

Fluorescence Lifetime Imaging (FLIM) and Micro Spectroscopy of Yb-doped materials

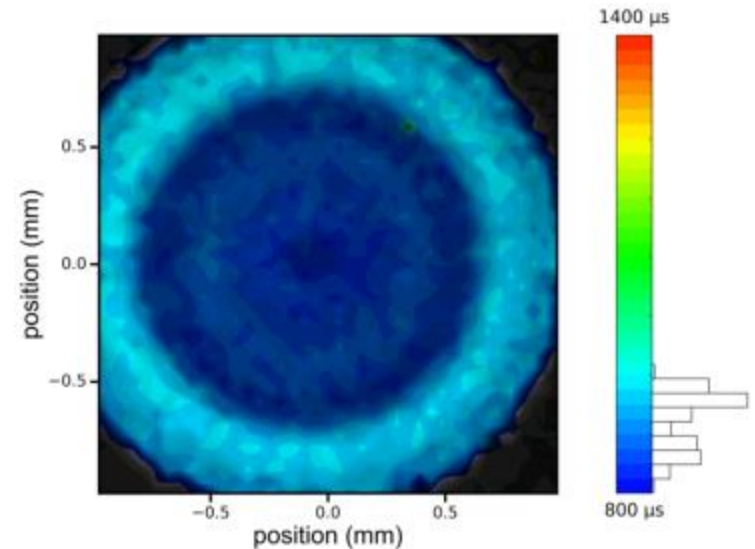


Fraunhofer
IOF

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Gerrit Feldkamp

Thomas Schreiber



Motivation

- Fraunhofer IOF produces Yb-doped material: preforms for fiber fabrication
- Before: measurement of refractive index and concentration possible
- We want to add another monitoring for drawn fibers and preforms
 - Detection of flaws
 - Spectral Yb characteristics
 - Calculation of emission cross section with Füchtbauer-Ladenburg eq.

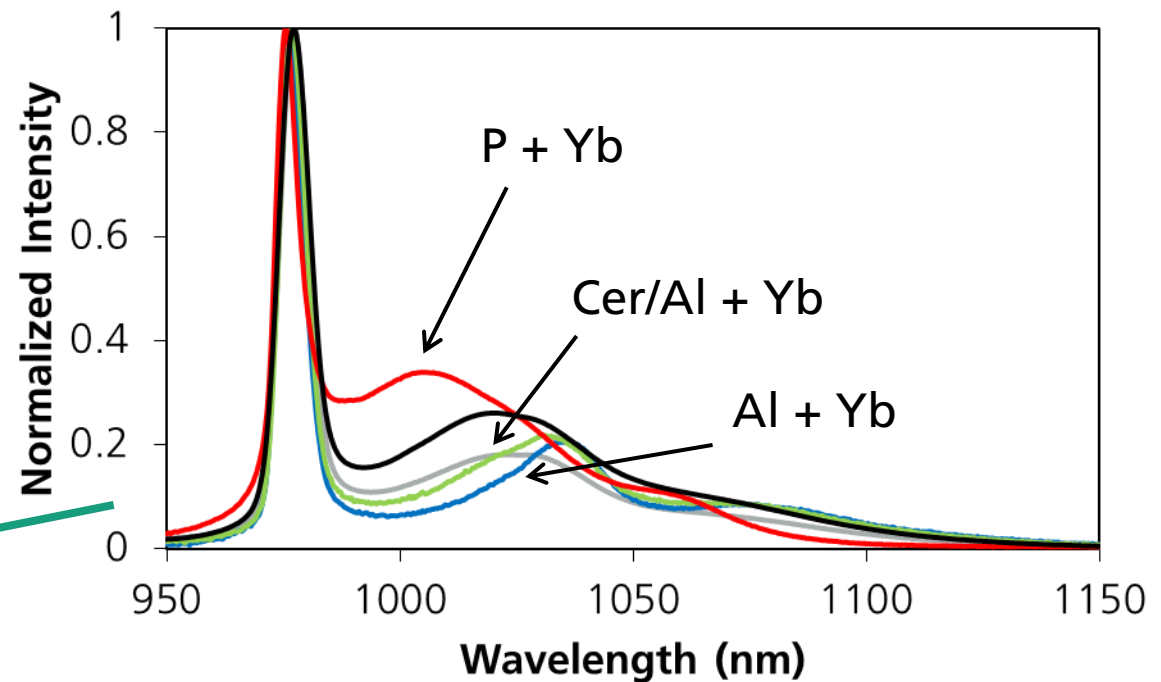
$$\sigma_{em} = \frac{1}{8\pi n^2 c \tau} \cdot \frac{\lambda^5 I_{em}(\lambda)}{\int \lambda I_{em}(\lambda) d\lambda}$$

Motivation

- Yb fluorescence is sensitive to its chemical environment
 - Phosphorus, Cerium, Aluminium as co-dopants
 - Spectral profile changes

→ Calculation of emission cross section

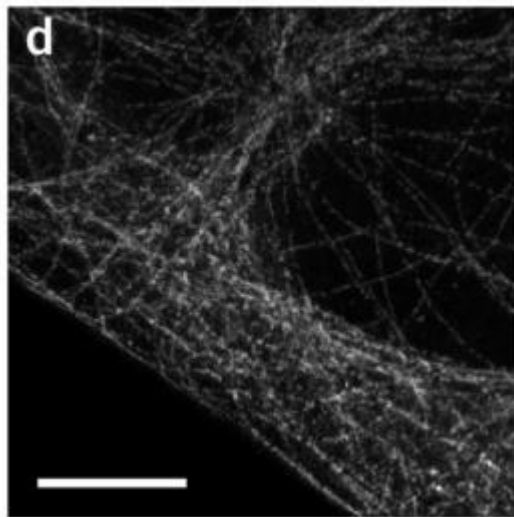
$$\sigma_{em} = \frac{1}{8\pi n^2 c \tau} \cdot \frac{\lambda^5 I_{em}(\lambda)}{\int \lambda I_{em}(\lambda) d\lambda}$$



FLIM principle

- Fluorescence Lifetime Imaging → FLIM

Fluorescence image

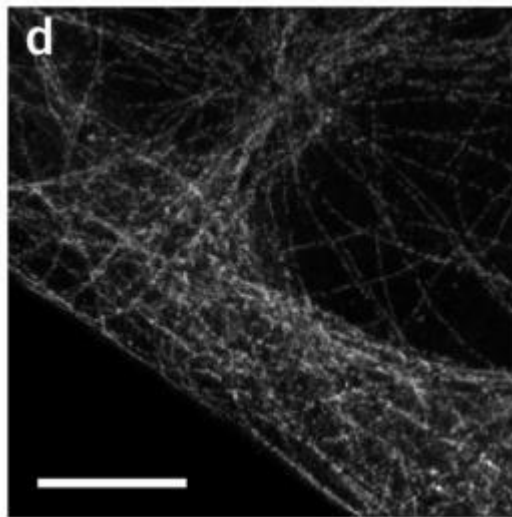


[1]

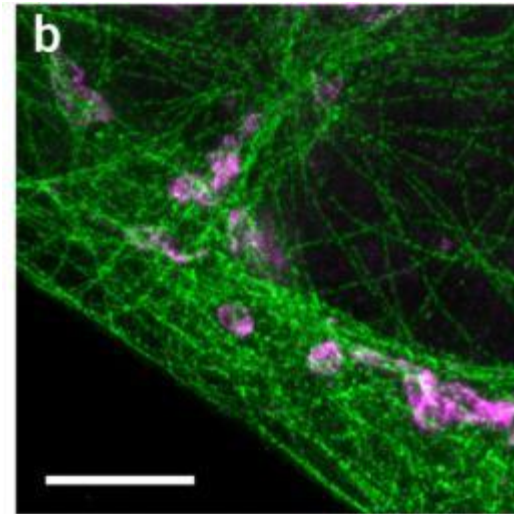
FLIM principle

- Fluorescence Lifetime Imaging → FLIM
- Lifetime gives additional contrast

Fluorescence image



FLIM



[1]

- Biological samples: $\tau \sim \mu\text{s} - \text{ns}$

[1] Niehörster, Thomas, et al. "Multi-target spectrally resolved fluorescence lifetime imaging microscopy." *Nature methods* (2016).

