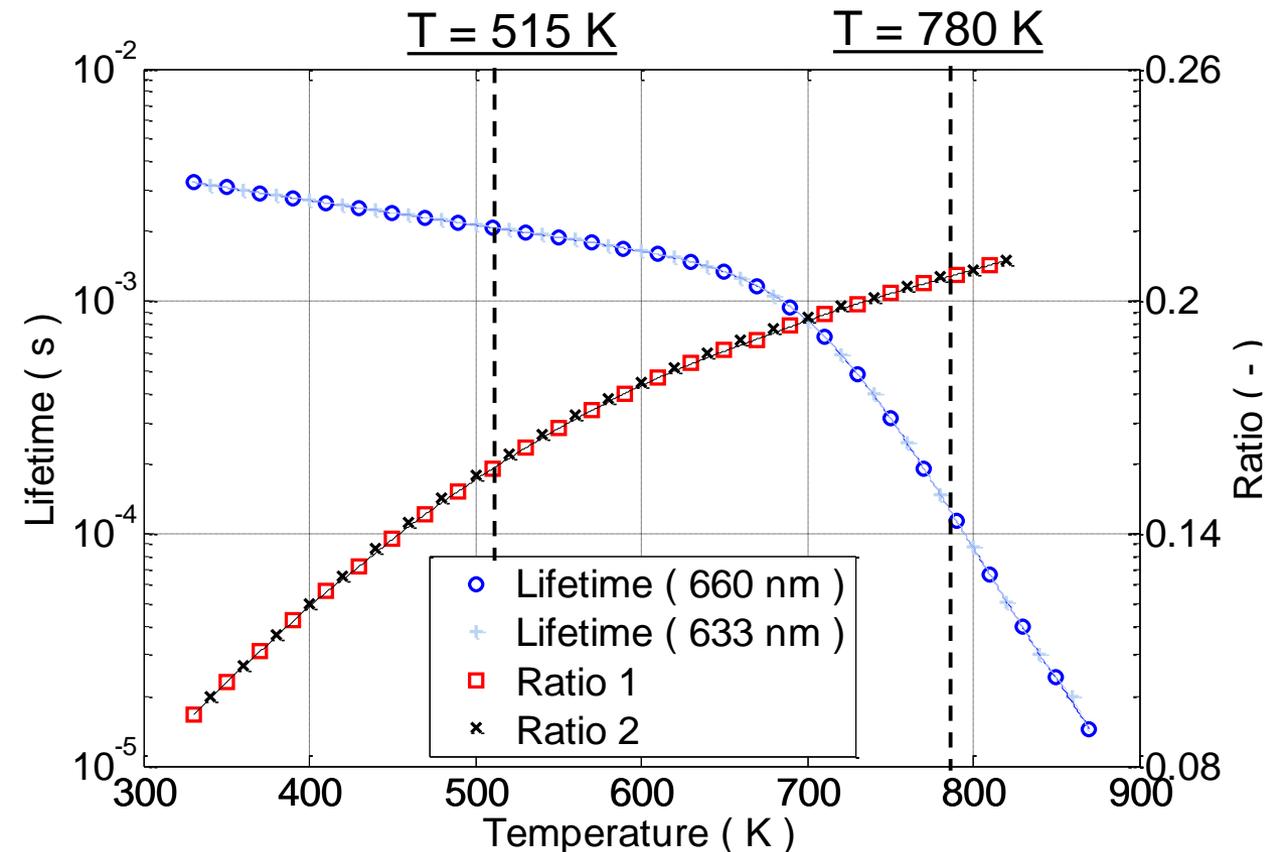
- Pixel-to-pixel (**spatial**) standard deviations



- 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

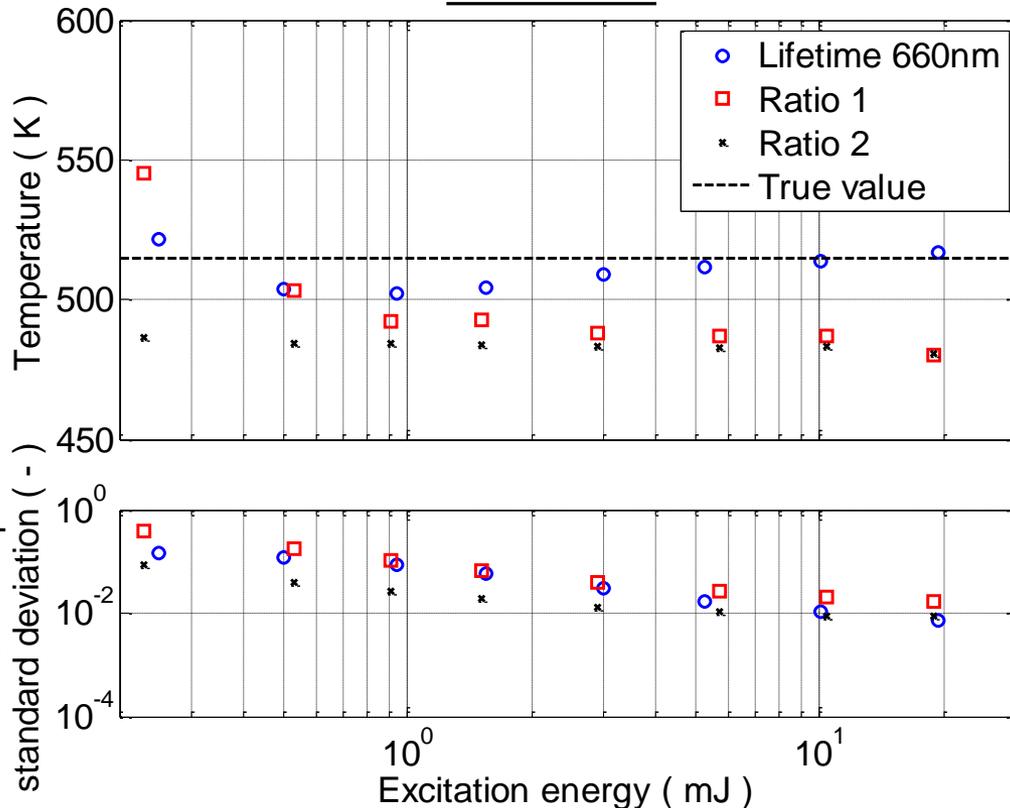


- Evaluated at two different temperatures corresponding to two different sensitivities in the temperature lifetime characteristic
- Calibration at fixed conditions and then parametric variation:
  - Energy variation
  - Changing settings in optical setup

# Results: Accuracy II

- Dependency on excitation energy at  $T = 515\text{K}$

$T = 515\text{K}$

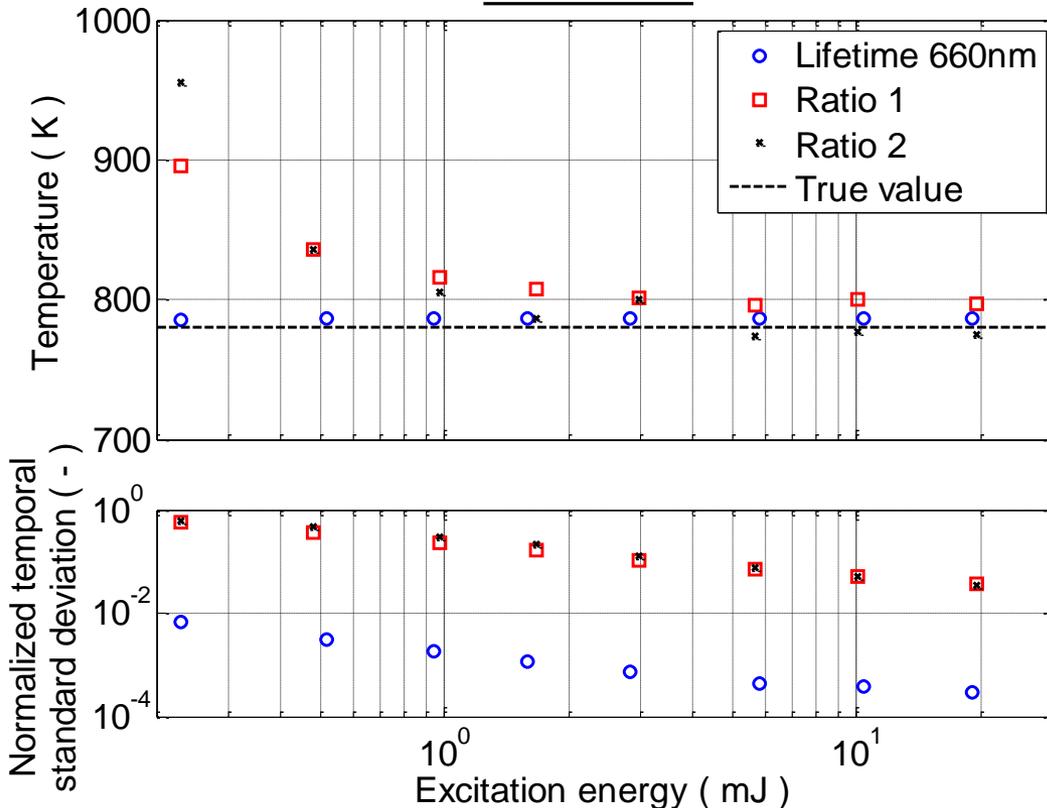


- Inaccuracy for too low excitations energies of lifetime approach well known (i.e. Brübach et al. 2008)
- Ratio 2 rather robust against energy variations, but inaccuracy over whole energy range

# Results: Accuracy III

- Dependency on excitation energy at  $T = 780\text{K}$

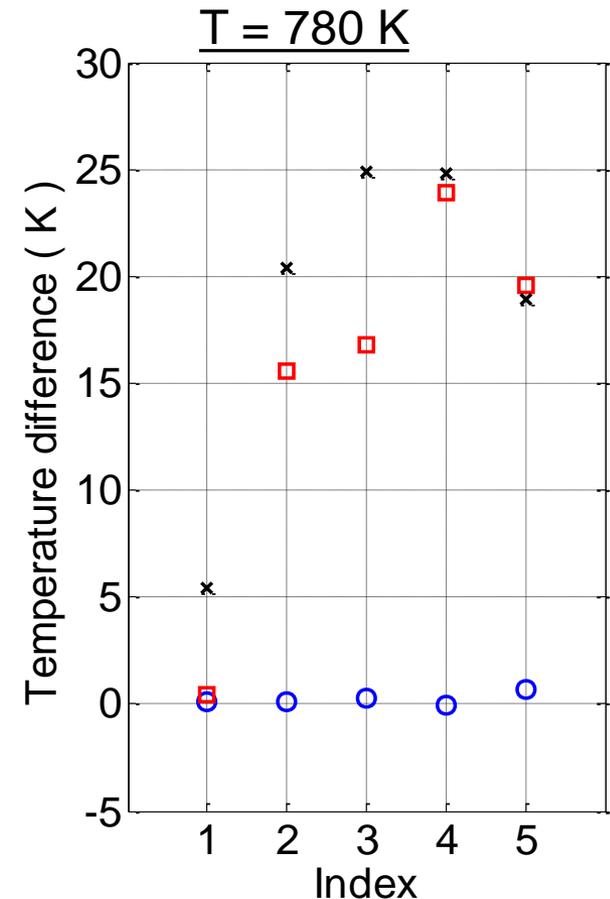
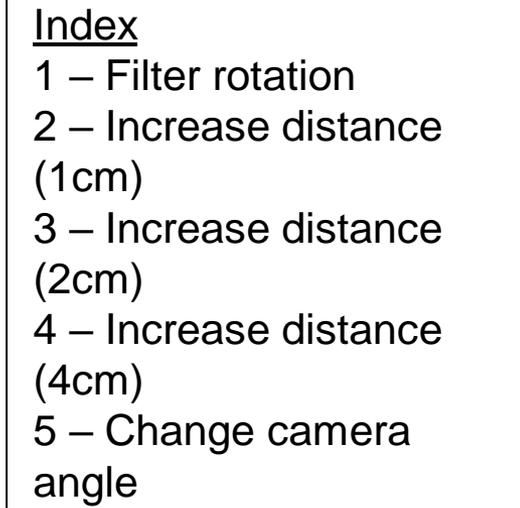
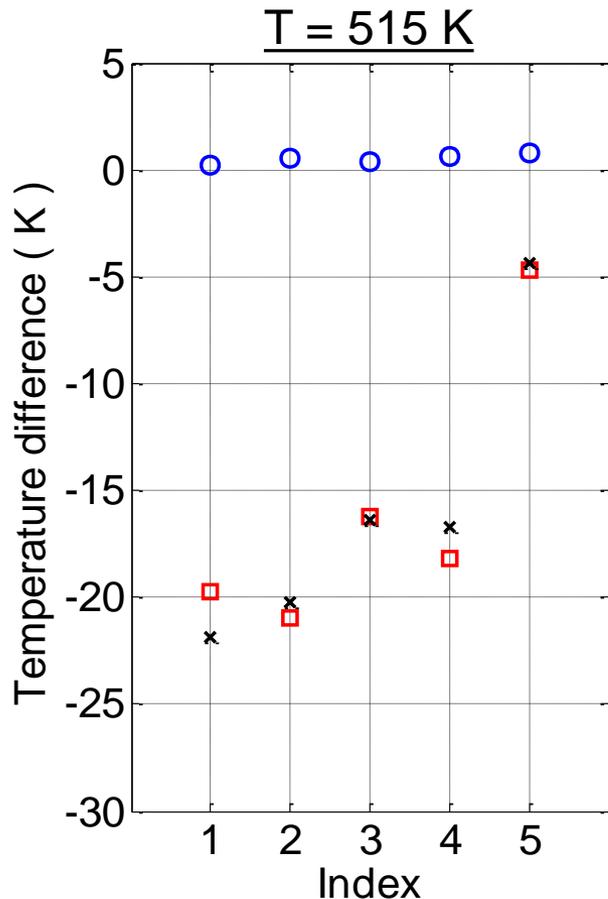
$T = 780\text{K}$



- Lifetime approach very accurate due to high sensitivity
- Ratio 1 & 2 show overestimation of more than 100 K at lower energies

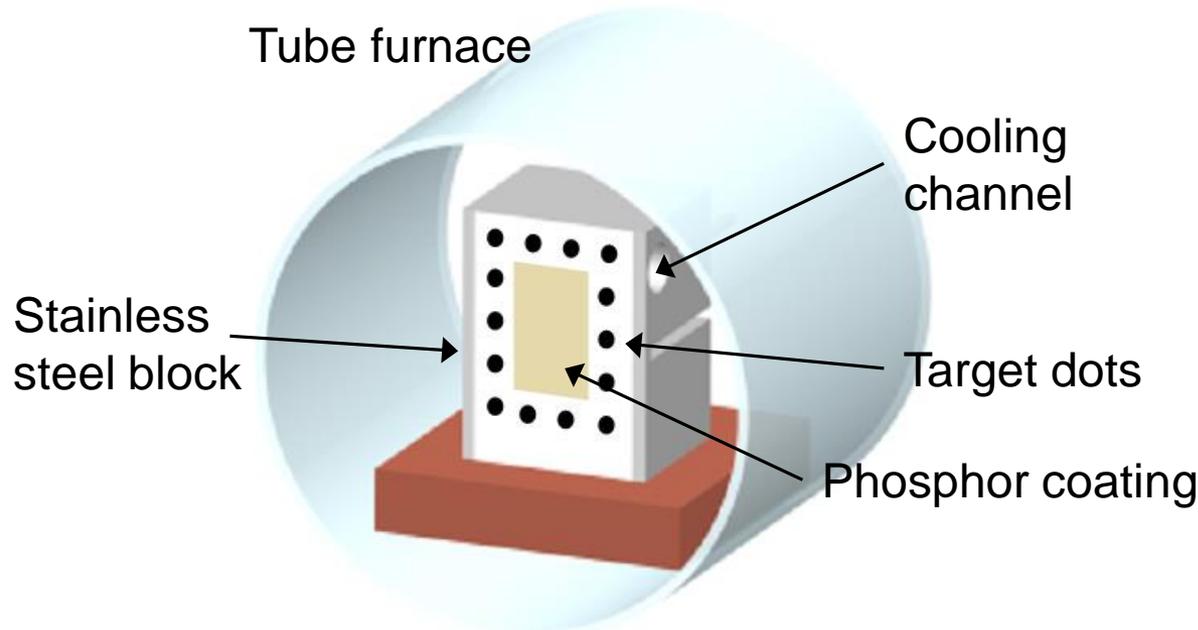
# Results: Accuracy IV

- Accuracy due to changes of settings in the optical setup



# Results: Cooling device I

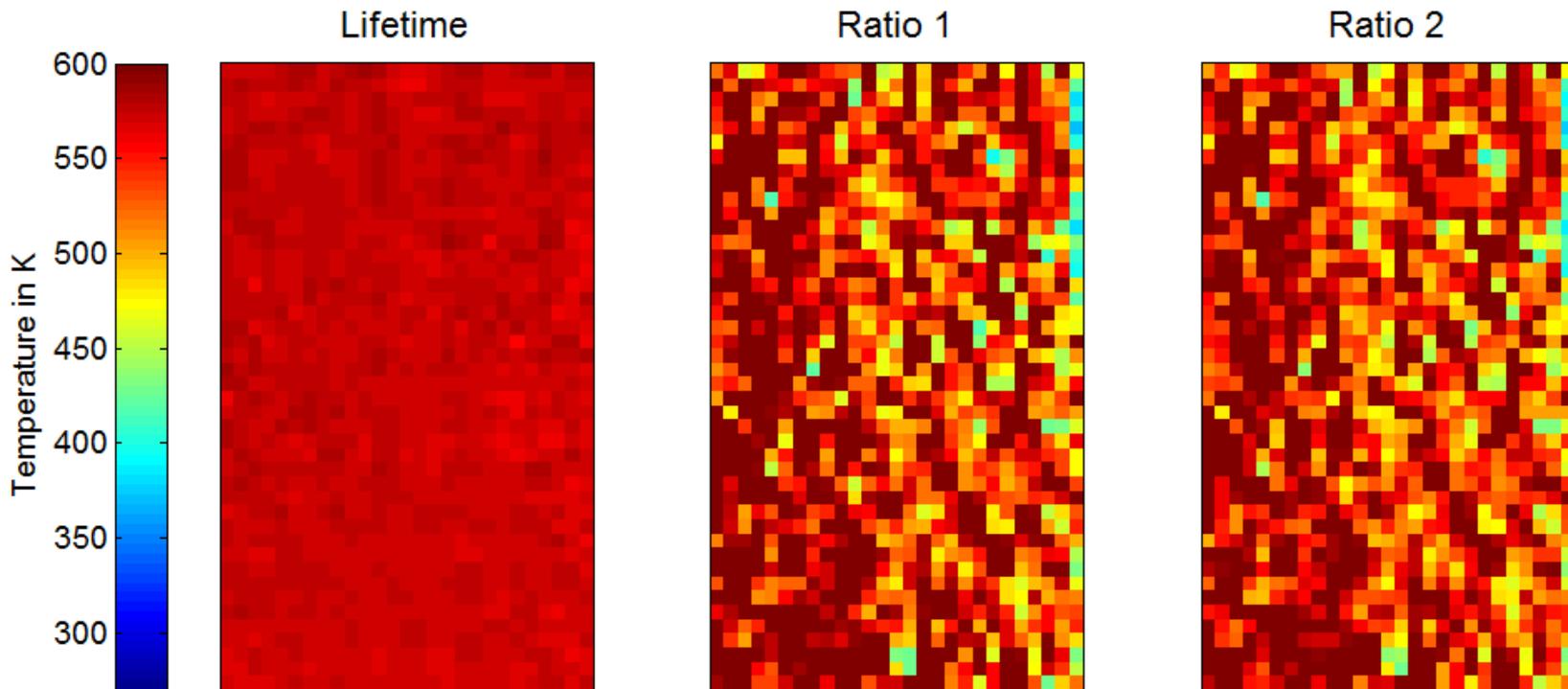
- Stainless steel block with cooling channel placed inside oven
- When block is at designated temperature, cold water is pumped through cooling channel
- For image mapping purposes target dots have been transferred to device



