

Exploring Ce³⁺:YAG glass ceramics by light sheet microscopy

**A. Herrmann, Y. P. Hartmann, J. Burkard, M. Hofmann,
U. Brokmann, E. Rädlein**

Institute of Materials Science and Engineering,
Technische Universität Ilmenau, Ilmenau, Germany

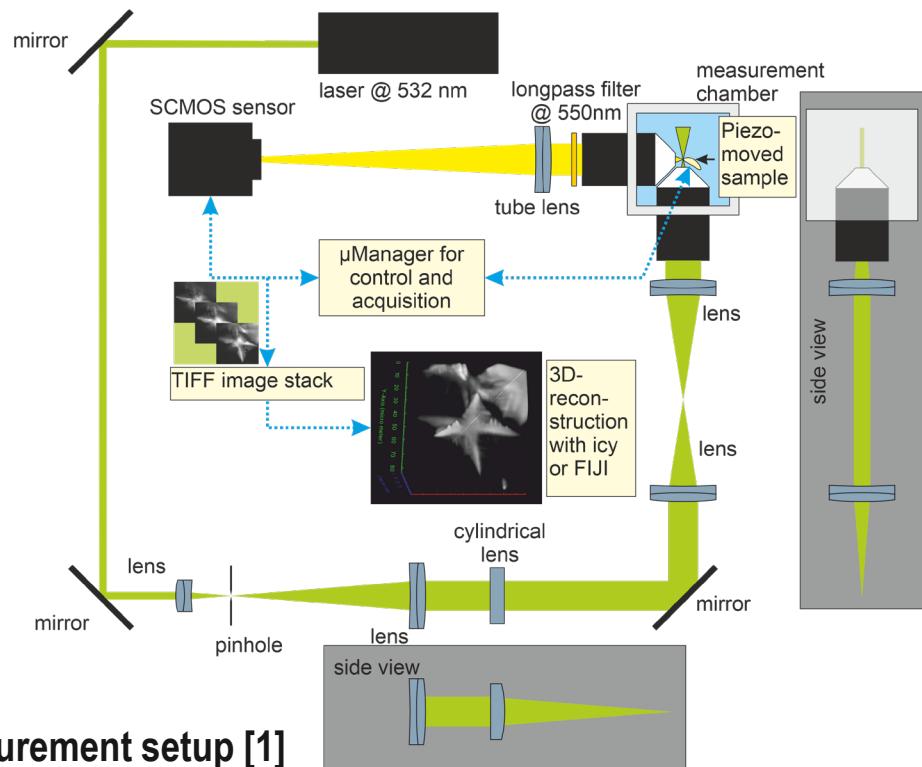
**60th Ilmenau Scientific Colloquium: Living Glass Surfaces XII,
07. September 2023**

- explore the crystall/glass interface by light-sheet fluorescence microscopy incl. fluorescence spectroscopy & fluorescence lifetime measurements
- sample: Ce:YAG glass ceramics as a model for material inhomogeneity
⇒ use the fluorescence of crystall and glass phases as structural probe
- work in progress:
determine boundaries of the method / push it to its limits
⇒ probable future application in material characterization
 - e.g. phase separated glasses
 - photo-active glasses

Light-sheet microscopy

Light-sheet fluorescence microscope (LSFM):

- excitation: laser @ 532 nm, 25 mW (405 nm, 50 mW)
- laser beam formed into a „laser sheet“ by a cylindrical lens
⇒ only a thin slide of the sample is irradiated
- fluorescence light from the sheet is filtered and detected by SCMOS-sensor
⇒ fluorescence picture or by spectrometer
⇒ emission spectrum



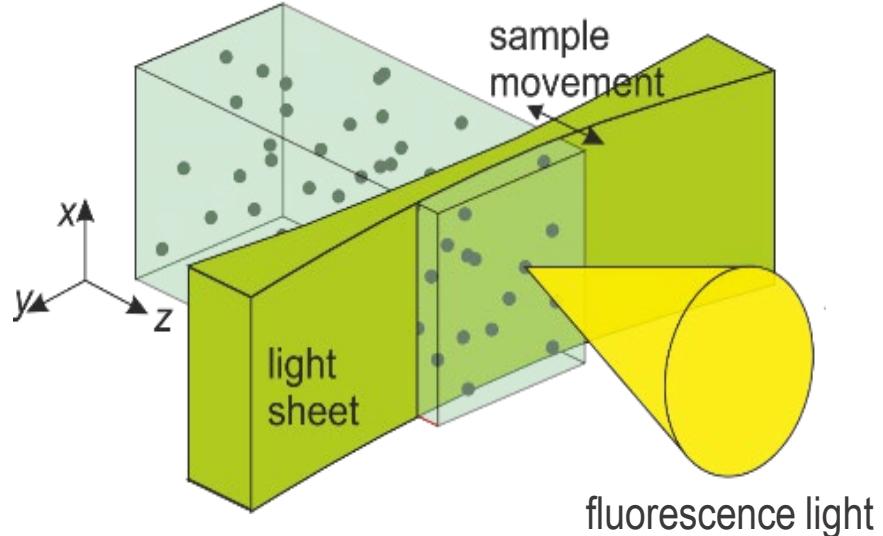
Light-sheet microscopy

Light-sheet fluorescence microscope

(LSFM):

