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# New developments of MCVD technology for the preparation of efficient laser fibers

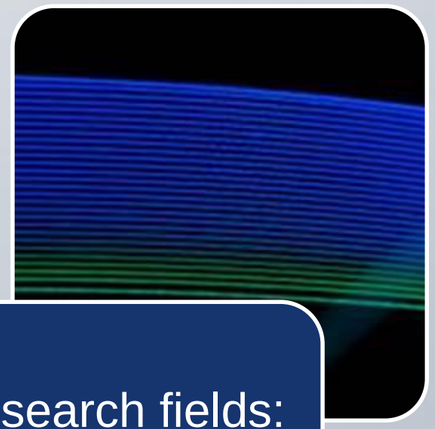
Florian Lindner
Fiber optics, optical fiber technology

**F. Lindner**, C. Aichele, J. Dellith, A. Dellith, A. Schwuchow, M. Leich, A. Scheffel, F. Froehlich, S. Unger, K. Schuster, A. Kriltz, H. Bartelt

# New developments of MCVD technology for the preparation of efficient laser fibers

Increasing fields of applications for modern optical fibers

Challenges to the material properties and the processing technology of fiber optics



Research fields: fiber amplifiers, fiber light sources, fiber sensor elements

Requires new and adapted properties of optical fibers

New glass materials for optical fibers - new material processing technologies

Understanding and improvement of state of the art materials and technologies

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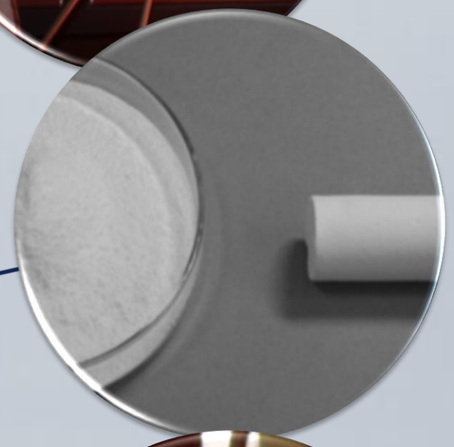
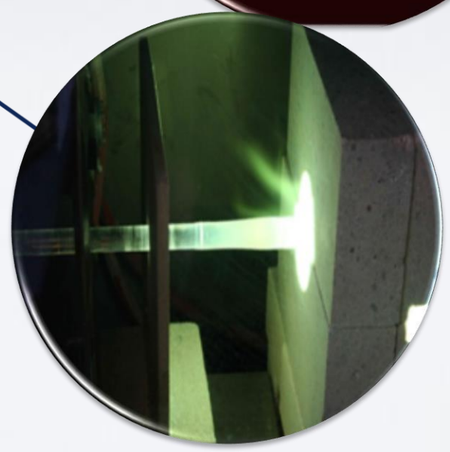
# Principle ways for preform fabrication

Glass melting



Reactive powder sinter process (REPUSIL)

Gas phase processes (oxidation: MCVD, PCVD)



Gas phase processes (hydrolysis: OVD, VAD)

# Preform fabrication – comparison of the different processes

| | Material quantity (active doped) | Purity / Fiber loss (active doped) | Homogeneity / Refractive index distribution | Doping elements |
|---|---|---|---|---|
| **MCVD solution doping** | ☹ $\varnothing_{Core}$ 1.8 mm | ☺ 5 dB/km | ☹ Layer structures, dip | 😐 RE, Al, P, F, B, Ce |
| **MCVD gas phase doping of RE and Al** | 😐 $\varnothing_{Core}$ 8 mm | 😐 15 dB/km | ☺ Excellent homogeneity | 😐 RE (Yb, Tm), Al, (Ce) |
| **REPUSIL Powder sintering technique** | ☺ $\varnothing_{Core}$ 15 mm | 😐 15 dB/km | 😐 Short-periodic index fluctuations | 😐 RE, Al, F, B, Ce |
| **Melt glasses (SAL)** | ☺ $\varnothing_{Core}$ >15 mm | ☹ 500 dB/km | ☹ Striae, RI fluctuations | ☺ RE, Al, P, F, B, Ce |

K. Schuster, S. Unger, C. Aichele, F. Lindner, S. Grimm, D. Litzkendorf, J. Kobelke, J. Bierlich, K. Wondraczek and H. Bartelt, “Material and technology trends in fiber optics”, Adv. Opt. Techn. 3 (4), 447–468 (2014)

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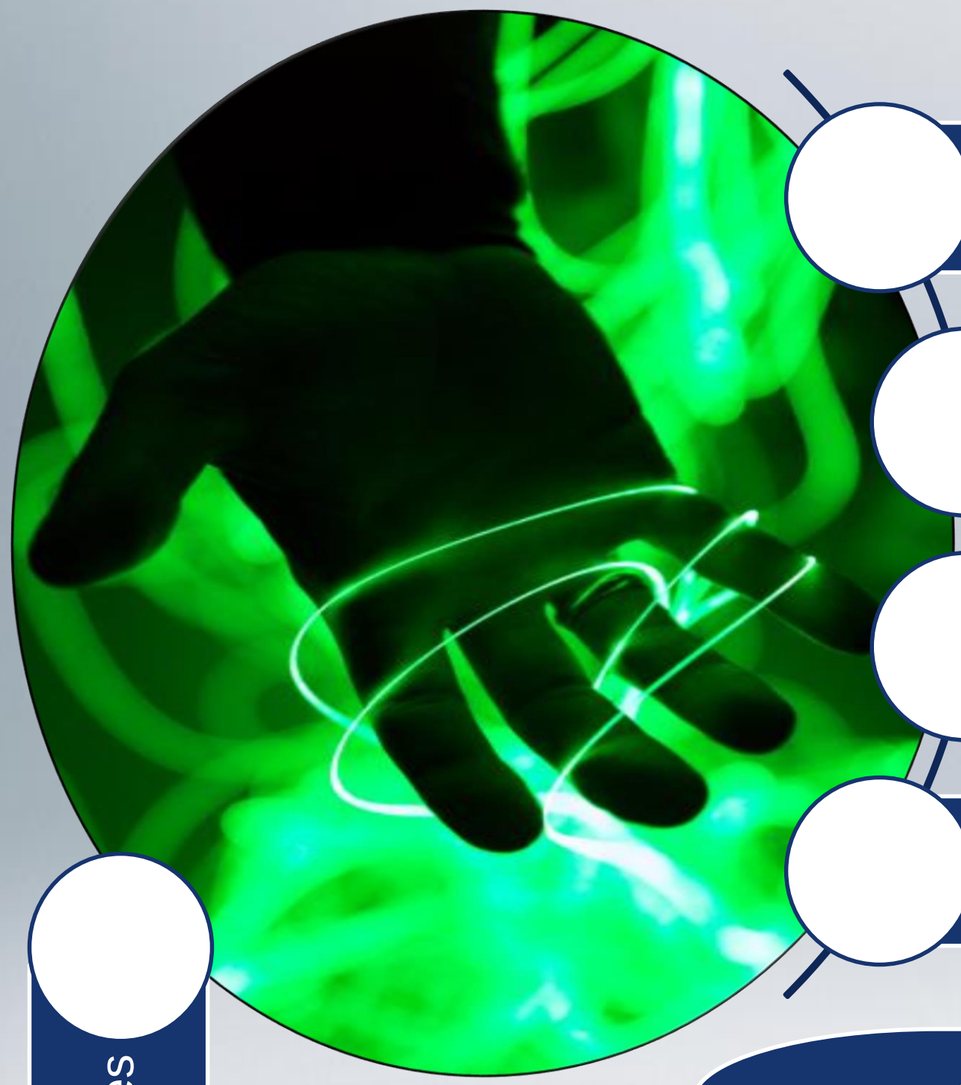
# Modified Chemical Vapor Deposition (MCVD) in combination with the solution doping