*Kissel, T., PhD Thesis, TU Darmstadt, 2011*

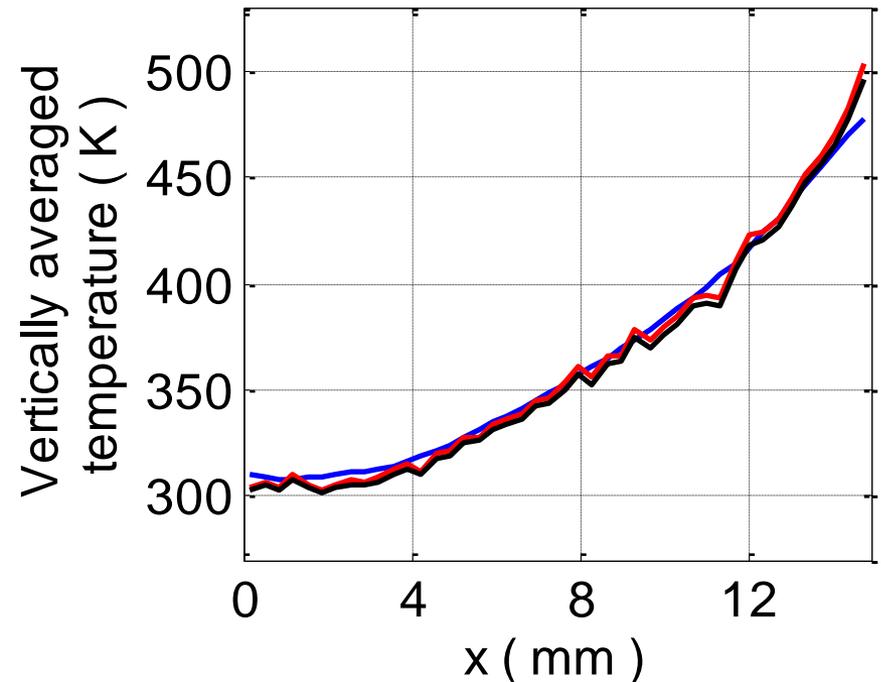
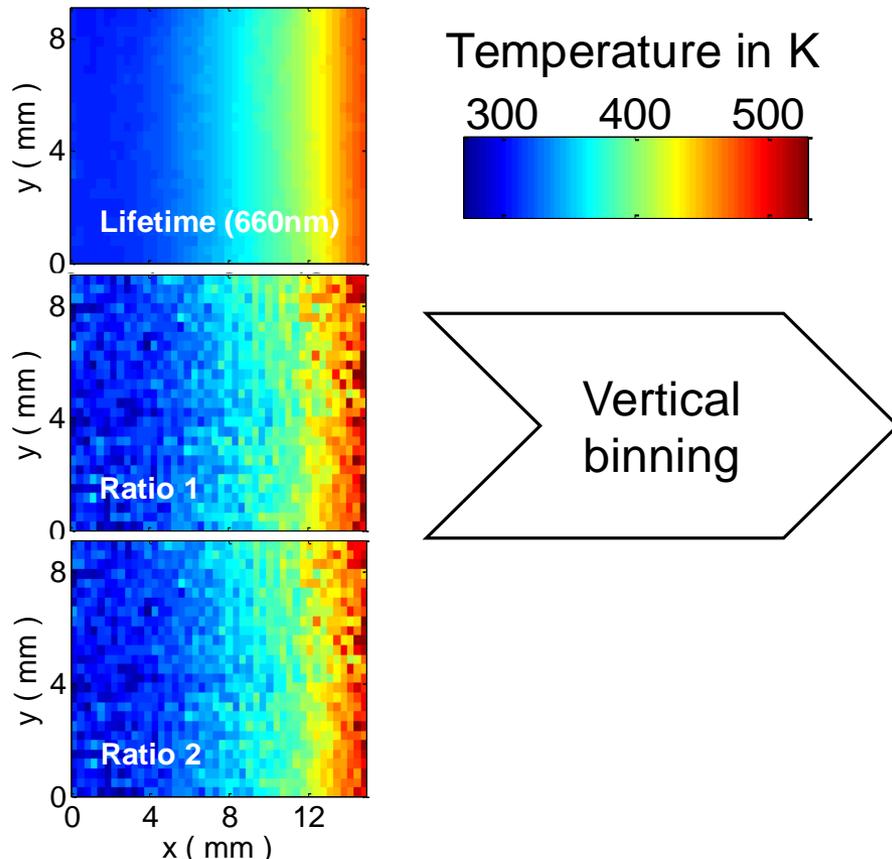
# Results: Cooling device II

- Transient Experiment
- Same set of data for all three methods



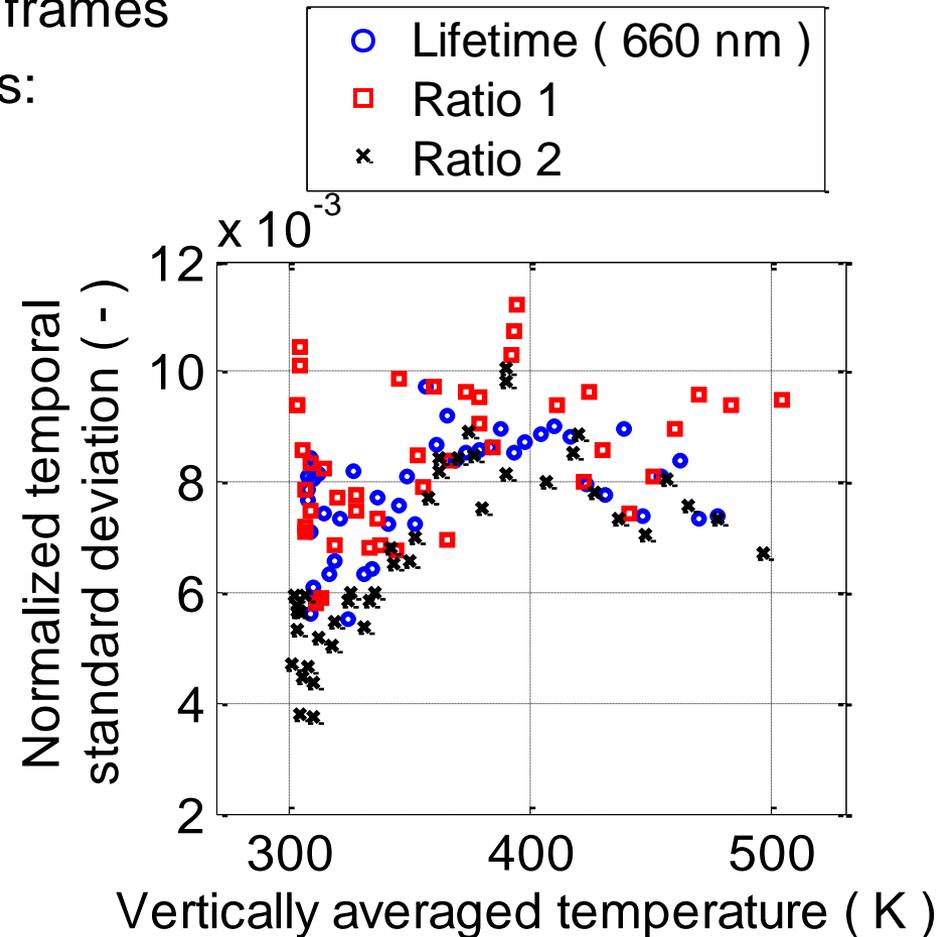
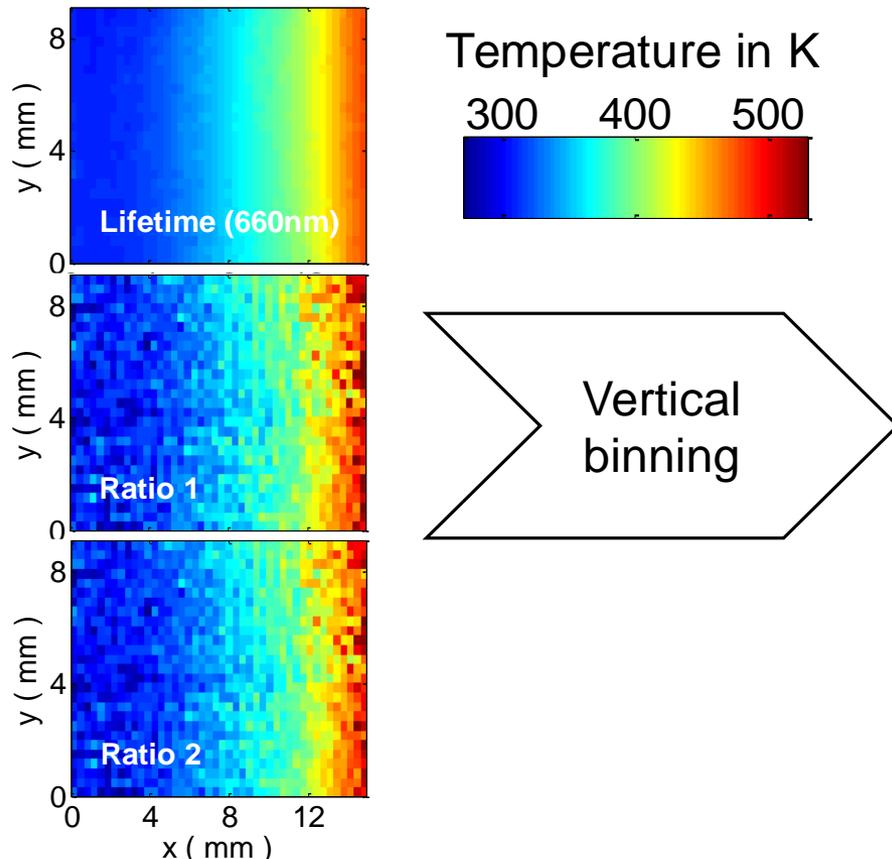
# Results: Cooling device III

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



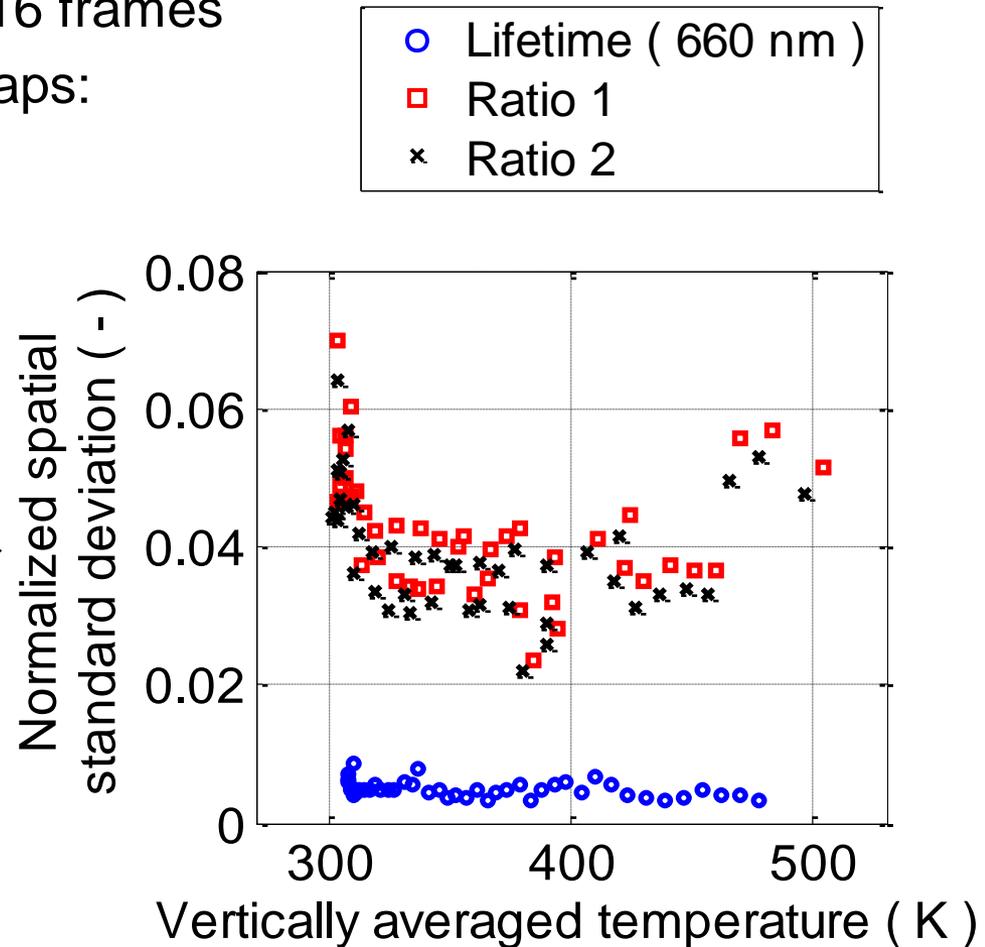
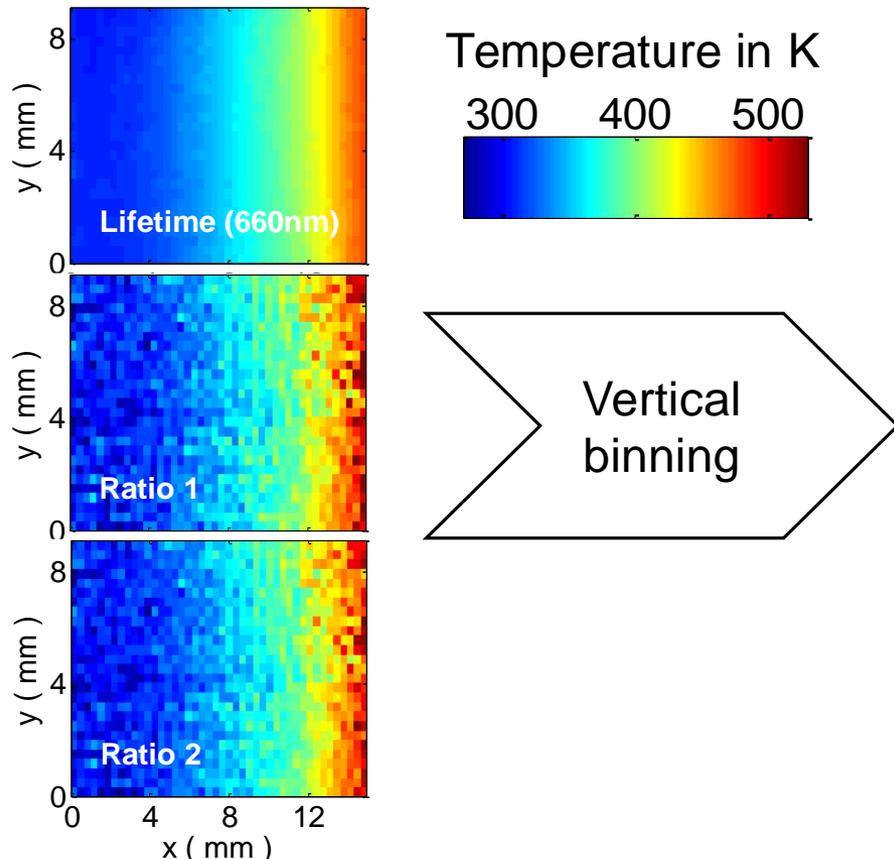
# Results: Cooling device III

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



# Results: Cooling device III

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



# Summary & Conclusions

Comparison of  $\text{Mg}_4\text{FGeO}_6$ : 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**

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

For **details** see: Brübach, Pflitsch, Dreizler, Atakan. Prog. Energy Combust. Sci. 39, 37 – 60 (2013)



# 1. Introduction

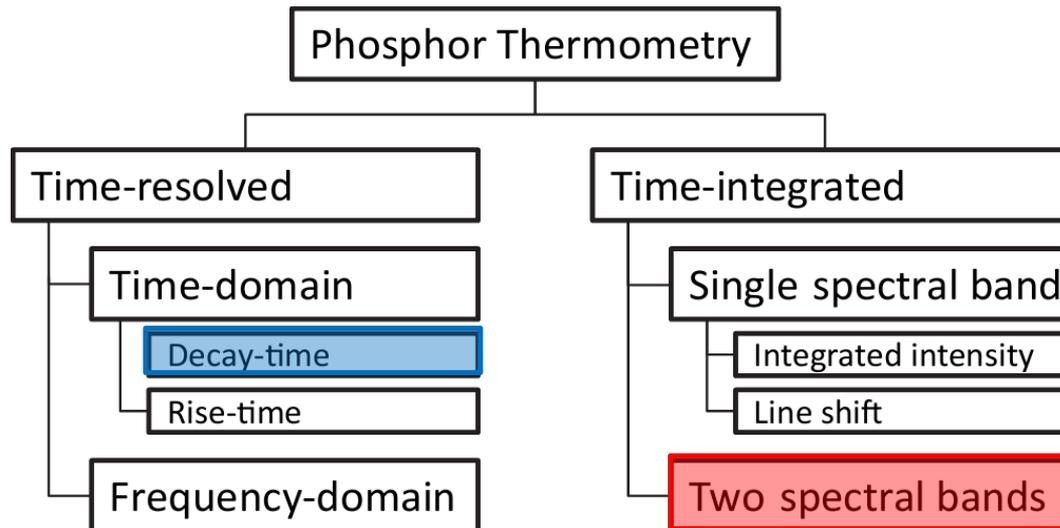
2. Measurement chain / Error sources

3. Applications

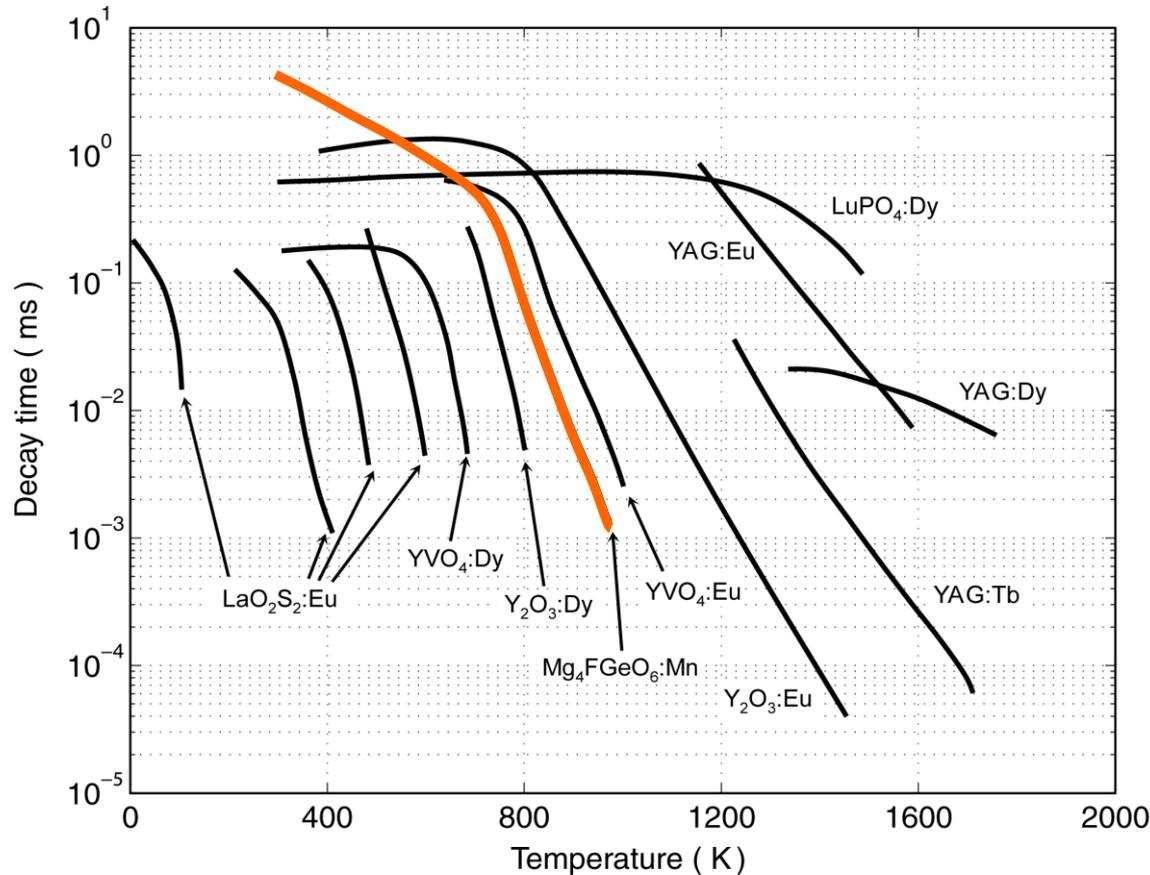
4. Summary

# 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:





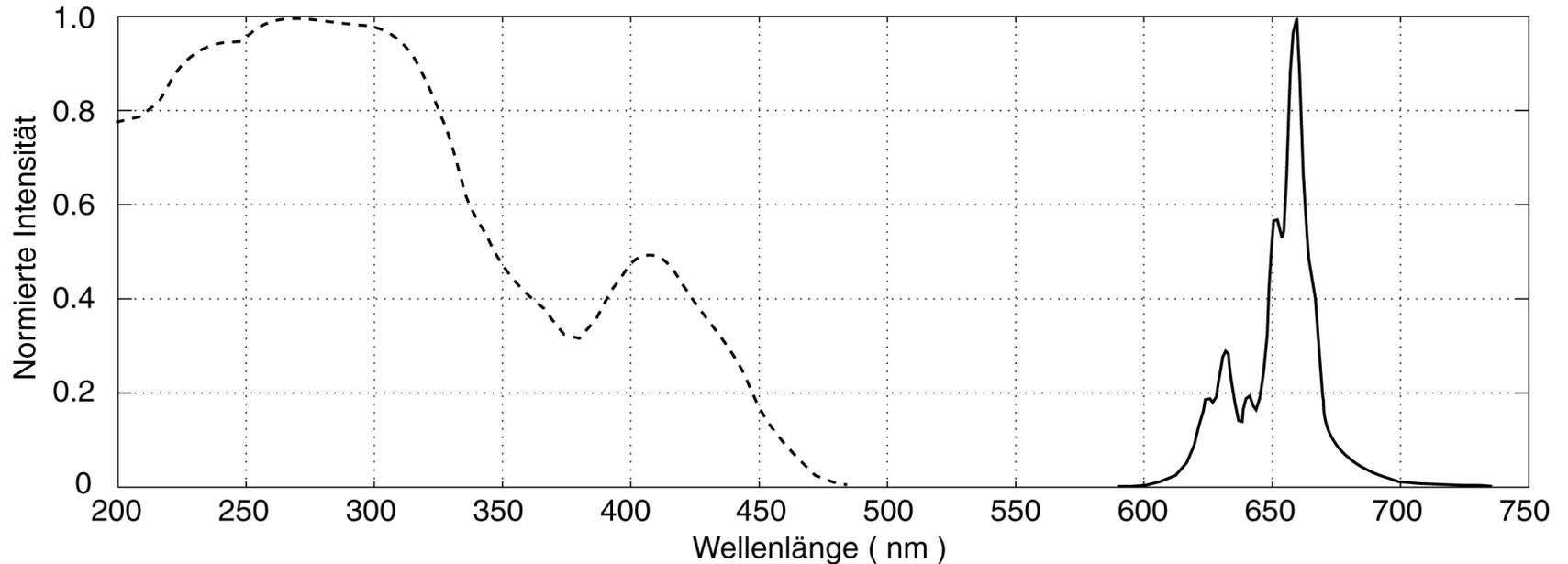
Excitation

Possible by standard laser wavelength



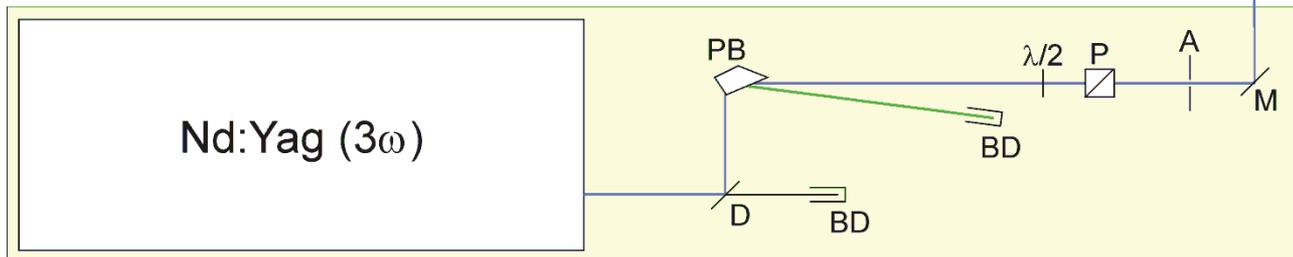
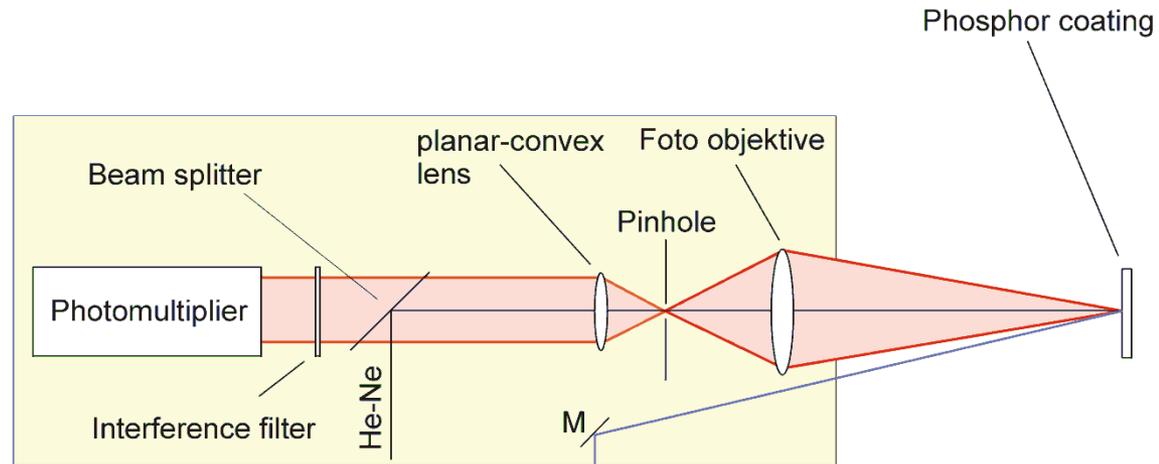
Emission