- excitation: laser @ 532 nm
(405 nm)
- laser beam formed into a „laser sheet“
by a cylindrical lens
 - ⇒ only a thin slide of the sample is irradiated
- fluorescence light from the sheet is
filtered and detected by SCMOS-sensor
 - ⇒ fluorescence picture
 - or by spectrometer
 - ⇒ emission spectrum

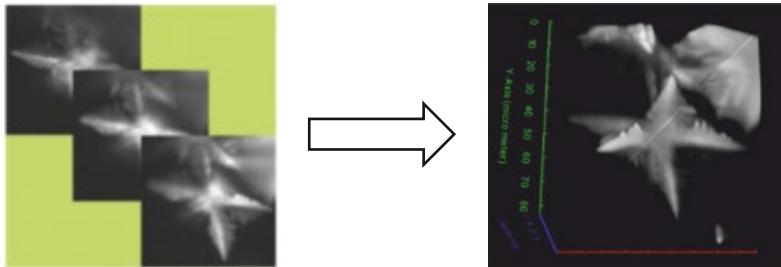
sample with
fluorescent crystallites



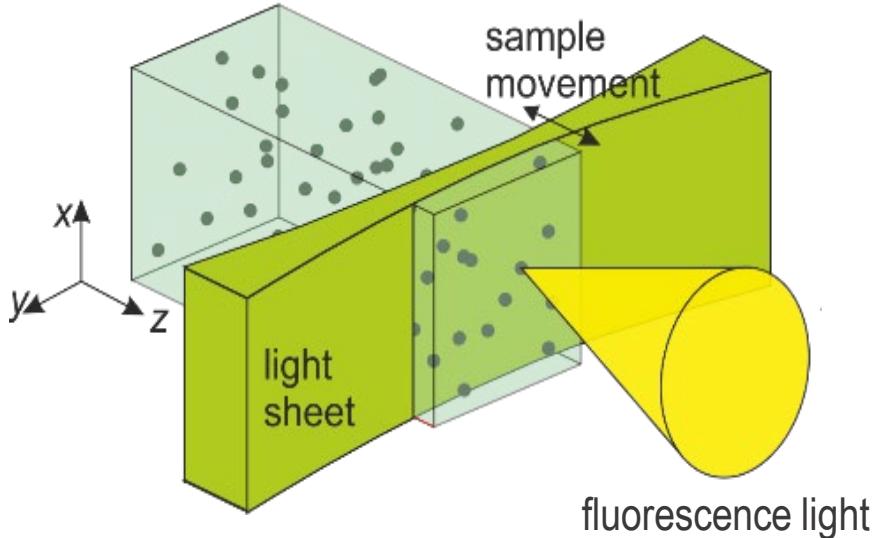
Light-sheet microscopy

Light-sheet fluorescence microscope (LSFM):

- fluorescence light from the sheet is filtered and detected by SCMOS-sensor (or spectrometer)
- moving the sample: different light sheets/ stacks of light sheets
⇒ 3D picture of the sample



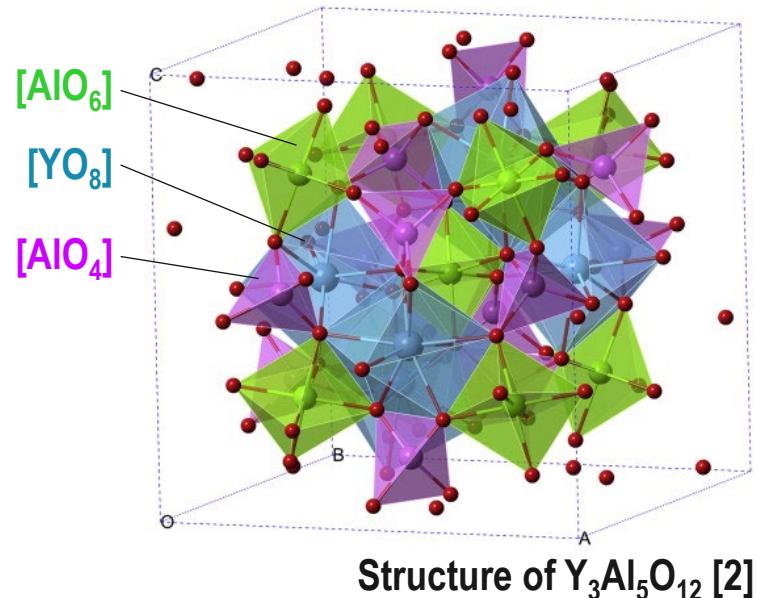
sample with
fluorescent crystallites



Ce:YAG glass ceramics

Yttrium Aluminum Garnet (YAG)

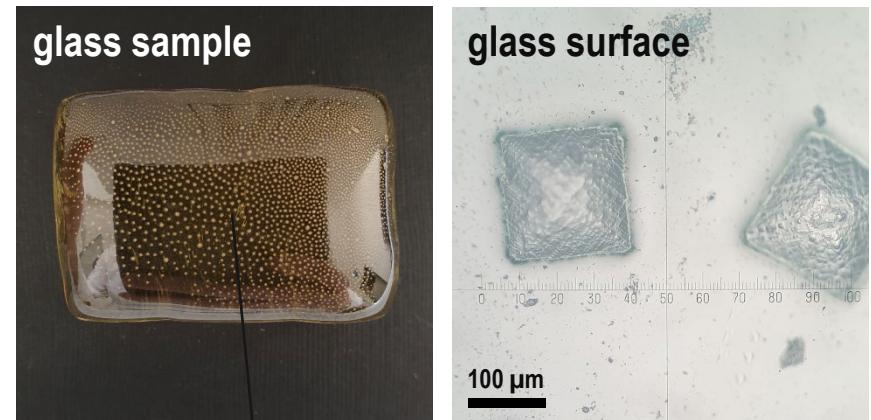
- $\text{Y}_3\text{Al}_5\text{O}_{12}$, cubic, colorless
- refractive index: 1.833
- melting temperature: 1950 °C
- chemically very stable
- very good solubility for rare earth ions
⇒ Ce^{3+} doping (yellow fluorescence)
- precipitation from a glass (previous project)
⇒ Ce:YAG glass ceramics [3]