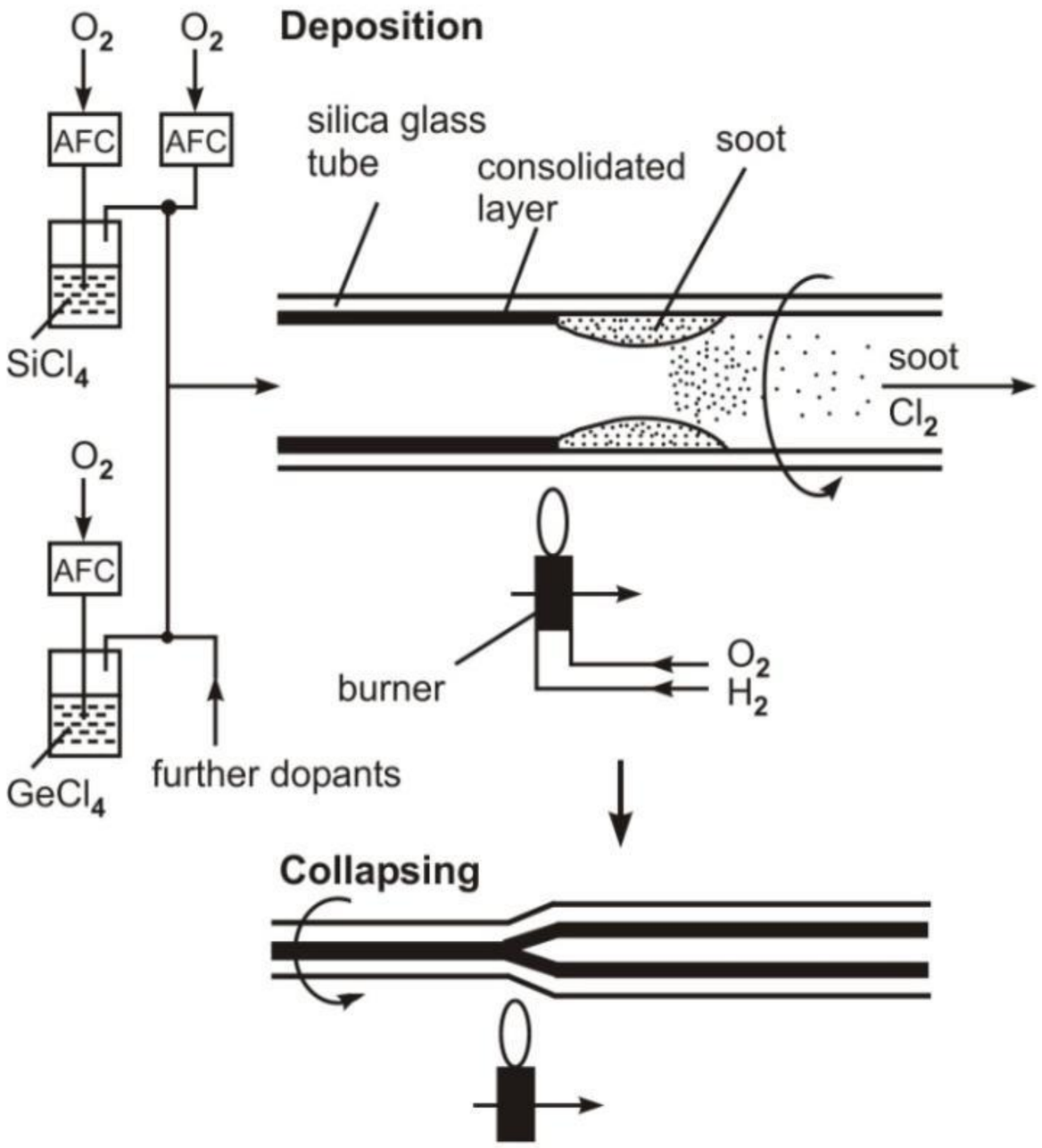


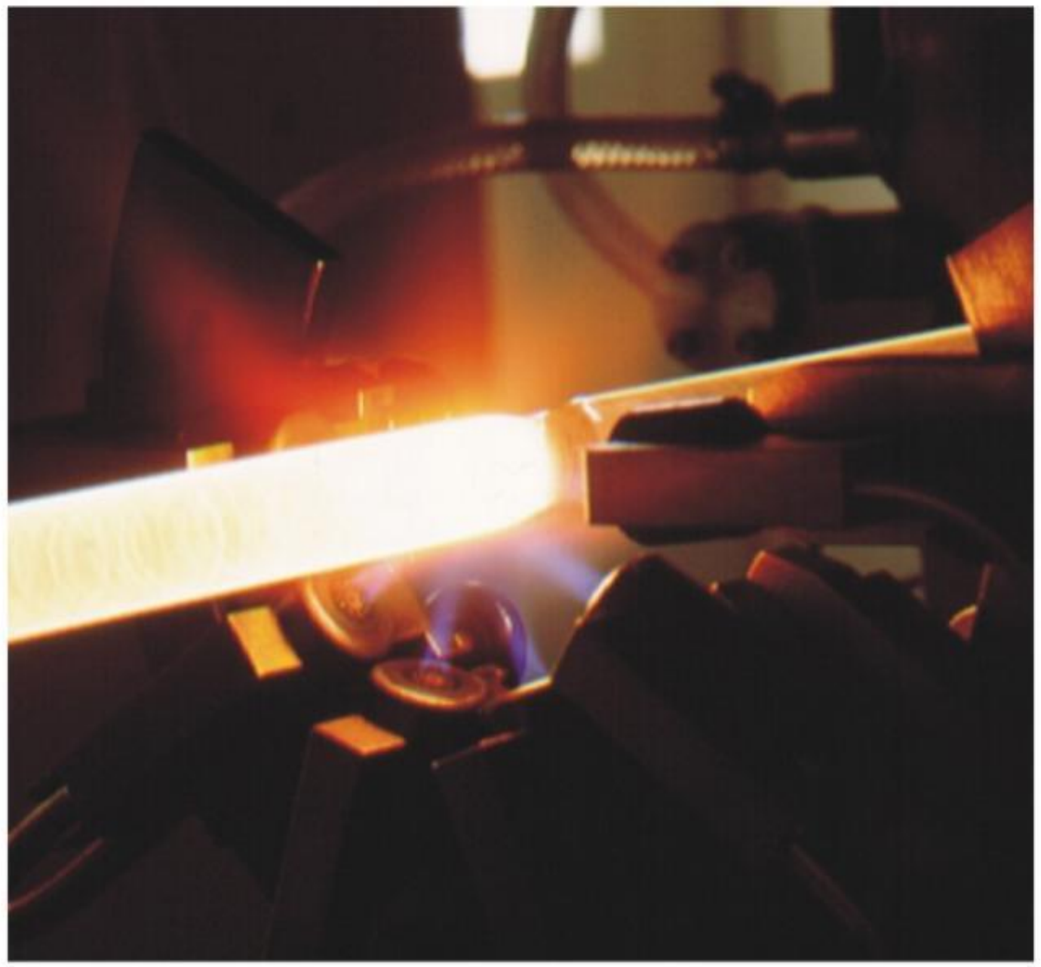
- Leading technology in manufacture rare earth (RE)-doped high power silica laser fibers
- Most important dopants here RE: ytterbium (Yb) and co-dopants: phosphorus (P) and aluminum (Al)
- (P : Al) = (1 : 1)
  properties: low NA, low photodarkening losses
- Limitations concerning the geometry, doping and refractive index homogeneity

**Challenges**

- Optimize P incorporation into the silica network
- Understanding of the processes on the atomic level
- Overcome limitation of conventional MCVD

# Preform fabrication - modified chemical vapor deposition (MCVD)



Silica glass tube with the burner

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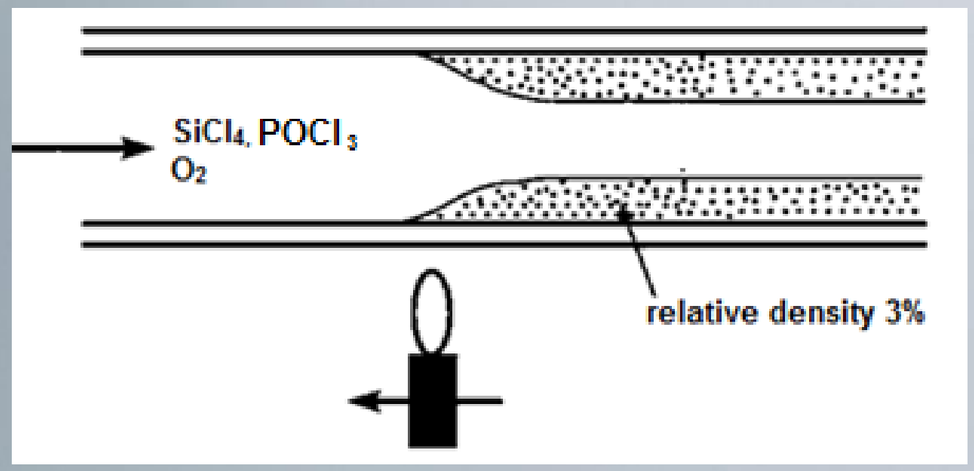
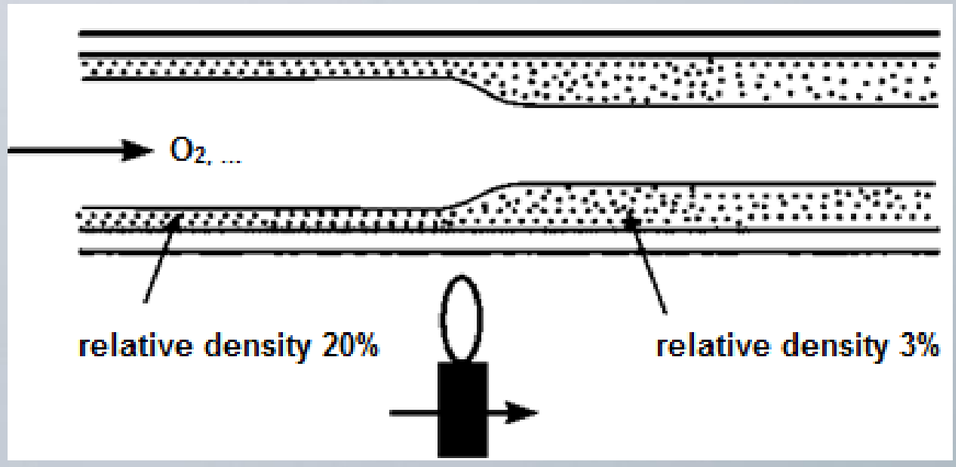
# Preform fabrication - MCVD in combination with solution doping

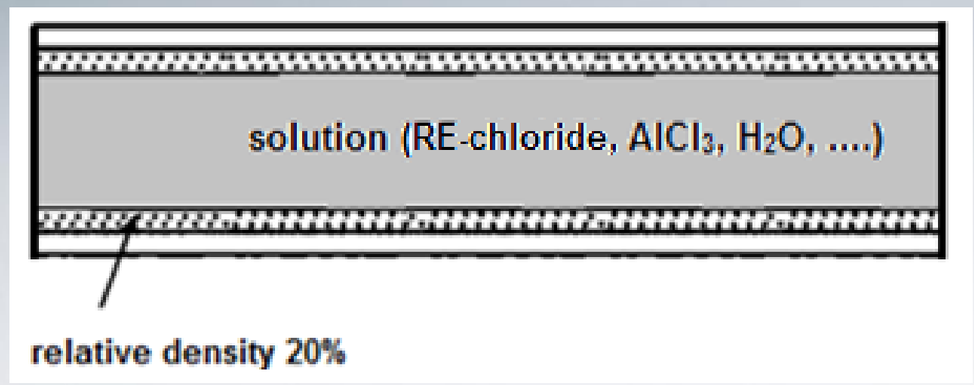
1. Step: reverse deposition of $P_2O_5/SiO_2$ soot layers



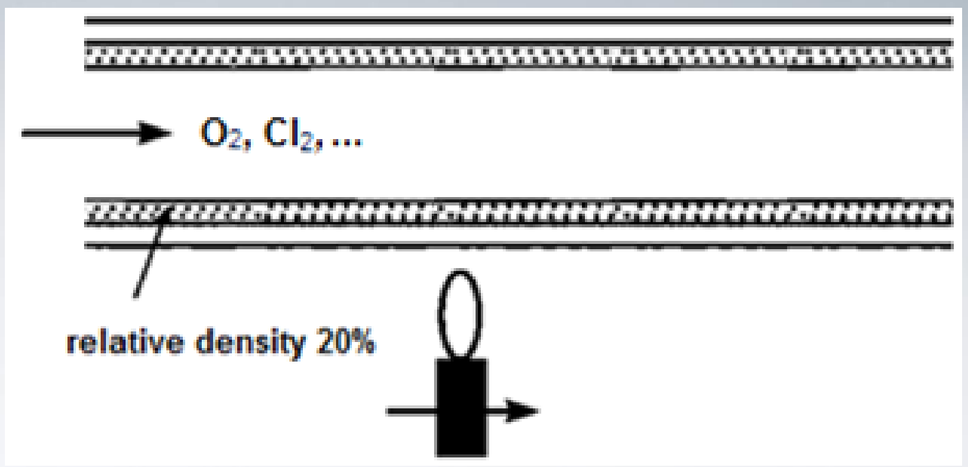

2. Step: pre-sintering to a relative density $d_r \sim 0.2$




3. Step: penetration with solution




4. Step: drying and chlorination

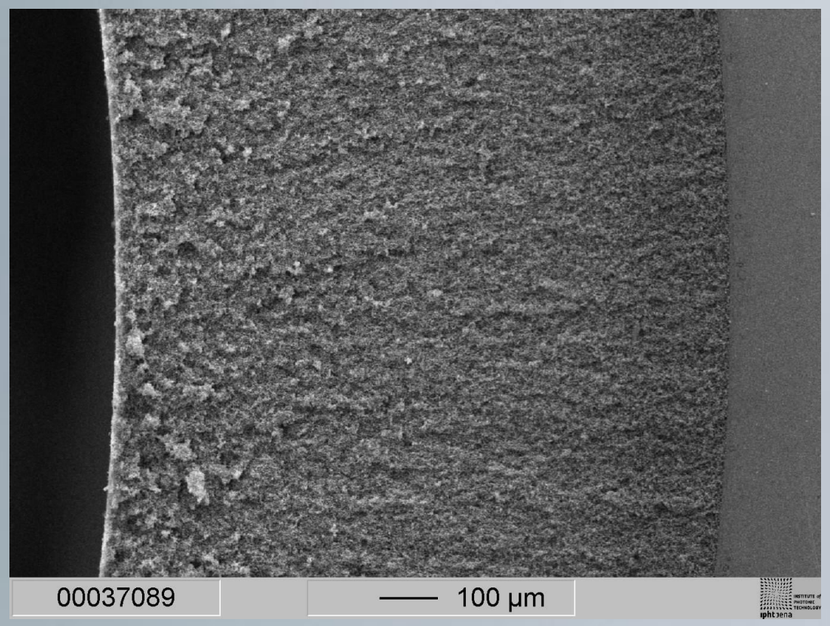


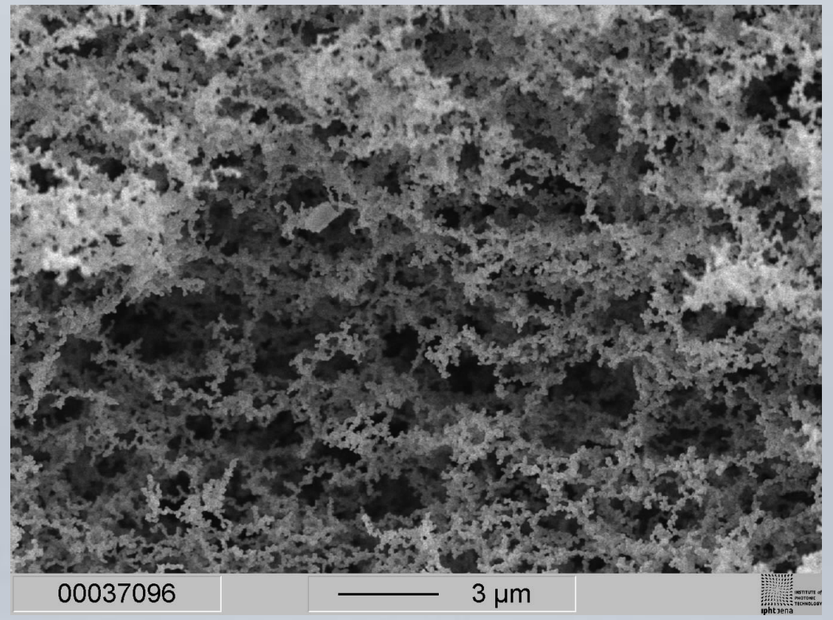

5. Step: vitrification

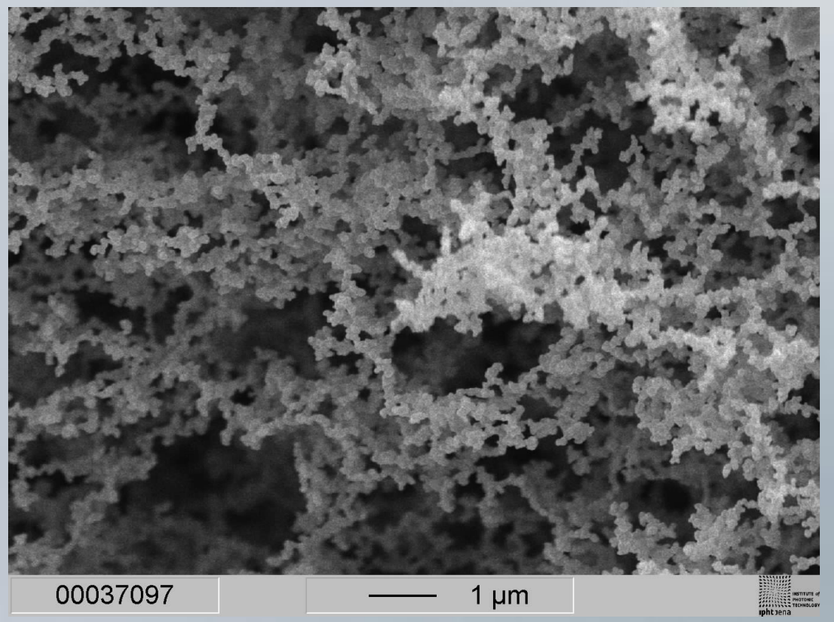
# Analysis of phosphorus doped soot material