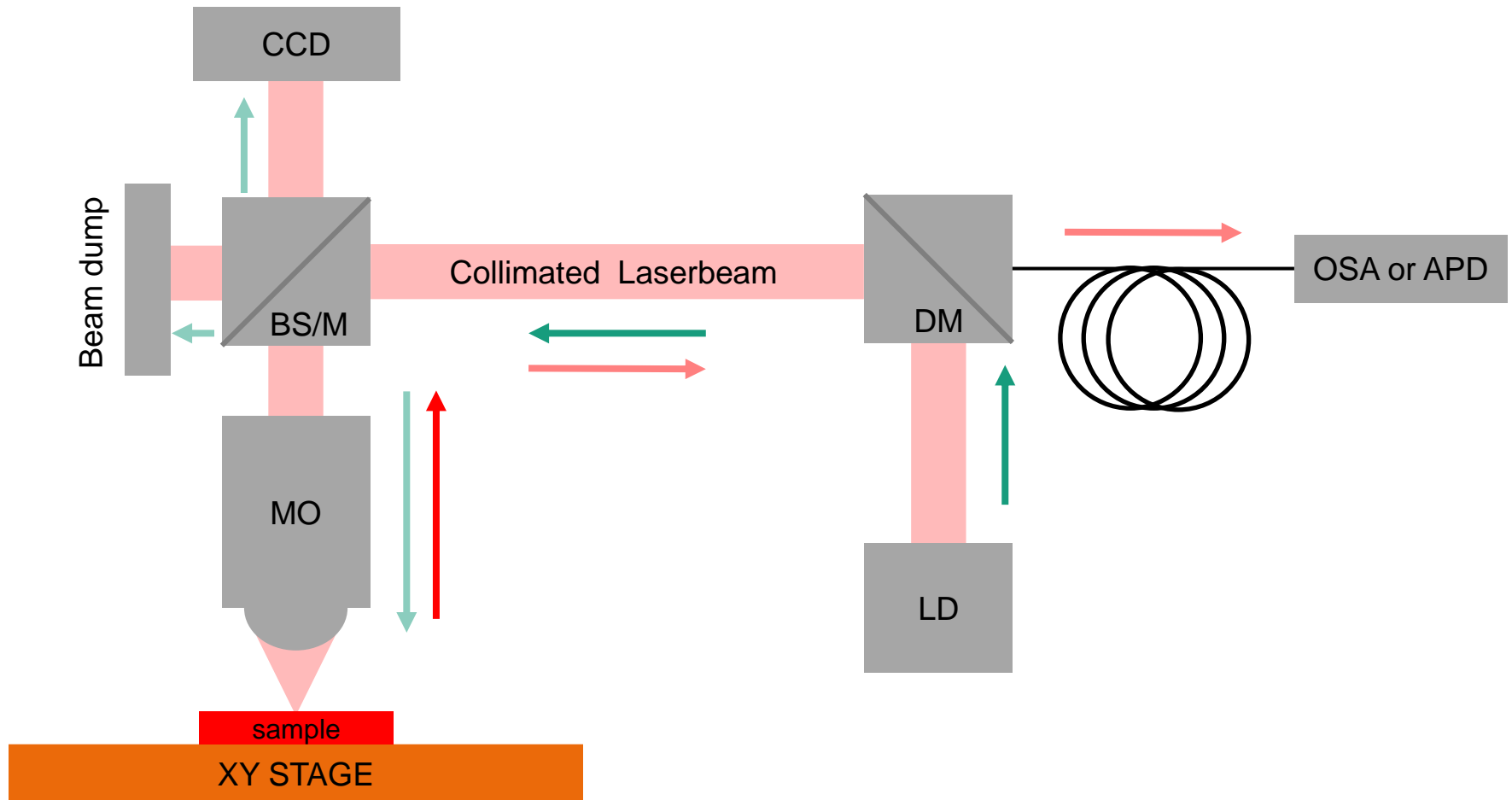
Motivation: FLIM principle

- Rare-earth doped laser materials
 - Typical lifetimes: ~ms
- Previously shown for Er-doped material[2]
- This talk: Yb doped laser material
 - Pump at 910nm
 - Characteristic fluorescence emission
 - Spectrum
 - lifetime



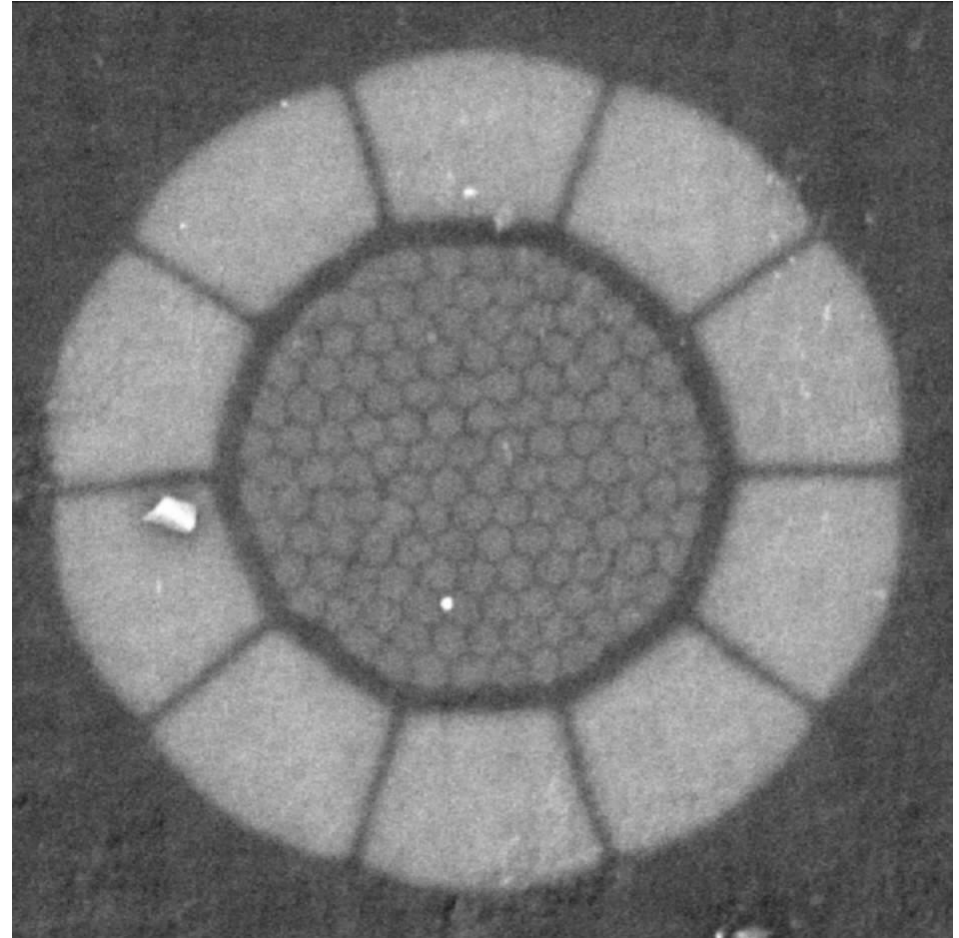
Fabrication of Yb doped silica

Setup



First measurements of Fluorescence on Yb:doped fiber

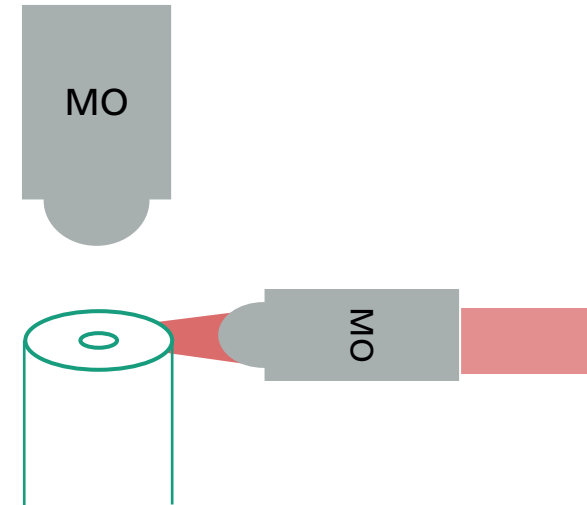
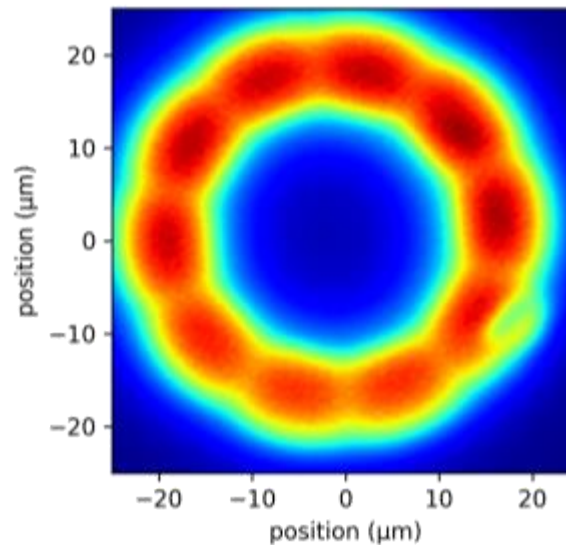
- RMO fiber
 - SEM image
 - Ring with 10 doped parts
 - Center non-doped
- This fiber used for Proof of principle test
- Presented scanning setup compared to light sheet microscopy



Proof of principle

- Lightsheet microscopy
 - One slice illumination
 - one frontal microscopic image

