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Special optical fibres with elevated alumina content for high temperature or medical applications

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1. Applied Fibre Technology at BFH

1.1 Our group

BFH **IAP**

Dr. Sönke Pilz
Co-Head AFT Lab

Dunia Blaser-Lóp
PhD Student

Dr. Manuel Ryser
Co-Head Fibres and Fibre Lasers

Dr. Dirk Spangenberg
Optics Expert

Pascal Hänzi
PhD Student

MSc Martin Hochstrasser
Chemistry Expert

Prof. Dr. Valerio Romano
Head of Group

Ass. Prof. Dr. Alexander Heidt
Head Nonlinear Fibre Optics

1. Applied Fibre Technology at BFH

1.2 Key elements of Optical Fibre production

- Fibre production needs:
  - Optical materials that are **suited for assembling a preform**
  - **Preform**
    - Scaled copy of the fibre (fibre:125um until 500um), its size ranges from: 10mm until several tens of centimetres (industrial side)
  - **Drawing tower**
  - **Set-up** for analysis

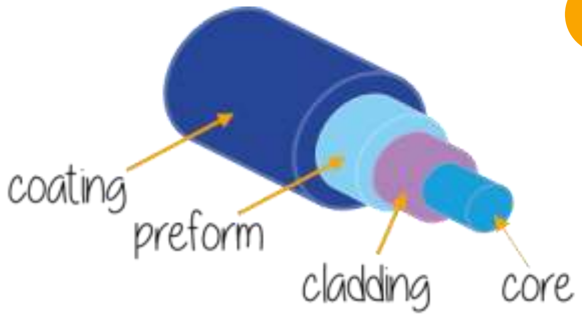
- Along with **IAP** the *Applied Fibre Technology group* has all key elements to produce fibres:
  - Granulated Silica methods for fibre production
  - Equipment to assemble and pre-treat preforms
  - Access to a drawing tower at IAP to draw fibres
  - and labs to handle and analyse fibres as well as to build systems (lasers etc.) from the drawn fibres

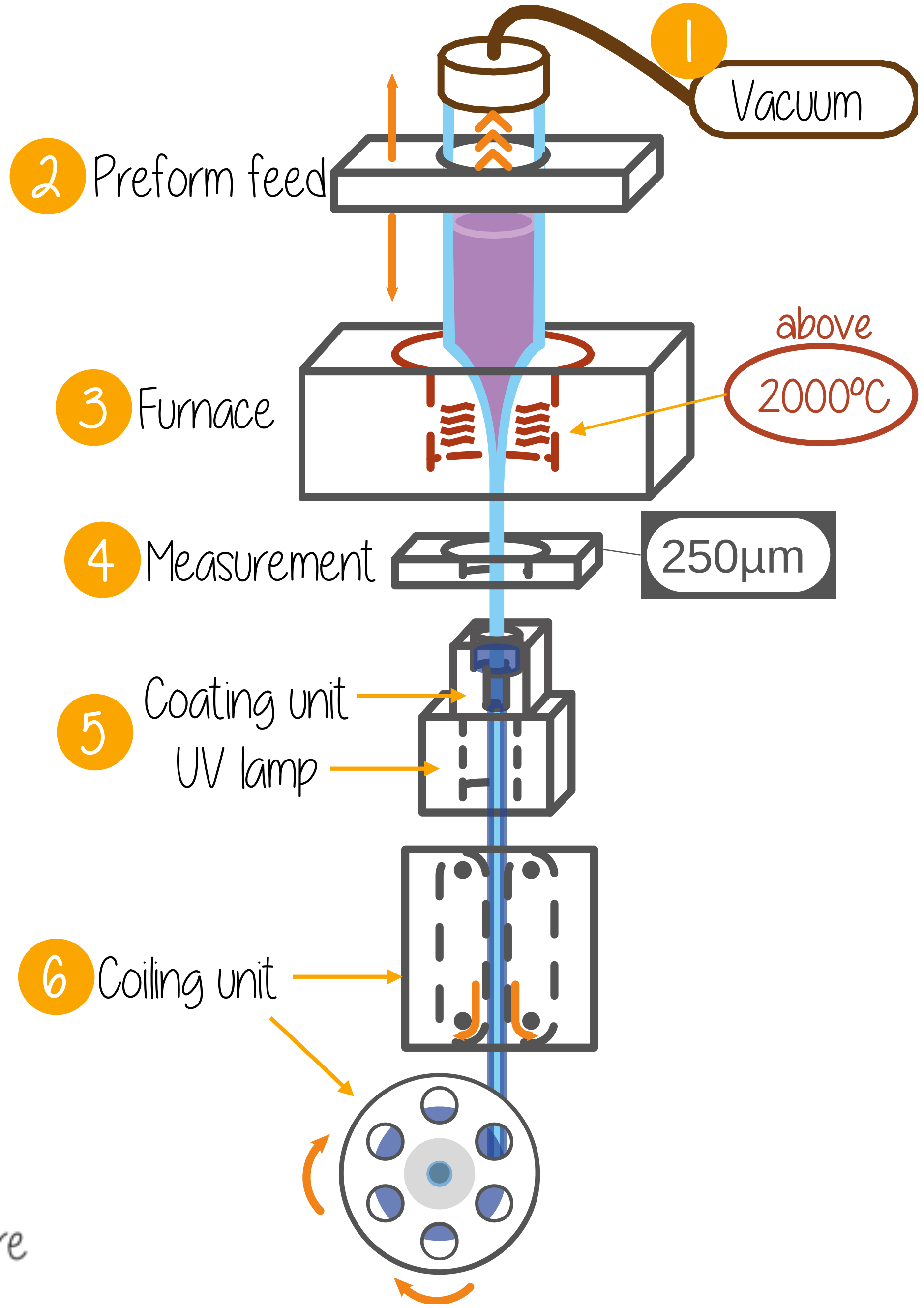
1. Applied Fibre Technology at BFH

1.3 Expertise of our group

a) Drawing Tower at IAP - concept

- The drawing tower consists of 6 essential units for drawing a fibre:
  1. Vacuum
  2. Preform feed
  3. Furnace
  4. Measurement Unit
  5. Coating and UV lamp unit
  6. Coiling (capstan) unit