## SEM micrograph of the phosphorus doped silica layer

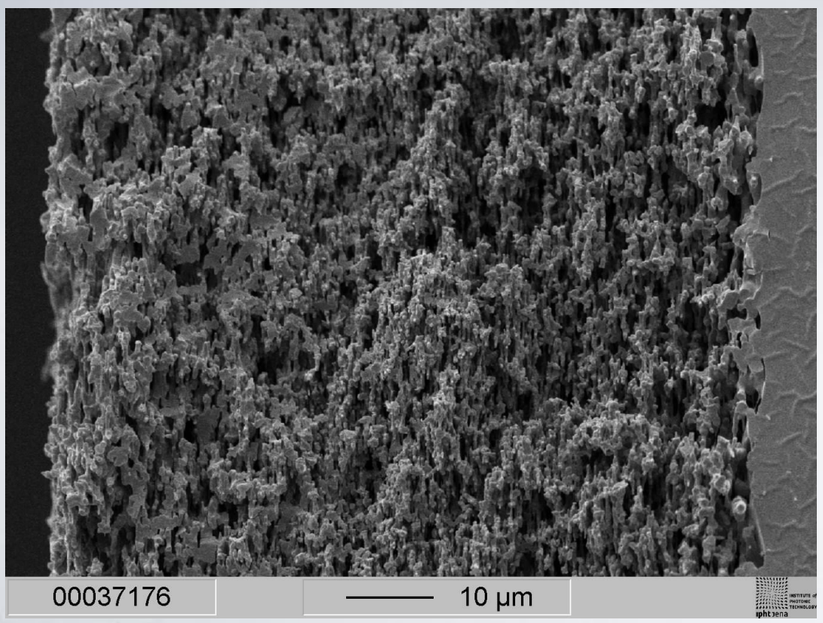
untreated ($x_{POCl3}/x_{SiCl4}$ = 0.3 ; relative density 0.03) *(1.Step)*

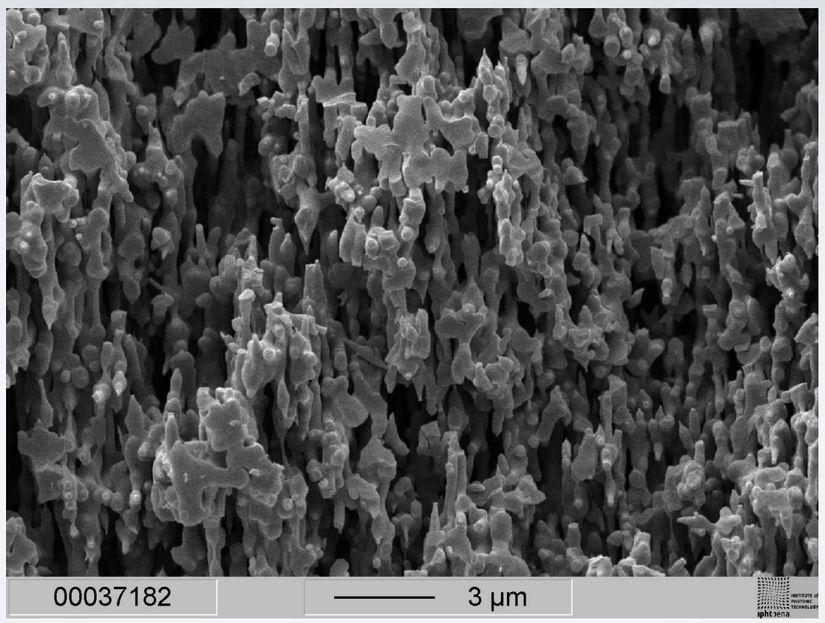


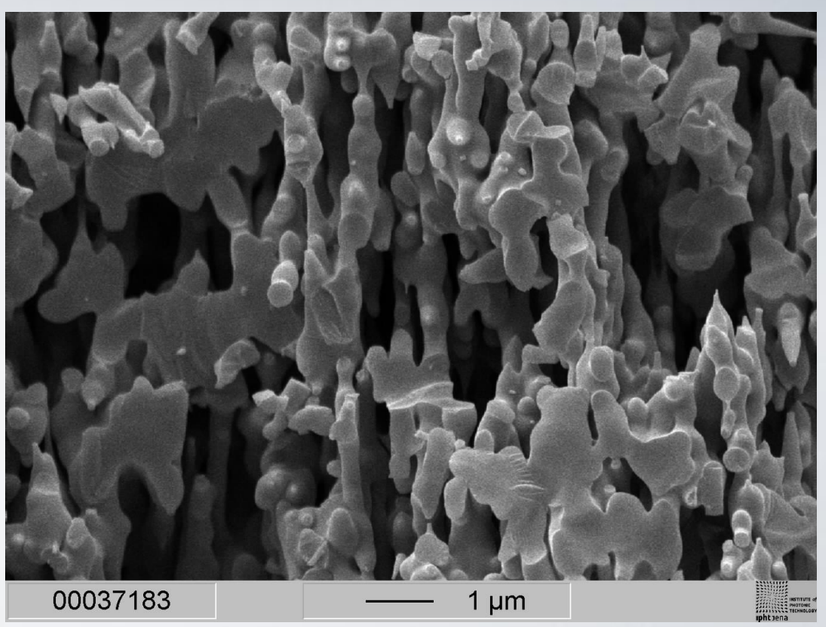




pre-sintered ($x_{POCl3}/x_{SiCl4}$ = 0.3 ; relative density 0.3) *(2.Step)*



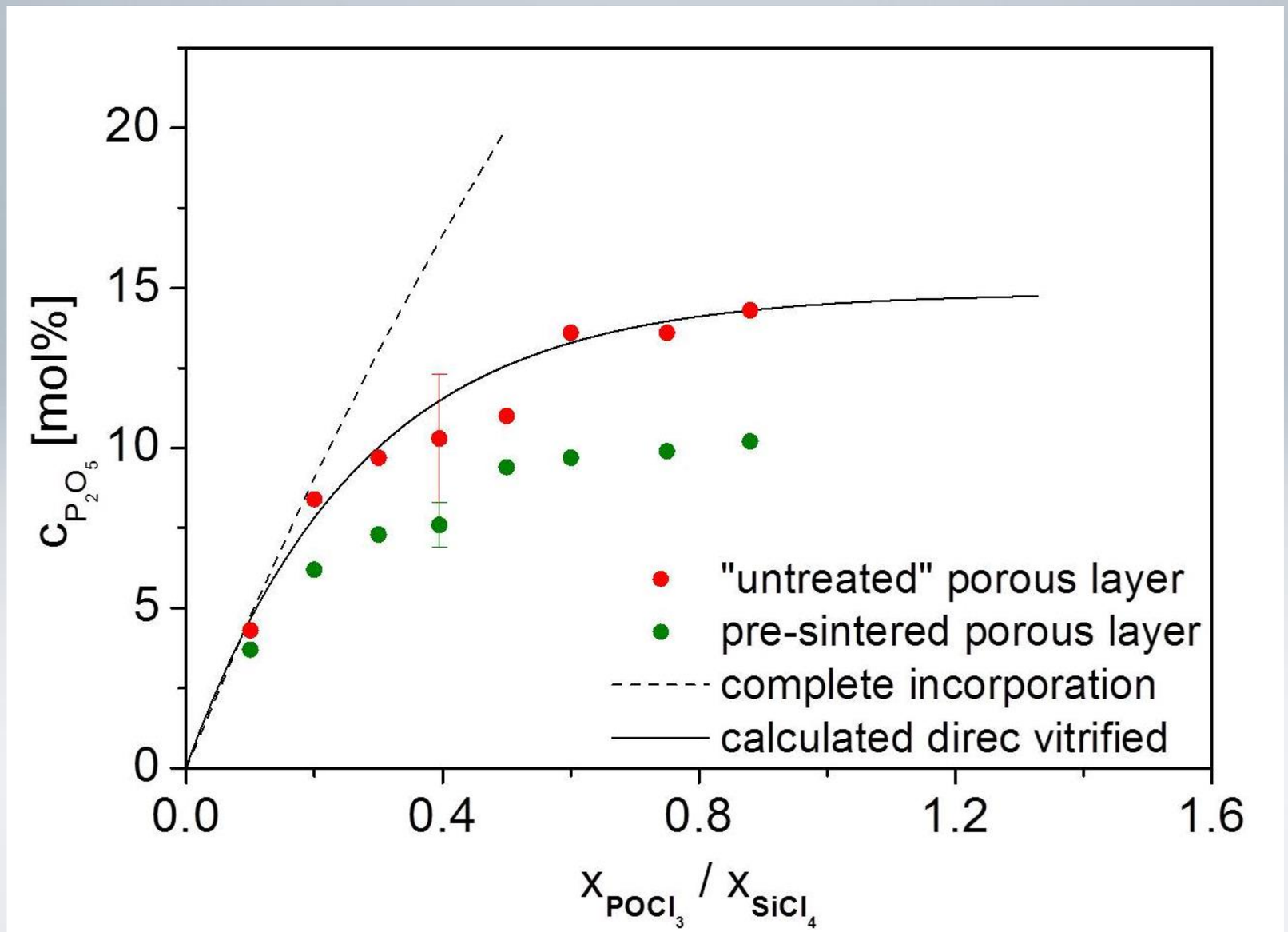



# Analysis of phosphorus doped soot material

## EDX-measurement

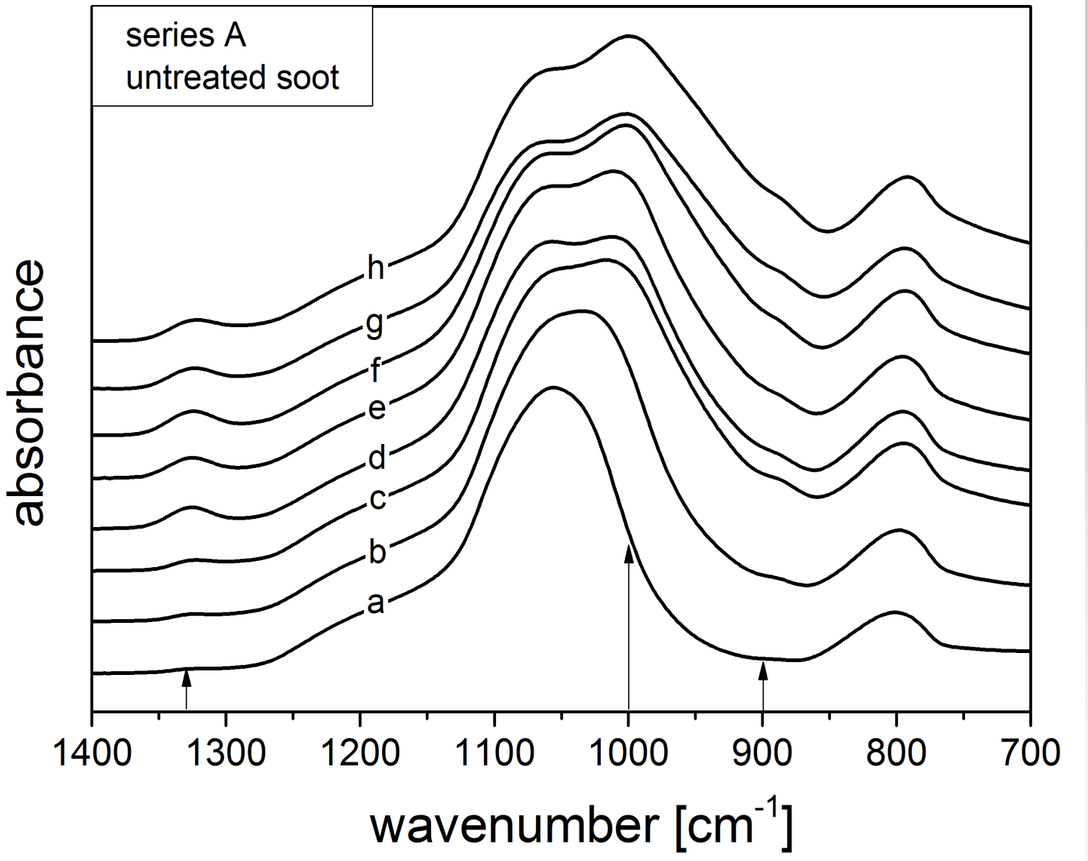
- Untreated soot layer and direct vitrify layer nearly same P-content
- Decreasing of P-content by pre-sintered (further P-evaporation)
- High measurement error in case of untreated soot (hygroscopicity)