may interfere with luminous combustion



# Experimental Setup – 0D

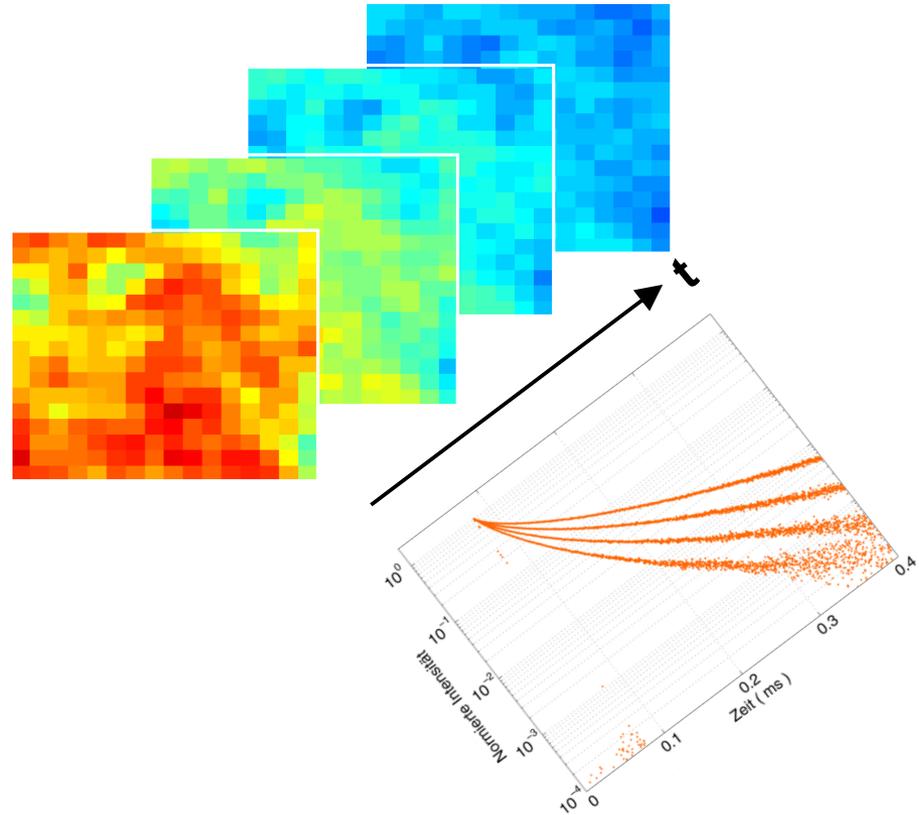
- A - Aperture
- BD - Beam dump
- D - Dichroitic mirror
- He-Ne - Helium neon laser
- M - Mirror
- P - Polarizator
- PB - Pellin-Brocca prism
- $\lambda/2$  - Halfwave plate



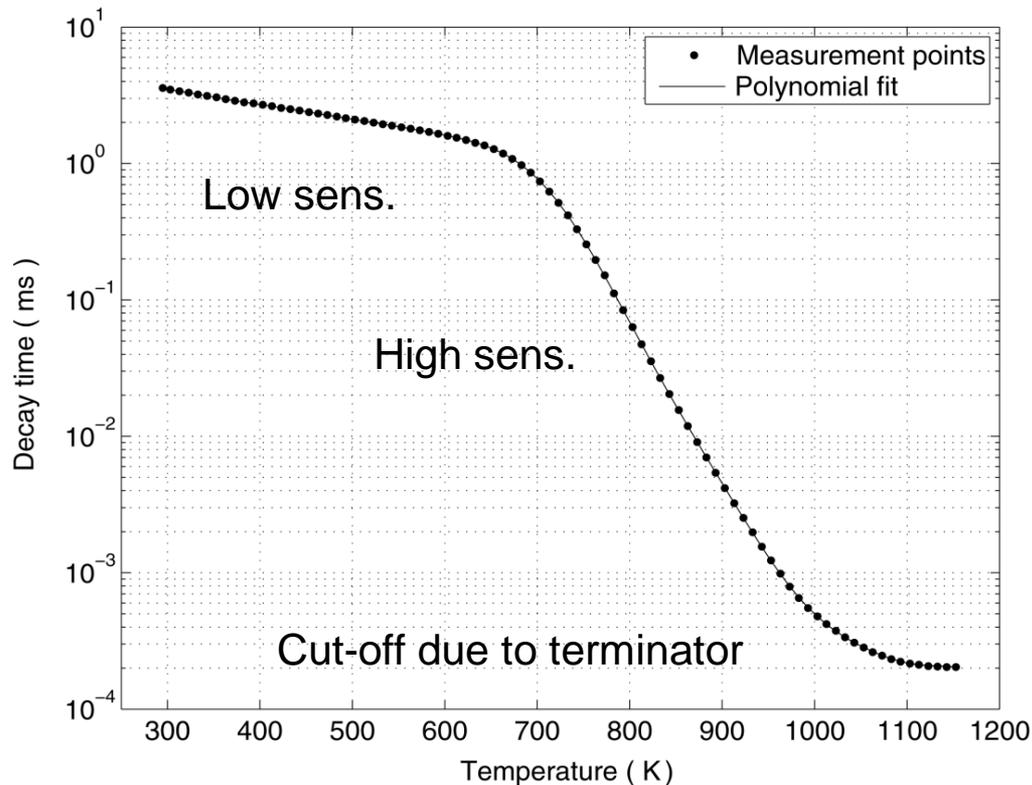
# Experimental Setup – 2D

## CMOS Kamera LaVision HSS6:

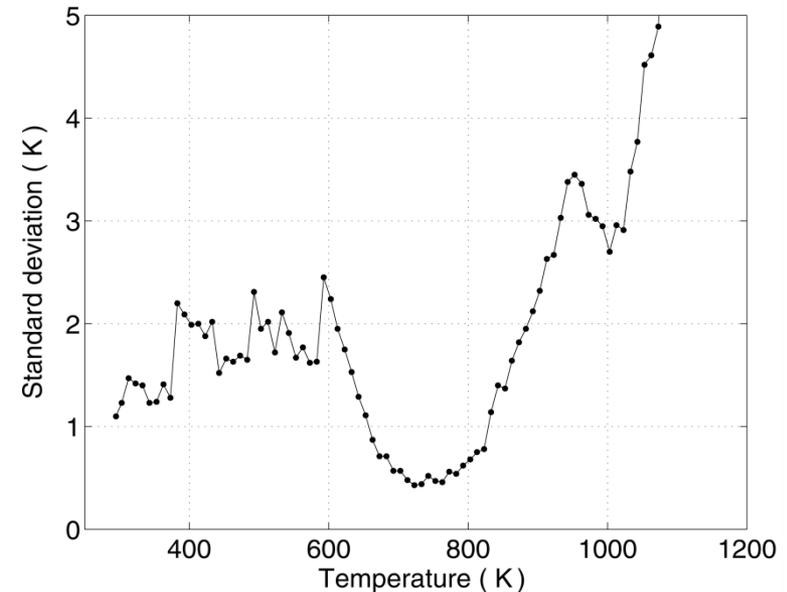
- Dynamic Range: 12 bit
- max. frame rate of 675 kHz at a resolution of 64 x 16 Pixels



# Calibration in well-controlled environments (oven)



- Phosphor:  $\text{Mg}_4\text{FGeO}_6:\text{Mn}$
- Substrate: Stainless Steel (1.4301)
- Gas phase pressure: 1 bar (air)
- Detection: PMT at 500  $\Omega$

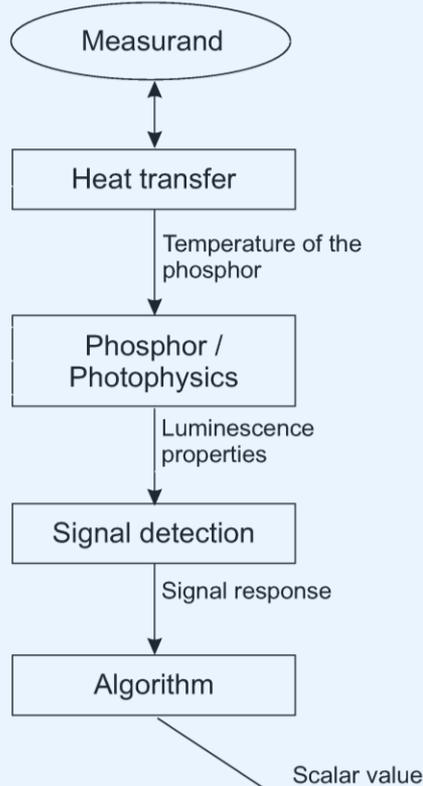




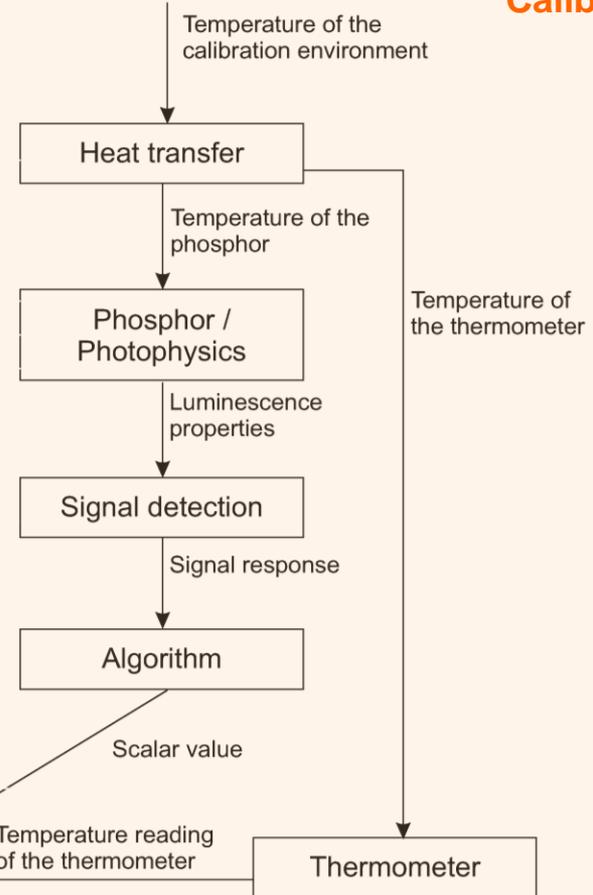
1. Introduction
- 2. Measurement chain / Error sources**
3. Applications
4. Summary

# Measurement chain

## Actual Measurement

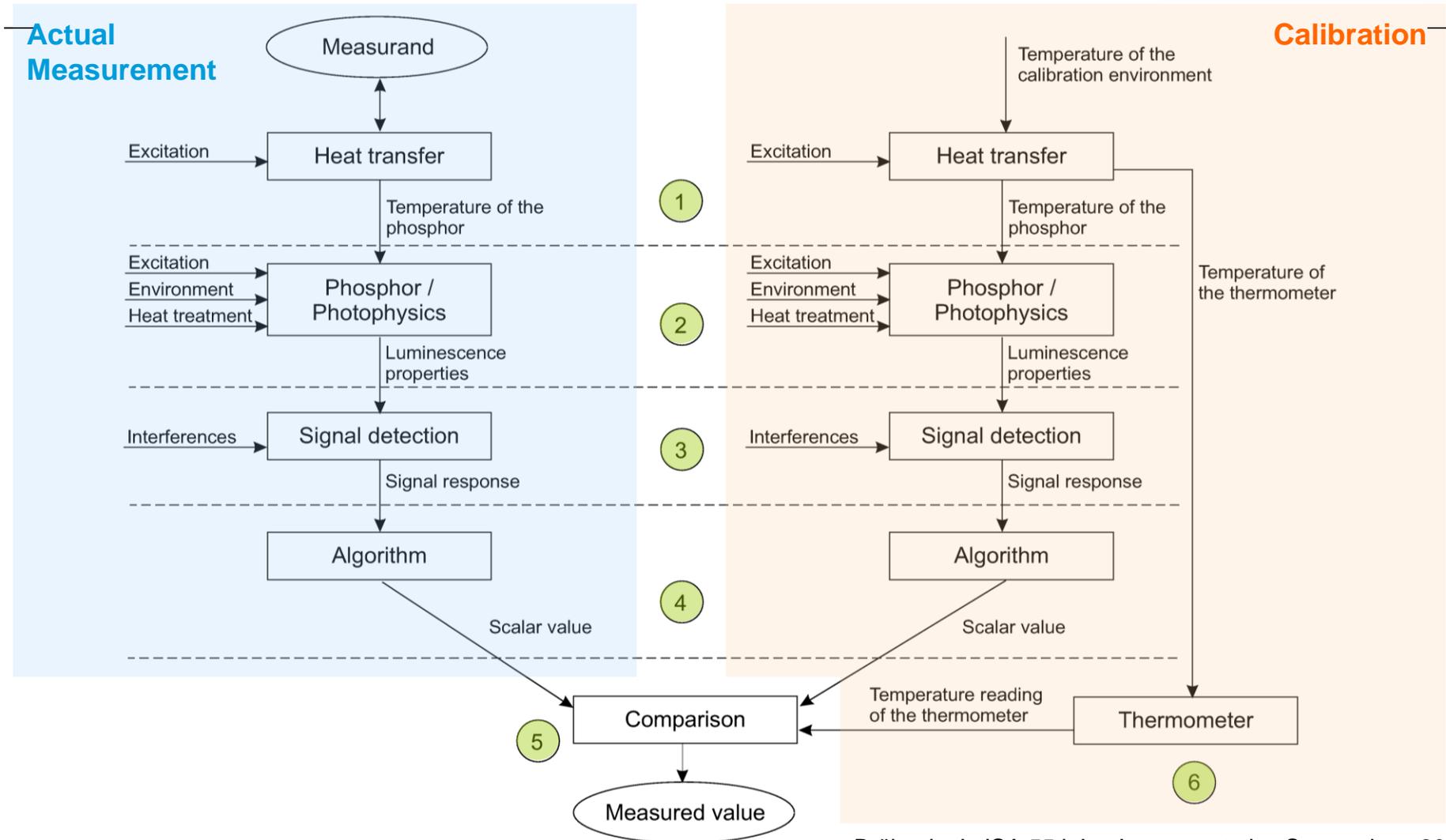


## Calibration

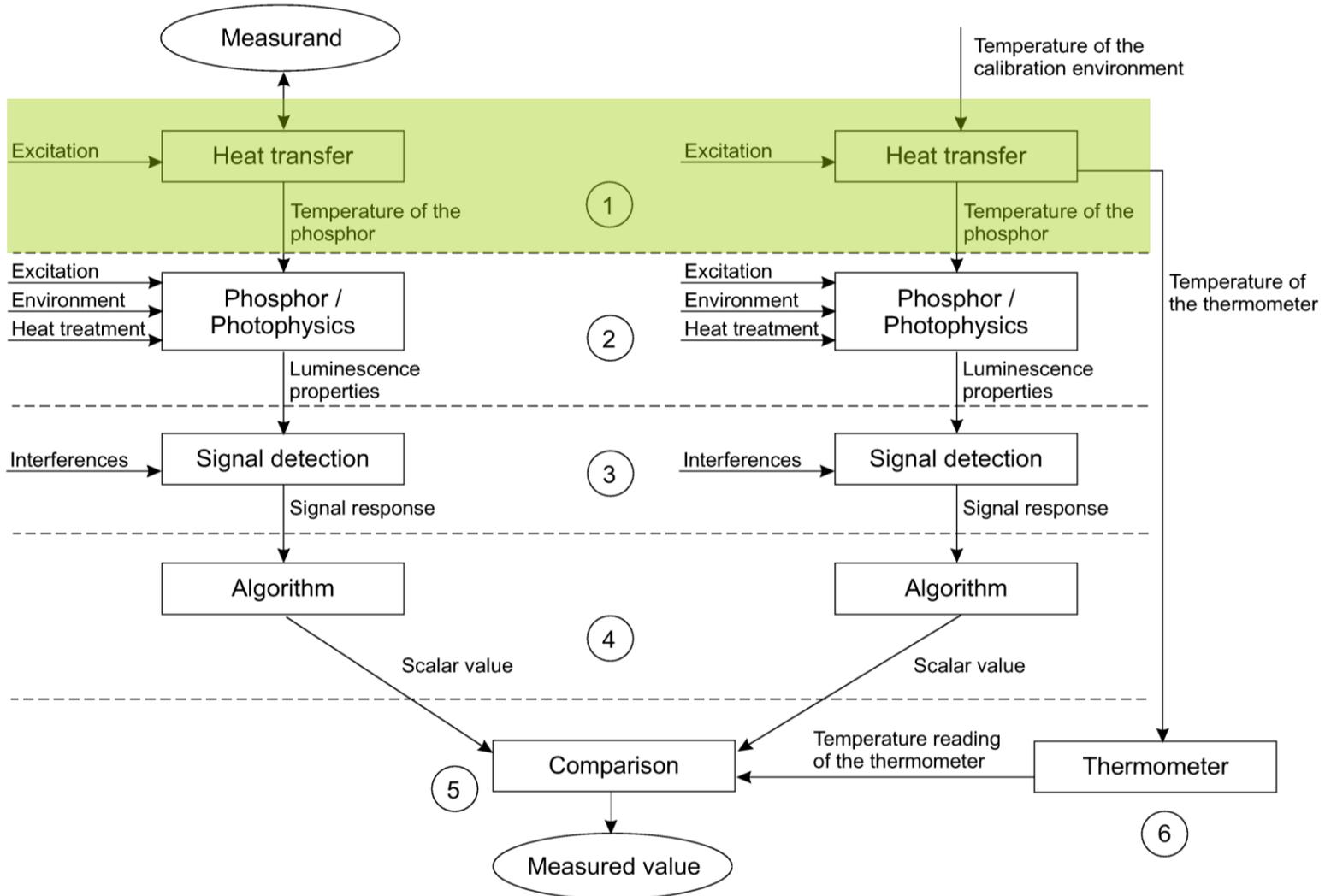


Measured value

# Measurement chain



# Error class 1: Thermal interactions



# Error class 1:

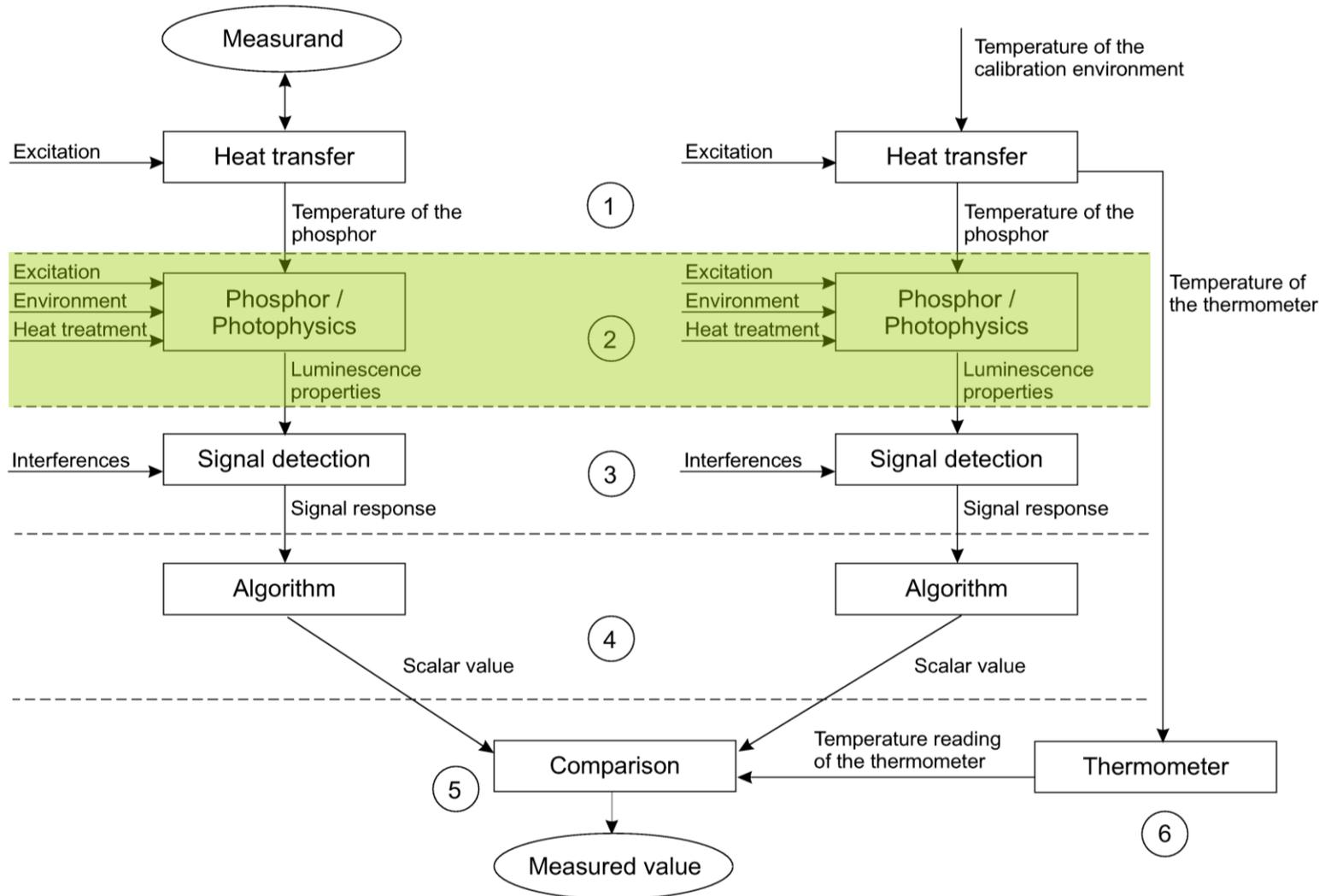
## Thermal interactions

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- 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