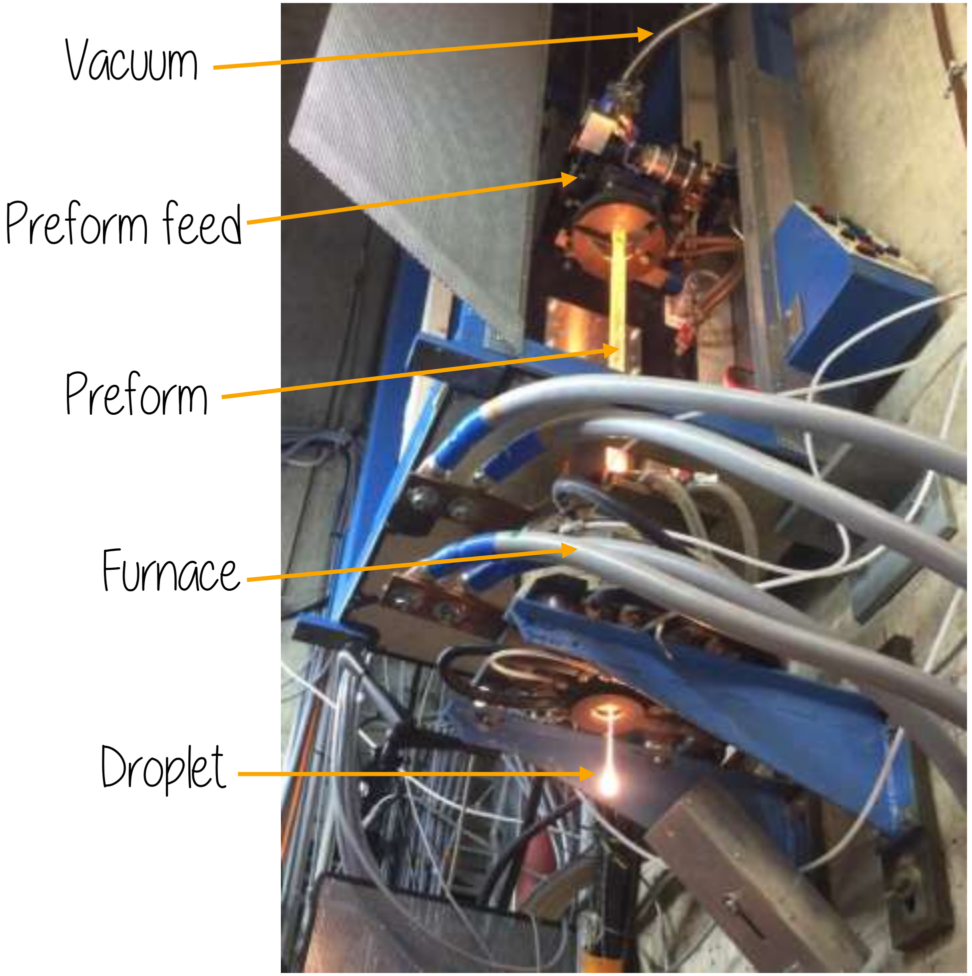


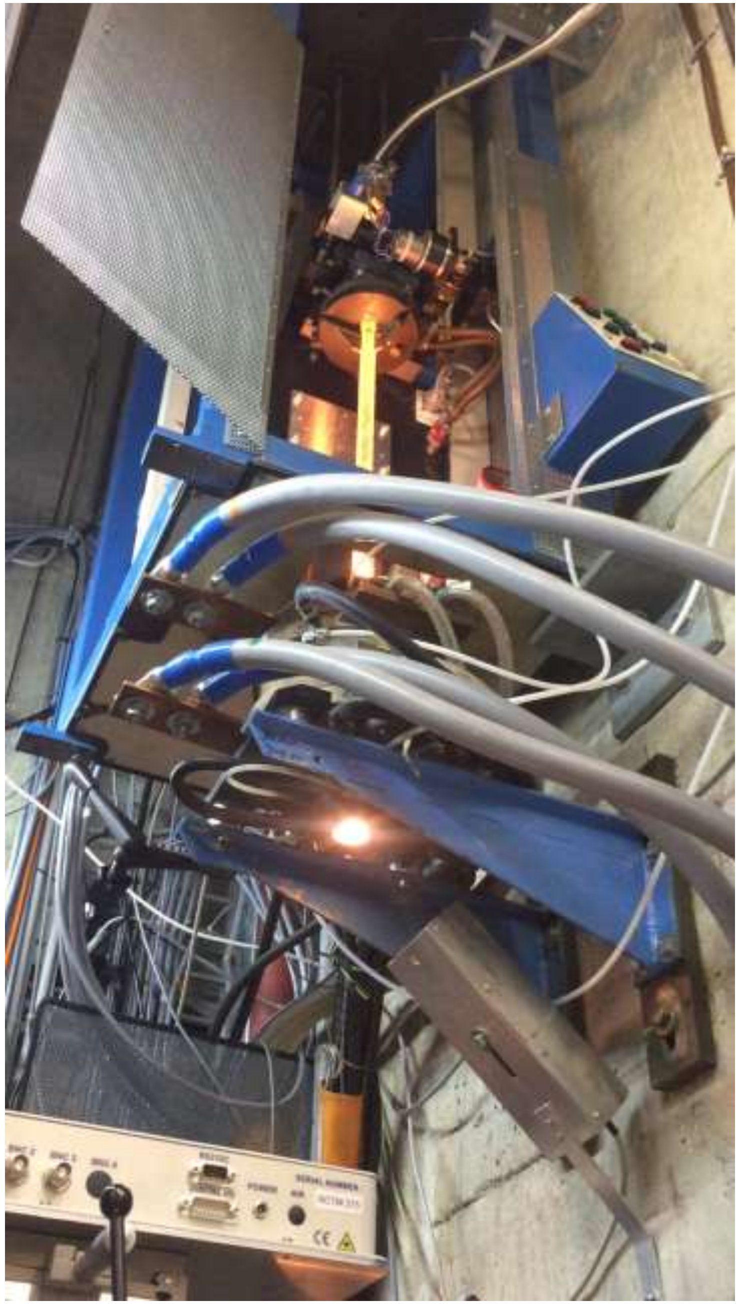


1. Applied Fibre Technology at BFH

1.3 Expertise of our group

b) Drawing tower at IAP





Close collaboration with
Institute of Applied Physics
of the University of Bern

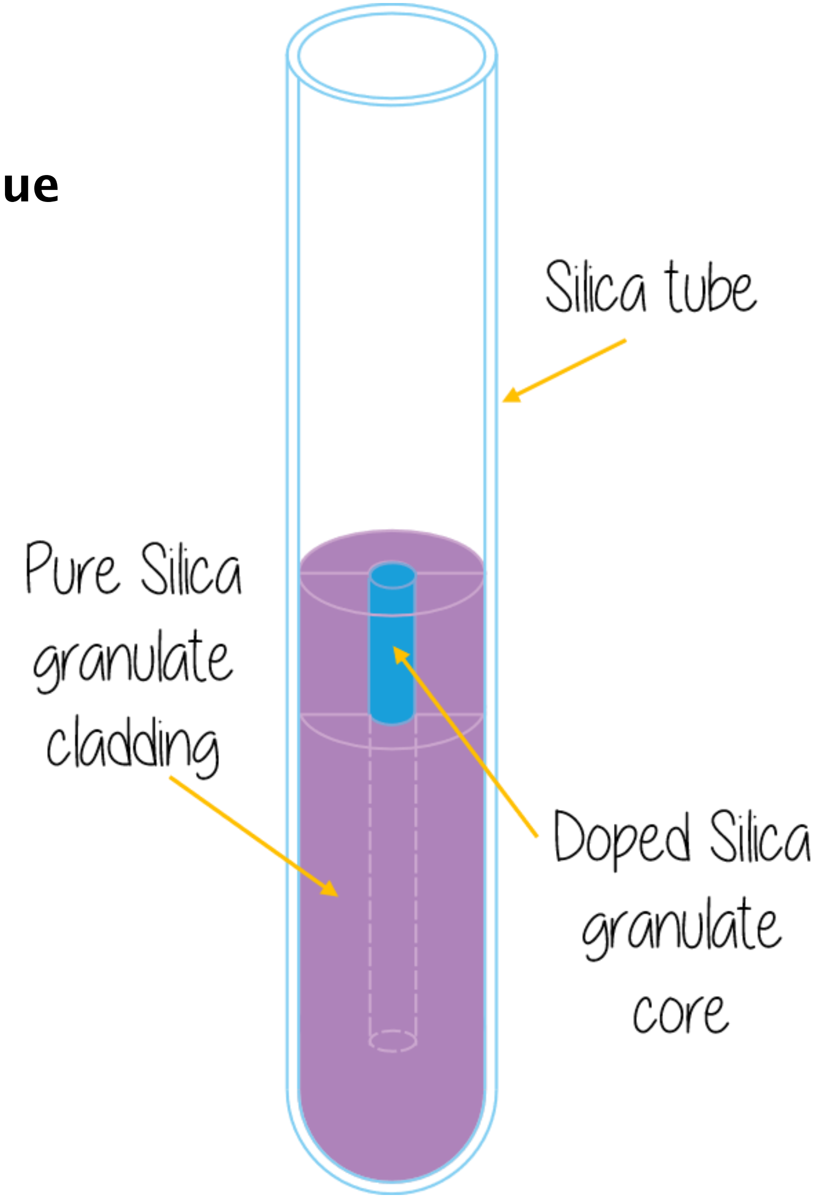
u^b UNIVERSITÄT BERN

1. Applied Fibre Technology at BFH

1.3 Expertise of our group

c) Granulated Silica method and Powder-in-tube technique

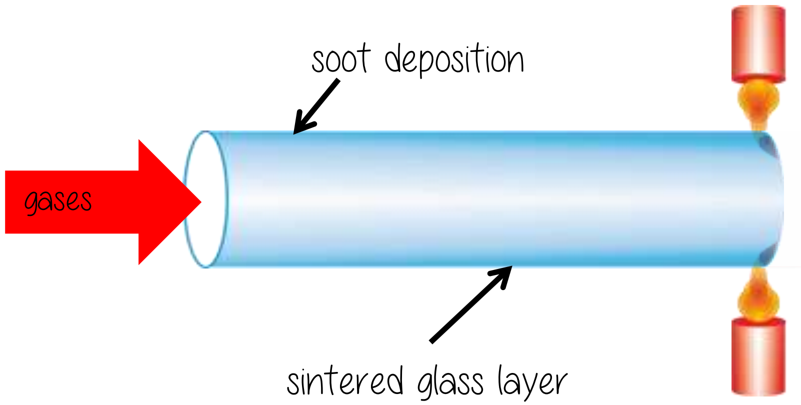
- Novel processes have been implemented in the conception and fabrication of such fibres:
  - **Powder-in-tube technique** combined along with
  - **Granulated silica powder method**
    Provides flexibility on the implementation of high dopant percentages of diverse elements, not suitable for MCVD methods.

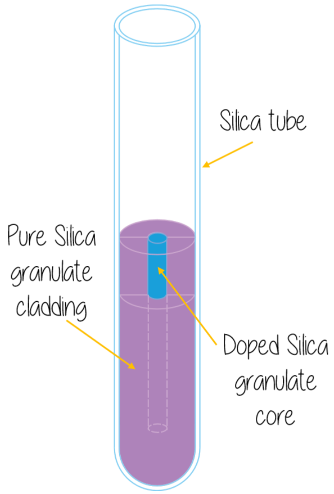


2. Motivation for developing the new method

2.1 Conventional Fibre Production and Limitations

Methods		Advantages	Disadvantages