Lightsheet microscopy

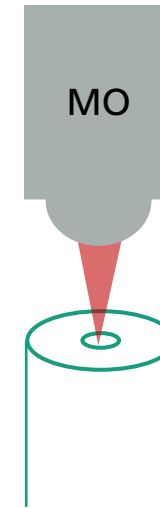
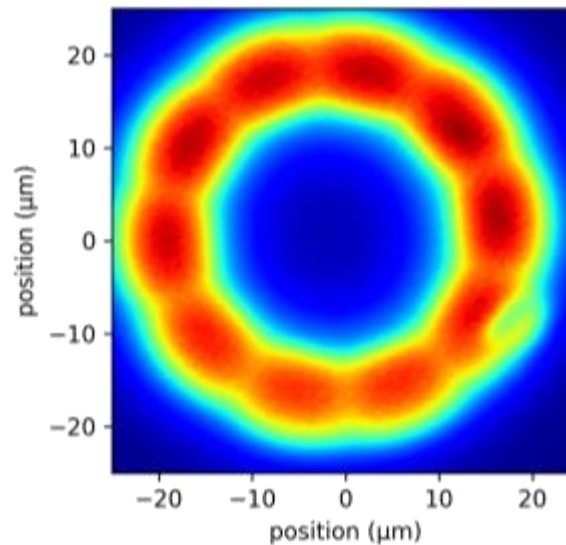


Proof of principle

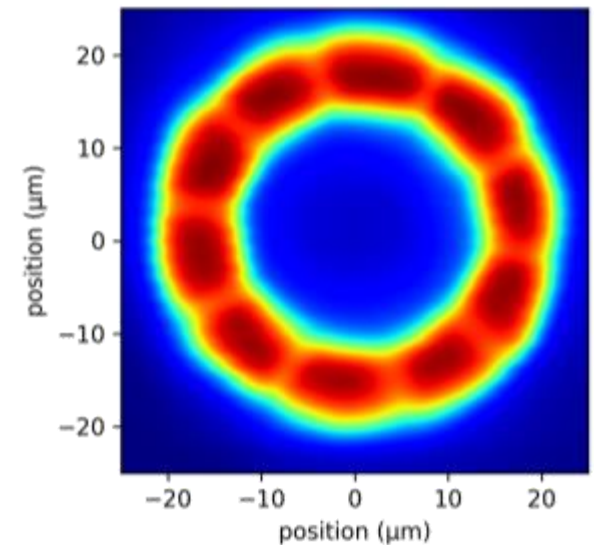
- Present setup
 - Point by point illumination
 - Point by point imaging
- Both setups show a similar image

- The setup works for imaging
- What is the advantage?

Lightsheet microscopy

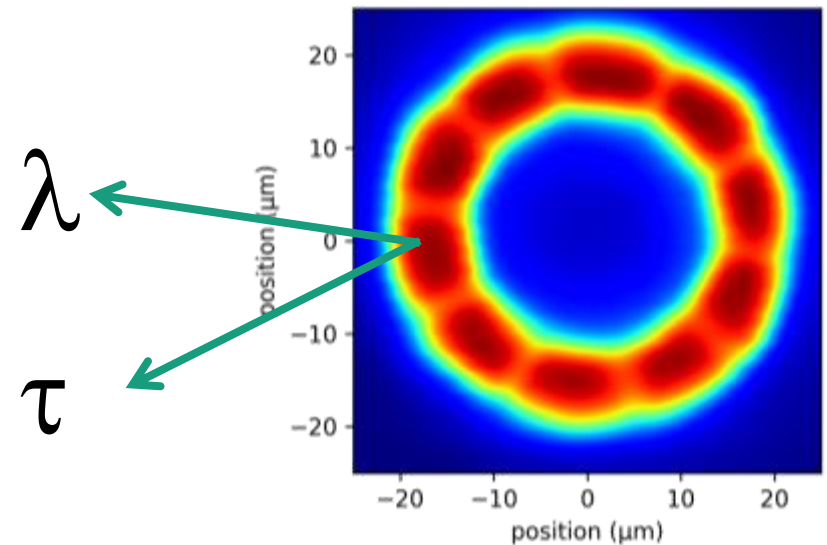


Present setup



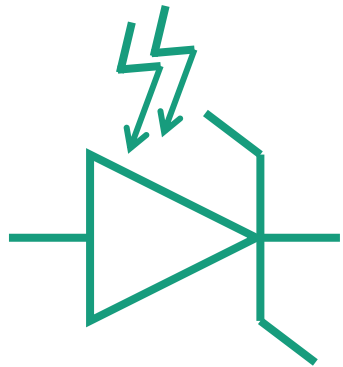
Proof of principle

- Scanning!!!
- In addition to intensity values
 - Lifetime
 - Spectrum
- This data is used for coloring the image