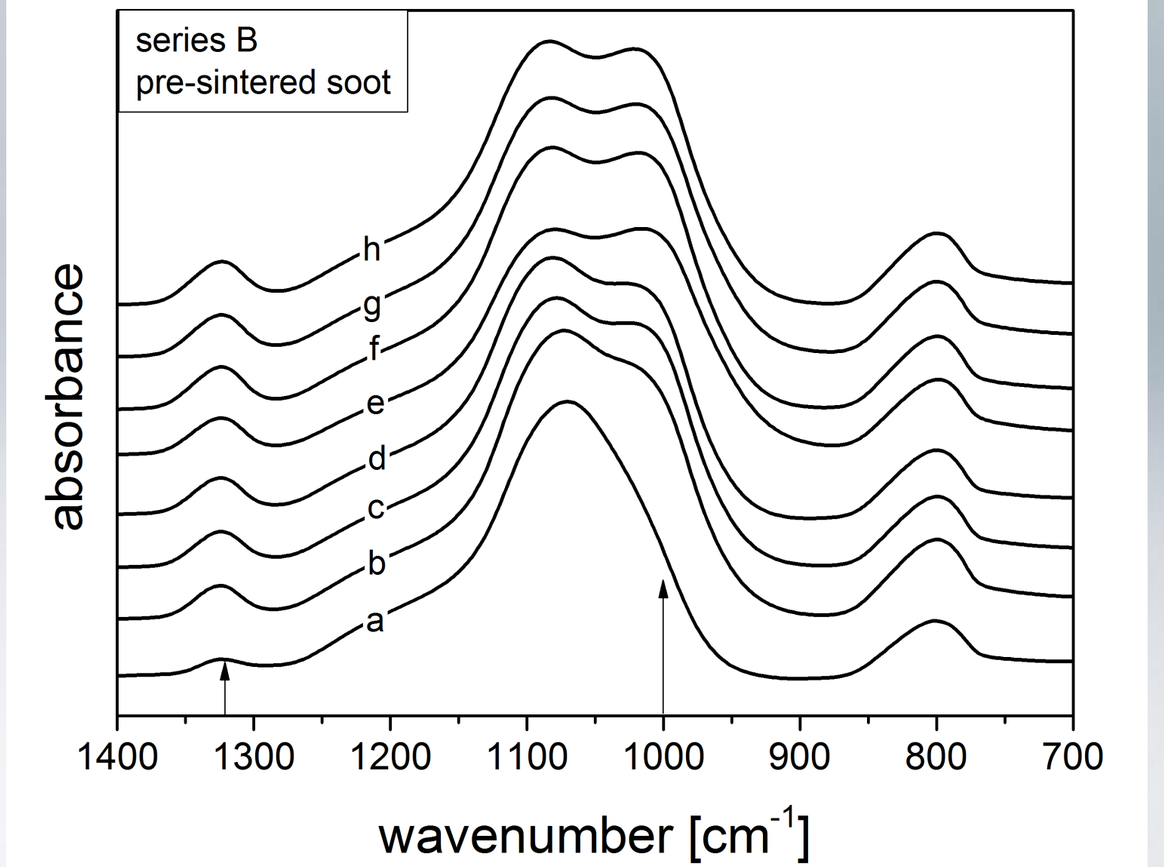


# Analysis of phosphorus doped soot material

## FTIR (ATR)-measurement of the soot material series

- Differences in the spectra of untreated and pre-sintered soot visible
- 1325 $cm^{-1}$ dedicated to P=O-band and 800 $cm^{-1}$ to Si-O-band
- High P-content → band at 1000 $cm^{-1}$ and 1325 $cm^{-1}$ increase
- Weak band at 900 $cm^{-1}$ only visible in untreated spectra

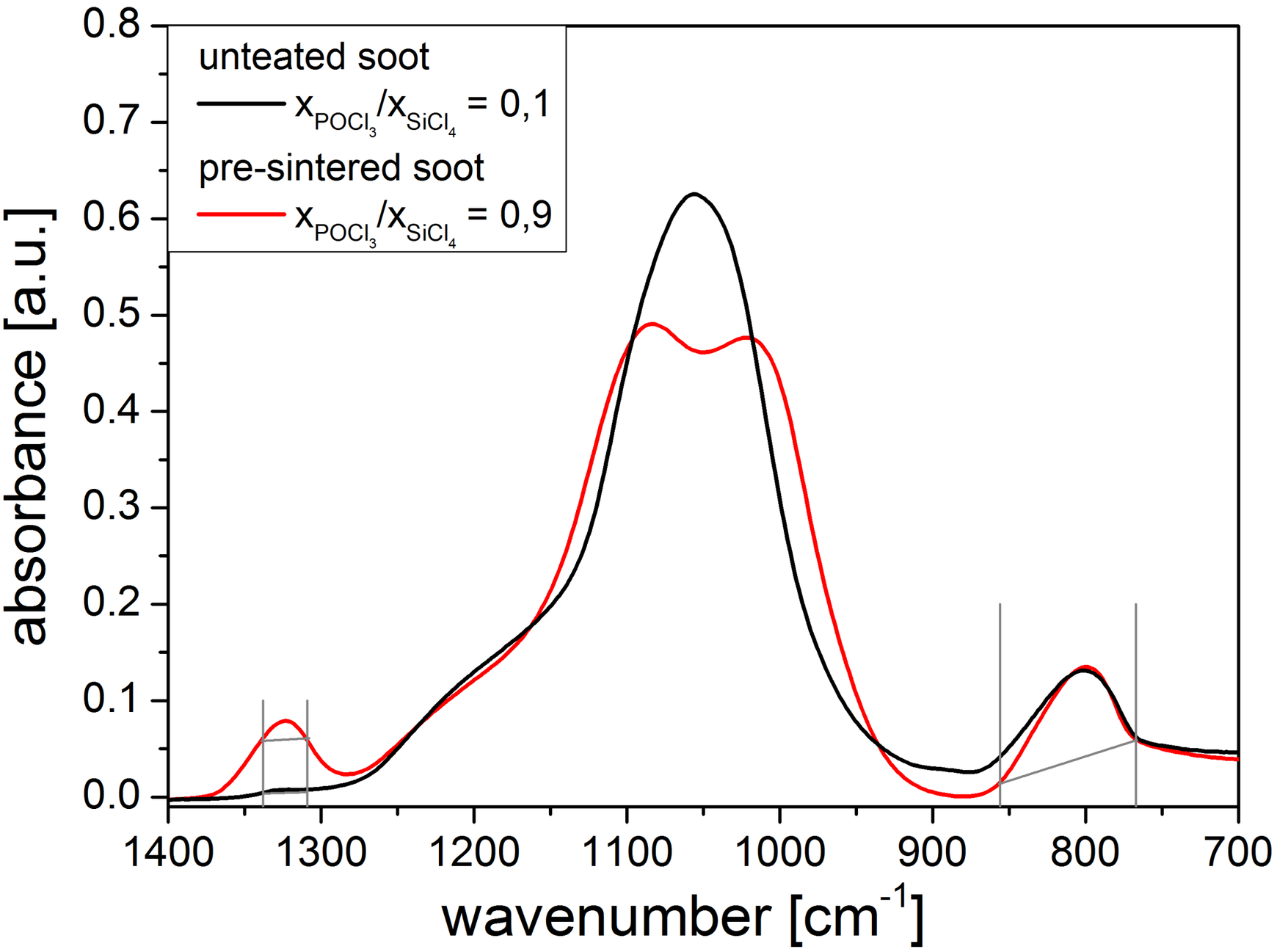



**F. Lindner, S. Unger, A. Kriltz, A. Scheffel, A. Dellith, J. Dellith, and H.Bartelt, "Phosphorus incorporation into silica during modified chemical vapour deposition combined with solution doping," Phys. Chem. Glasses: Eur. J. Glass Sci. Technol. B, vol. 56 (6), no. 6, pp. 278–284 (2015).**

# Analysis of phosphorus doped soot material

## Comparison of untreated and pre-sintered IR spectra




- Absorbance influenced by powder consistence
- Band analysis difficult because of the overlay of Si and P species
- Band area ratio of Si-O-Si band to P=O band for analysis of the IR spectra

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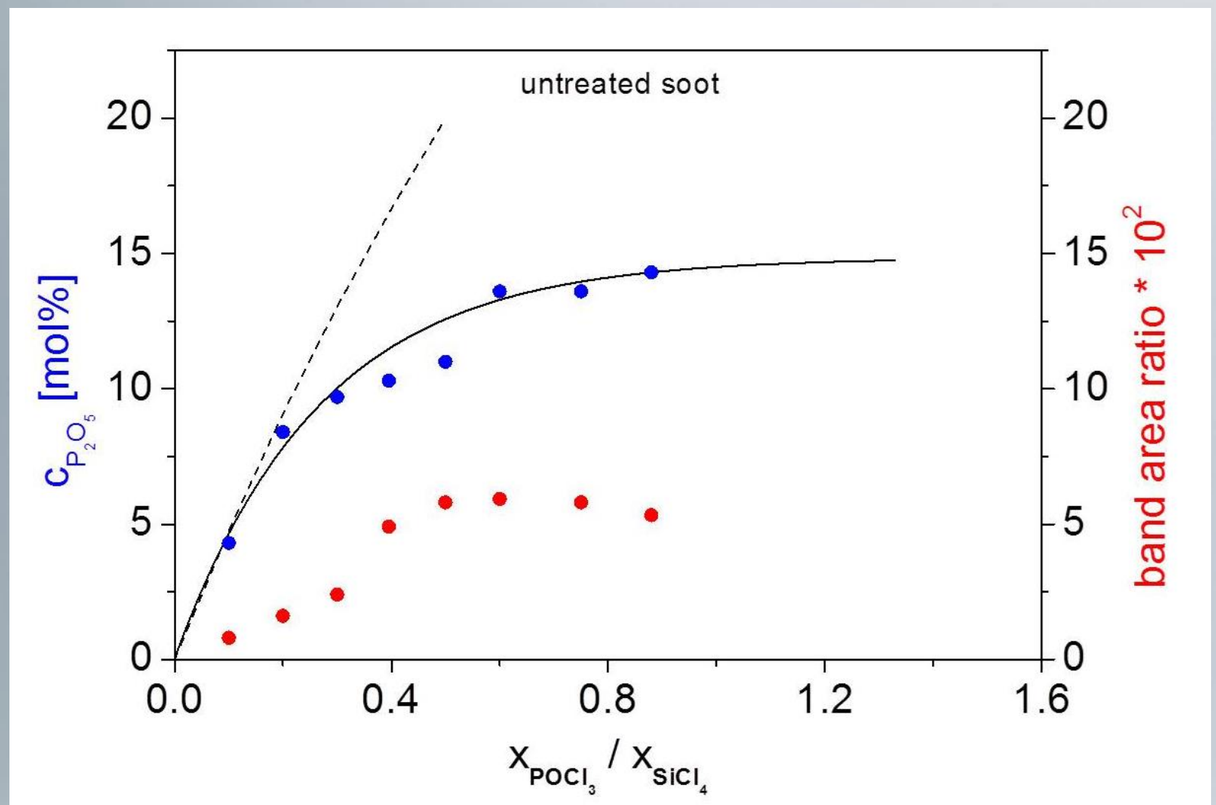
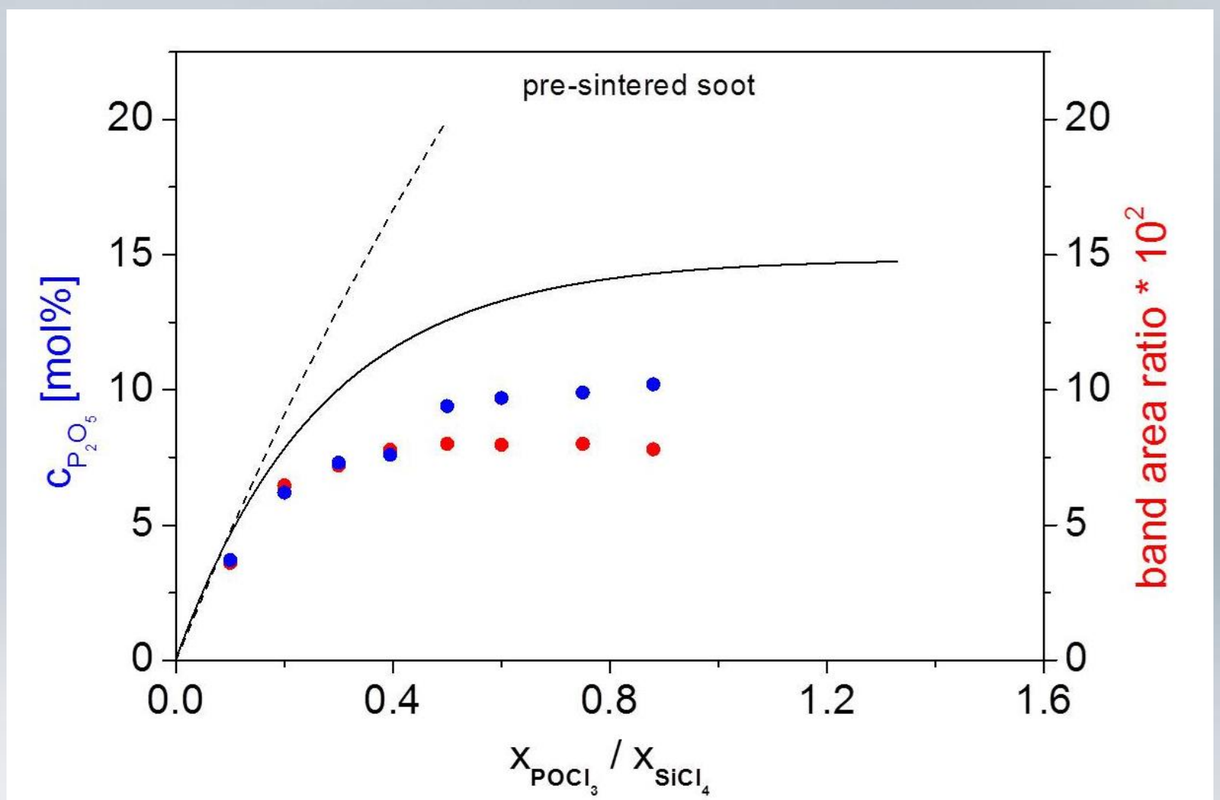
# Analysis of phosphorus doped soot material