Chemical Vapor Deposition	▪ Solution doping ▪ Gas Phase ▪ Aerosol ▪ Direct Nano particle Deposition(DND)	▪ Stable and well optimized technologies ▪ Uniform doping profile ▪ **Highest quality, very low scattering losses:** 0.6dB/km@1100nm 0.18dB/km@1550nm	▪ Difficult to make Large homogeneous Cores ▪ Increasing doping concentration (Stress & re-evaporation doping elements) ▪ Limited symmetries for structured fibers ▪ No non-silica materials ▪ relatively big technical effort/time consuming ▪ Limitation of doping concentration
Granulate Silica	• Powder Sintering • Sol-ger • Oxide	▪ High doping & Large Core ▪ Various structuring fibers ▪ Cost-effective & Simplicity ▪ Any powder ▪ Short period of time	▪ Intrinsic scattering loss due to powders ▪ Optimized vitrifying process needed depending on the rare earth powder





3. Granulated Silica Technique

3.1 Fiber Fabrication

Granulated Silica Method : Powder in tube with coarse grains

Granulated-silica method: our proposal to overcome limitations

- First mentioned by John Ballato and Elias Snitzer,
  - "Fabrication of fibers with high rare-earth concentrations for Faraday isolator applications," *Applied Optics*, vol. 34, no. 30, p. 6848, Oct. 1995