Ce:YAG glass ceramics

Ce:YAG glass ceramics

- taken from different project
- glass: yttrium alumino silicate
doping: 1 mol% CeO₂
- YAG crystallization at only 1040 °C [3]
- some surface crystallization of YAG after casting
 - ⇒ cubic crystals (‘pyramids’ at surface)
crystallite size 100-200 µm
 - ⇒ dendritic growth in sample volume
 - ⇒ crystals are Ce³⁺ doped (fluorescence)

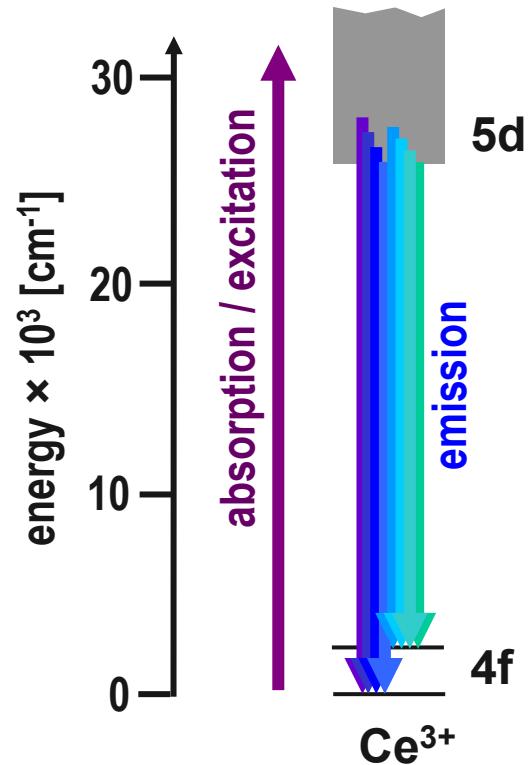


surface
crystallization

Ce³⁺ fluorescence

^{58}Ce : electron configuration [Xe] 6s² 4f²

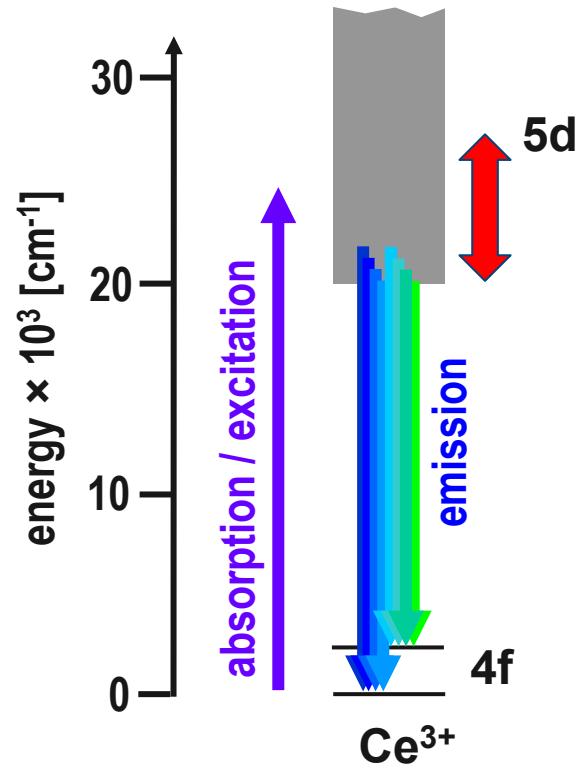
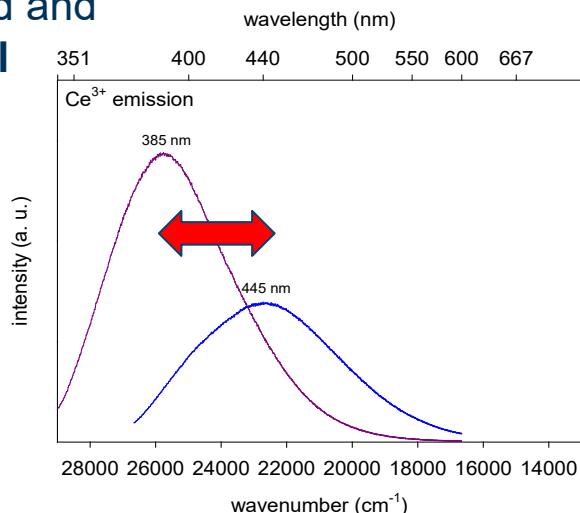
- 5d orbital: five broad overlapping energy levels
4f orbital: two narrow energy levels ('shielding')
- 5d-4f transitions very broad and
sensitive to host material
- excitation/emission in
most glasses: UV-blue