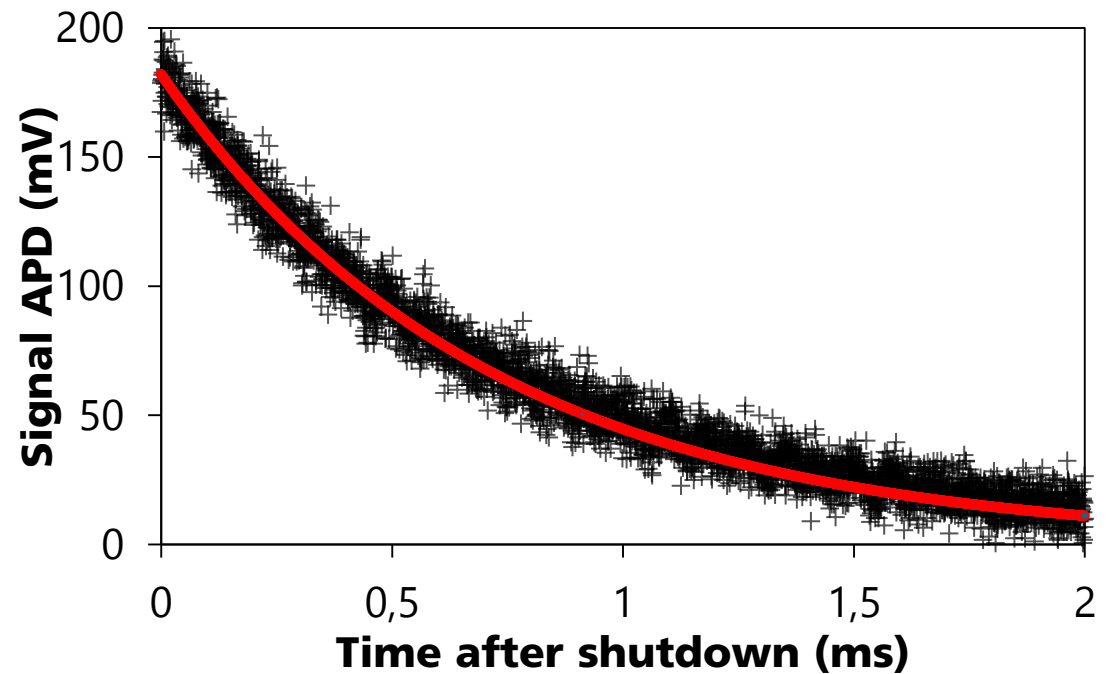


Data processing: Lifetime

- For each datapoint:
 - simple decay function to fit lifetime



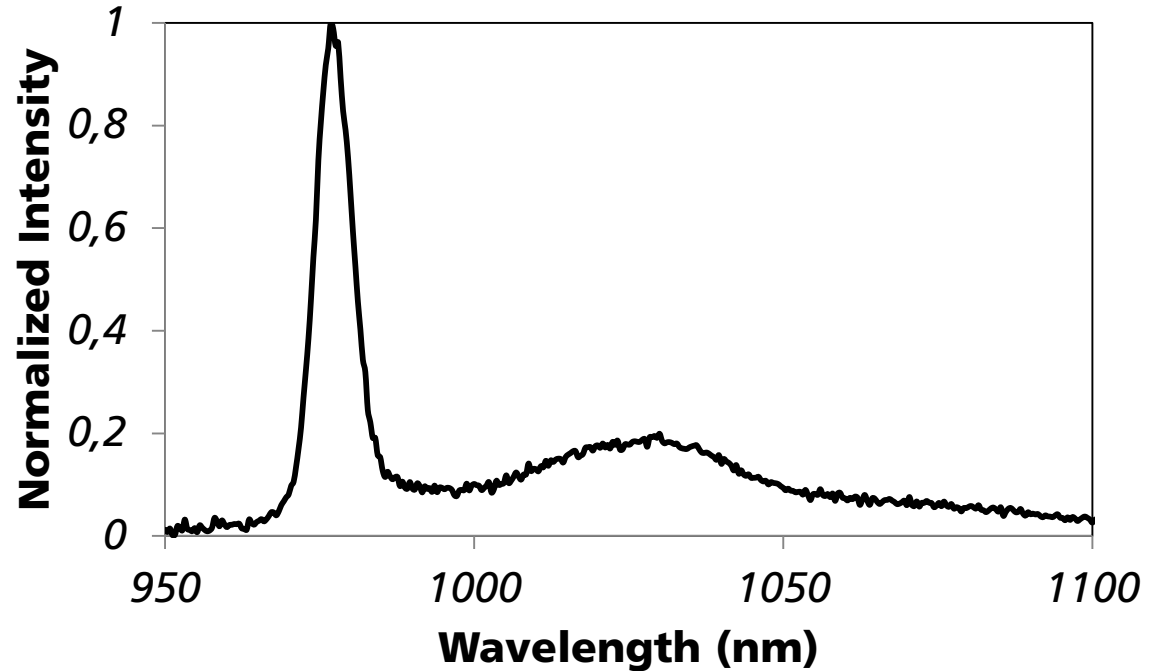
Avalanche Photodiode



Data processing: μ -spectroscopy

- For each datapoint:
 - Fluorescence emission spectrum acquired by OSA

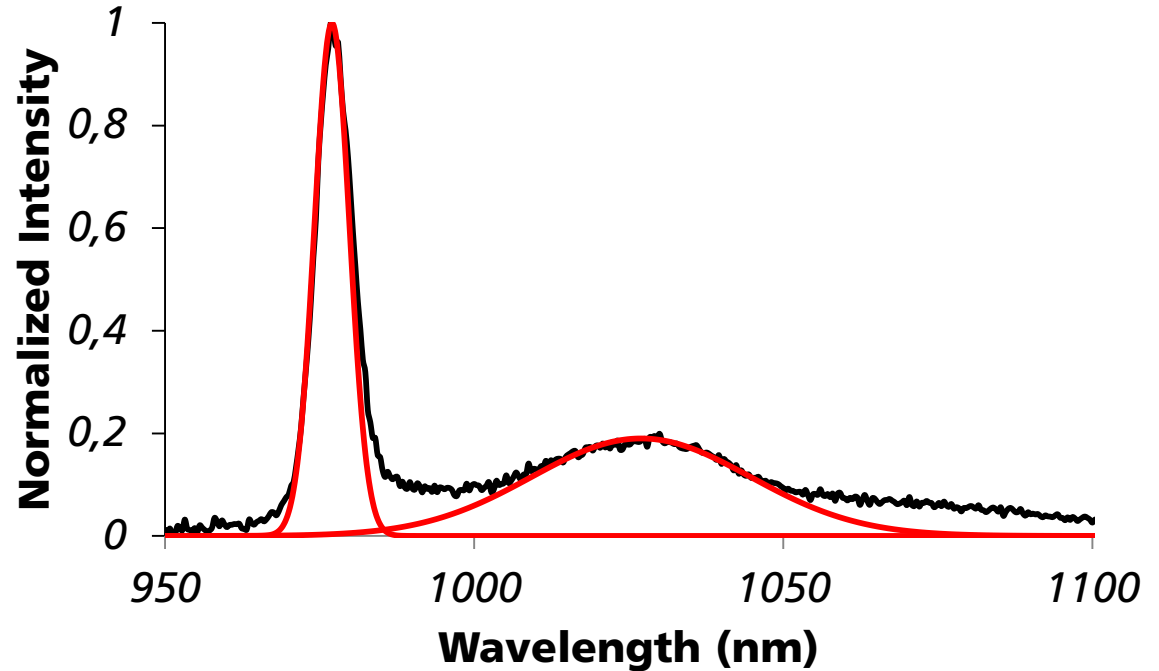
OSA →



Data processing: μ -spectroscopy

- Easy contrast values \rightarrow central wavelengths

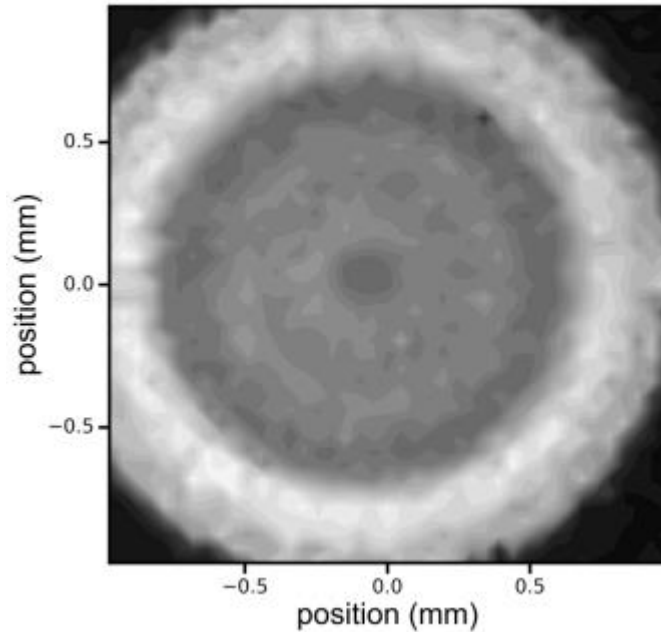
OSA \rightarrow



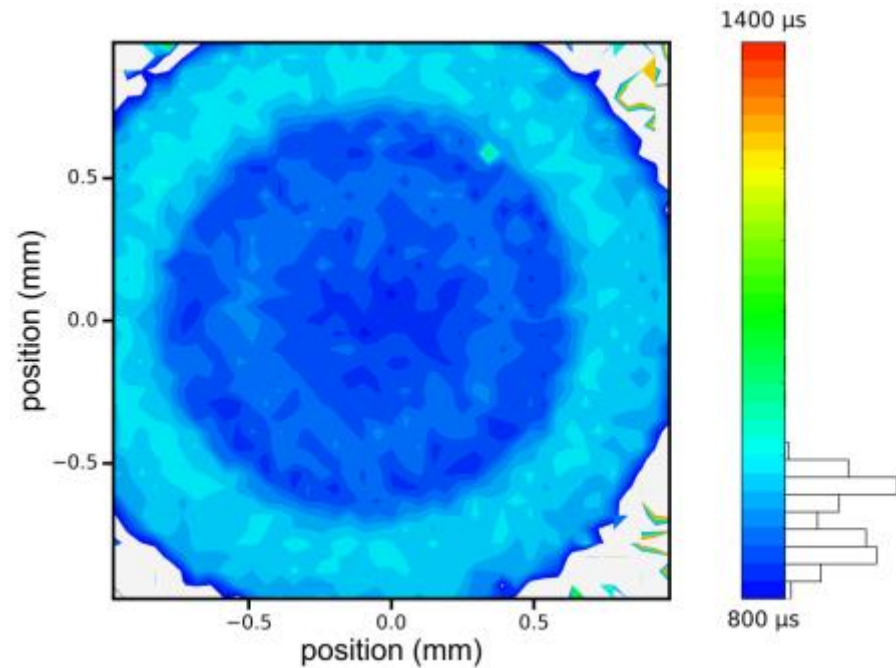
Data processing: Image creation

- Custom design preform
 - Ring and center different composition

Fluorescence Intensity map



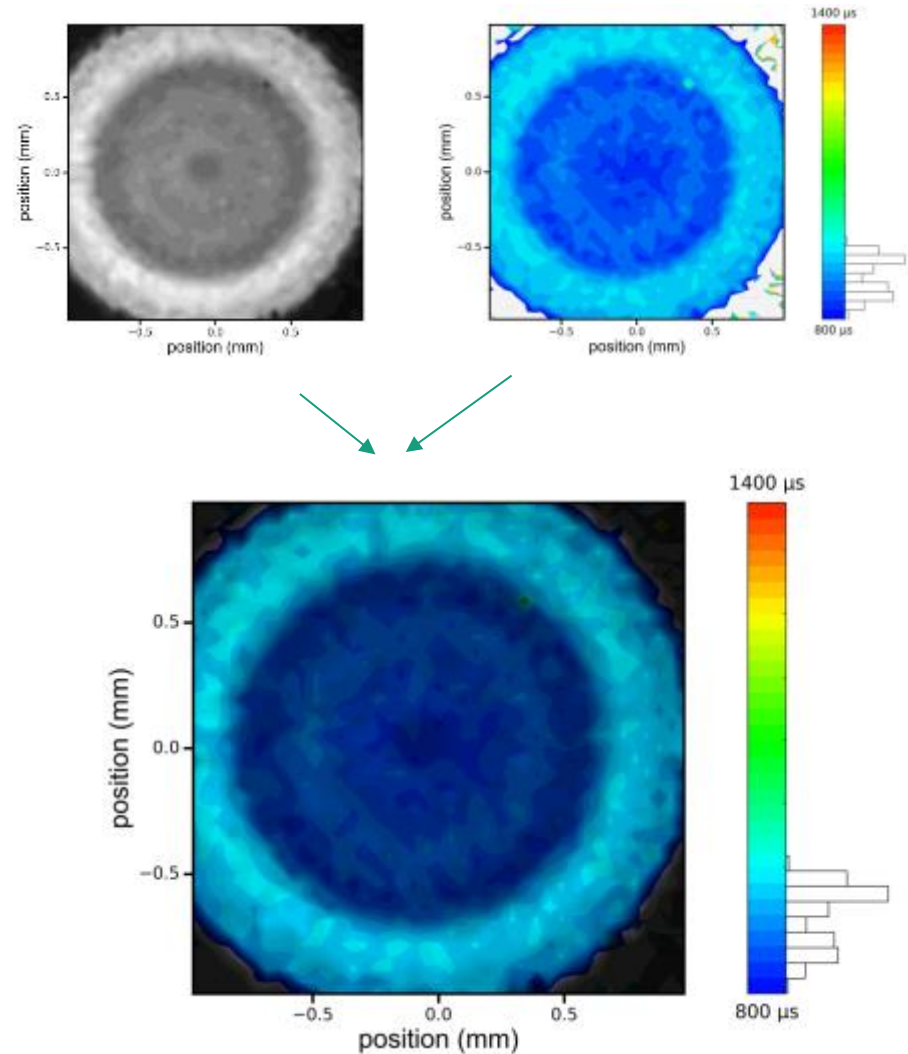
Fluorescence Lifetime map
(or spectral data)