- 1996 - IAP: Production of RE doped glass for spectroscopy
- 2004 - IAP: undoped microstructured fibres (after)
- 2008 - IAP: RE-doped fibres
- 2008 - IAP: Metal and transition metal doped fibres
- 2011 - IAP/APRI/BFH: Granulated silica and travelling laser vitrification
- 2013 - BFH/APRI/IAP: sol-gel GS + travelling small zone laser vitrification
- 2014 - BFH/Reseachem: Improvement of doped sol-gel glass
- 2015/2016 - Every powder grain is doped based on the Sol-Gel process → homogeneous Yb/Al/P dopants distribution
- 2017/2018 - Reduction losses for Sol-Gel derived fibers due to additional (laser-based travelling small zone) vitrification Sol-Gel Benchmark Yb/Al/P: 0.2dB/m @633nm
- 2019 - Yb-doped silica fibers with different Al and P concentration (RI tailoring)
- 2020 - "Benchmark" Yb/Al/P-doped silica fiber with a slope efficiency of 55-61% and output power of 125W

3. Granulated Silica Technique

3.2 Principle of Vitrification

Optional Vitrification Step
for Reducing bubbles and scattering losses

Tube Setup › **Filling the Core Sand** › **Filling the Clad sand** › **Reforming Core** › **Vitrification**