Ce³⁺ fluorescence

^{58}Ce : electron configuration [Xe] 6s² 4f²

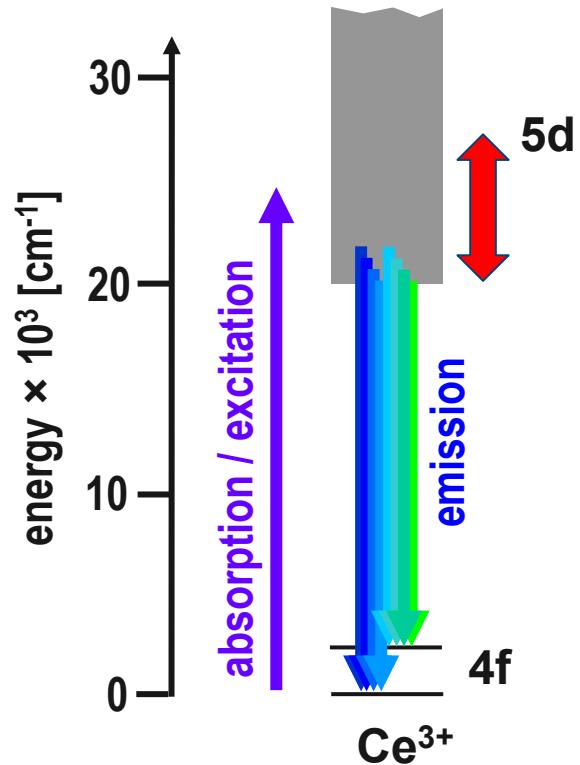
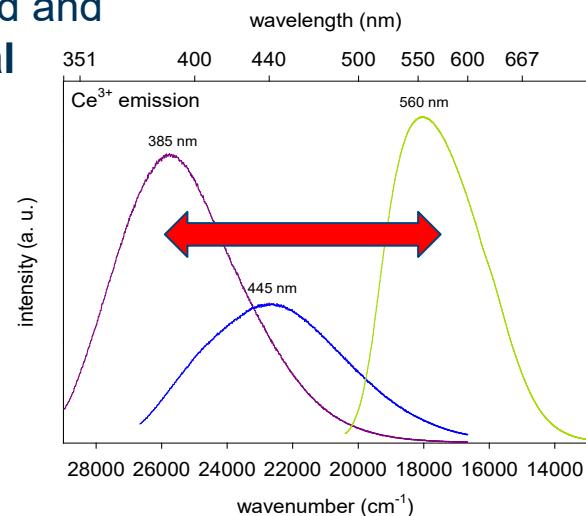
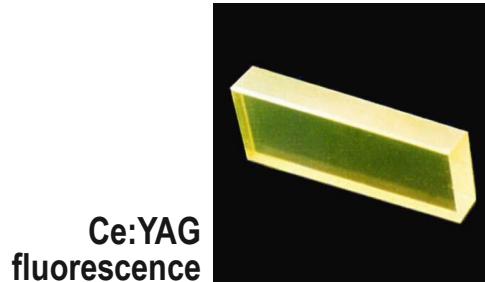
- 5d orbital: five broad overlapping energy levels
4f orbital: two narrow energy levels ('shielding')
- 5d-4f transitions very broad and
sensitive to host material
⇒ absorption and emission
shift with host material [4]



Ce³⁺ fluorescence

^{58}Ce : electron configuration [Xe] 6s² 4f²

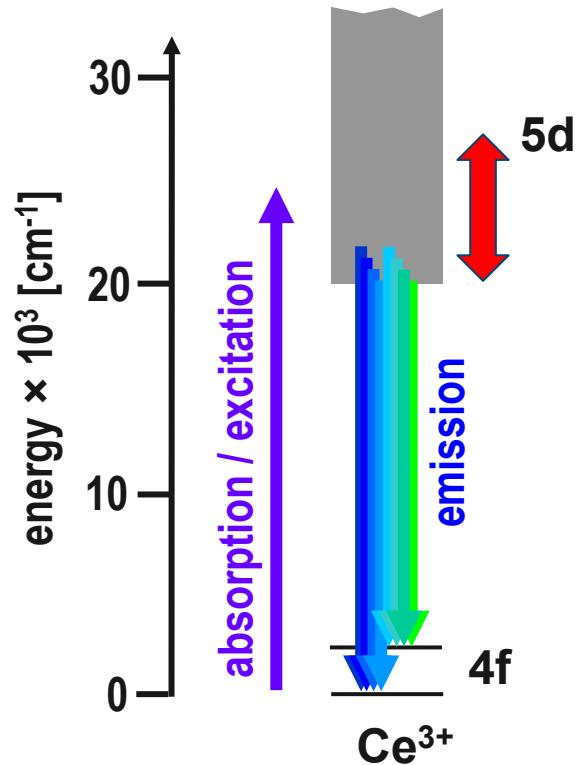
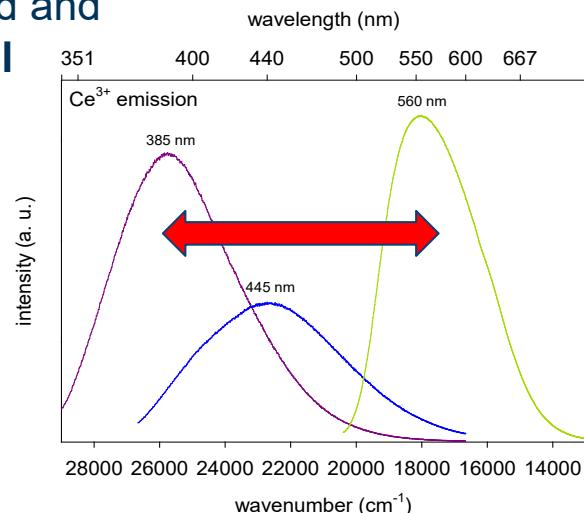
- 5d orbital: five broad overlapping energy levels
4f orbital: two narrow energy levels ('shielding')
- 5d-4f transitions very broad and
sensitive to host material
⇒ absorption and emission
shift with host material [4]