## Band area ratio




- Includes only P=O → strong difference in untreated soot




- Corresponds well in case of pre-sintered soot with P-concentration

Technological advantage → Estimate the P content in the Soot → Optimize penetration

# Preform fabrication – comparison of the different processes

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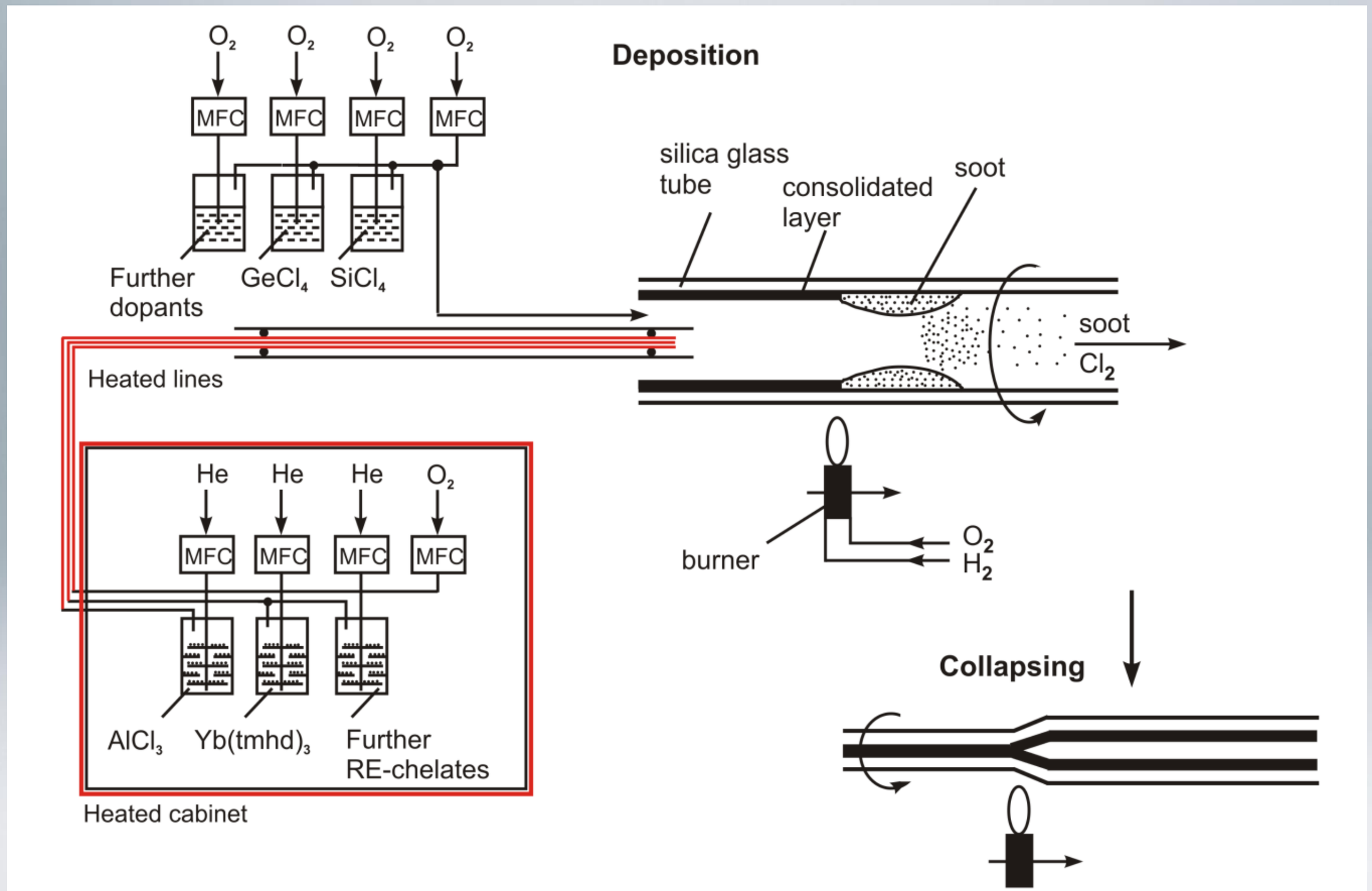
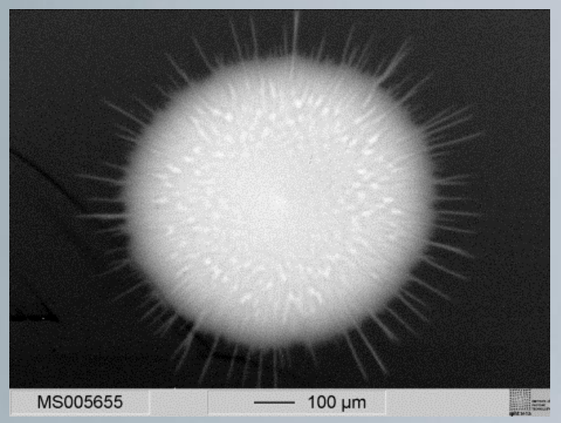
# Preform fabrication - MCVD process with gas phase doping




Special equipment for chelate delivery by OptaCore (Slovenia)

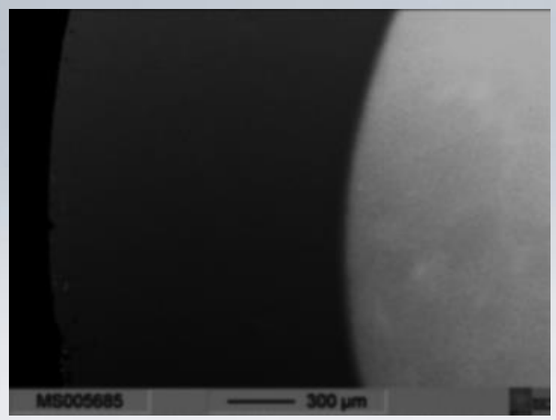
# Refractive index and concentration distributions

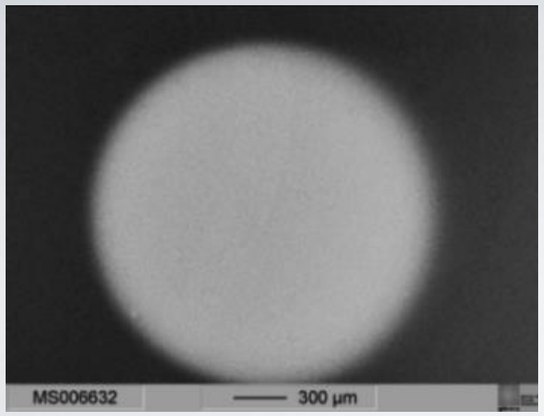
- homogenous radial distributions of refractive index change (Δn) and dopant concentrations of Al ($c_{Al_2O_3}$) and Yb ($c_{Yb_2O_3}$)
- without layer structure