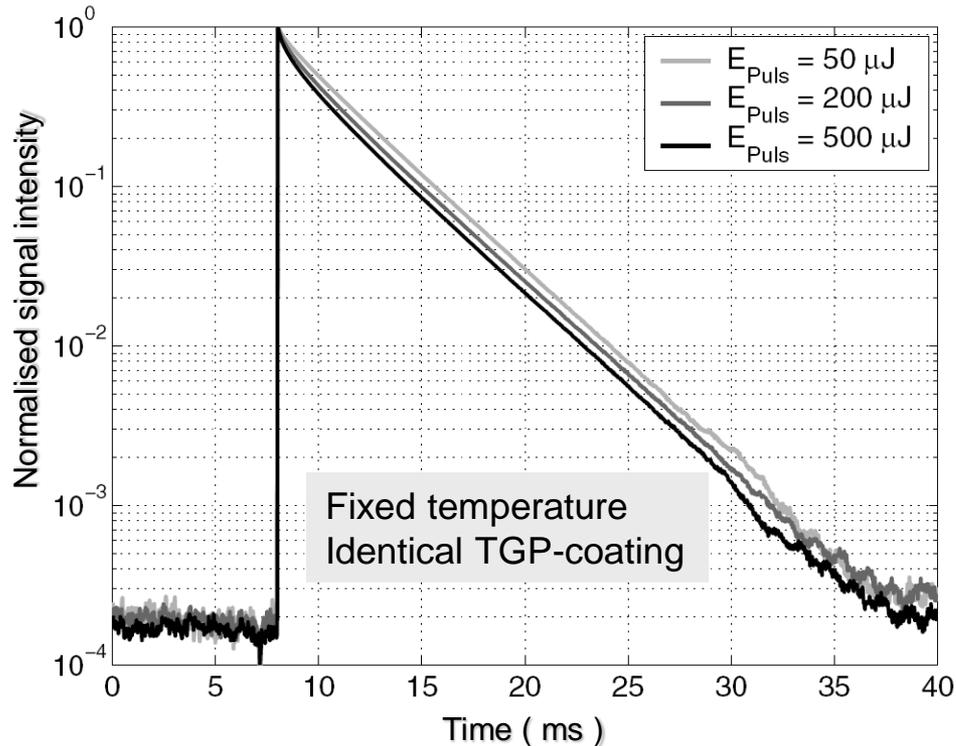
# Error class 2:

## 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:

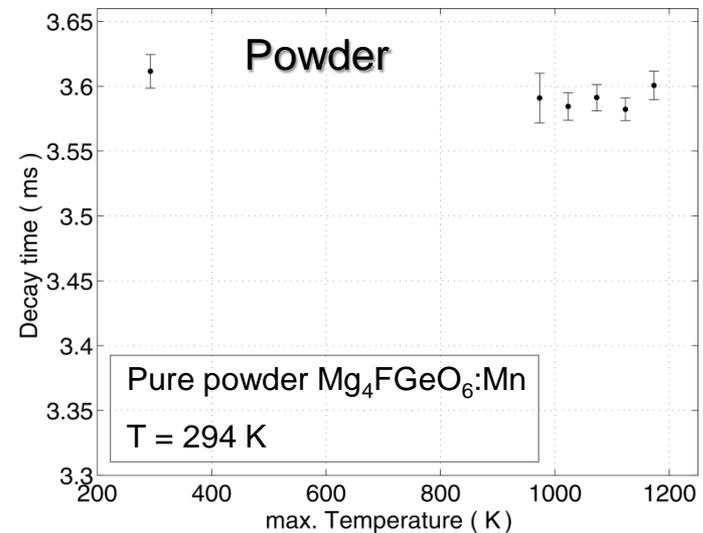
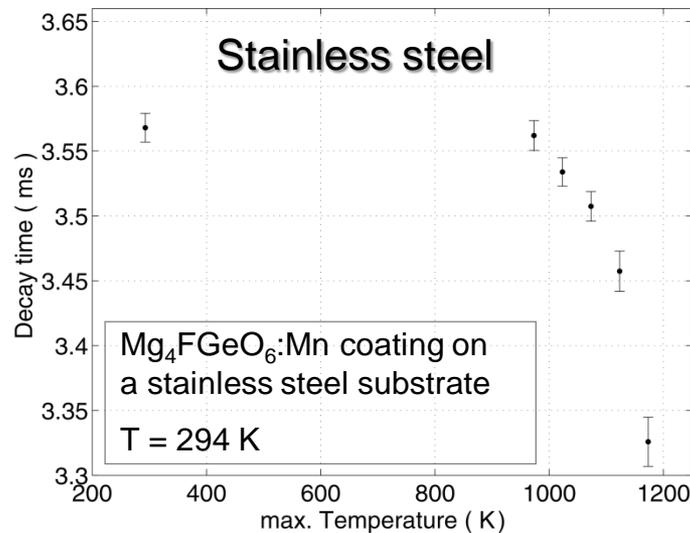
## Excitation irradiance



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

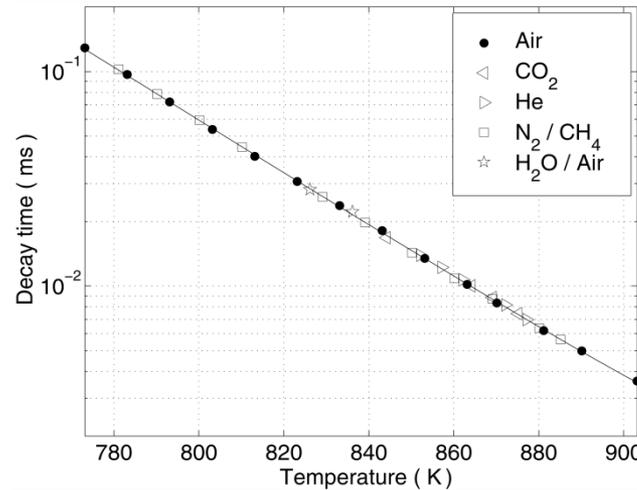
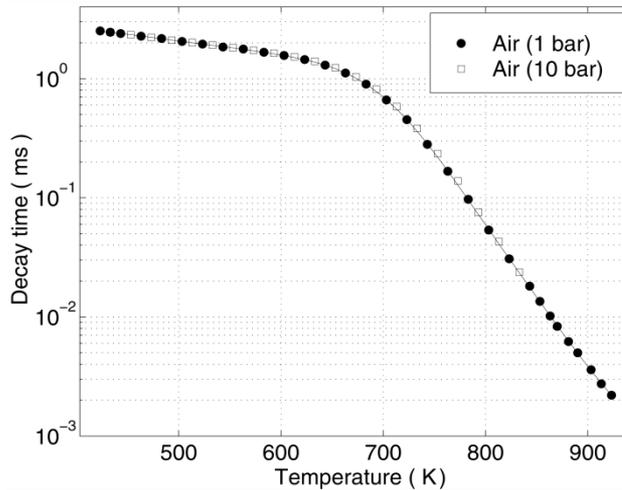
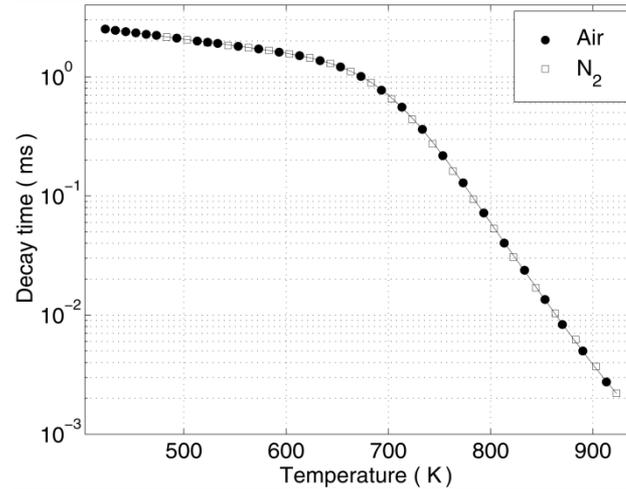
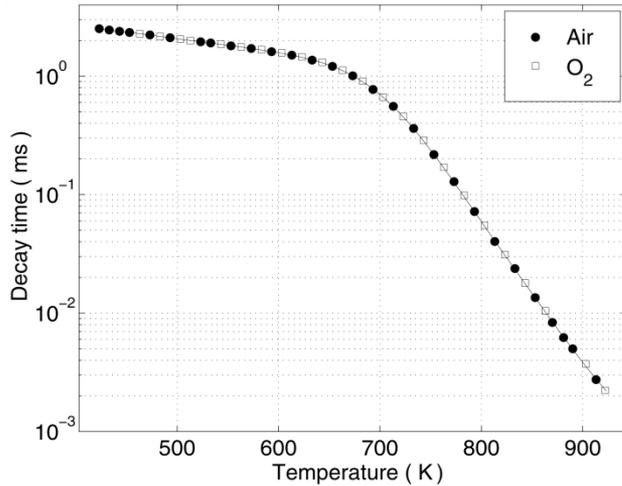
## Error class 2:

### Decay time @ 294 K after heat treatment



- ➔ Dependency on:
- Substrate material
  - Duration of the heat treatment
  - Heat treatment temperature
- ➔ Diffusion process between phosphor und substrate

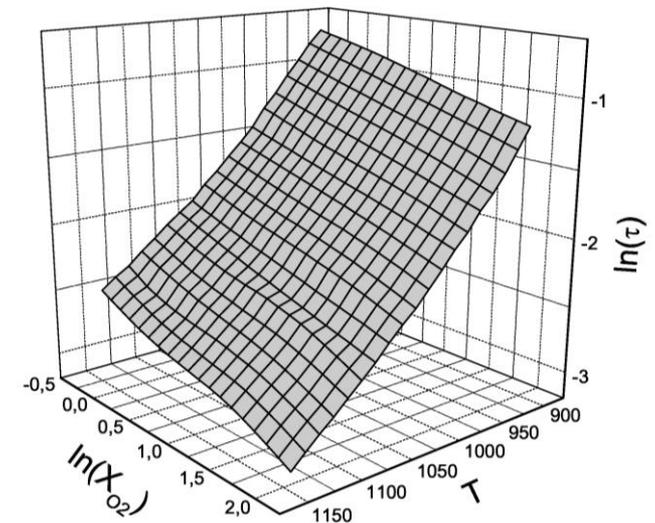
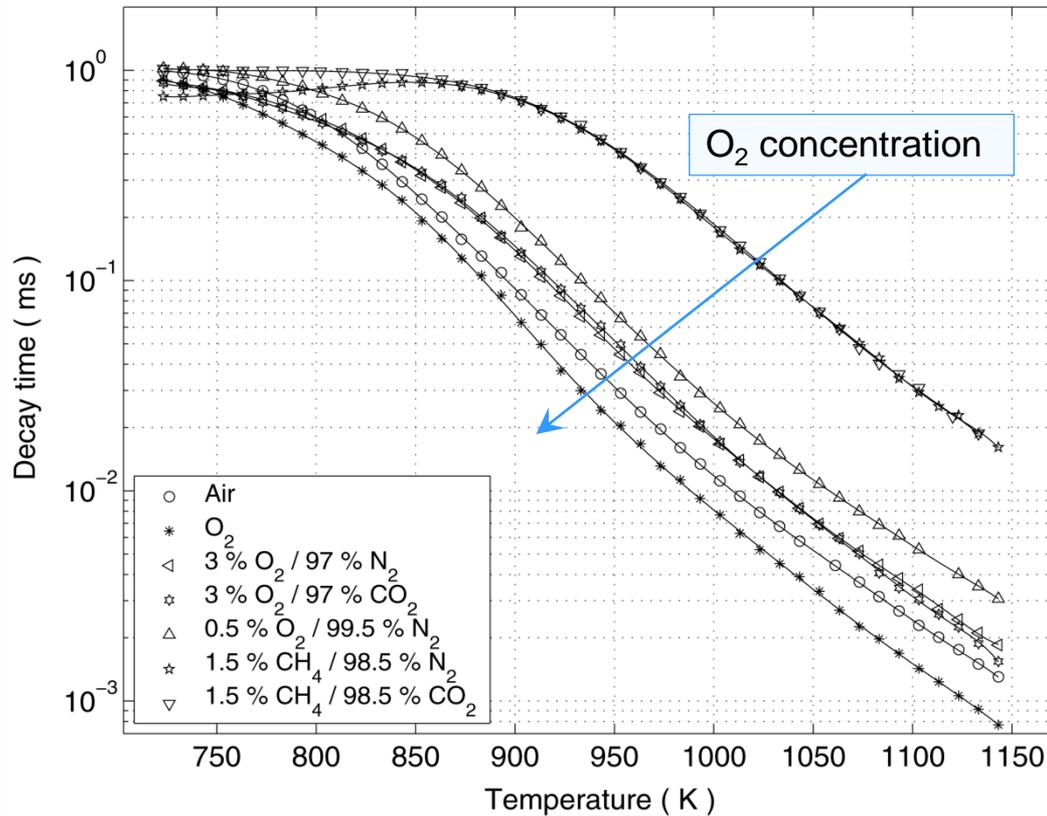
# Error class 2: Surrounding gas phase