A narrow pure silica tube embedded wit h a larger silica tube

Doped silica sand is introduced into the narrow tube

Pure silica sand is introduced into the interspace between the two tubes

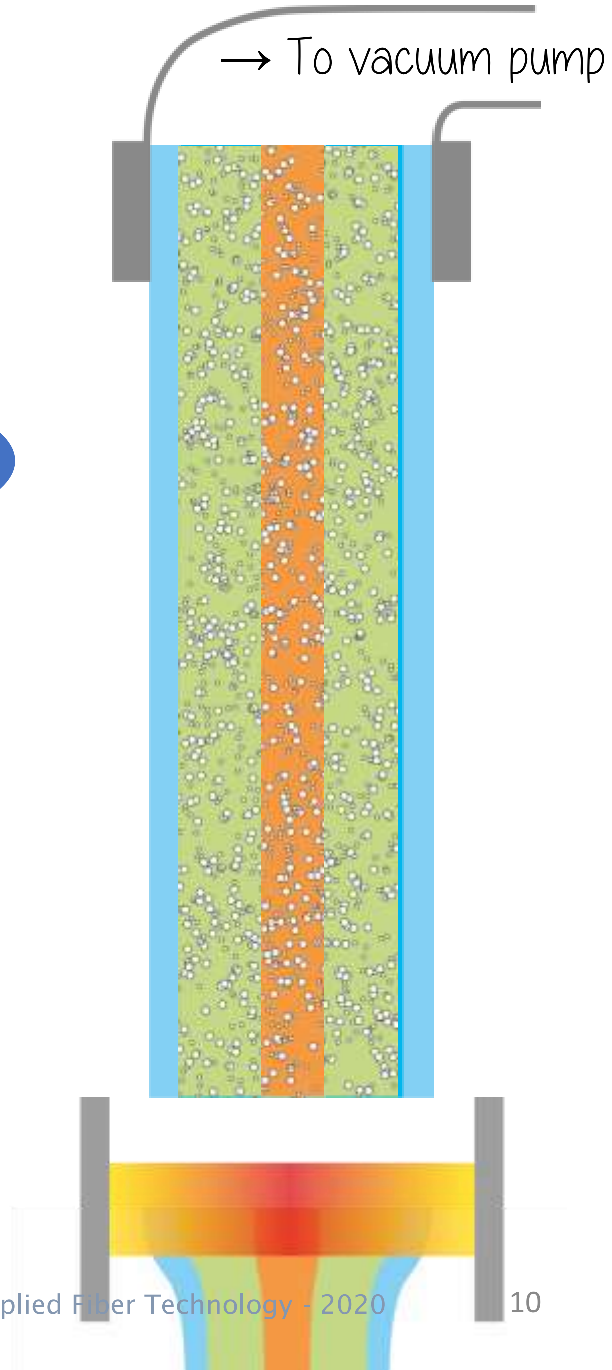
The narrow silica tube is retired

The structure is evacuated and heated with a cleaning gas treatment

Sand is vitrified, all solid preform is obtained

Important parameter: Grain-size
Similar small grain size
better diffusion and less refractive index-fluctuations

Small Enough → evacuation difficult



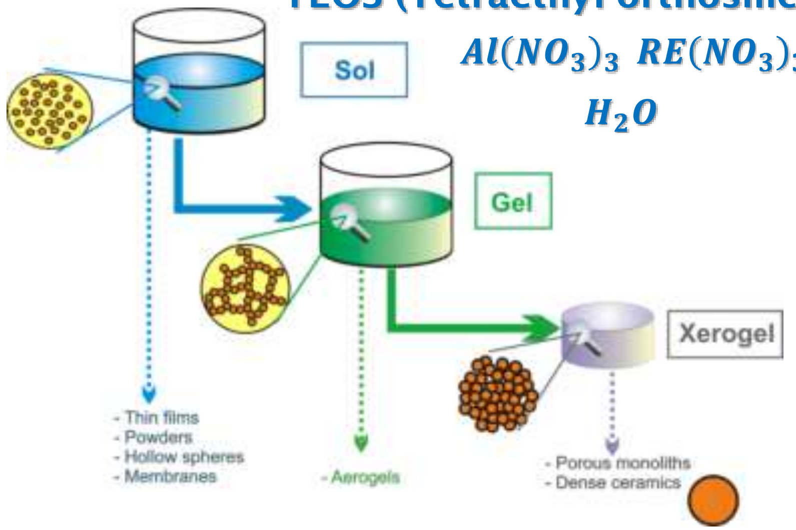
3. Granulated Silica Technique

3.3 Improving scattering losses: Sol-gel

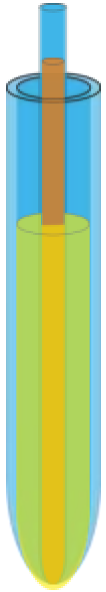
TEOS (Tetraethyl orthosilicate)

$Al(NO_3)_3$ $RE(NO_3)_3$

H_2O



→ **Produce granulate:** All grains are homogeneously doped →



→ **Vitrification** →

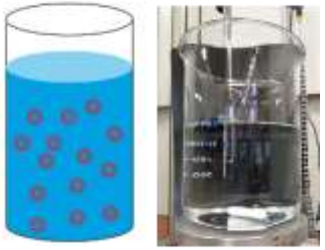
- ✓ Flexibility of dopant content - any water or ethanol soluble dopant can be dissolved homogeneously
- ✓ Flexibility of choosing processing temperatures (200°C - 2000°C)
- ✓ Very cost-effective
- ✓ Rapid prototyping and manufacturing

- ✗ Wet chemical process → OH groups (large Si OH absorption @ 950nm, 1240nm, 1390nm)
- ✗ Possible scattering losses

3. Granulated Silica Technique

3.4 From Sol-Gel to Granulate: One possible route to improve homogeneity

Solution of Precursors: «sol»



Mixing of precursors in the liquid phase (colloidal dispersion)