Ce³⁺ fluorescence

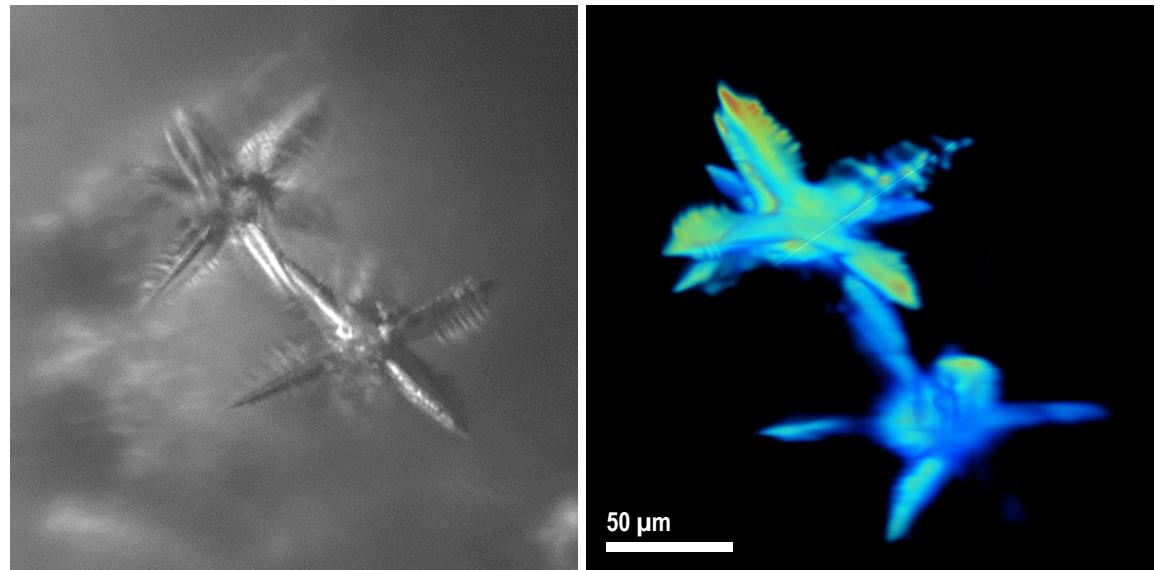
^{58}Ce : electron configuration [Xe] 6s² 4f²

- 5d orbital: five broad overlapping energy levels
4f orbital: two narrow energy levels ('shielding')
- 5d-4f transitions very broad and
sensitive to host material
 - ⇒ absorption and emission shift with host material [4]
 - ⇒ **changes in the emission spectra at the crystal/glass interface?**



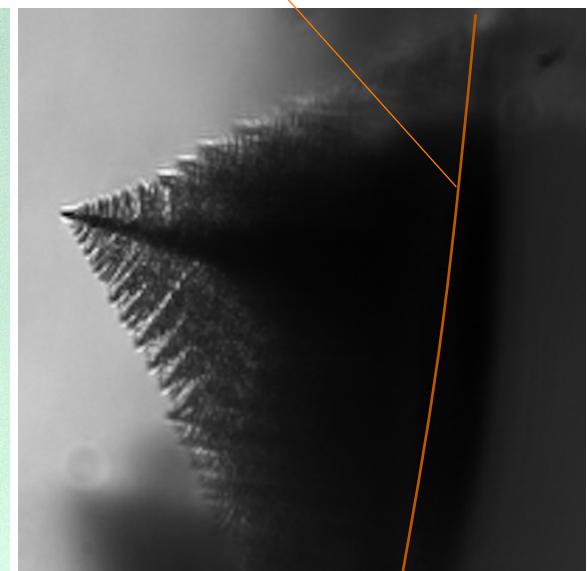
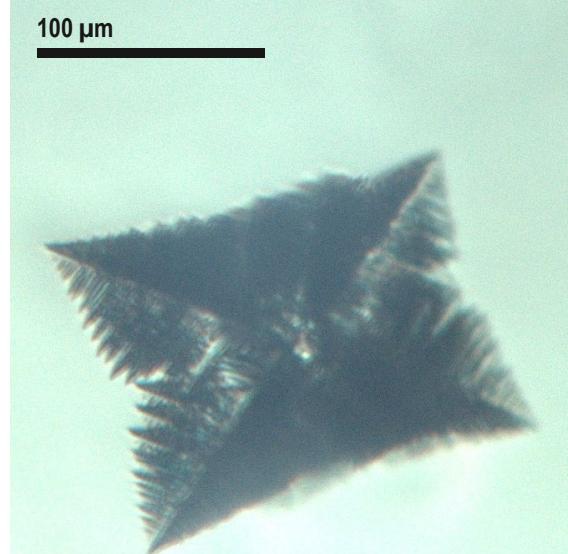
LSFM microscopic/fluorescence pictures