## Mg<sub>4</sub>FGeO<sub>6</sub>:Mn

No significant dependency  
on the composition and  
pressure of the gas phase

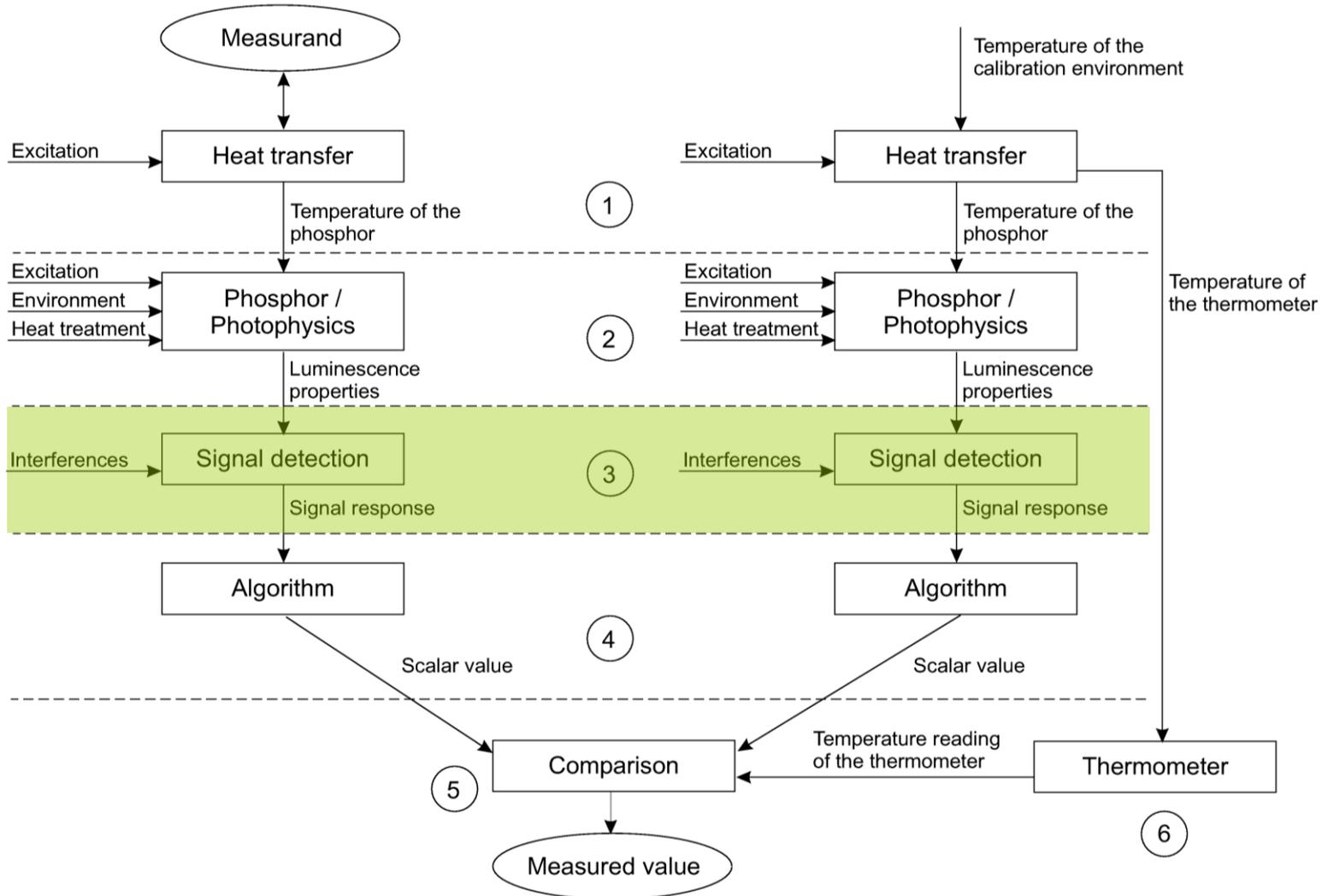
# Error class 2: Surrounding gas phase



**Y<sub>2</sub>O<sub>3</sub>:Eu**

Dependency on the oxygen  
concentration

# Error class 3: Signal detection



# Error class 3:

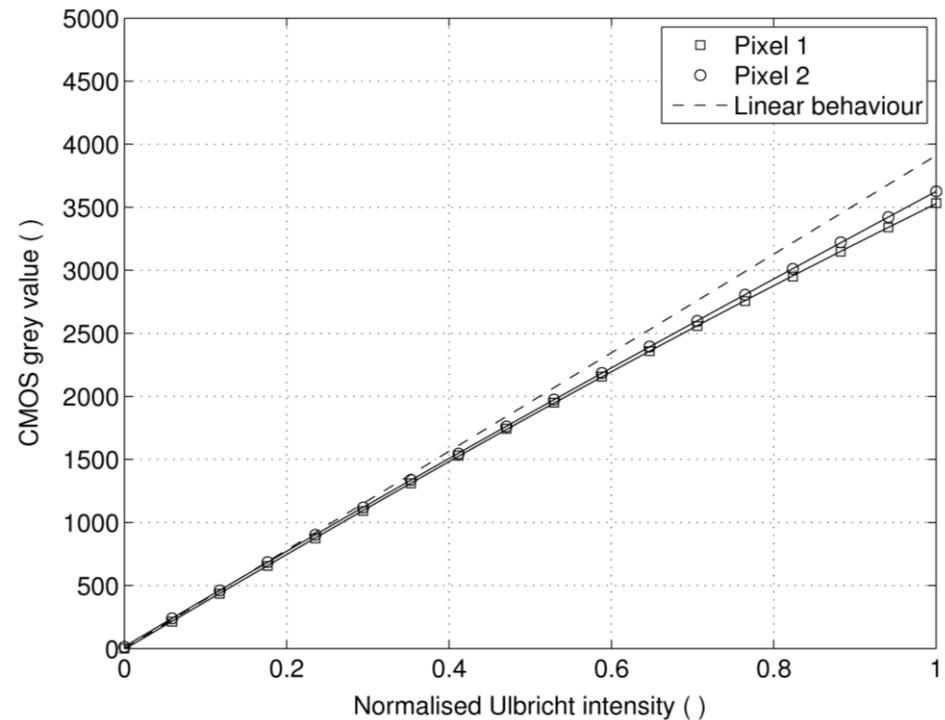
## Signal detection – overview

- 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

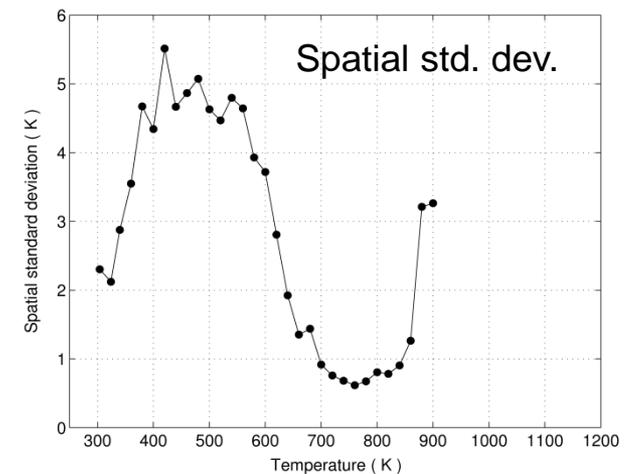
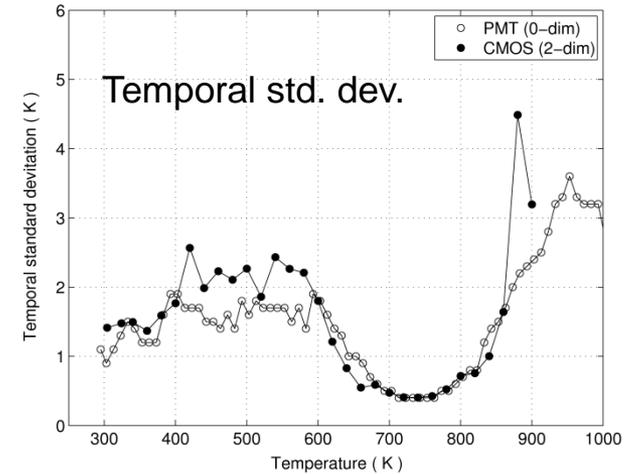
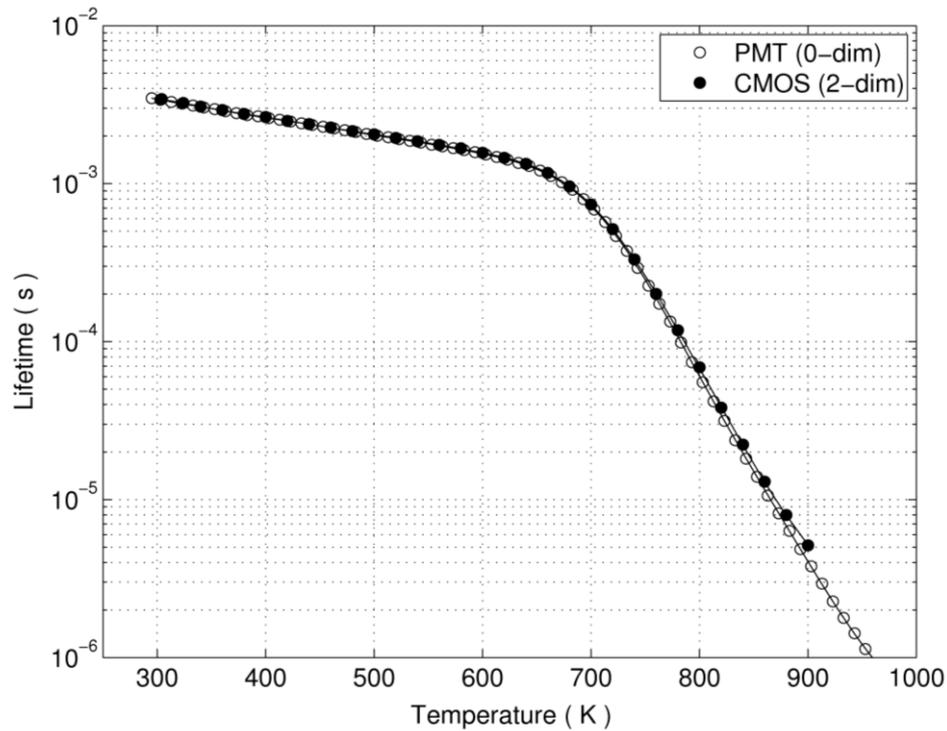
- Photomultiplier unproblematic
- CMOS camera shows problems regarding
  - Linearity
  - Pixel-to-pixel homogeneity
  - Offset stability



Kissel, T.; Baum, E.; Dreizler, A.; Brübach, J.: Appl. Phys. B, 96:731–734, 2009.

# Error class 3: Non-linear detector behaviour

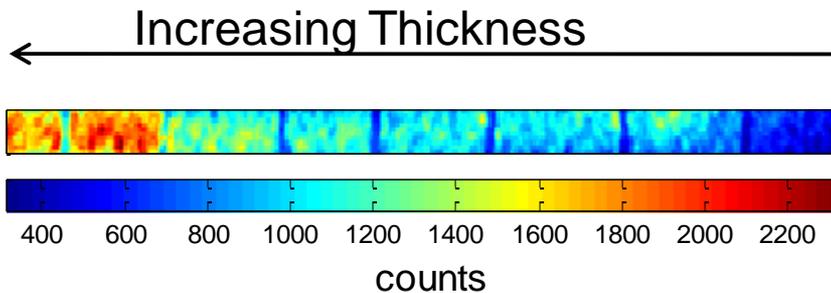
➔ Correction function for each pixel  
fixes the problem



# Error class 3:

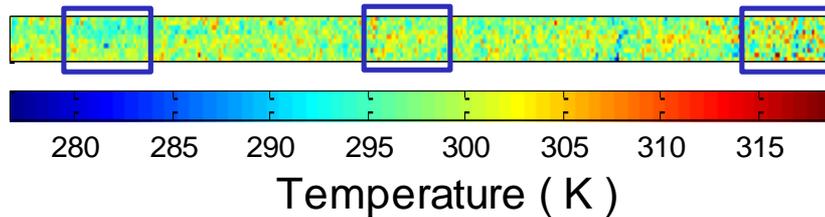
## Coating thickness and CMOS frame rate

### Variation of coating thickness and luminescence intensity



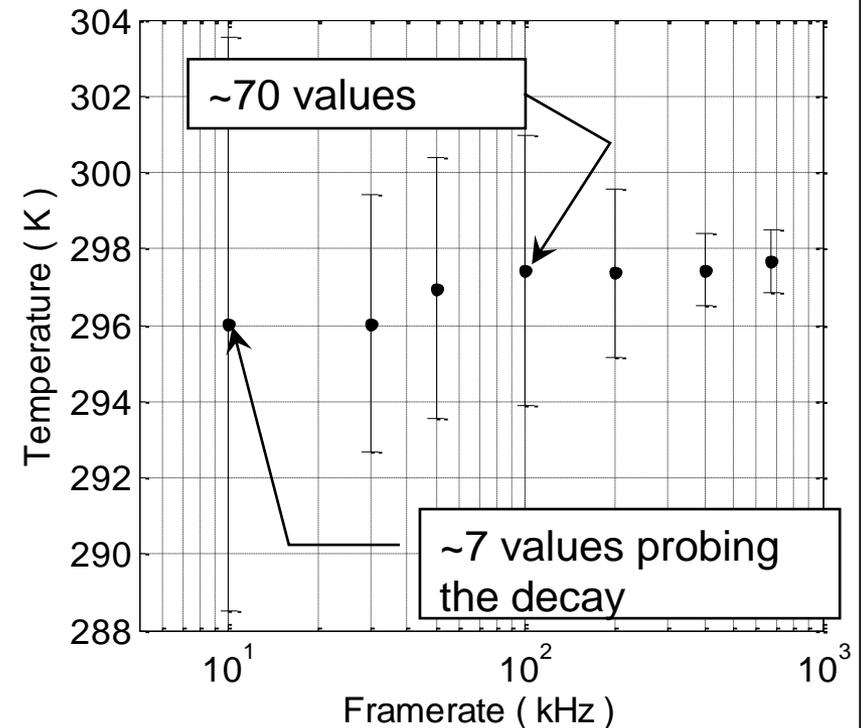
### Resulting Temperature:

299,4 K ±3,0 K	298,0 K ±3,3 K	297,4 K ±5,7 K
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No significant influence!

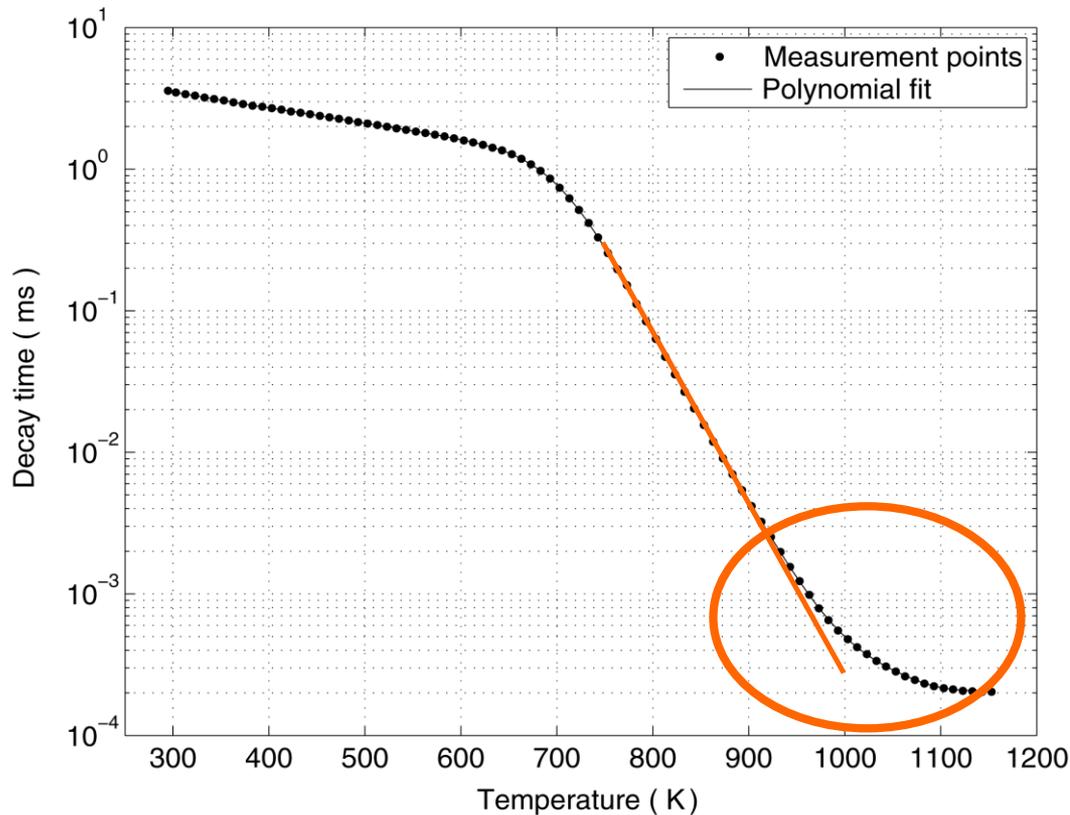
### Variation of frame rate:



Minimum of standard deviation at  
high temporal discretisation.

# Error Class 3:

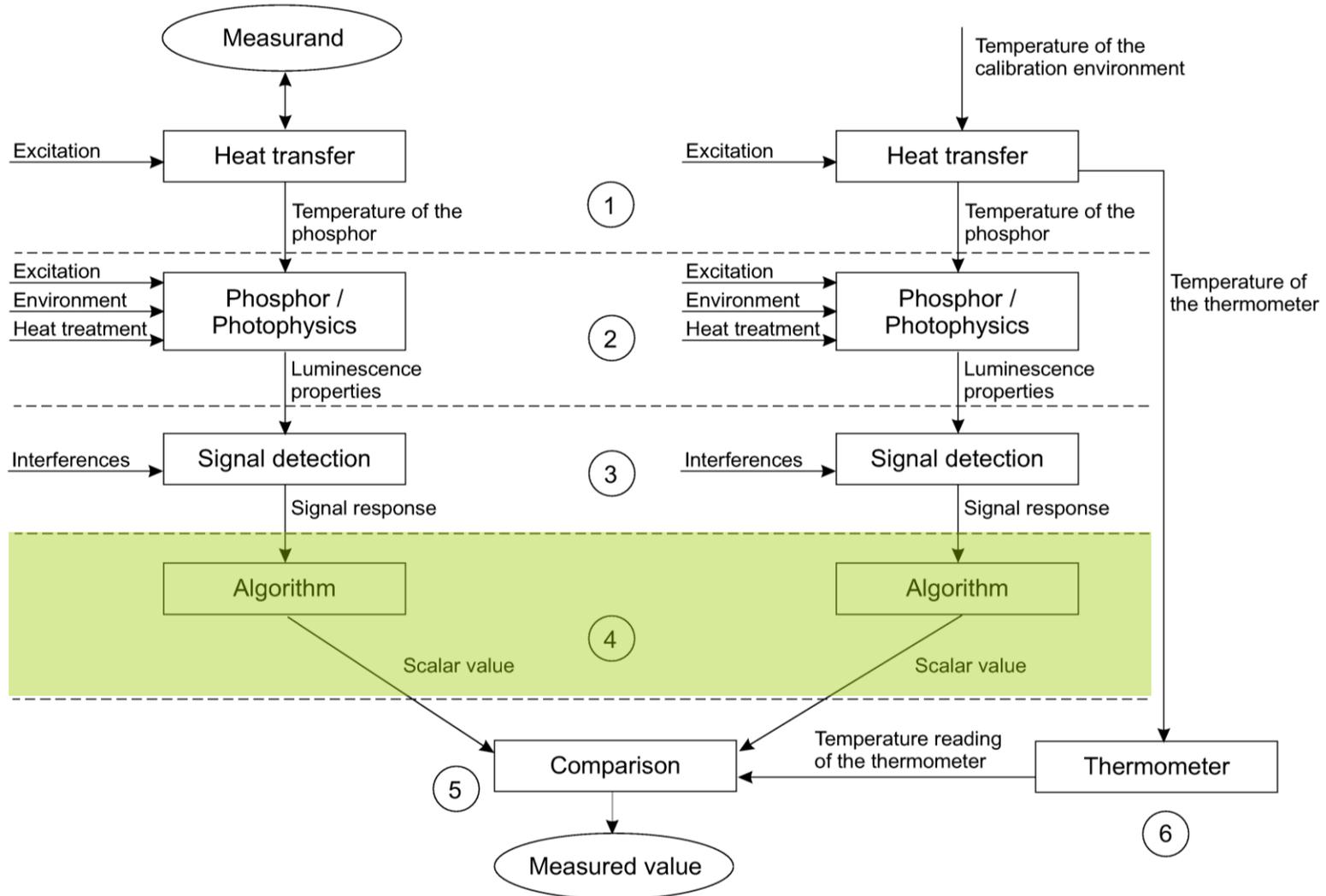
## Parameters impacting the transfer function



- ➔ Modified low pass character of the detection system (PMT) due to terminating resistor
- ➔ might strongly change the calibration curve, especially at low decay times

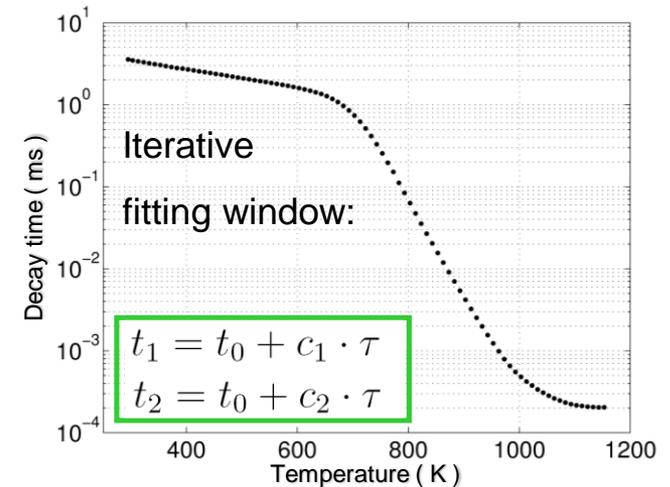
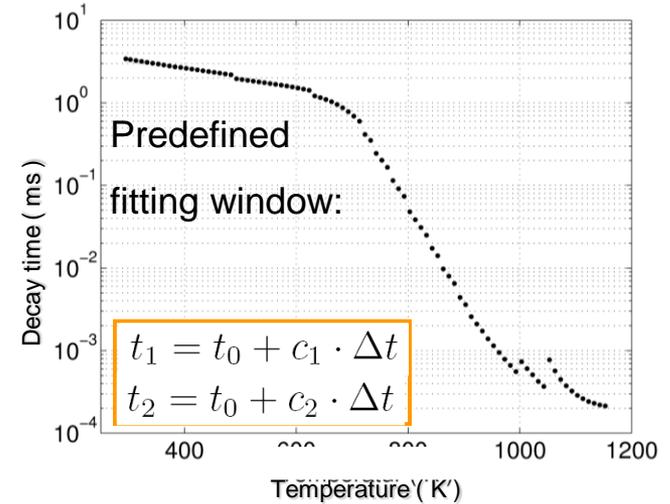
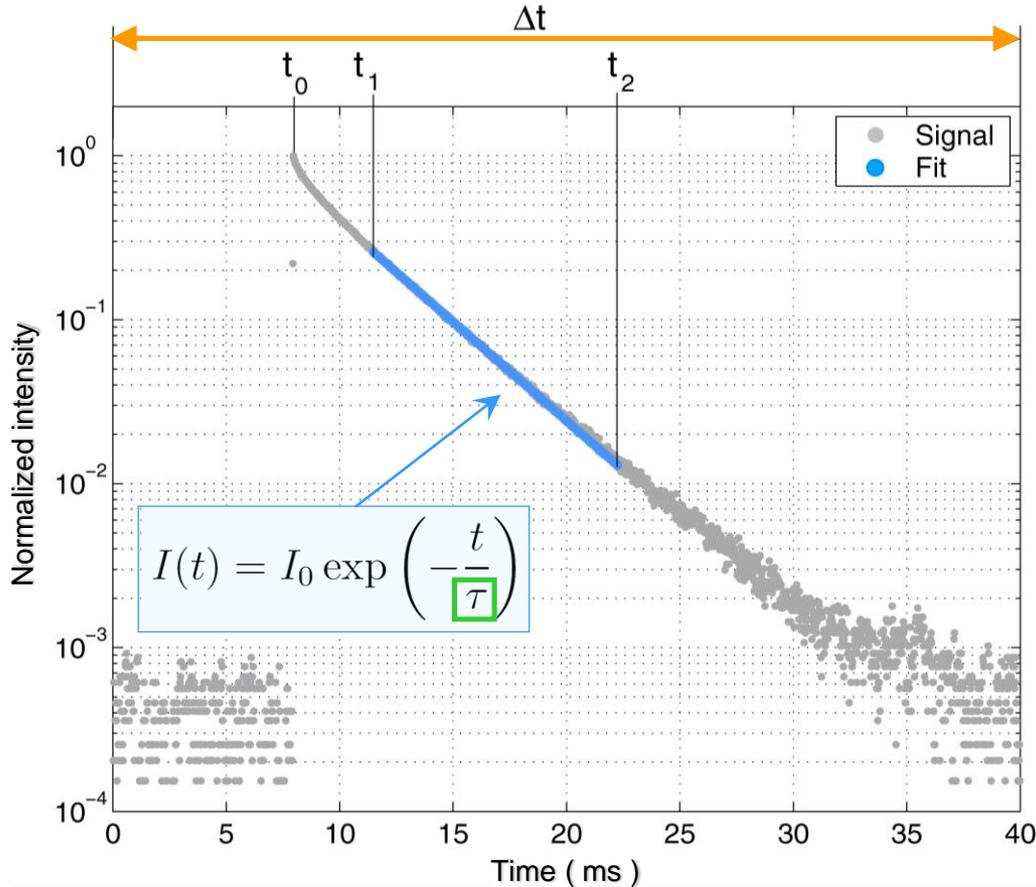
# Error class 4:

## Algorithm for the data reduction



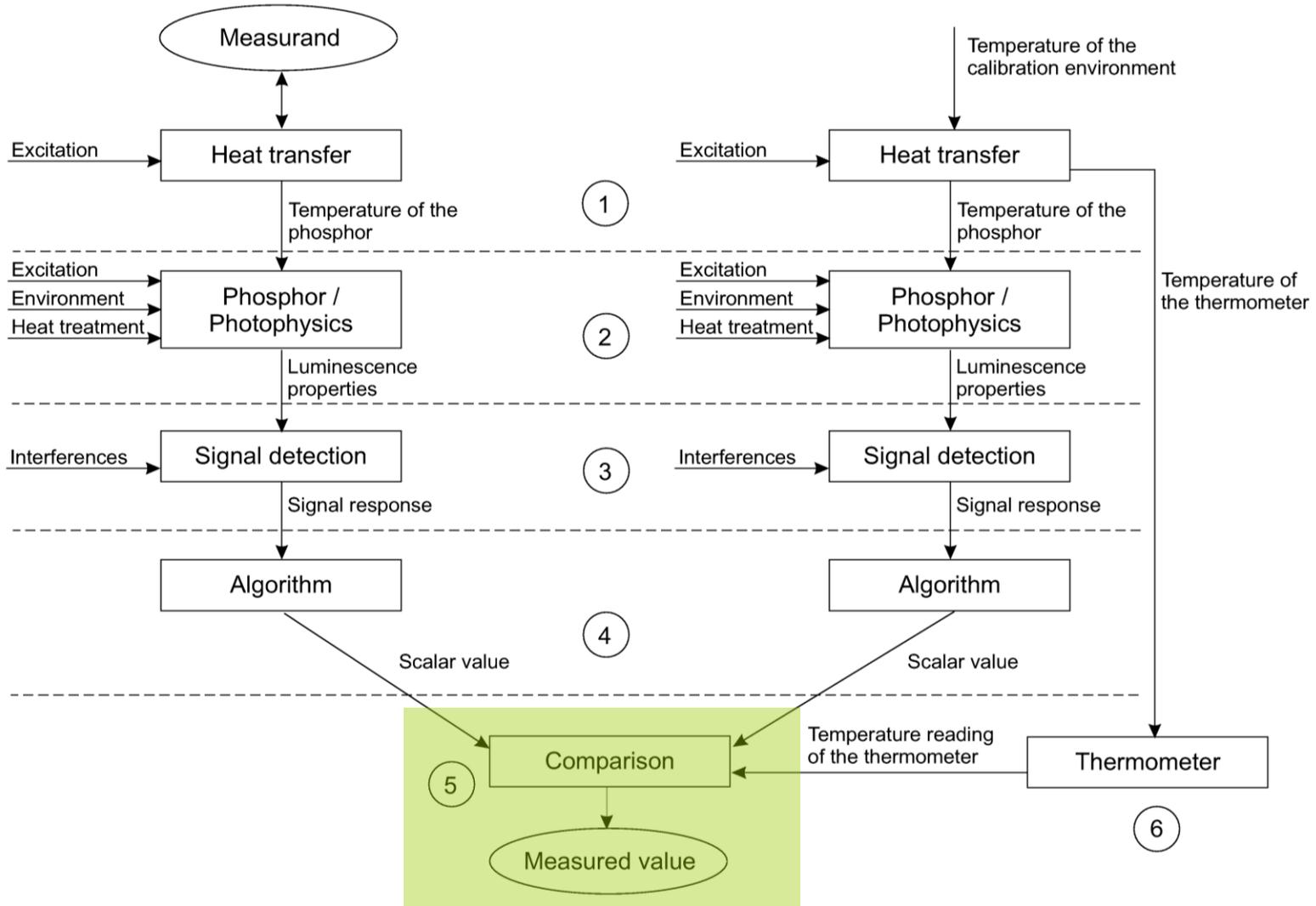
# Error class 4: Algorithm for the data reduction

➔ **Problem:**  
Decay characteristics are not single-exp.