Data processing: Image creation

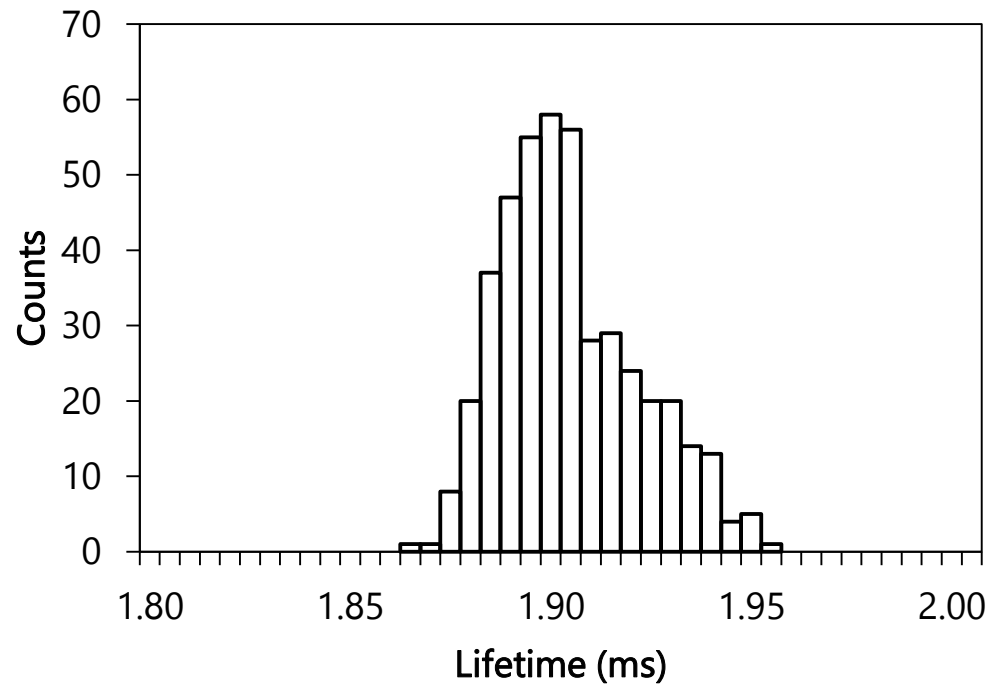
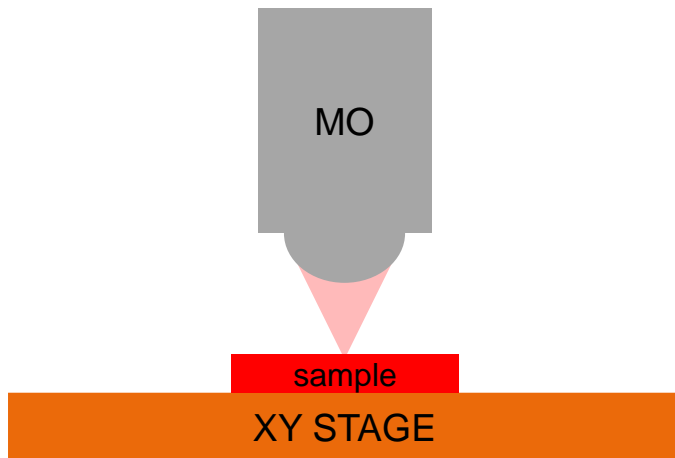
- FLIM image
 - Combined image
 - Similar for μ Spectroscopy

- Nice pictures...
are they reliable?



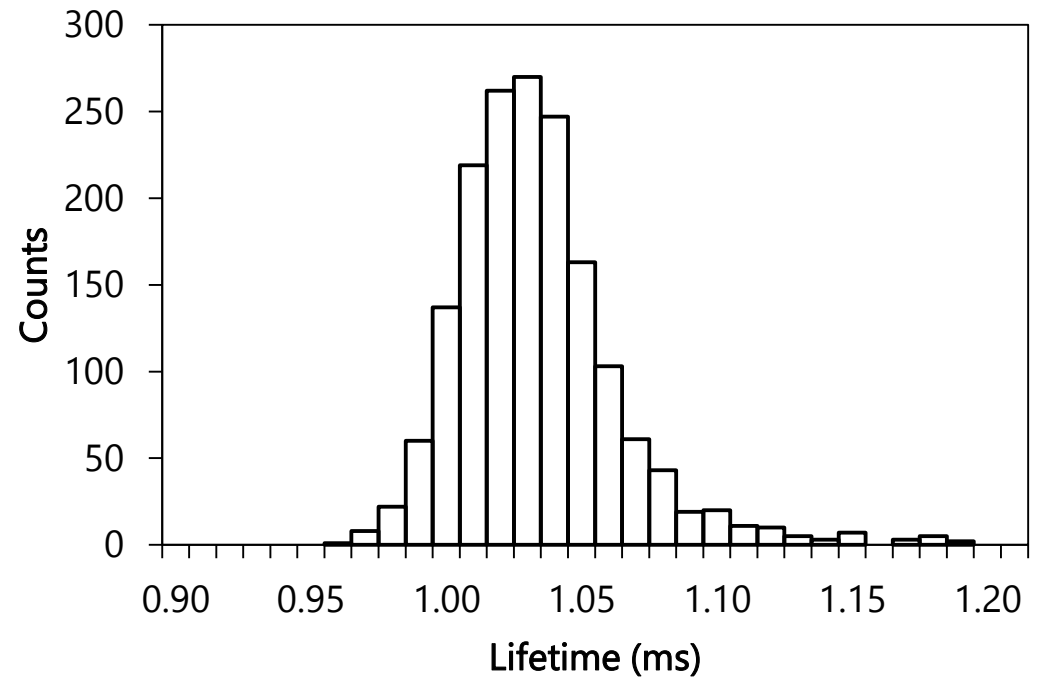
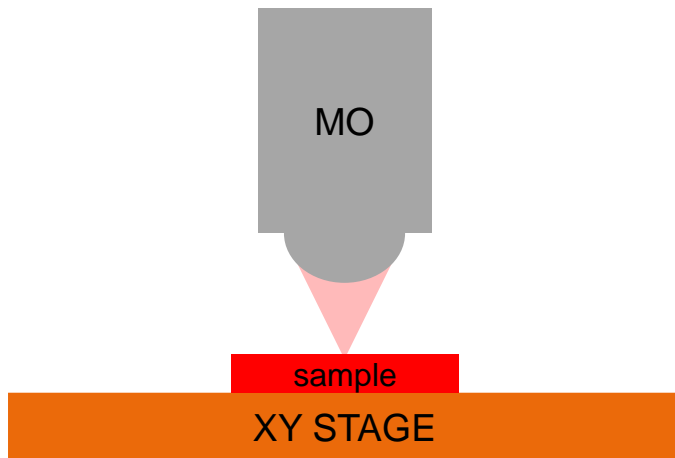
Validation: Lifetime

- 3 at% Yb:CaF₂
 - Reported lifetime of 1.9 ms \pm 0.1ms
 - Our setup 1.90 ms \pm 0.03ms