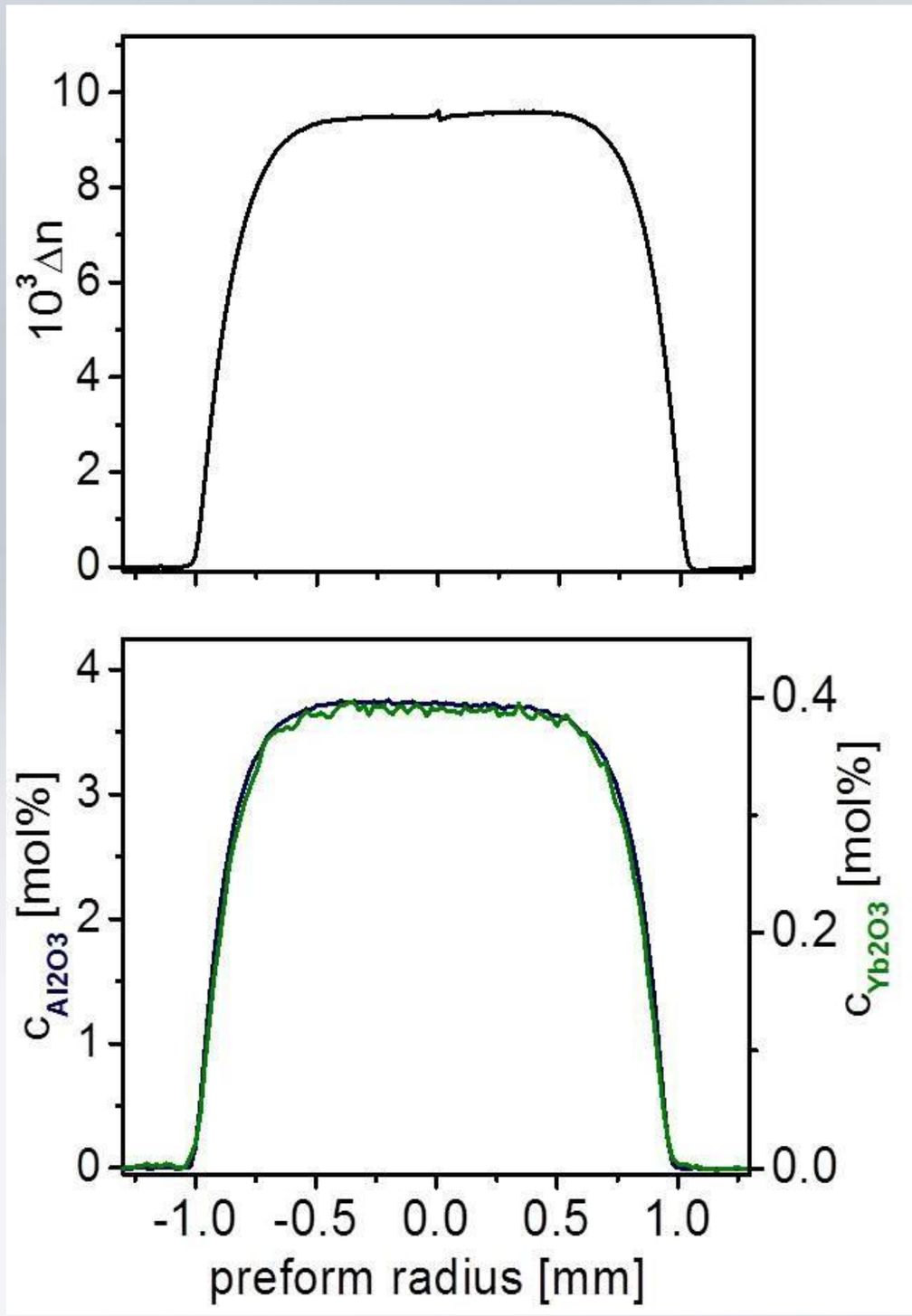

MCVD/ solution doping

REPUSIL

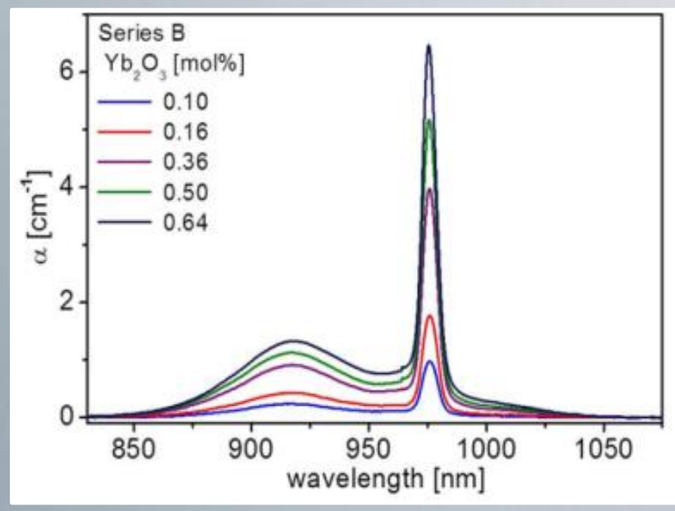


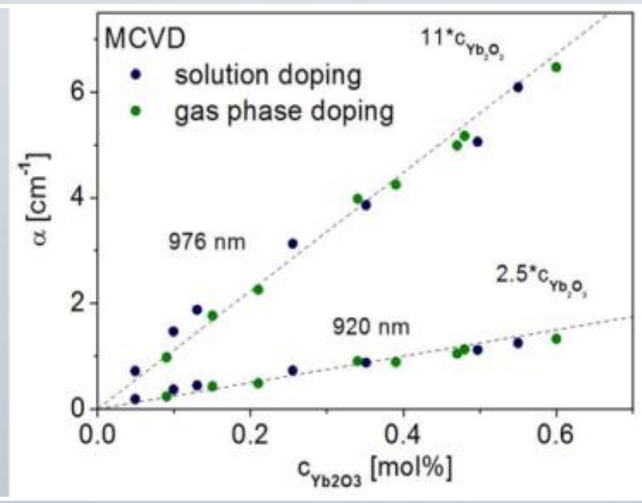




MCVD/ gas phase doping



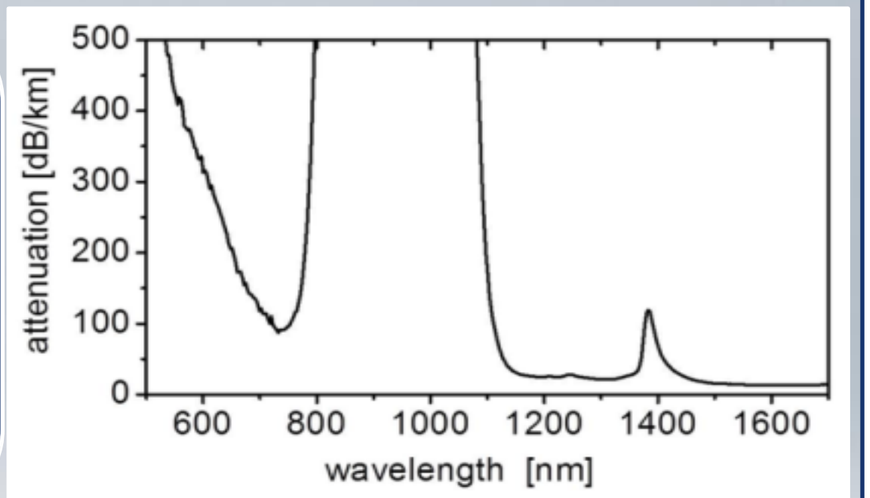
# Optical properties of the material




## Yb-absoption

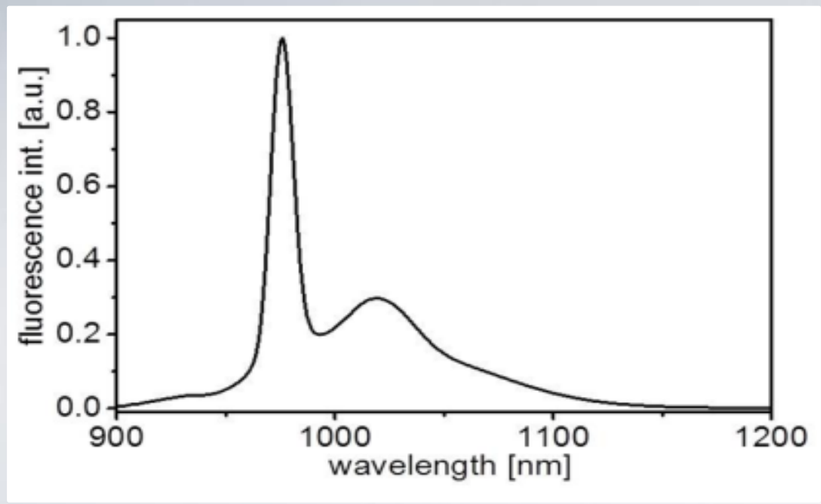
typical $Yb^{3+}$-related absorptions




## Fiber attenuation

background loss at 1200 nm (~20 dB/km) is still slightly increased

OH content in the range of 1.5 ppm and 5 ppm




## Spectral and time resolved fluorescence

typical emission spectra

fluorescence lifetimes of the $Yb^{3+}$ion: 780-840 µs

**Correspond well with Yb-doped alumosilicate preform samples using the MCVD/ solution doping technique**

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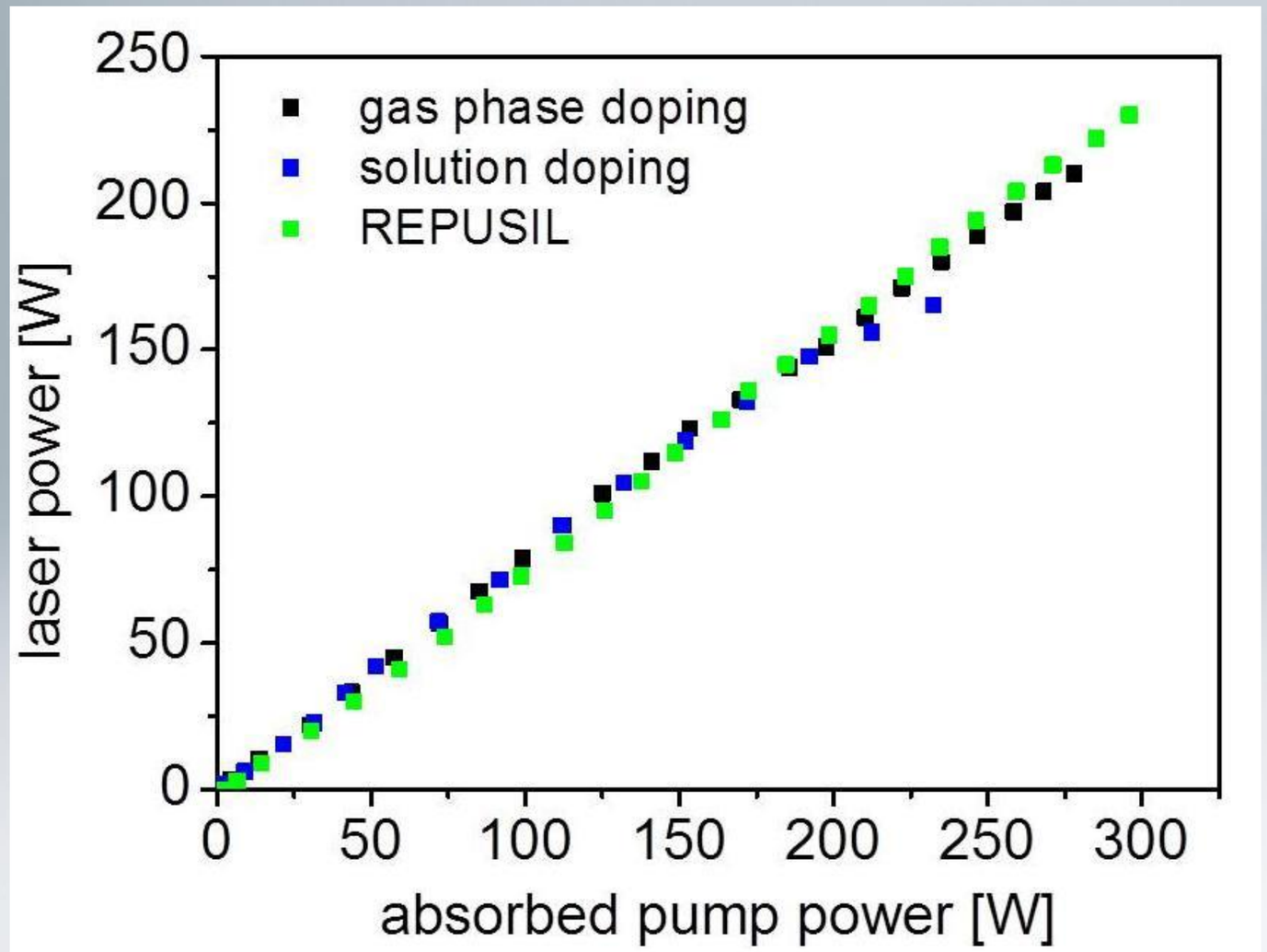
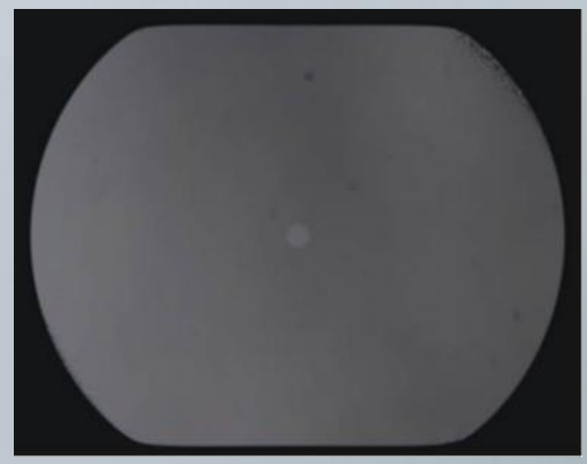
# Lasing behavior

- characterized in a cw laser set-up (pumping at 976 nm)
- excellent laser performance, output power > 200 W, slope efficiencies of 80%



core composition:
1.7 mol% $Al_2O_3$
0.18 mol% $Yb_2O_3$

cladding : core
180/230 : 10 µm = 20

- similar to Yb-doped silica fibers prepared using MCVD/solution doping or REPUSIL technique

Unger, S., Lindner, F., Aichele,C., Leich, M., Schwuchow, A., Kobelke, J., Dellith, J., Schuster, K. and Bartelt H., "Highly efficient Yb-doped silica laser fiber prepared by gas phase technology," Laser Phys. 24 (3), 035103 (2014).

# Summary

- IR-measurement of the Sootmaterial enable an optimization of the solution doping technique
- Provides information of the glass structure
- Active properties of gas phase doping specimen (absorption and emission spectra, fluorescence lifetime) correspond well with similar MCVD/solution doping samples
- Similar Yb/Al doped fibers of Repusil, gas phase doping and MCVD/solution doping show same excellent laser performance (output power > 200 W,  slope efficiencies of around 80%)
- Combination of different techniques or prepared materials enable new fiber design and application for optical fibers (multifilament, pedestal and LMA (large mode area) structures)