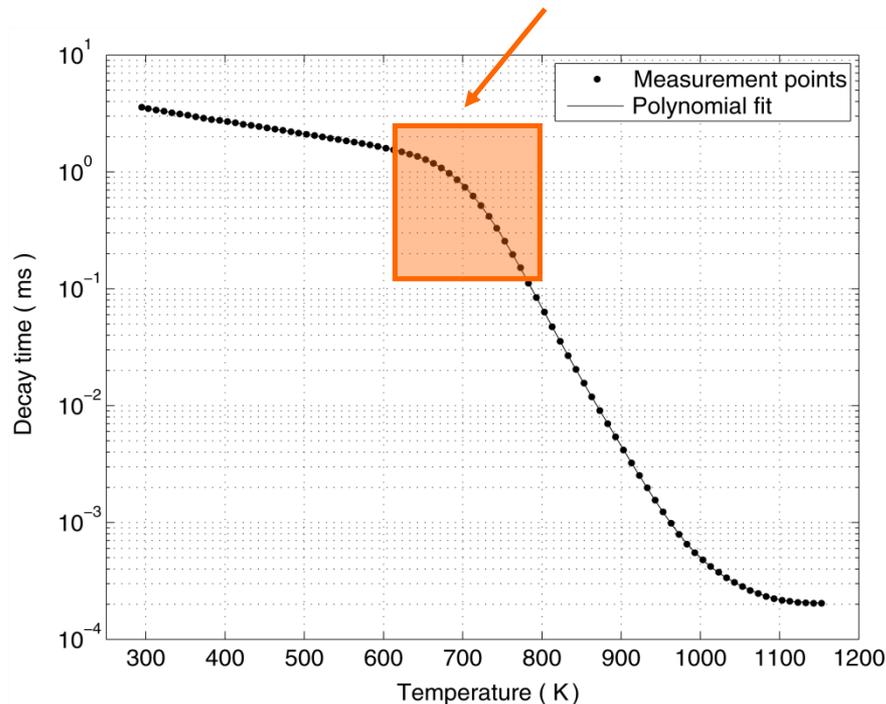
Brübach, J.; Janicka, J.; Dreizler, A.: Opt. Laser Eng., 47(1):75–79, 2009.

# Error class 5: Comparison of the scalar values



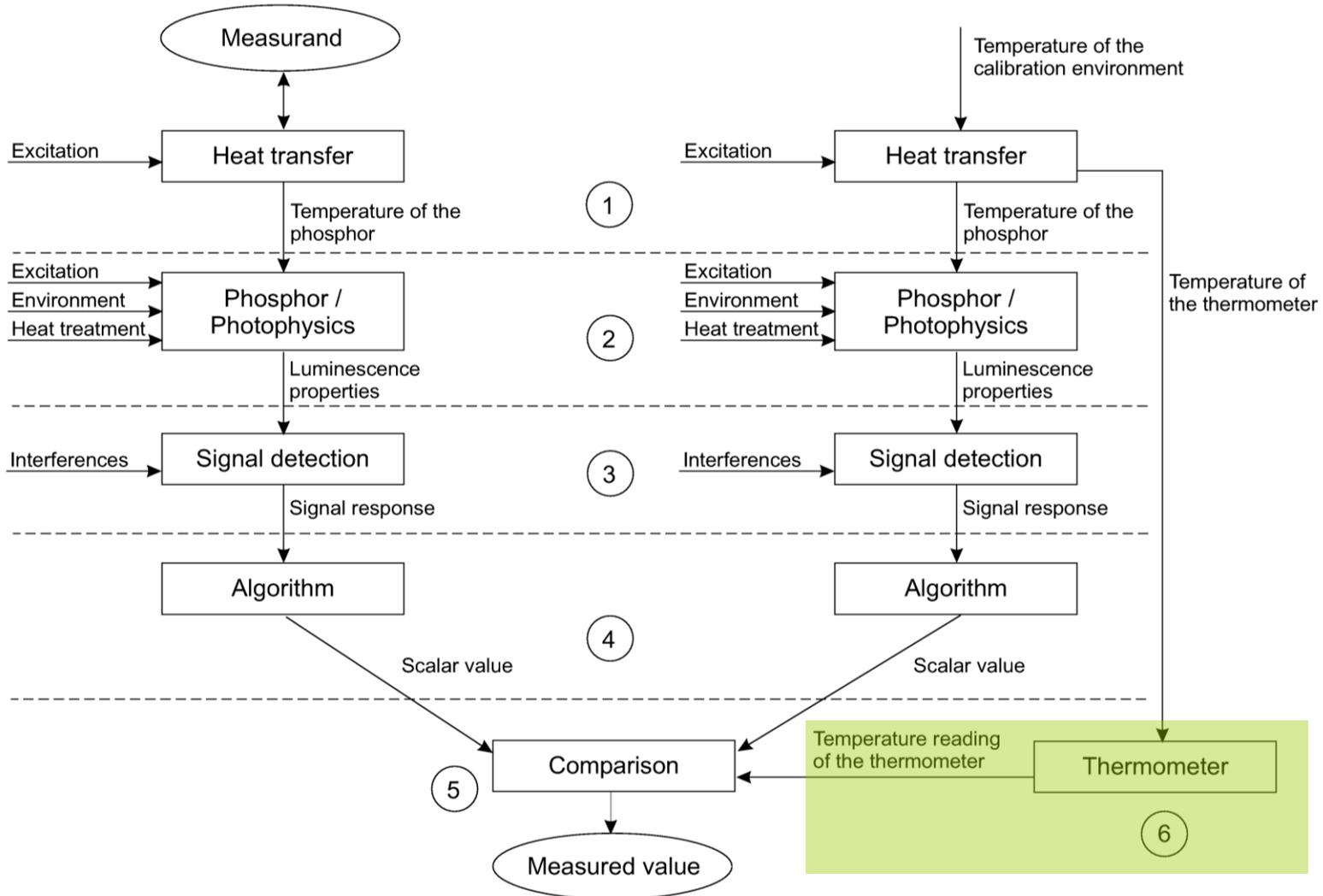
# Error class 5: Comparison of the scalar values

- 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  $\Delta T = 20\text{K}$  or even  $\Delta T = 10\text{K}$  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



## Systematic Error:

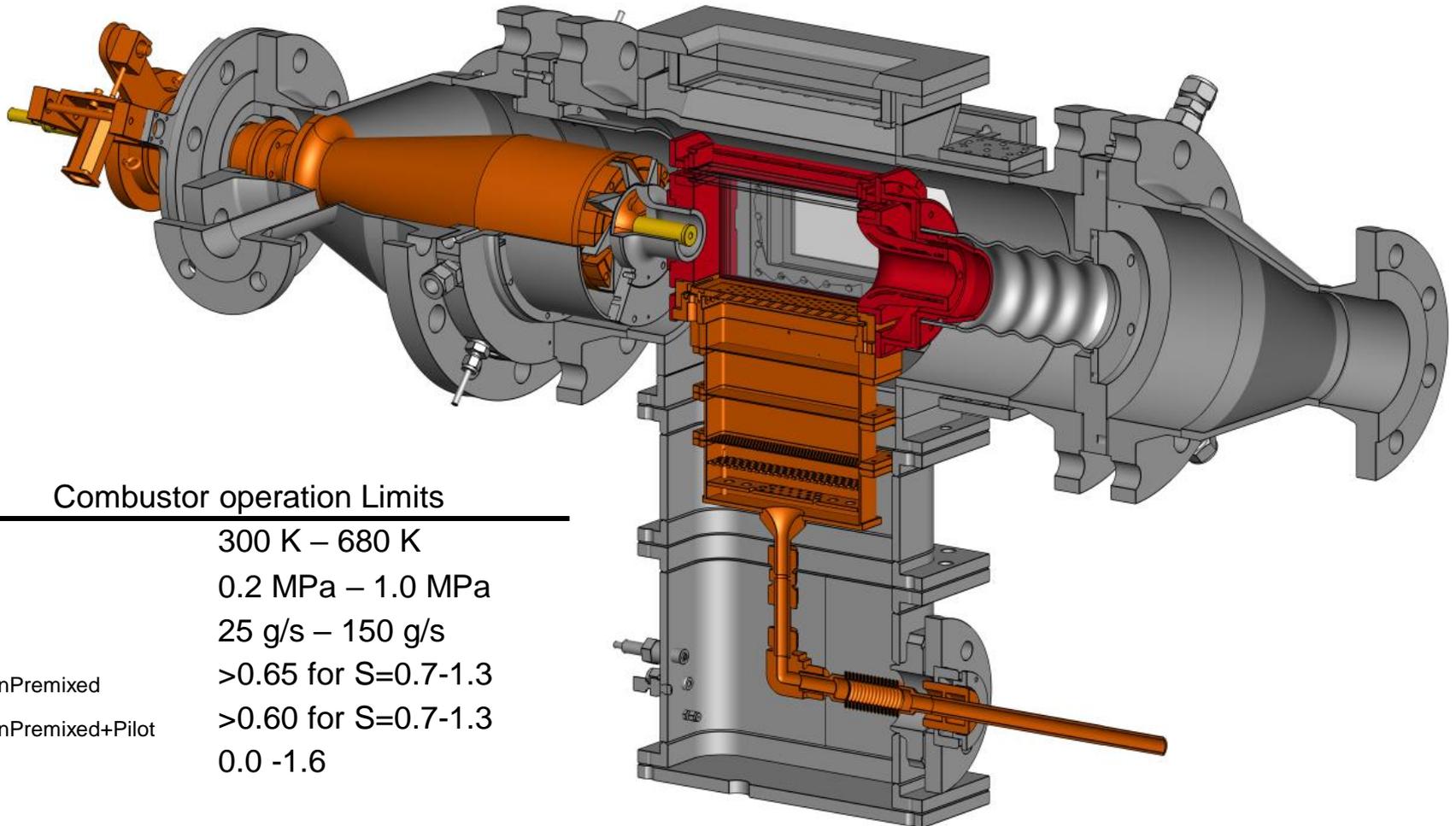
Provided a careful practice:  
→ Systematic Error < 1 %

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

## Statistical Error:

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

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



## Combustor operation Limits

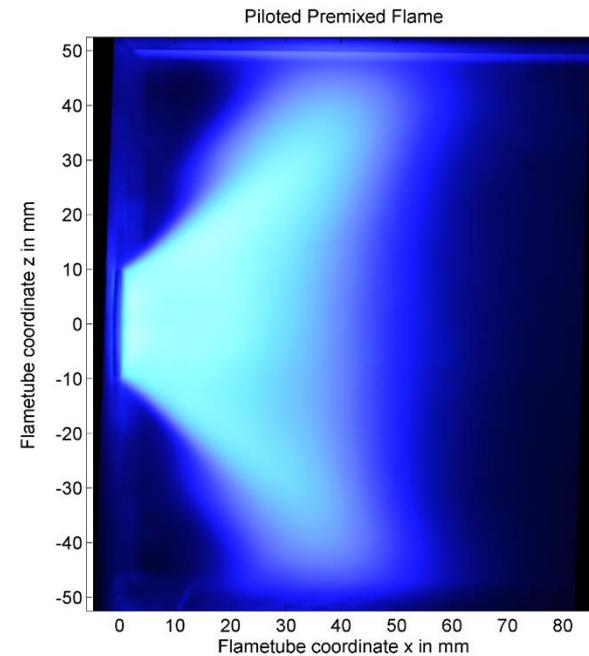
$T_3$	300 K – 680 K
$P_3$	0.2 MPa – 1.0 MPa
$m_3$	25 g/s – 150 g/s
$\Phi_{\text{LeanPremixed}}$	>0.65 for $S=0.7-1.3$
$\Phi_{\text{LeanPremixed+Pilot}}$	>0.60 for $S=0.7-1.3$
$S$	0.0 -1.6

Herrmann et al. Dreizler. Flow Turb. Combust. (2019) <https://doi.org/10.1007/s10494-018-9999-y>

- Standard operation point

## Operation conditions

$T_3$	623K
$P_3$	0.25 MPa
$\dot{m}_3$	0.030kg/s
$\Phi_{\text{LeanPremixed}}$	0.65/0% Pilot
$\Phi_{\text{LeanPremixed+Pilot}}$	0.65/10% Pilot
$S$	0.7
$\dot{m}_5$	0.0125 kg/s
$T_5$	623 K



Low-pass (490nm ) filtered  
Chemiluminescence images from piloted and  
premixed flames

# Selected results: wall thermometry

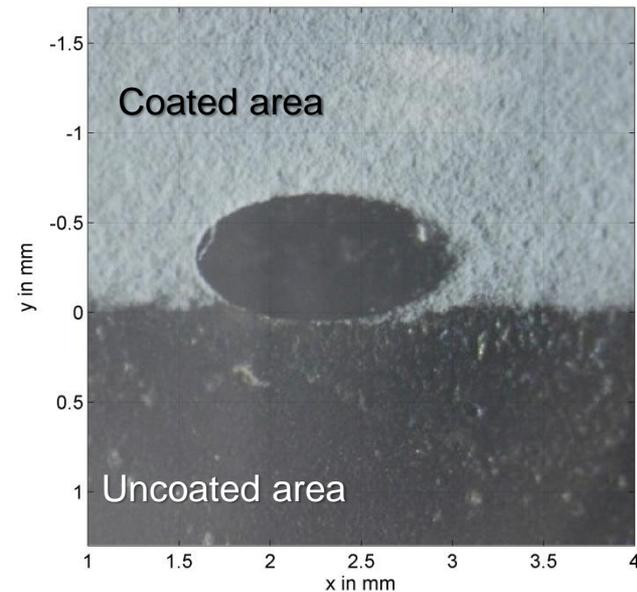
- 2d wall temperature fields

Magnified view of the coated liner.

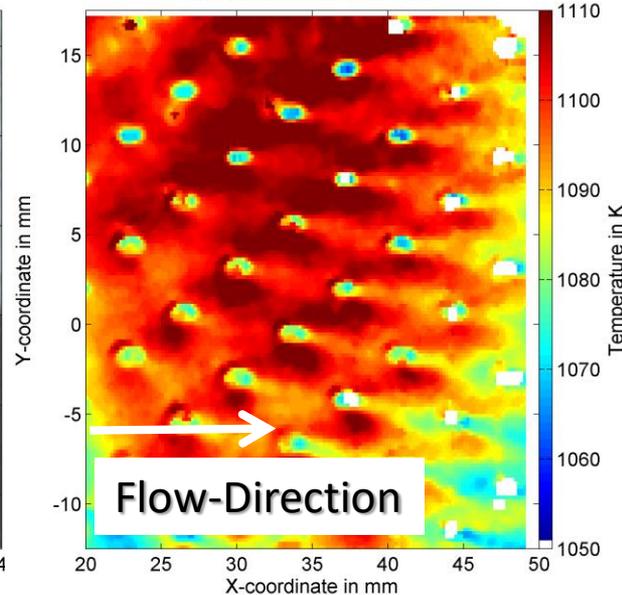
Coating thickness  $t$   $10\mu\text{m} < t < 50\mu\text{m}$

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

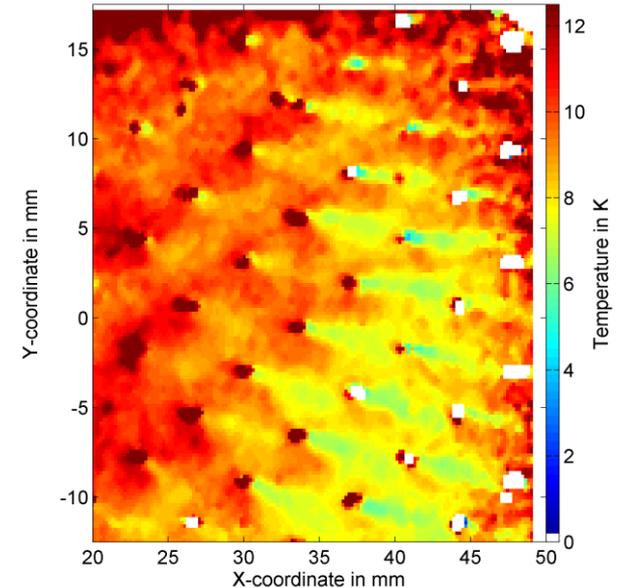
Coated effusion hole



Mean temperature in FOV4

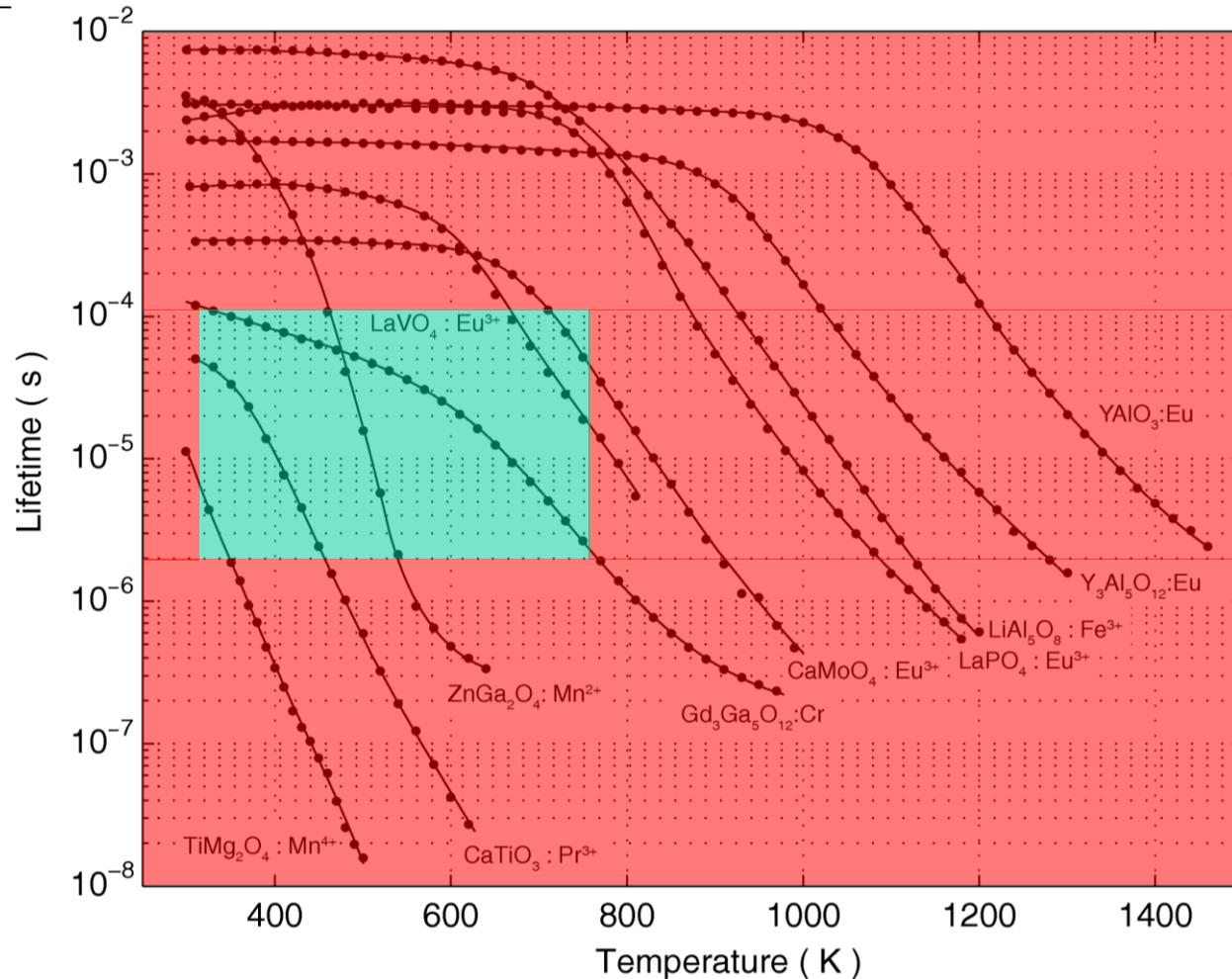


Standard deviation in FOV4



# Optically accessible combustion engine

CMOS

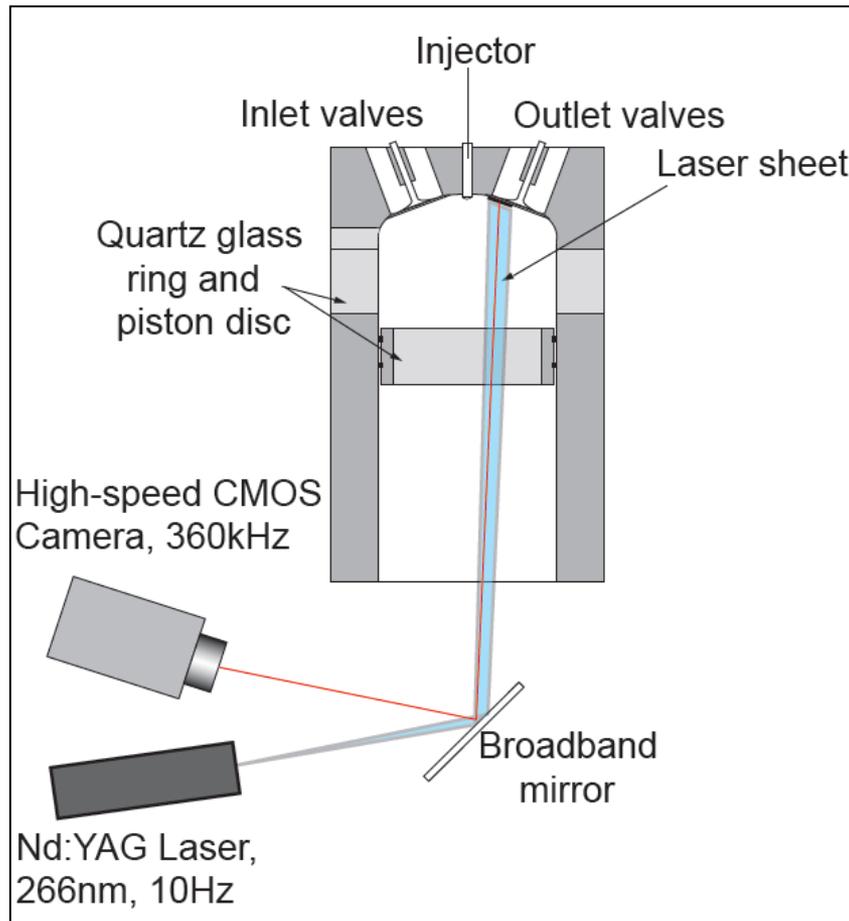


Fuhrmann, N. ; Kissel, T. ; Dreizler, A. ; Brübach, J. : Meas. Sci. Technol., Meas. Sci. Technol.22 045301 (2011)

Brübach, J. ; Kissel, T. ; Frotscher, M. ; Euler, M. ; Albert, M. ; Dreizler, A. : J. Lumin., 131, 559 – 564 (2011)

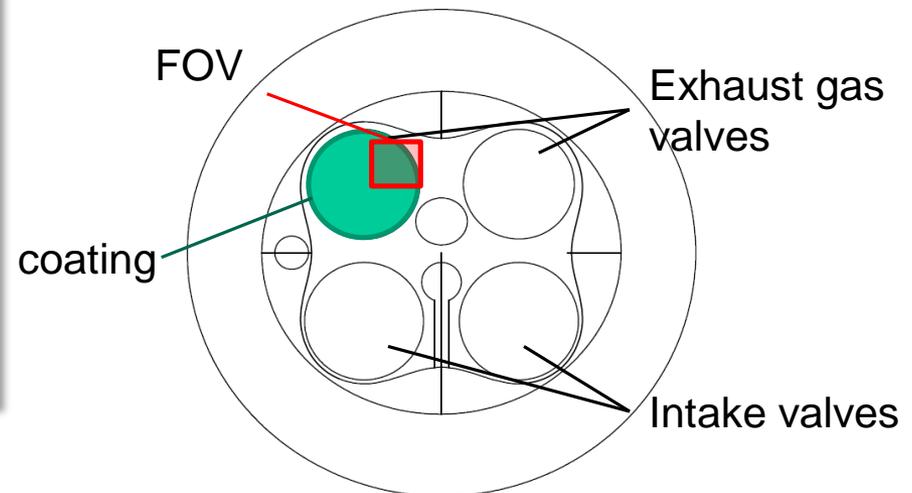
# Optically accessible combustion engine

See Appl. Phys. B 2011, Fuhrmann et al.