- preliminary results [5]: samples with volume crystallization
⇒ dendritic Ce:YAG crystals size \sim 100-200 μm
- excitation 532 nm: fluorescence well detectable
⇒ 3D pictures



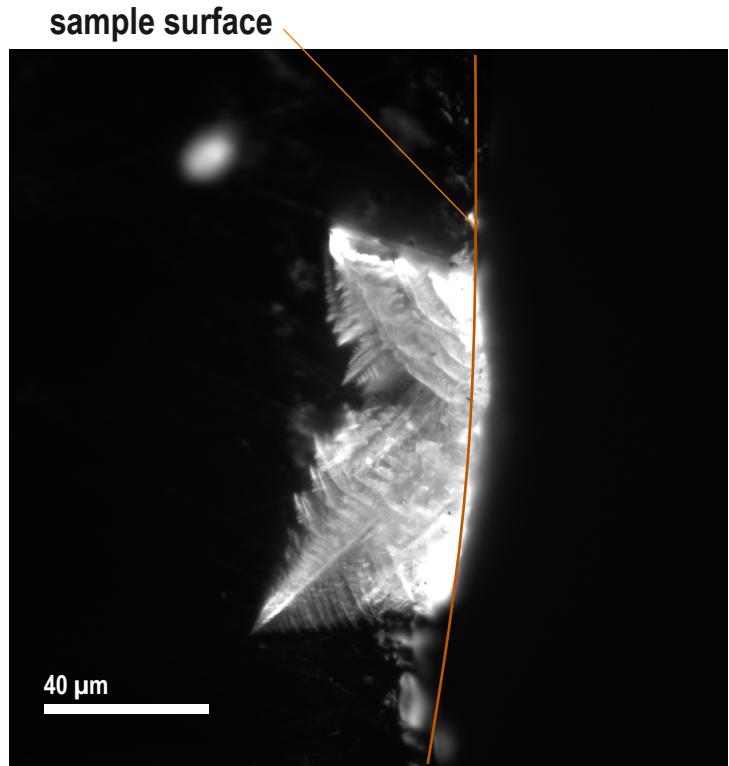
LSFM microscopic pictures

- new samples:
mostly surface crystallization
+ some volume crystallization
- ⇒ dendritic Ce:YAG crystals
size ~100-200 µm
- ⇒ single dendrites visible



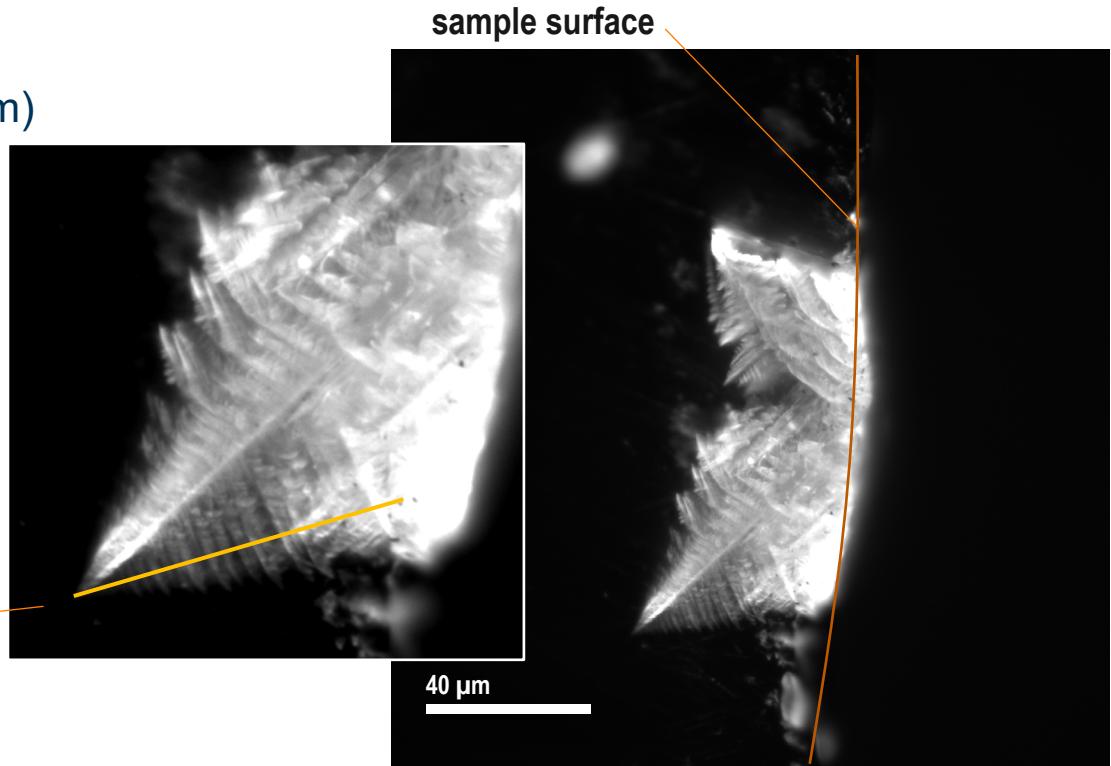
LSFM: spatial resolution
(luminescence picture, Ex.: 532 nm)

- new samples:
mostly surface crystallization
+ some volume crystallization
- ⇒ dendritic Ce:YAG crystals
size ~100-200 µm
- ⇒ single dendrites visible