Validation: Lifetime

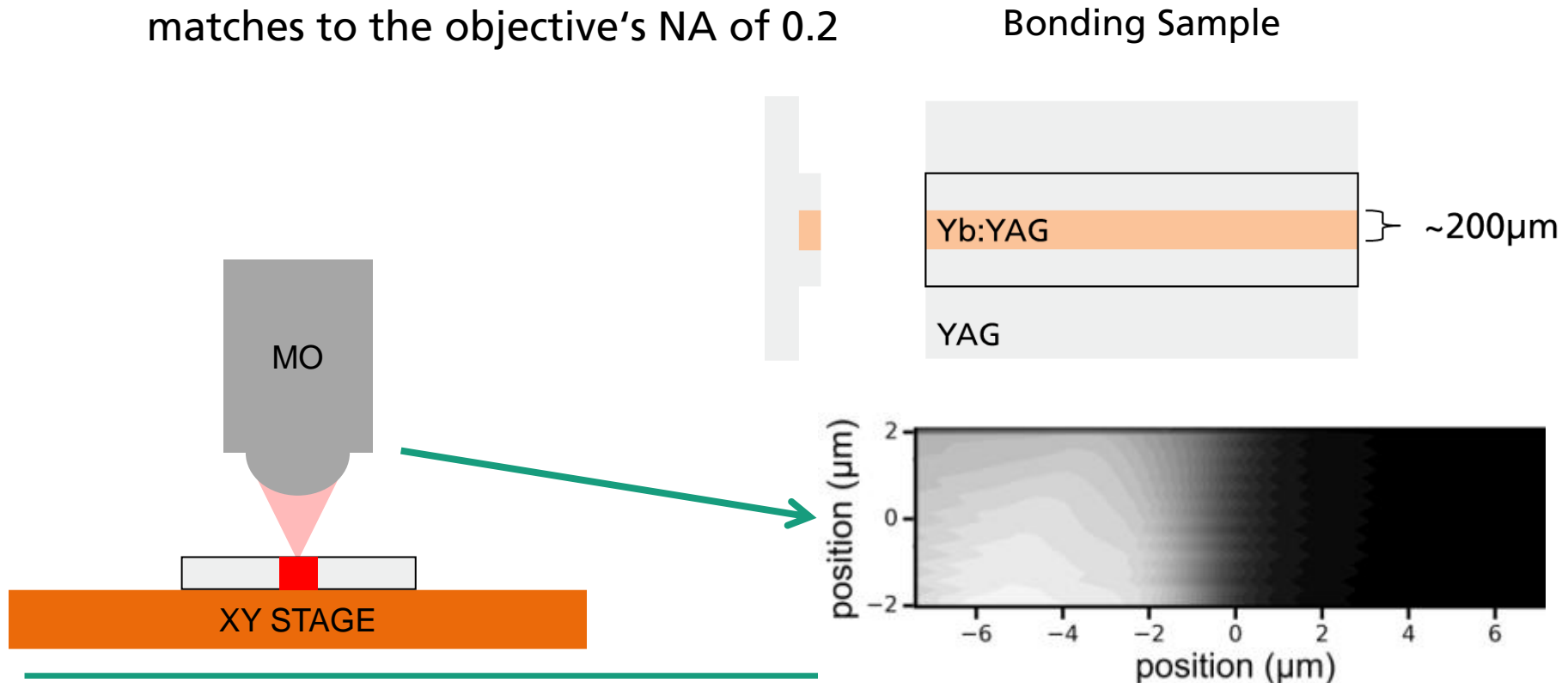
- 1 at% Yb:YAG
 - Reported lifetime of $1.0 \text{ ms} \pm 0.1 \text{ ms}$
 - Our setup $1.03 \text{ ms} \pm 0.05 \text{ ms}$



Validation: Resolution

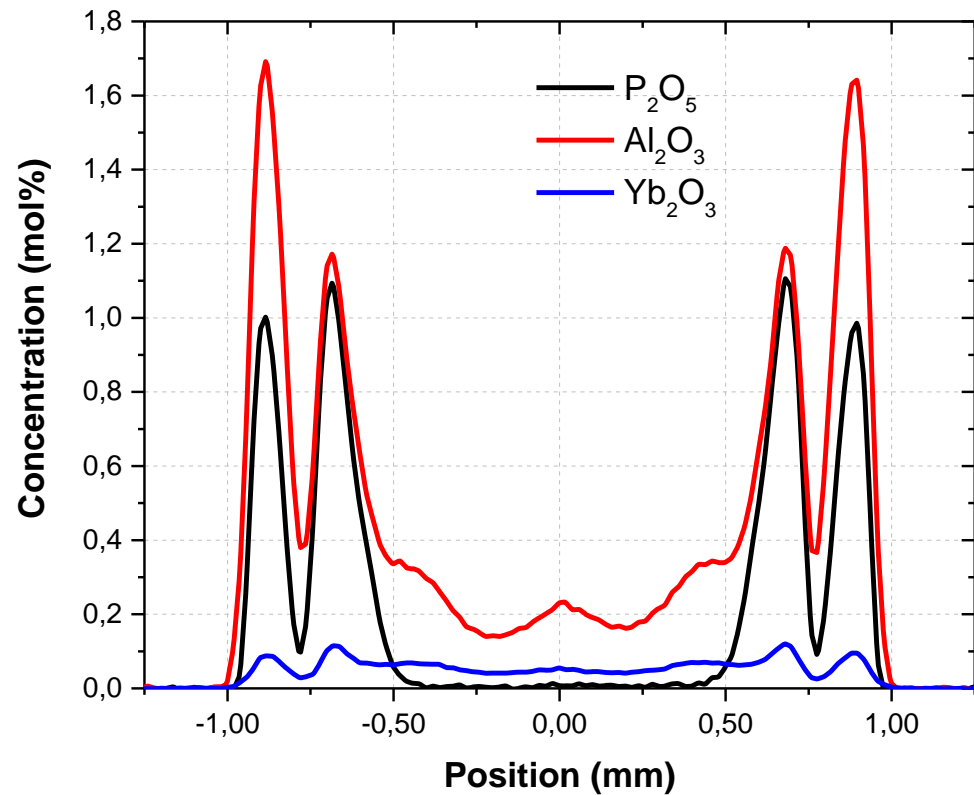
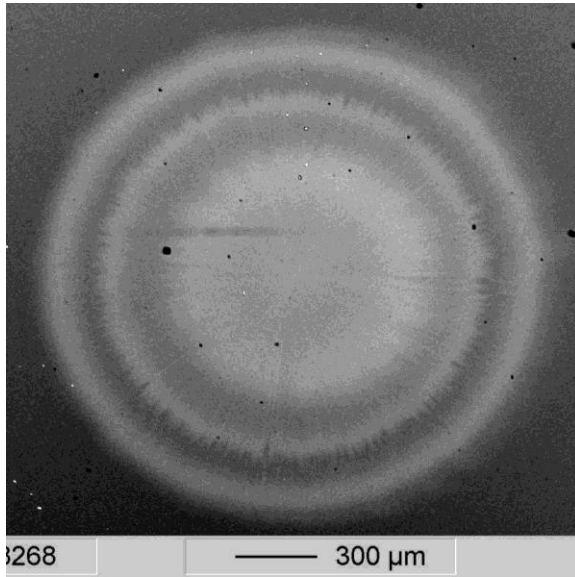
■ 1 at% Yb:YAG

- edge detection of Yb:YAG bonded to non-doped YAG
- **Spatial Resolution(10%-90%) of $4\mu\text{m}$**
matches to the objective's NA of 0.2



Example 1: Preform inspection

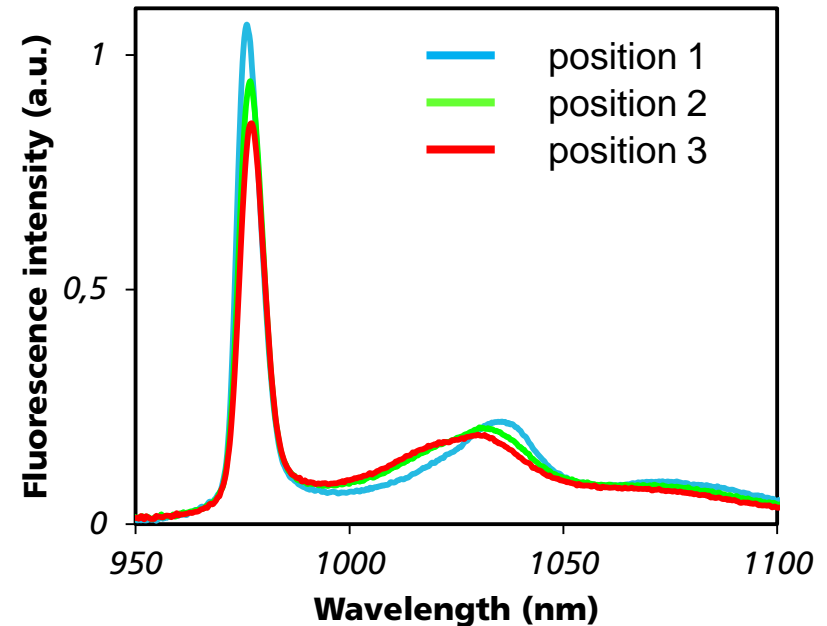
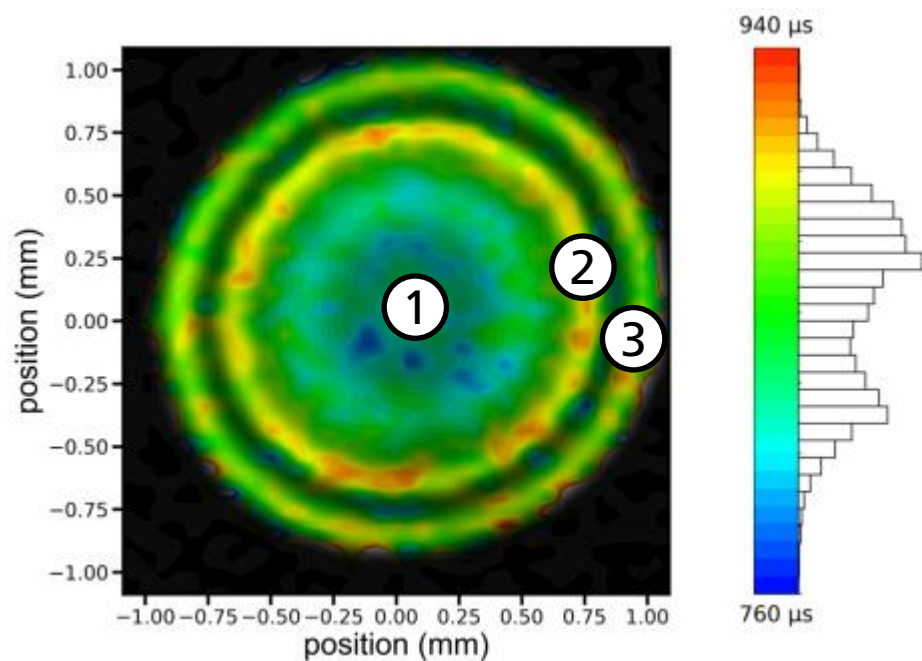
- Custom preform with multilayer structure
- EPMA measurement