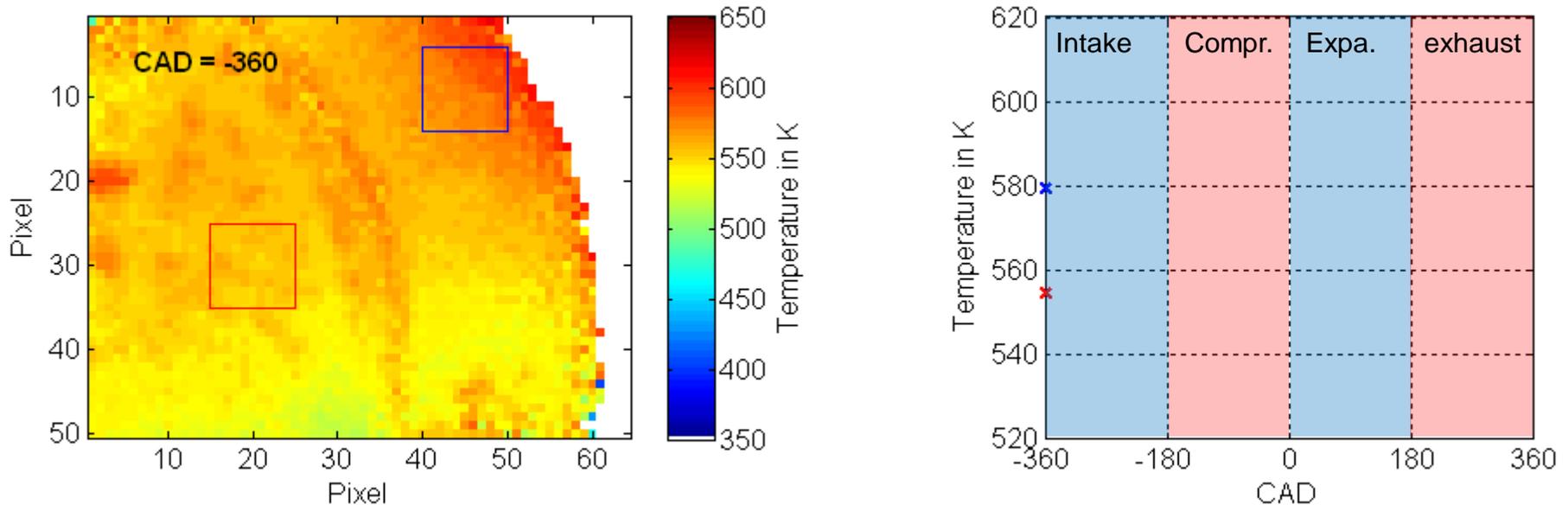
## Temperatur at exhaust valve:

- DI-ICE @ 2000 min<sup>-1</sup>
- Coating: Gd<sub>3</sub>Ga<sub>5</sub>O<sub>12</sub>:Cr + Ufalux Ofenlack
- Spatial resolution: 188 μm/Pixel @ 64x64 Pixel (360 kHz framerate)



## Fired engine operation:

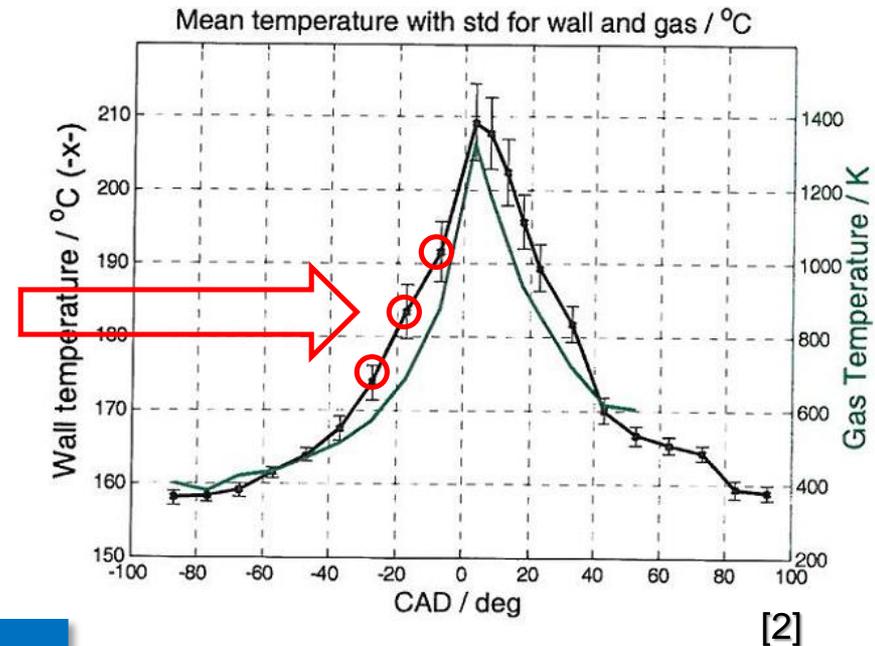
- Temperature measurement at selected CAD



N. Fuhrmann, M. Schild, D. Bensing, S.A. Kaiser, C. Schulz, J. Brübach, A. Dreizler: *Two-dimensional cycle-resolved exhaust valve temperature measurements in an optically accessible internal combustion engine using thermographic phosphors*; Applied Physics B, DOI 10.1007/s00340-011-4819-2.

# 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

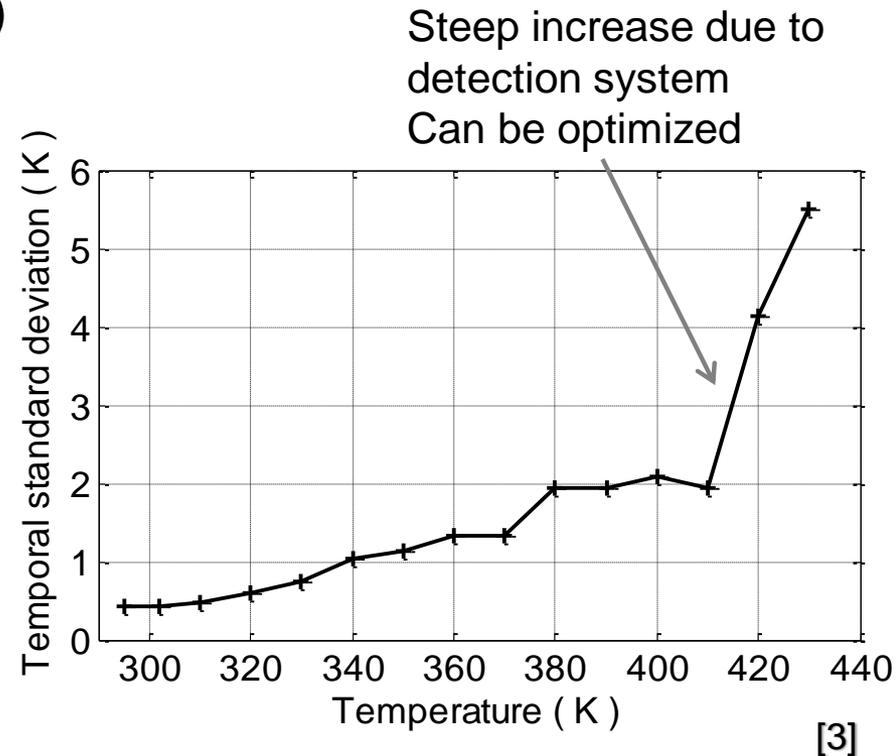
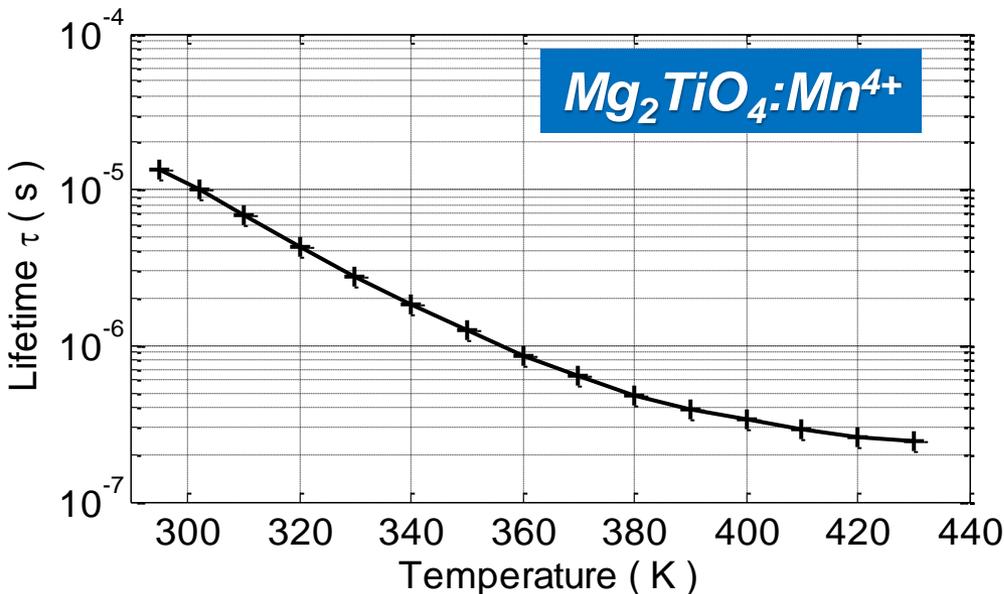


High speed phosphor thermometry

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  $167\mu\text{s}$  ( $1^\circ\text{CA}$  at 1000 rpm)



# Influence of laser-induced heating

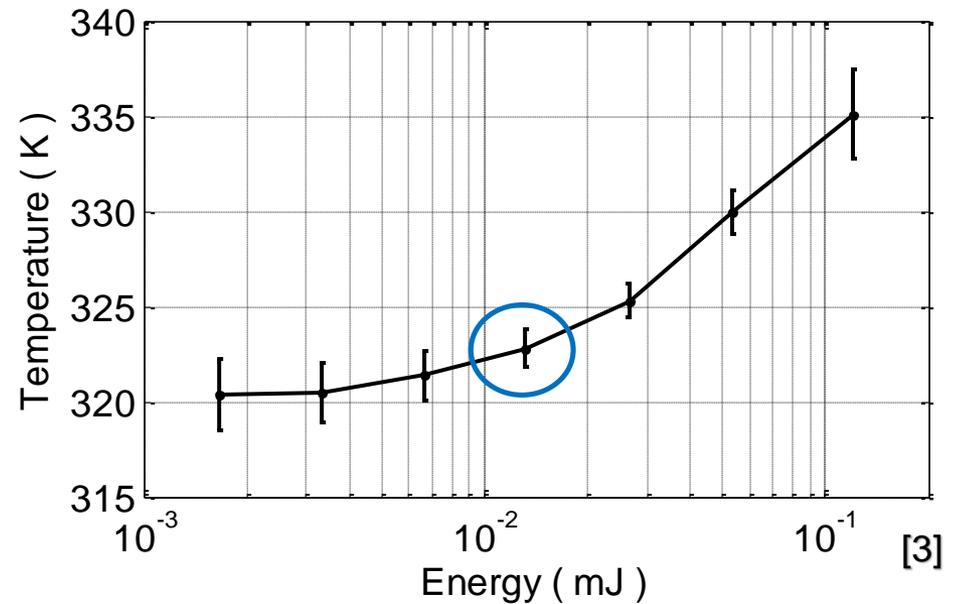
- Challenges when using High Speed Phosphor Thermometry:
  - → Laser-induced heating effects

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

Optimum at  $E = 13\mu\text{J}$  :

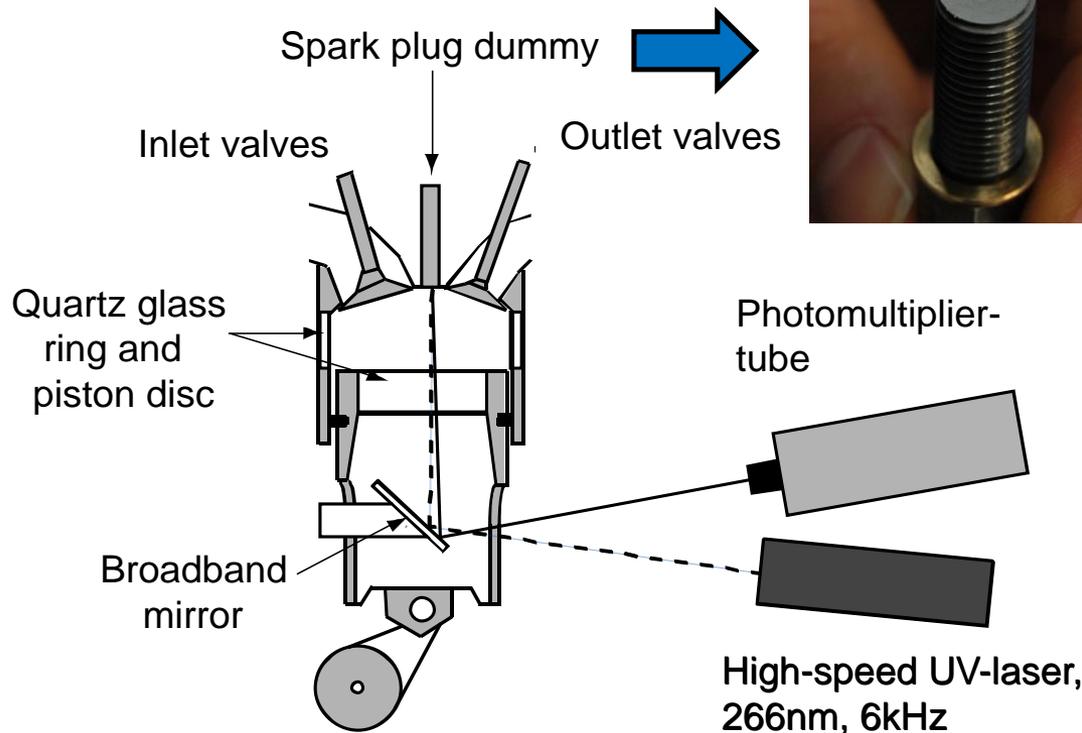
Standard deviation below 1K

Systematic error of 2.5K



# Set-up and realization

## ■ Experimental Setup



## Coating

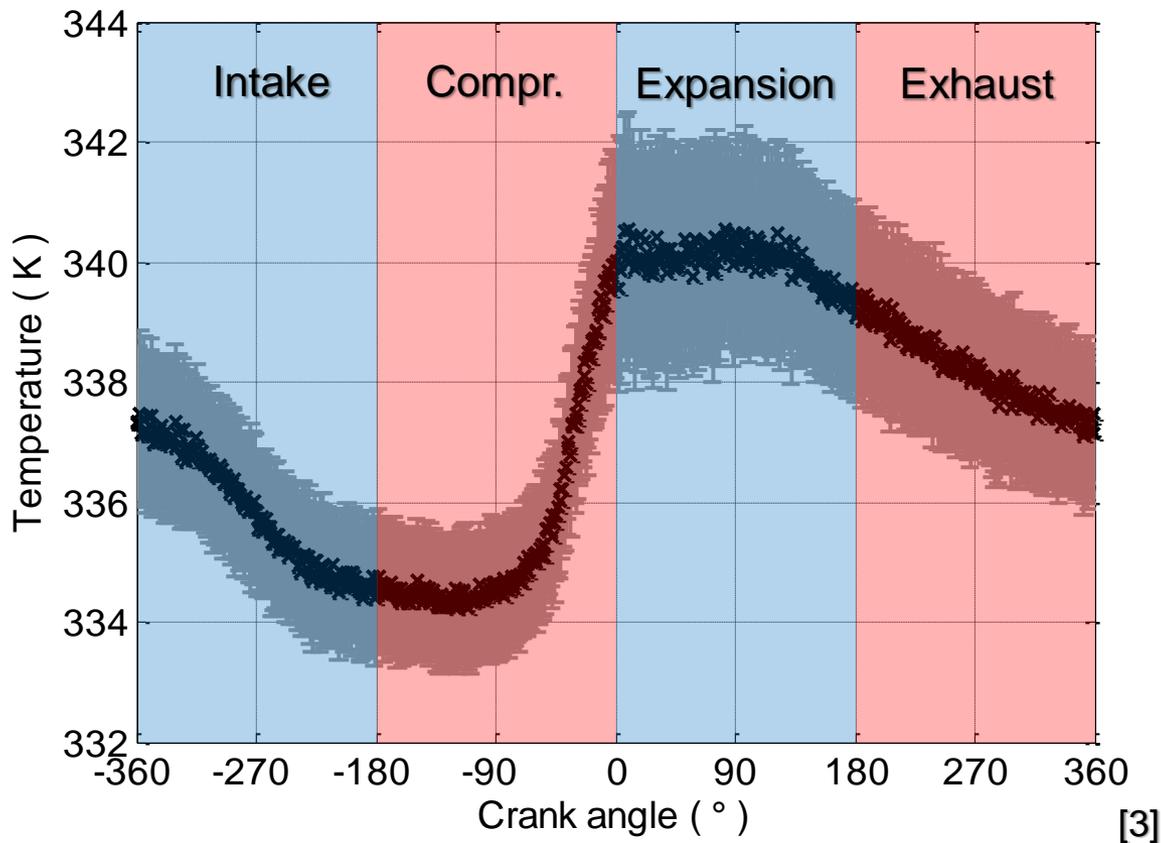
- Coated via airbrush
- Dispersion of binder and phosphor