hydrolysis & condensation

formation of gel

Gel

drying

removal of the liquid part

Porous powder

Homogeneously doped grains

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sintering

removal of OH-groups

Sintered block of powder

crushing & sieving

grain size selection

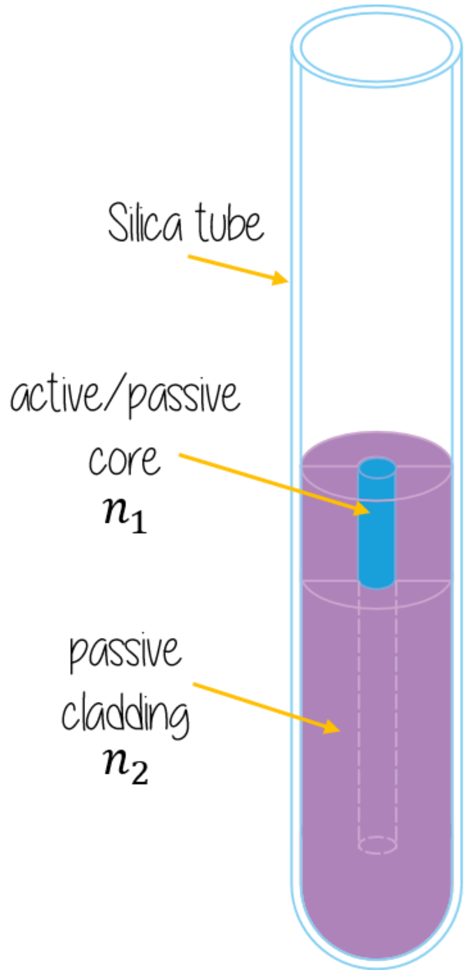
Granulate

grain size~100µm

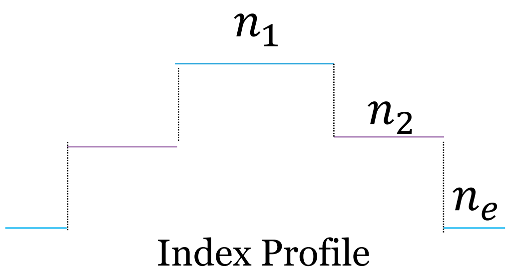
Wide variety of doped granulate

4. Example Fibres

4.1 Step Index fibre



Light is confined and guided within the core due to the **total internal reflection** effect, a fibre must have a **lower index** of refraction in the **cladding** than the one in the core.



4. Example Fibres

4.2 Single core – different dopants

Doping concentration [at.%]	
AL_2O_3	1.3
Bi_2O_3	0.1
Er_2O_3	0.02
Nd_2O_3	0.01
SiO_2	98.57

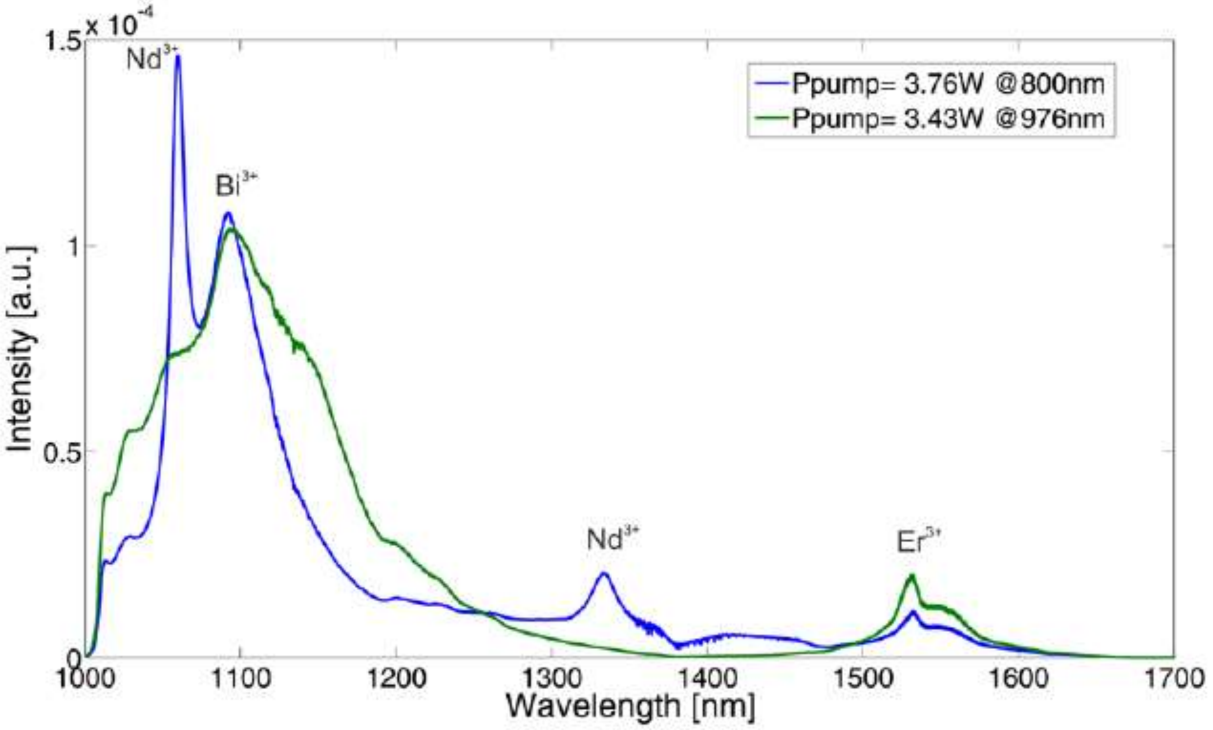


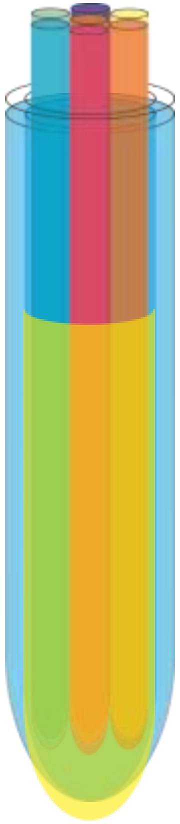
Figure 8: Comparison between the IR-fluorescence spectra of a 9.96m long fiber piece for a 800nm ($P_{pump,800}$=3.76W) and a 976nm ($P_{pump,976}$=3.43W) pump wavelength.