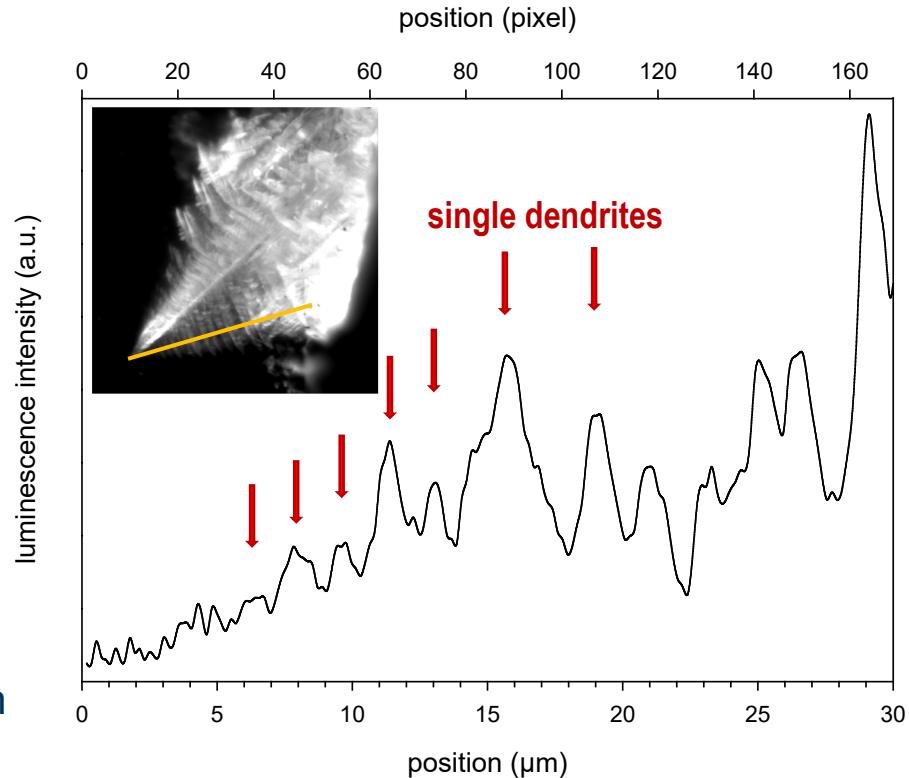
LSFM: spatial resolution
(luminescence picture, Ex.: 532 nm)

- new samples:
mostly surface crystallization
+ some volume crystallization
- ⇒ dendritic Ce:YAG crystals
size \sim 100-200 μm
- ⇒ single dendrites visible
- ⇒ intensity profile



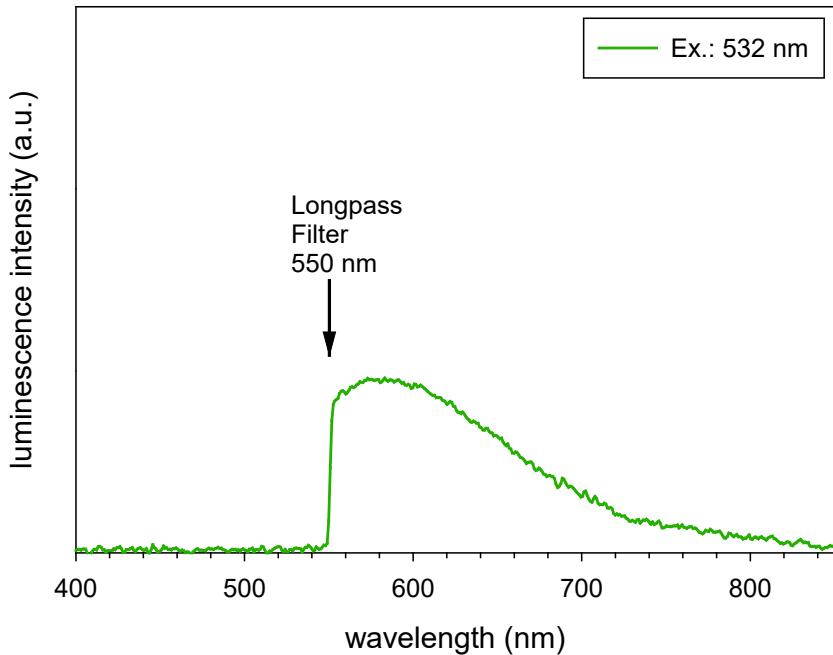
LSFM: spatial resolution
(luminescence picture, Ex.: 532 nm)

- new samples:
mostly surface crystallization
+ some volume crystallization
- ⇒ dendritic Ce:YAG crystals
size \sim 100-200 μm
- ⇒ single dendrites visible
- ⇒ intensity profile
- ⇒ distance between dendrites \sim 1.5-2.0 μm