SEM of preform

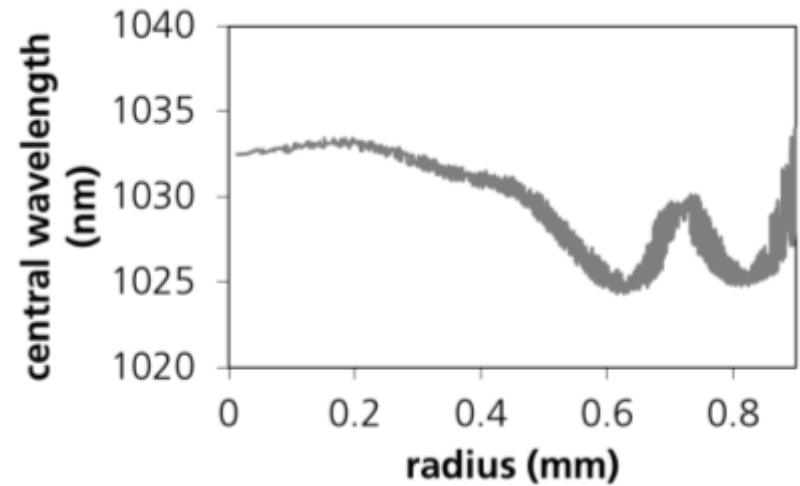
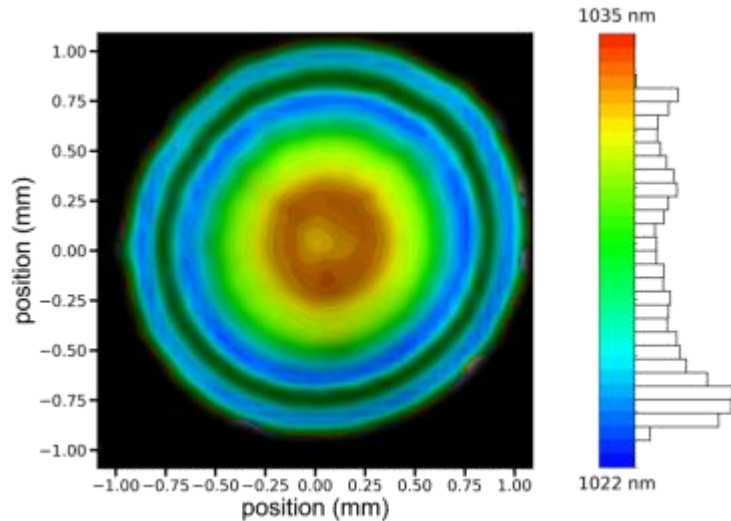
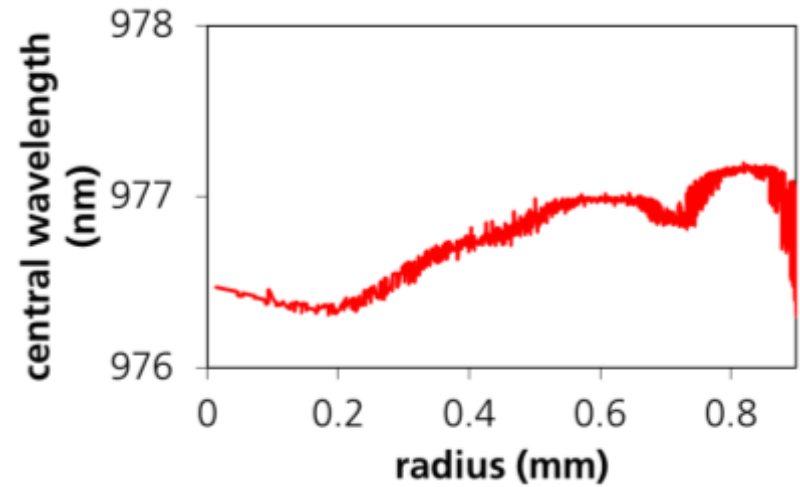
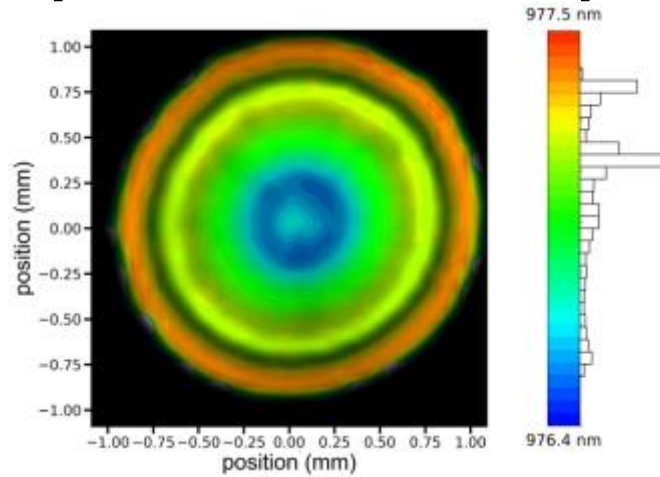
Example 1: Preform inspection

- 3 spots
- Different spectra



FLIM of preform

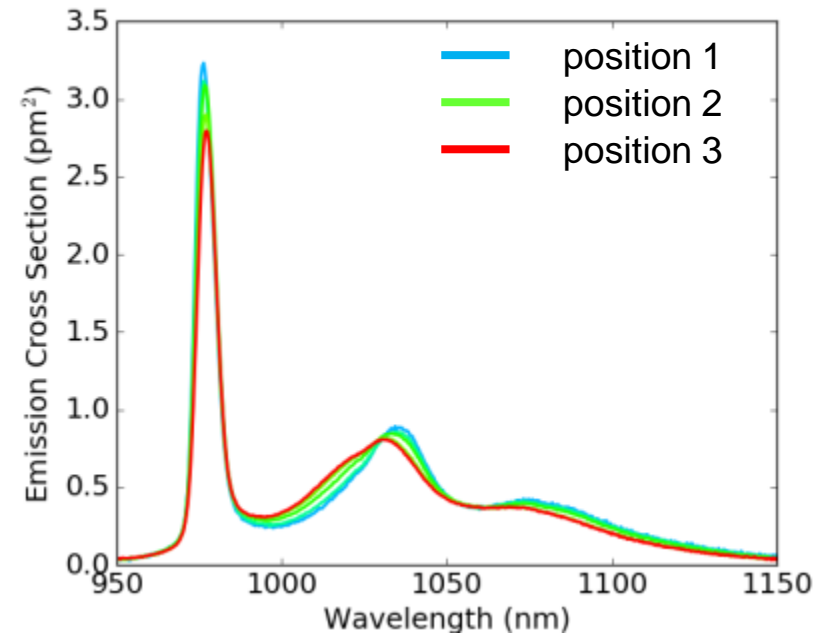
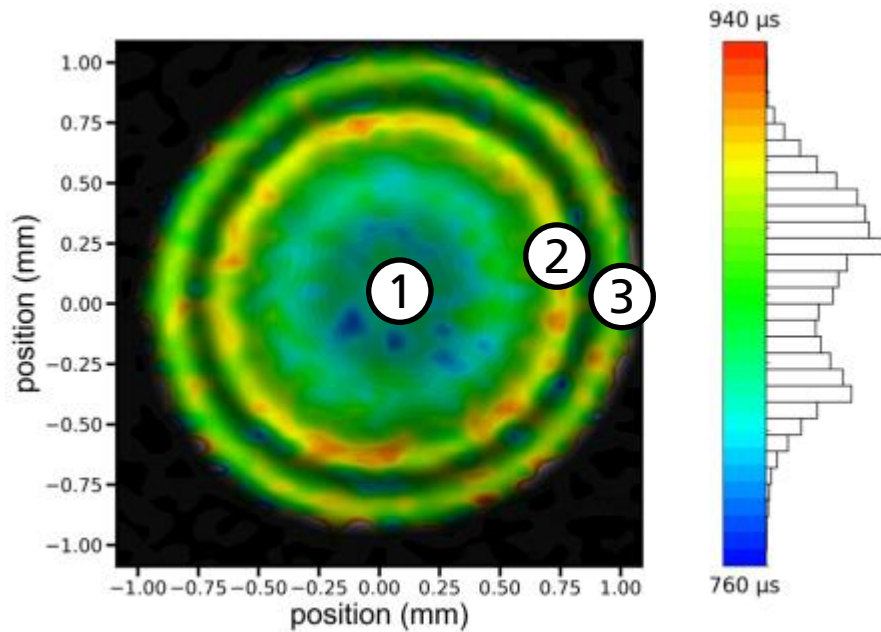
Example 1: Preform inspection