# Thank you for your attention!

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EUROPÄISCHER SOZIALFONDS

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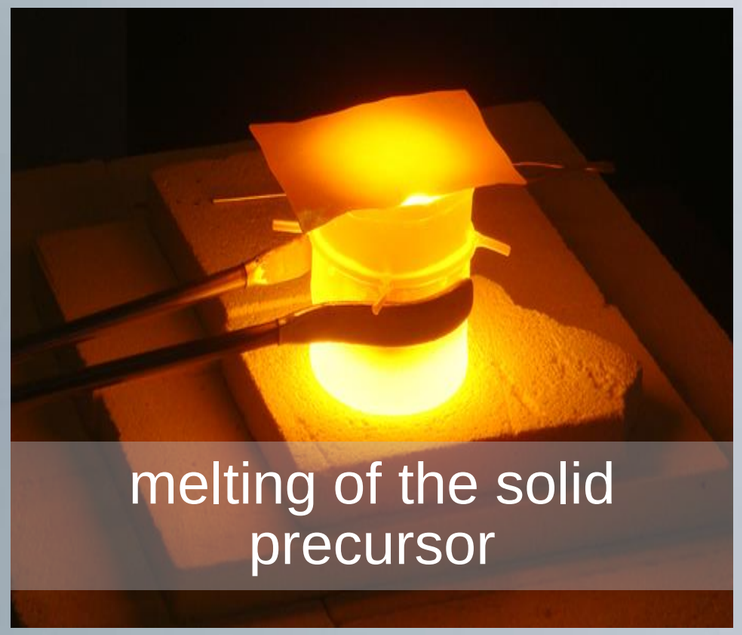
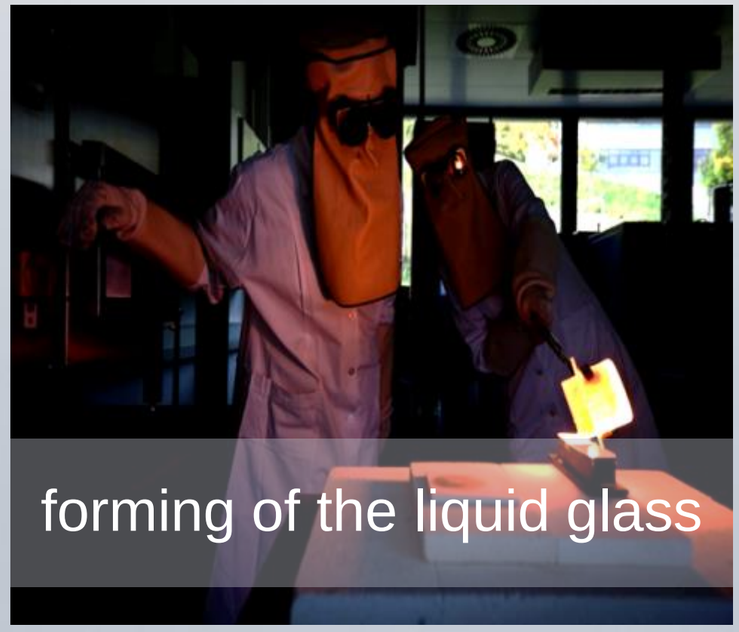
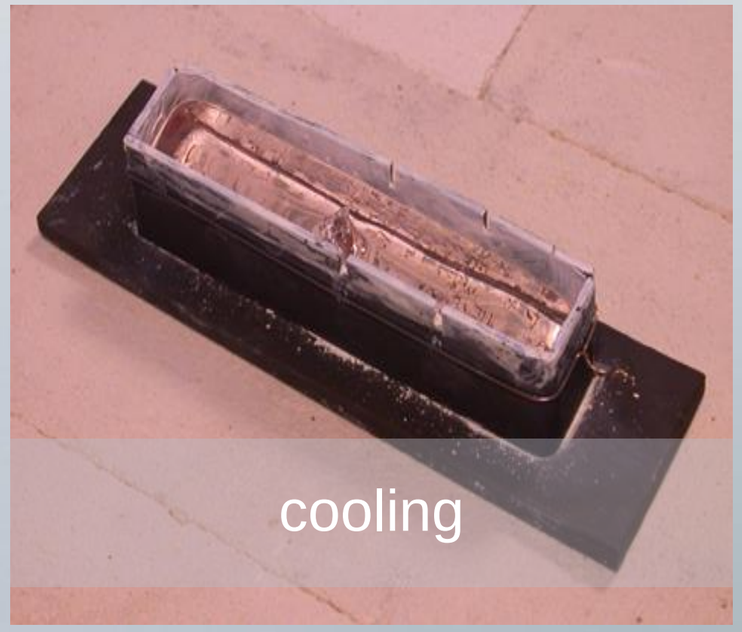
# Preform fabrication – Glass melting


melting of the solid precursor


forming of the liquid glass


cooling


polishing


grinding and polishing


drill out and polishing

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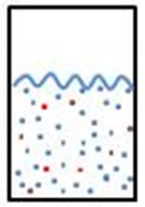
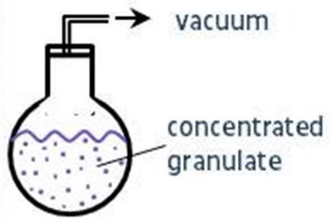
# Preform fabrication – REPUSIL

## 1. Powder Doping

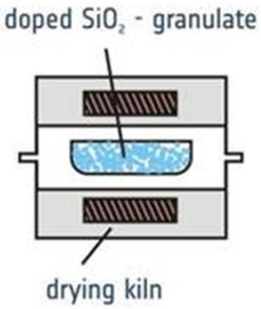



Reactive addition of dopants

## 2. Conditioning of the Granulate




Concentration and granulation

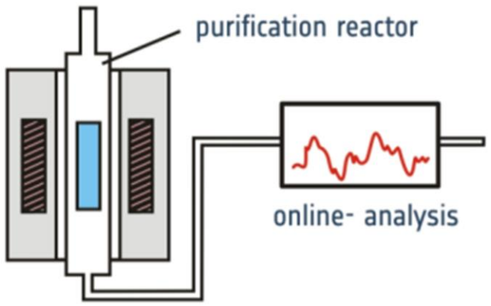



Thermal drying of the granulate

## 3. Shape Forming of Granulates

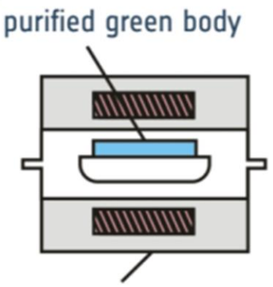

Manufacture of green bodies from the granulates

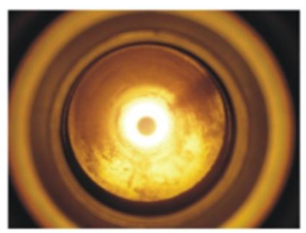
## 4. Chemical Purification




Chemical purification of the green body with online process control by IR-spectroscopy

## 5. Sintering

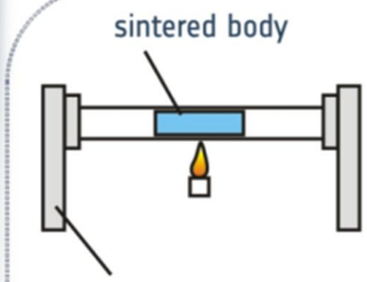


view into the sintering furnace

Sintering of the green body in define ambiance

## 6. Flame Vitrification




flame vitrification with hydrogen / oxygen torch

Flame vitrification of the sintered body

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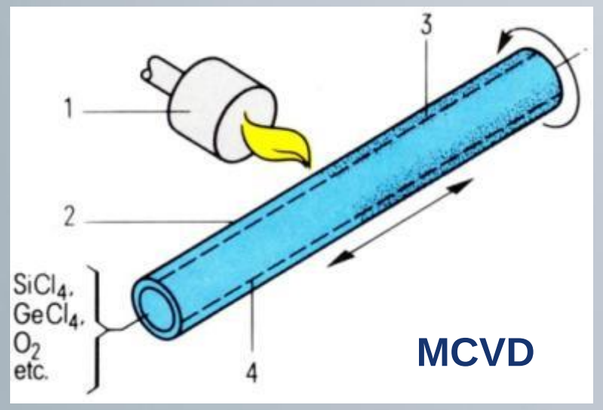
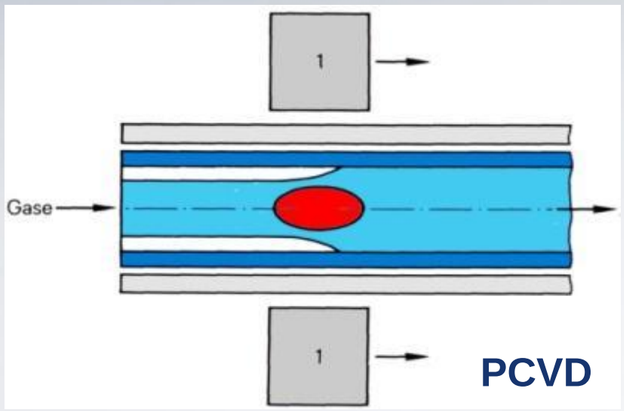
# Preform fabrication – Gas phase deposition

## Inside deposition

### thermal / oxidation

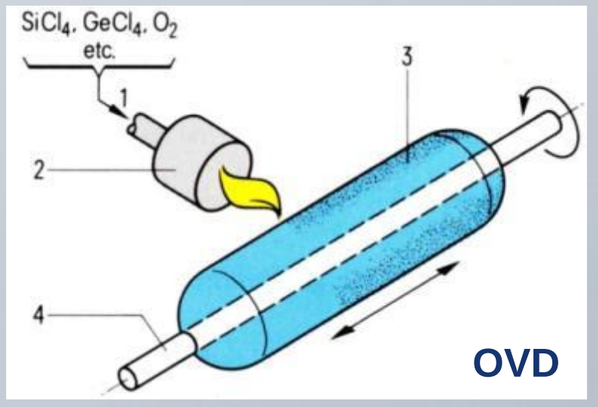


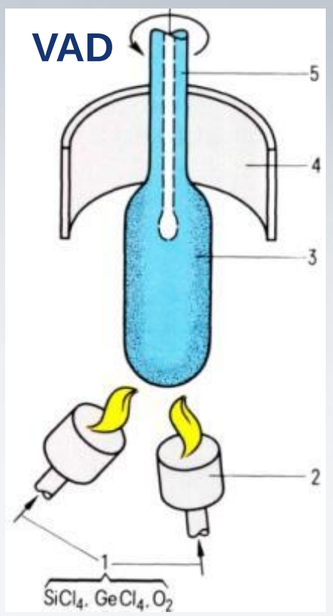

### plasma / oxidation

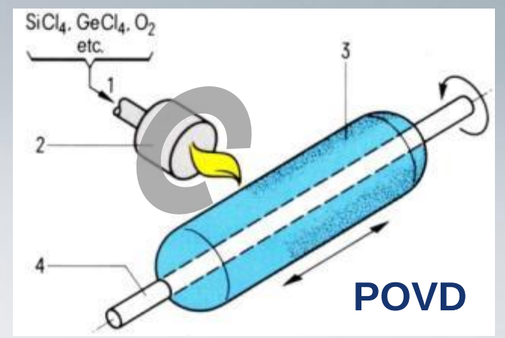



## Outside deposition

### thermal / hydrolysis



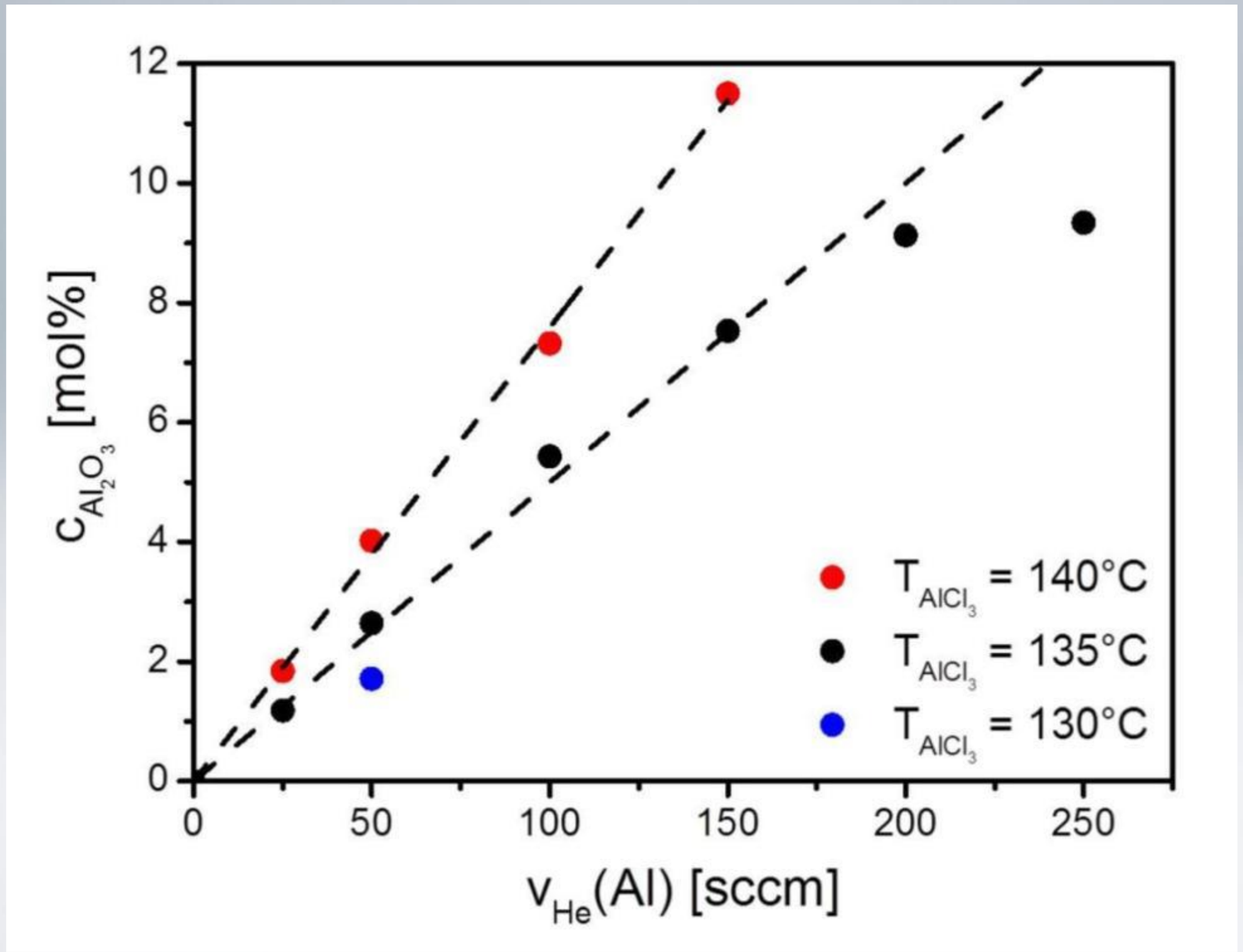




G.Mahlke and P. Gössing, „Lichtwellenleiterkabel“, Siemens Aktiengesellschaft, Berlin und München, 3. Auflage (1992).