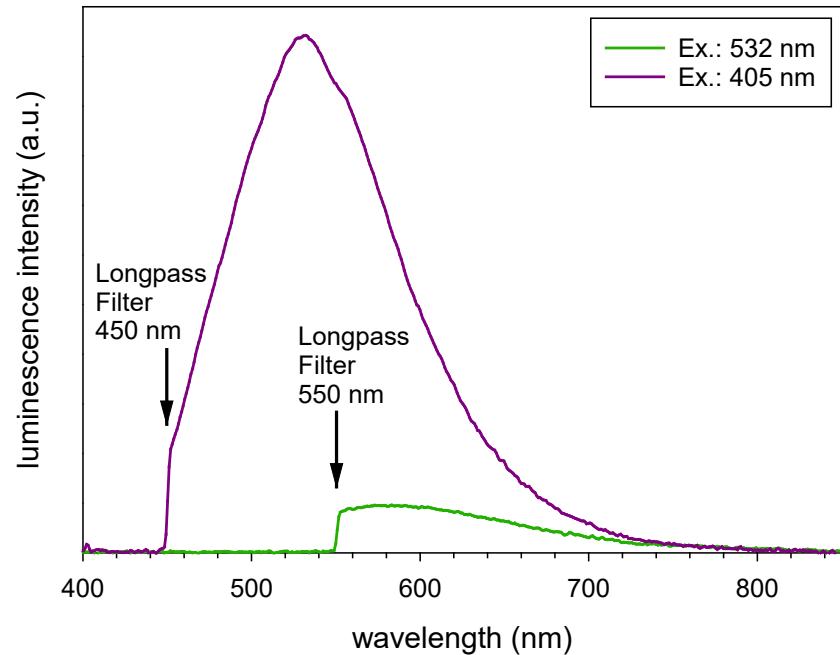
LSFM emission spectra

- 532 nm laser:
low fluorescence intensity
550 nm filter cuts peak maximum
⇒ not suitable to detect peak shift



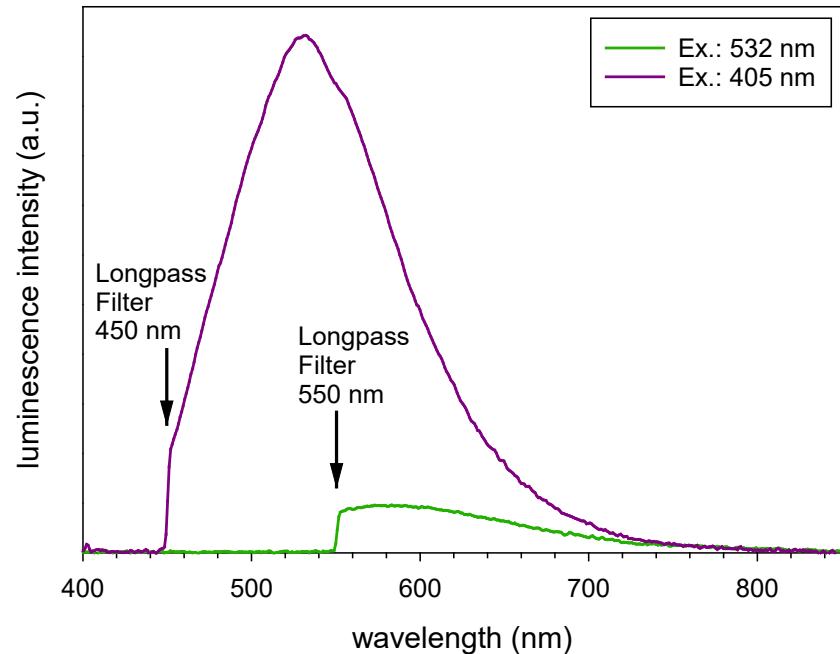
LSFM emission spectra

- 532 nm laser:
low fluorescence intensity
550 nm filter cuts peak maximum
⇒ not suitable to detect peak shift
- 405 nm laser:
much higher fluorescence intensity
good S/N ratio



LSFM emission spectra

- 532 nm laser:
low fluorescence intensity
550 nm filter cuts peak maximum
 \Rightarrow not suitable to detect peak shift
- 405 nm laser:
much higher fluorescence intensity
good S/N ratio
- measurements at different crystal positions
 \Rightarrow but: peak position constant
 \Rightarrow no peak shift at crystal/glass interface
detectable



- LSFM can be used to create 3D pictures of the precipitated crystals
- spatial resolution $\sim 1\text{-}2 \mu\text{m}$
- fluorescence spectra could be measured with 532 and 405 nm excitation
(532 nm not suitable, latter has much better S/N ratio)
- up to now no shift of the emission spectra detected at the interface crystal/glass

Outlook:

- improvement of the spatial resolution (better fitting optics for 405 nm)
- scanning the samples at higher accuracy
- fluorescence lifetime measurements at different spots in the glass ceramics
- UV laser (fluorescence of glass and YAG phases detectable)

References

- [1] P. Pitrone, J. Schindelin, L. Stuyvenberg et al.: OpenSPIM: an open-access light-sheet microscopy platform. *Nature Methods* 10 (2013) p. 598-599.
- [2] Z. Yanchun, X. Huimin, F. Zhihai: Theoretical Investigation on Mechanical and Thermal Properties of a Promising Thermal Barrier Material: $\text{Yb}_3\text{Al}_5\text{O}_{12}$, *Journal of Materials Science & Technology* 30 (2014) p. 631.
- [3] X. Xia: Optimization of a glass composition for synthesis of Ce^{3+} :YAG crystallites: MA thesis, TU Ilmenau, 2021.
- [4] A. Herrmann, H. A. Othman, A. A. Assadi, M. Tiegel, S. Kuhn, C. Rüssel: Spectroscopic properties of cerium-doped aluminosilicate glasses, *Optical Materials Express* 5 (2015) p. 720.
- [5] M. Hofmann, A. Herrmann, U. Brokmann: Light-sheet fluorescence microscopic probing of silicate materials, *tm - Technisches Messen* 89 (2022) p. 447-454.

**Thank you for your attention!
Vielen Dank für Ihre Aufmerksamkeit!**