## Engine

- Motored at 1000rpm
- No injection and ignition
- Compression ratio 8.5:1

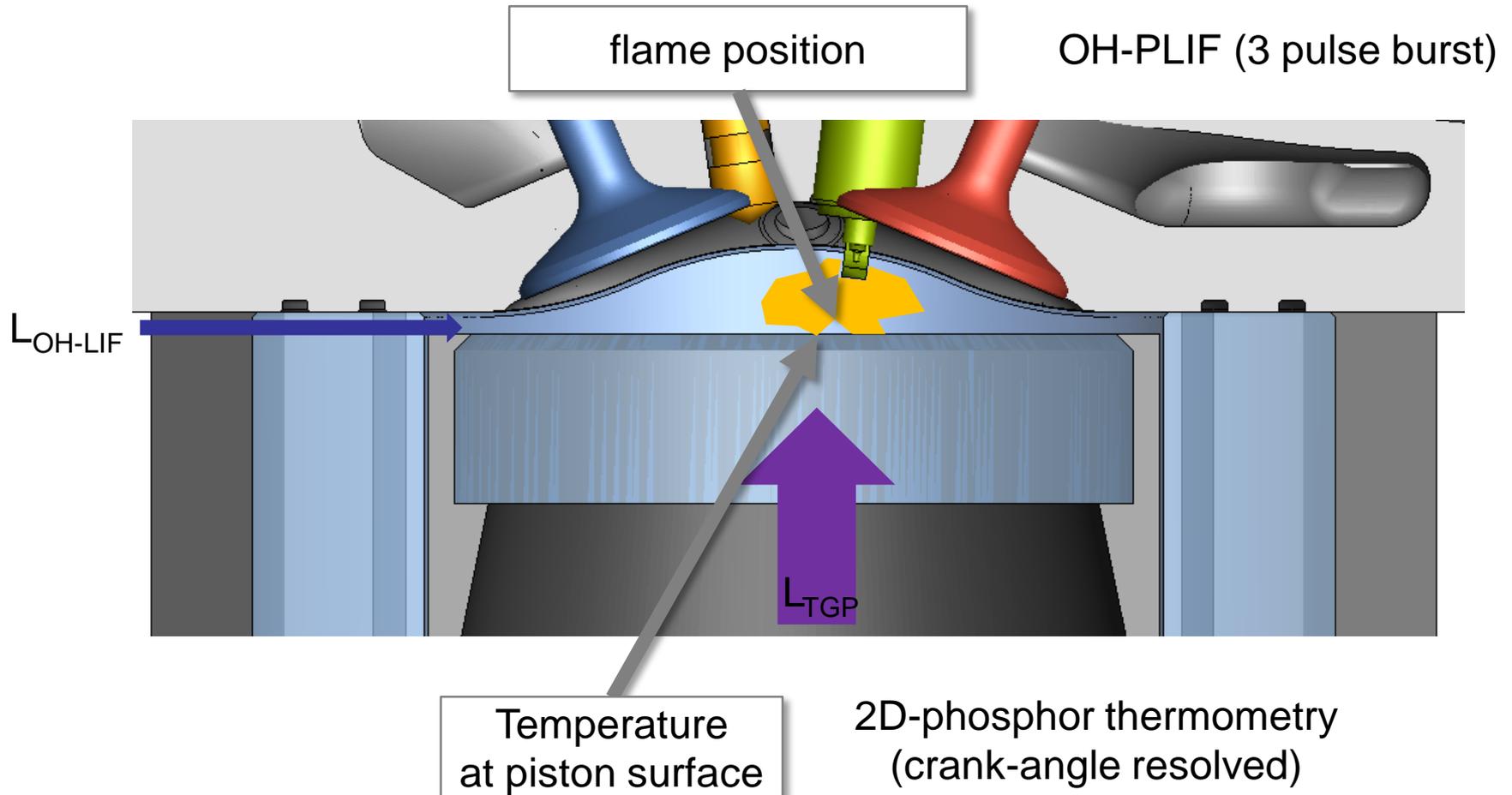
# Results for motored engine (no combustion)

- 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

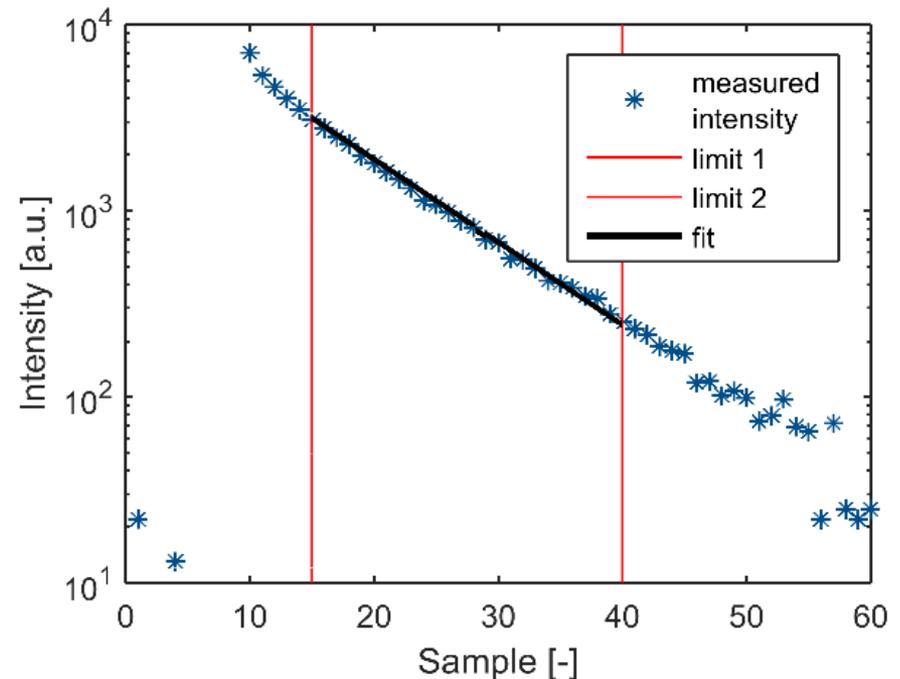
# Wall temperature measurements during flame wall interactions in IC engine



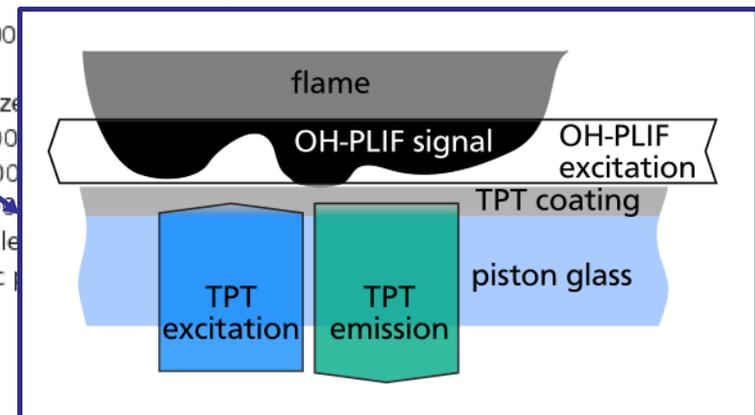
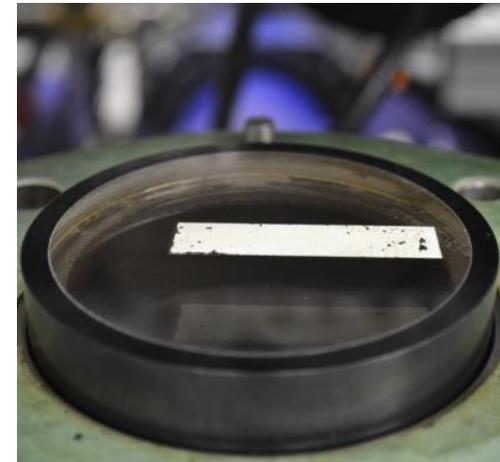
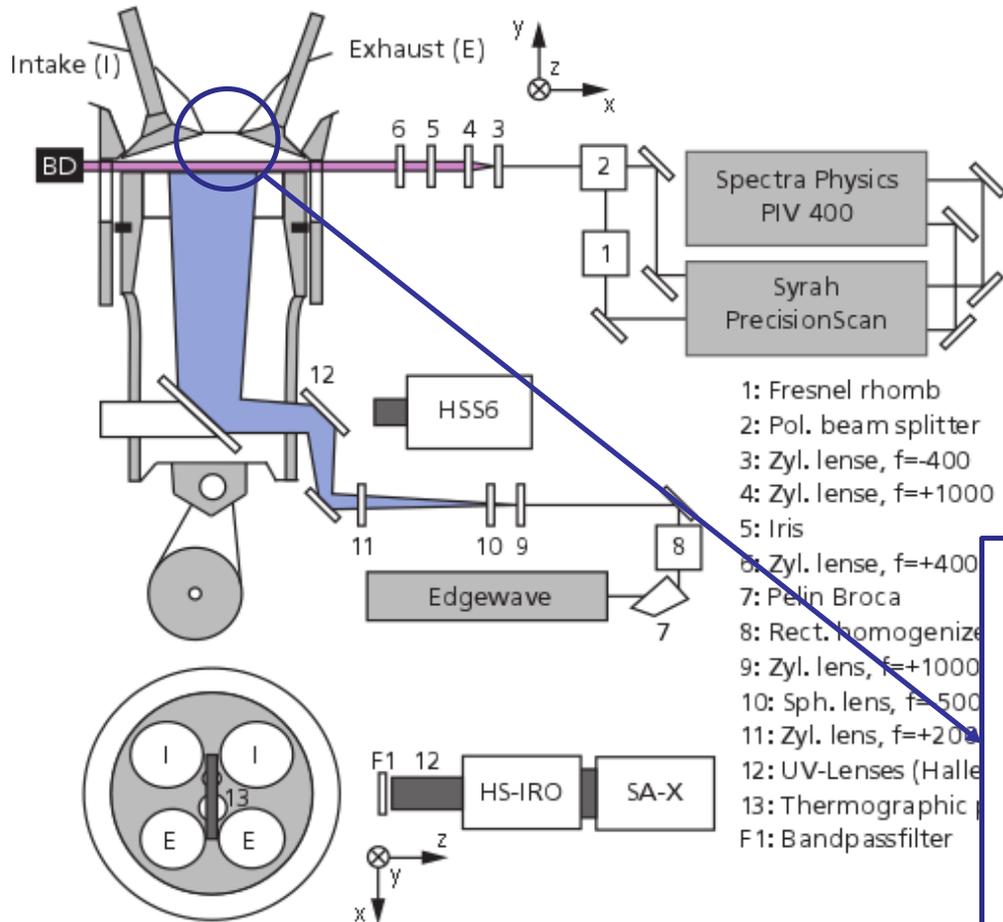
Ding et al. Dreizler, Böhm. Appl. Phys. B (2017) 123:110

# Phosphor thermometry

- Decay-time method
  - Ex-situ calibration
- Engine requirements
  - Fast temperature measurements ( $\sim 10\mu\text{s}$ )
  - High sensitivity, high SNR ( $\sim 5\text{K}$ )
- Suitable thermographic phosphor:  $\text{Gd}_3\text{Ga}_5\text{O}_{12}:\text{Cr,Cer}$

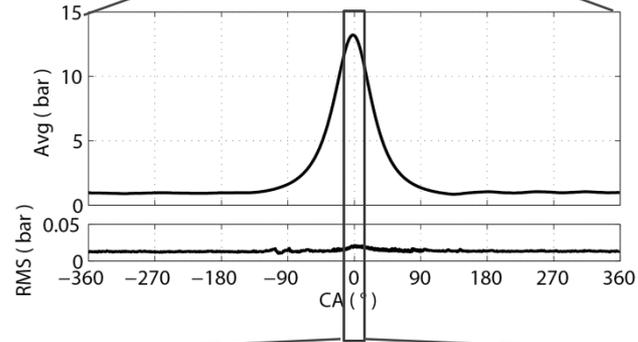
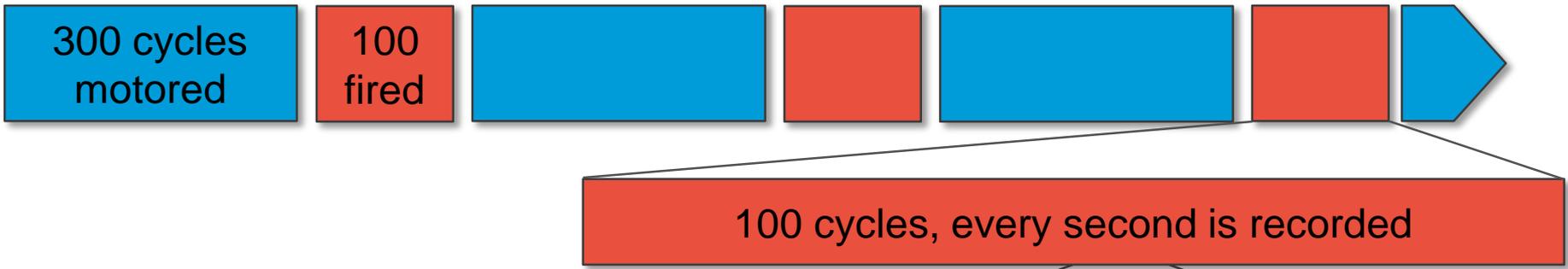


# Experimental setup for studying flame-induced heating



# Engine operational conditions

Heating up the engine for several runs → multiple runs recorded



HS-TPT

15 Images from -6.5 to 14.5 CAD aTDC

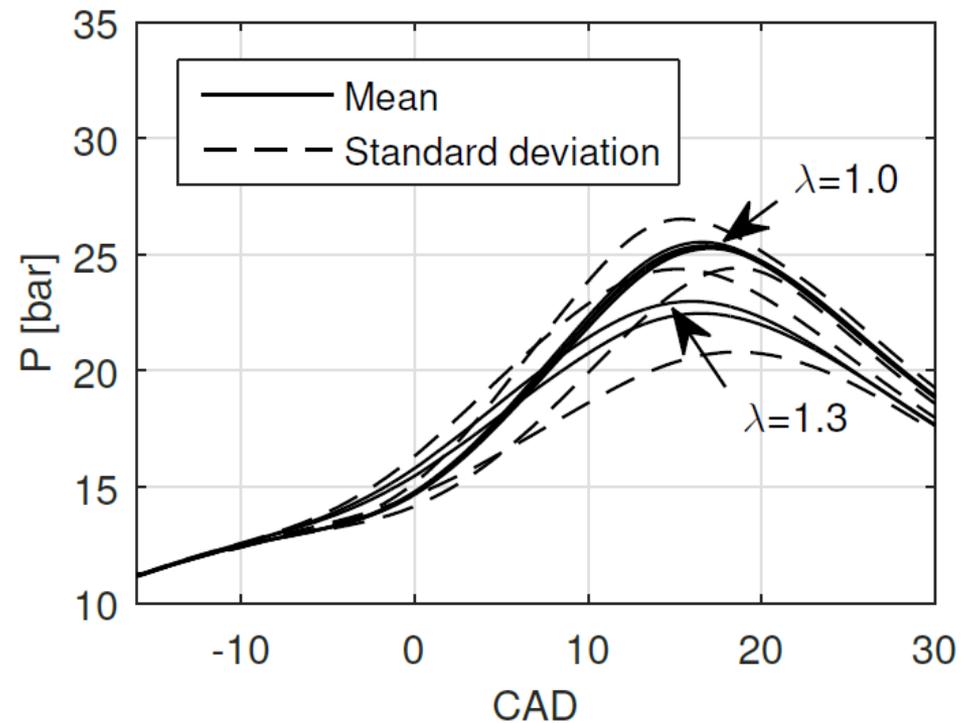
OH-PLIF

3 Images at -2, -0.5 and 1.0 CAD aTDC

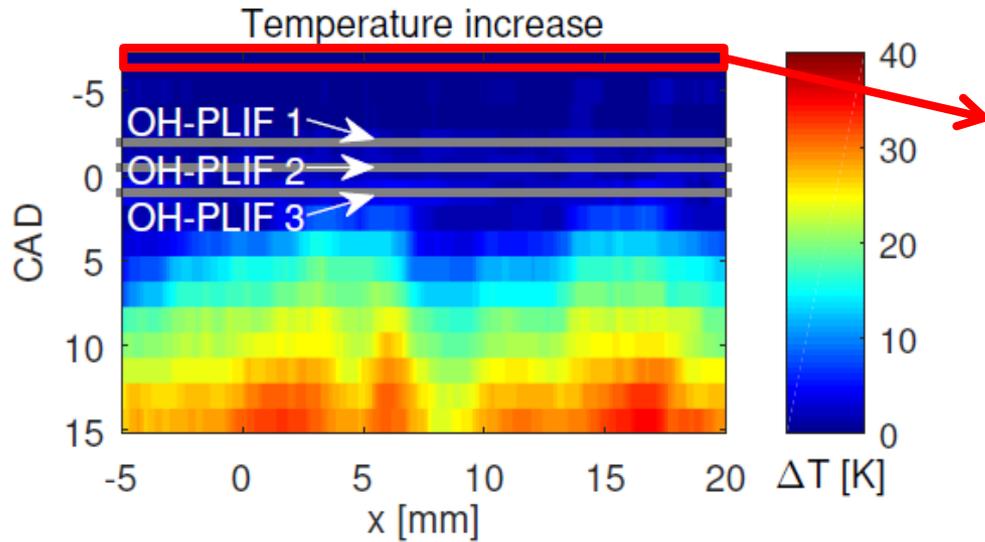
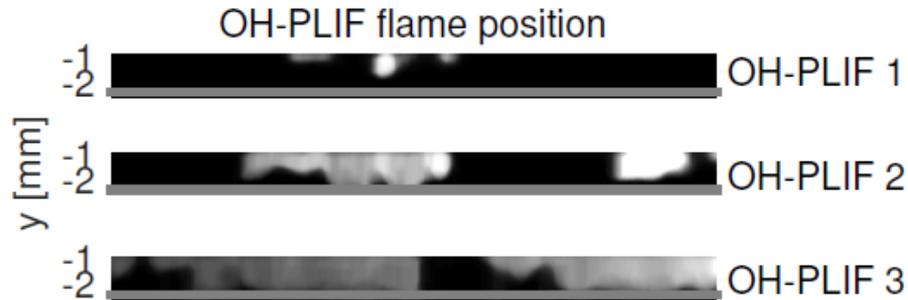
# Engine operational conditions

## Operating Point

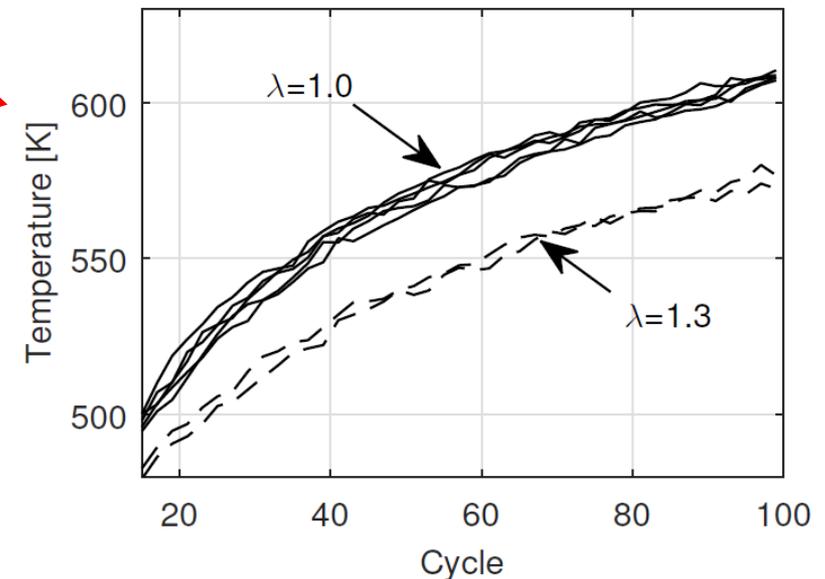
fuel	CH <sub>4</sub>
p <sub>in</sub>	950 mbar
T <sub>in</sub>	32°C
Speed	800 rpm
Imep	5.3 bar
lambda	1.0 and 1.3
t <sub>ignition</sub>	-11 and -16 CAD aTDC



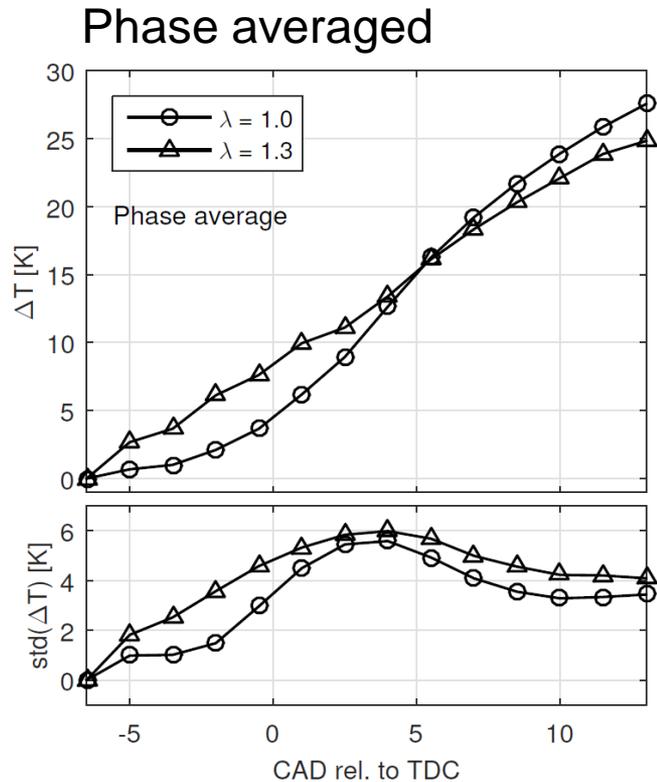
# Flame position and wall temperature



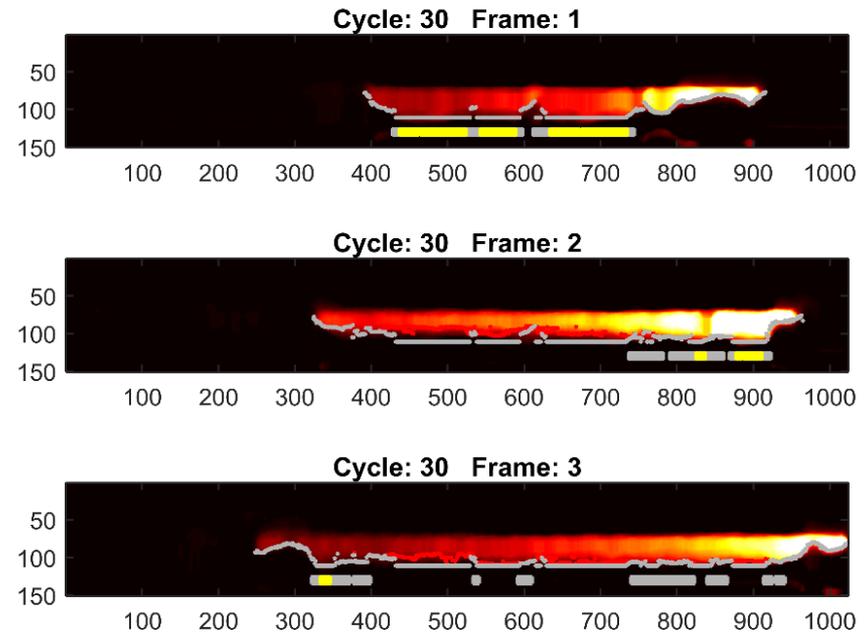
- coherence of flame impingement and wall temperature increase
- reproducible temperature evolution



# Temperature rise during flame-wall interaction



## Conditional averaged



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



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