- Pilz, S. et al. "Infrared broadband source from 1000nm to 1700nm, based on an Erbium, Neodymium and Bismuth doped double-clad fiber." (2012).

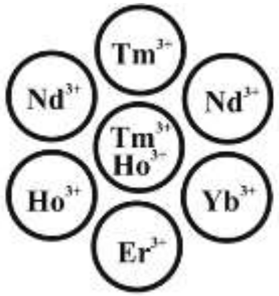
4. Example Fibres

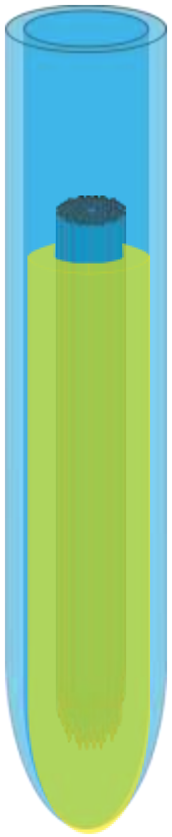
4.3 Multi-core (a dopant per core) and Microstructure Fibres

Rapid preform/fibre prototyping with Granulated Silica-Method

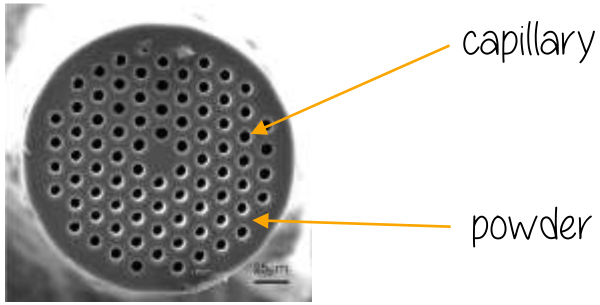


Rare-Earth doped Multicore fibres





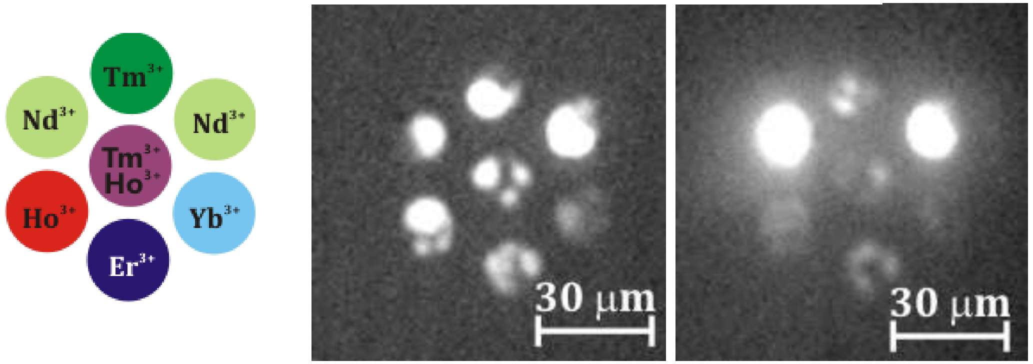
Micro-structured fibres (hybrid)