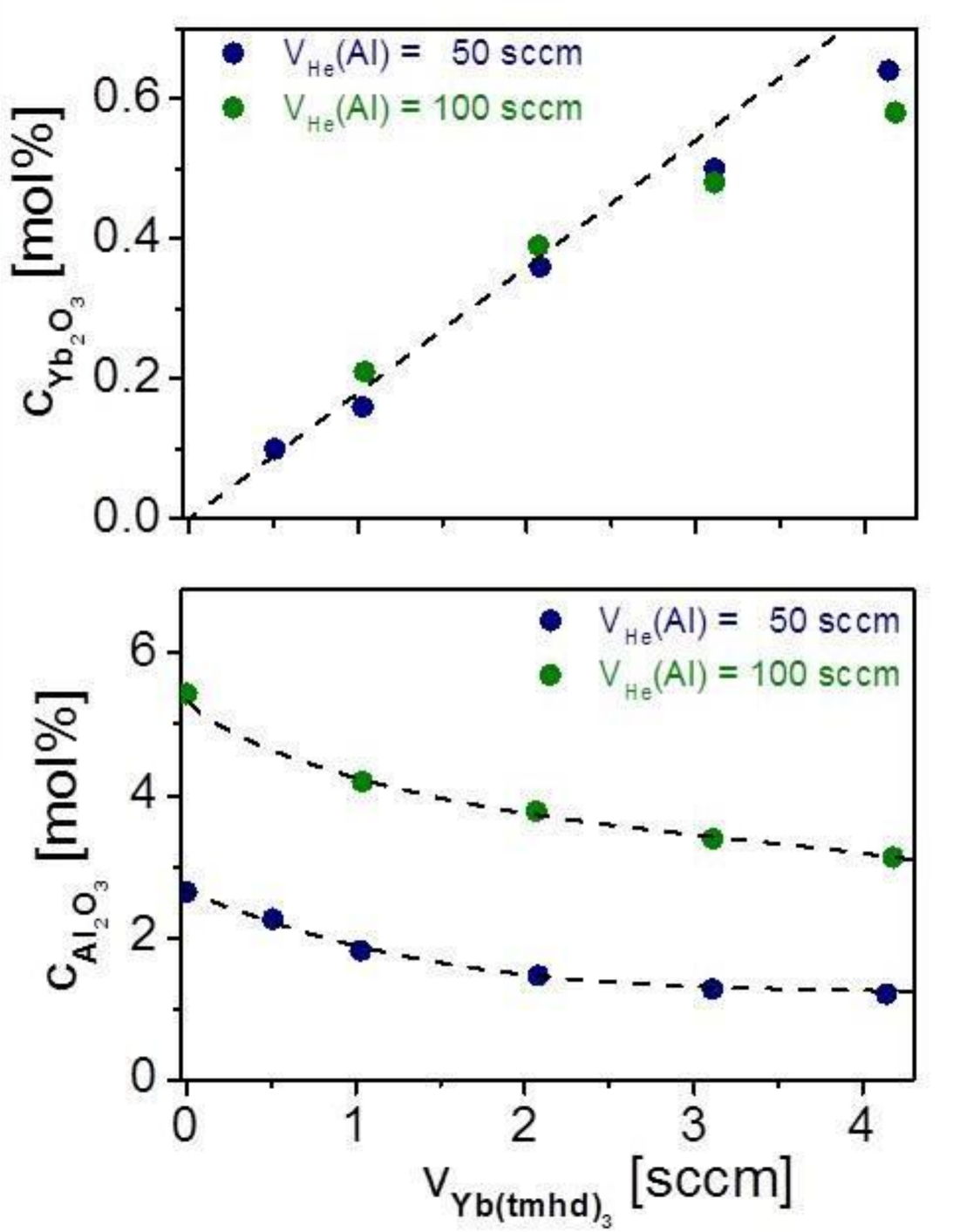
# Incorporation of Al into silica

Al content increases with increasing $T_{Al}$ and $v_{He}(Al)$

- $T_{Al}$ increase allow decrease of $v_{He}(Al)$ flow
- setting of specific Al content with $T_{Al}$ and $v_{AlCl_3}$



# Incorporation of Yb in combination with Al into silica




Yb content $c_{Yb_2O_3}$ increases practically linear with the gas flow of $Yb(tmhd)_3$ ($v_{Yb(tmhd)_3}$)

- Yb incorporation independent form the Al content level

Al content decreases continuously with increasing $v_{Yb(tmhd)_3}$

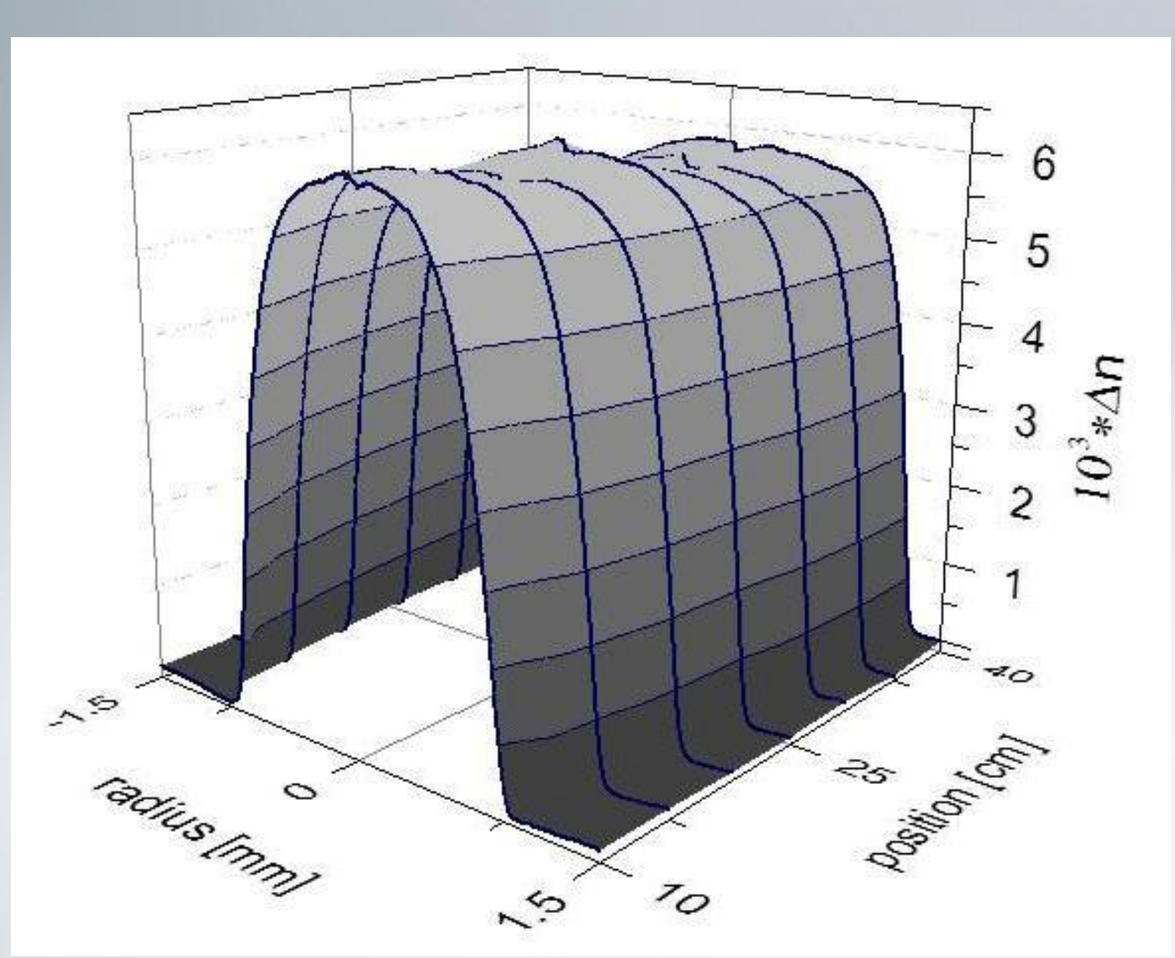
- Interaction between Yb and Al precursor

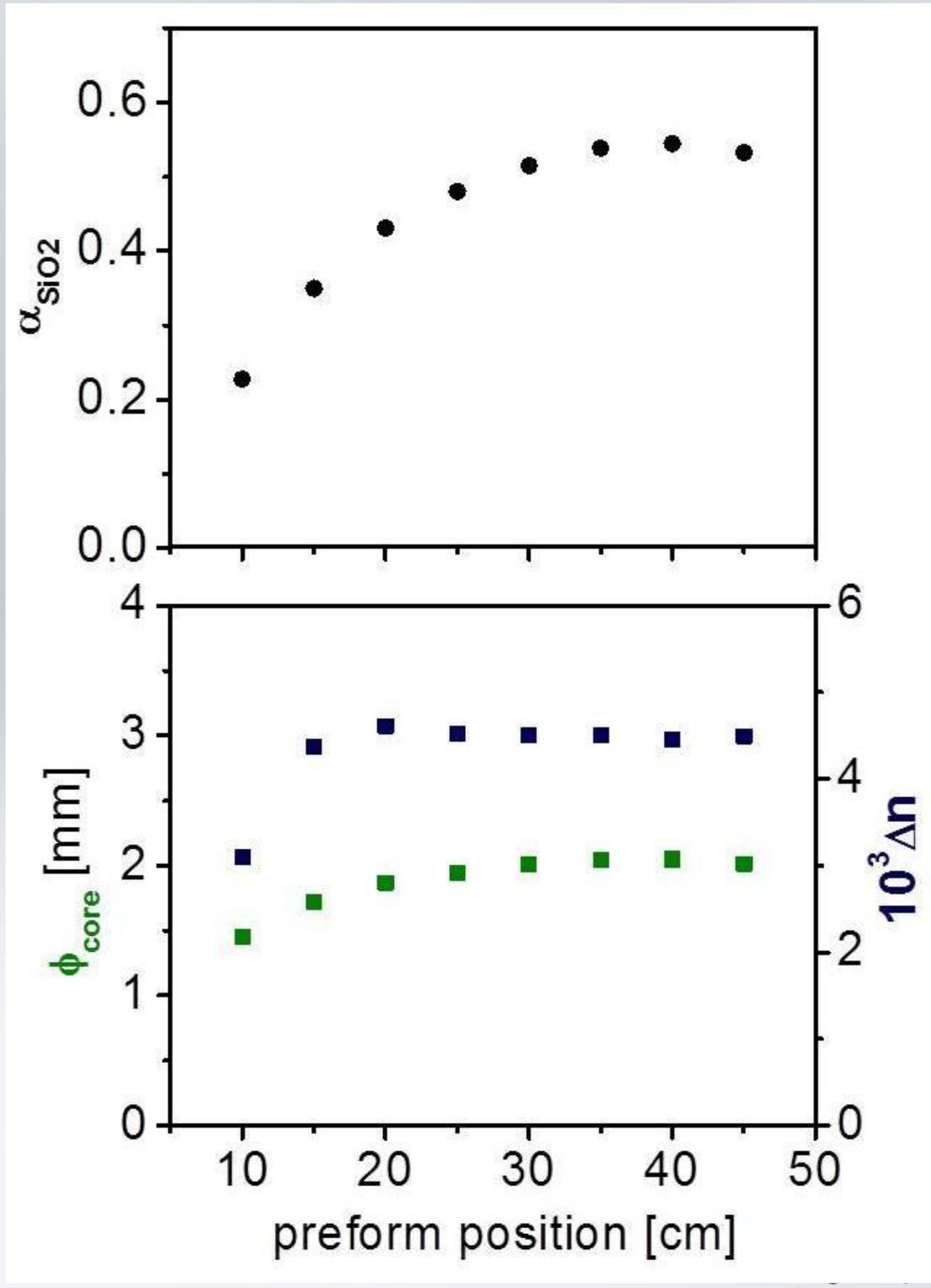
F. Lindner, C. Aichele, A. Schwuchow, M. Leich, A. Scheffel, S. Unger, „Optical properties of Yb-doped fibers prepared by gas phase doping", Proc. SPIE 8982 (2014)

# Longitudinal homogeneity / Deposition efficiency

refractive index, deposition efficiency and core diameter nearly constant from preform position 20 cm

- homogeneous Al or Al/Yb-doping of a length of 30 cm



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# Optical properties of Yb/Al-doped fibers prepared by gas phase doping