Example 1: Preform inspection

- Füchtbauer-Ladenburg-Equation
- 2D map of emission cross sections

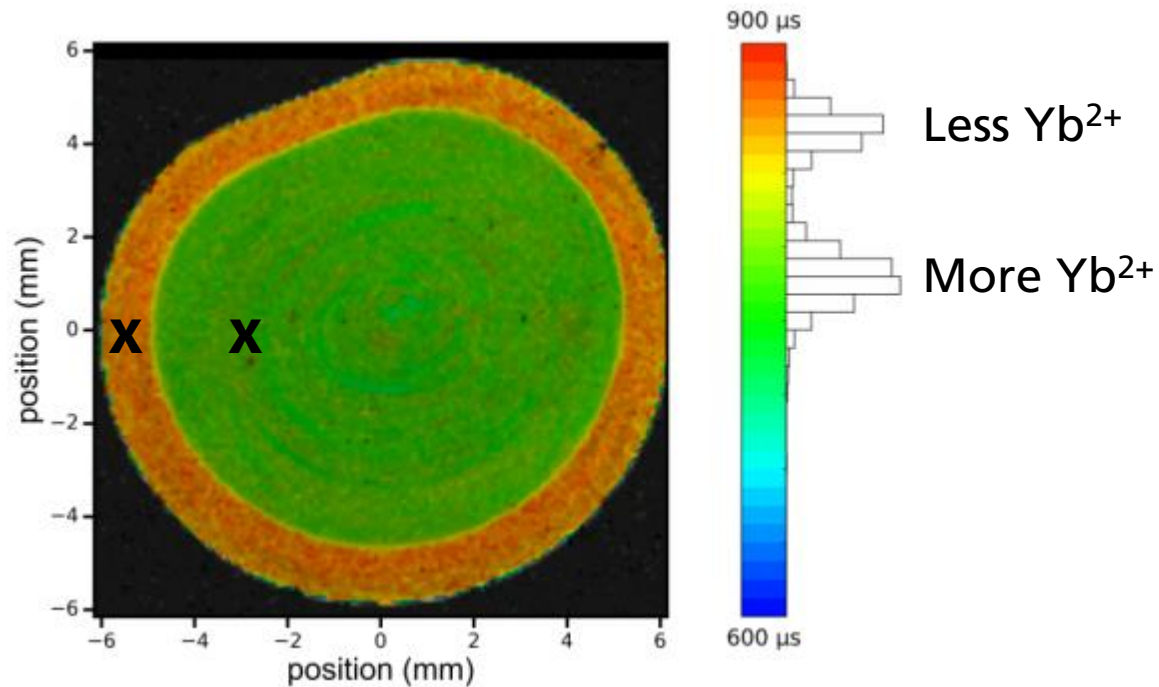
$$\sigma_{em} = \frac{1}{8\pi n^2 c \tau} \cdot \frac{\lambda^5 I_{em}(\lambda)}{\int \lambda I_{em}(\lambda) d\lambda}$$



Example 2: REPUSIL sample

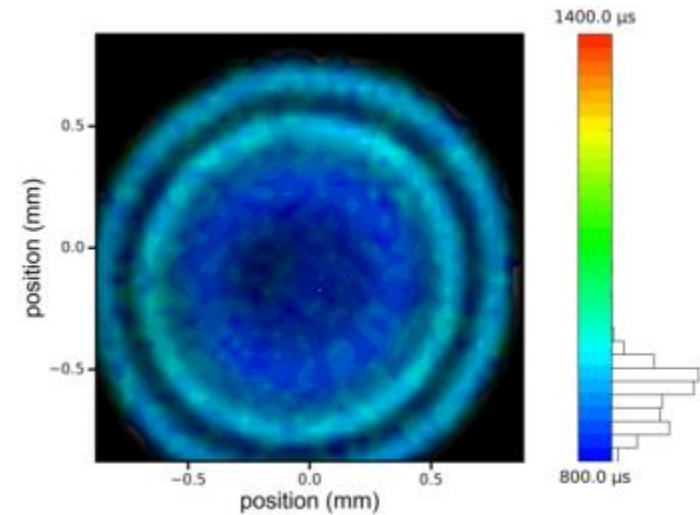
Effect of annealing process

- Effect of heat treatment on REPUSIL sample
- Outer circle contains less Yb^{3+} than inner portion



Summary

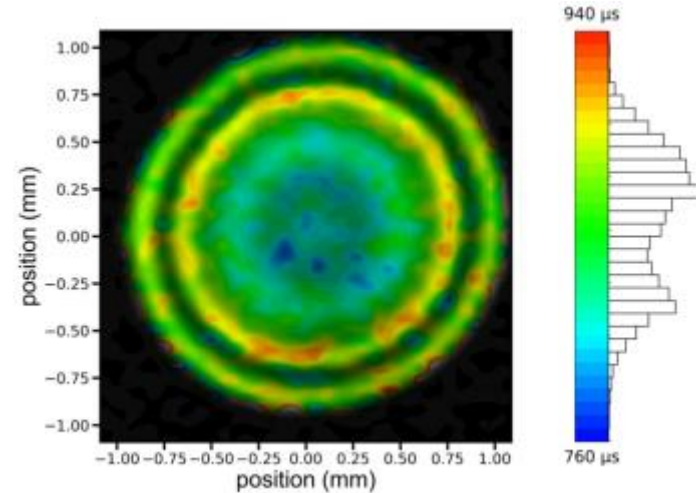
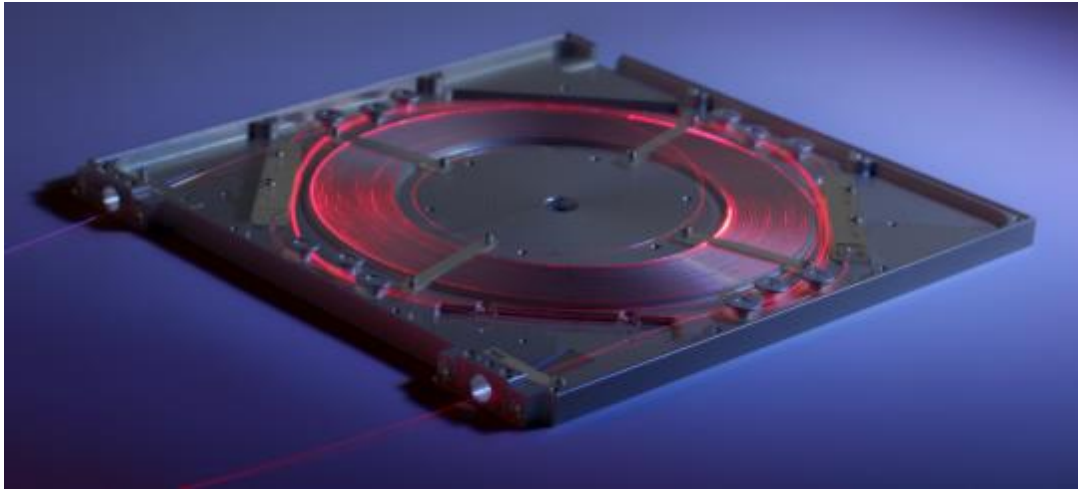
- Suitability and Resolution:
 - mapping of ROI
 - Size...fibers to preforms
 - One setup → all data
- Setup presented to detect Yb fluorescence
 - Validated lifetimes
 - Viable for monitoring Yb distribution
 - Lifetime and μ -spectroscopy add contrast to intensity-only pictures
- Shown Examples
 - Yb fluorescence is influenced by its chemical environment
 - Process related changes of Yb fluorescence
- Not shown
 - Photodarkening effects in Yb-doped fiber preforms detectable



Thank you, for your attention!

Special thanks to:

- Gerrit Feldkamp (Master Thesis)
- IPHT for kindly providing some of the shown samples
- and ...



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