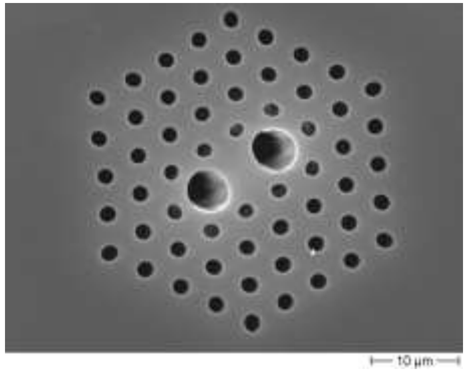
4. Example Fibres

4.4 Characterization of diverse Optical Fibres

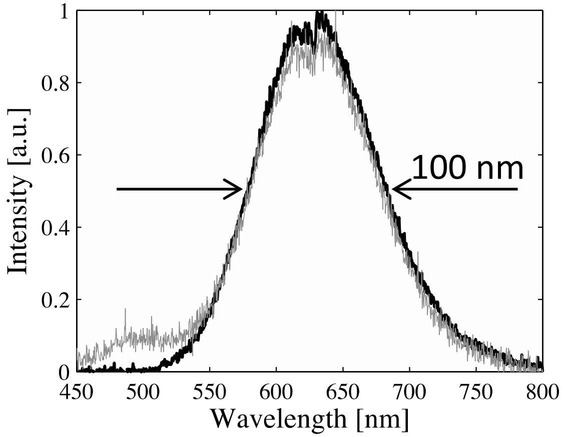
Rare-earth doped multicore fiber



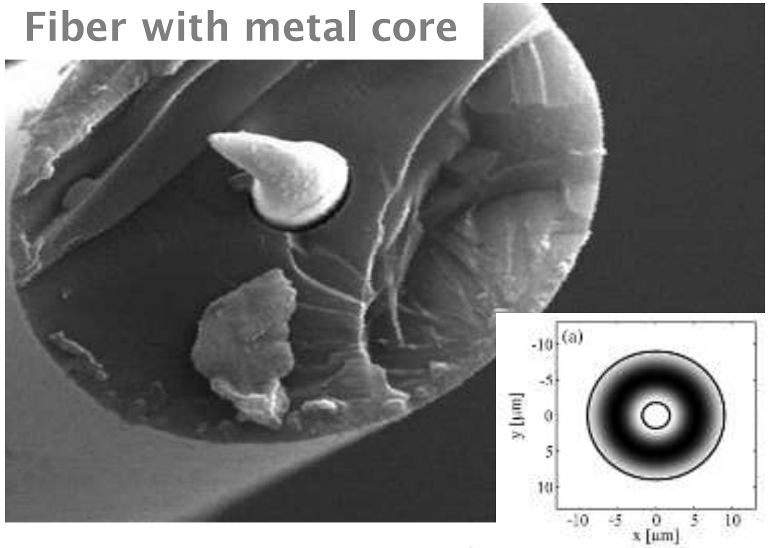
Microstructured fiber



Sb^{3+}-doped fiber



Fiber with metal core



Metal-doped fibers

5. Current research: high Alumina Oxide fibres

- **Motivation**
  - Transmission at longer wavelengths, between 2µm and 3µm is of **utmost importance for Medical Applications**
  - Fused Silica's OH content:
    - Transmission limitation in the mid-IR region
    - Safely used up to 1200°C

- **Solution**
  - Introduction of Sapphire or aluminium oxide (Al_2O_3) content
    - Optical transmission ranges from UV up to 5.5 µm
    - Considered a high-temperature material
    - Better suited with respect to optical and mechanical **resistance to elevated temperatures.**

5. Current research: high Alumina Oxide fibres

- **Fibre under development by Dunia Blaser**

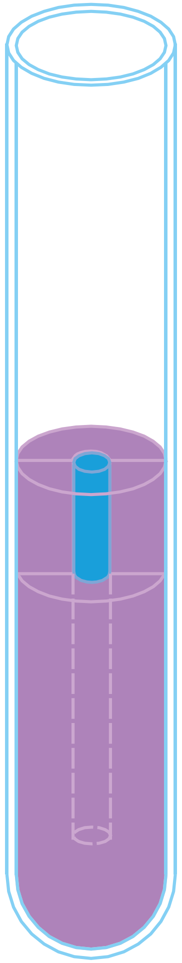
- **Challenge**
  - Sapphire in crystalline phase or Aluminium Oxide, stays solid up to 2050°C and then **suddenly** melts.
  - Its viscosity at slightly above 2050°C is comparable to the viscosity of water at room temperature.

Sapphire rods in Granulated silica powder

Conception of Green compacts

6. Elevated Alumina Fibres

6.1 Sapphire core rod inside SiO_2 powder cladding – first test



	active or passive	composition	base material	condition	Diameter *mm*	Length *mm*	region	refractive index
	Passive	SiO_2	Silitec SiO_2	Loose powder	18	130	Cladding	n_{p1}
	Passive	Sapphire	Sapphire	Rod	2	100	Core	n_{p2}
	Passive	Si = 100at.%	Silica tube	Envelope (tube)	18/21	-	-	n_{Si}

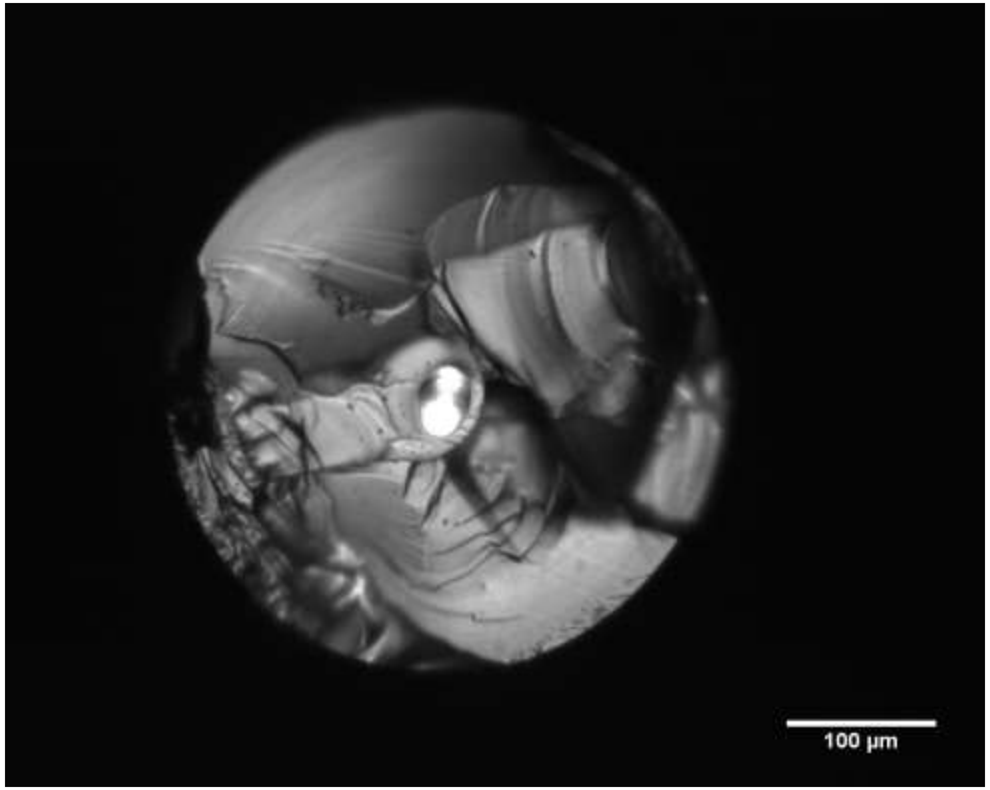
Drawing Temperature: 1990°C

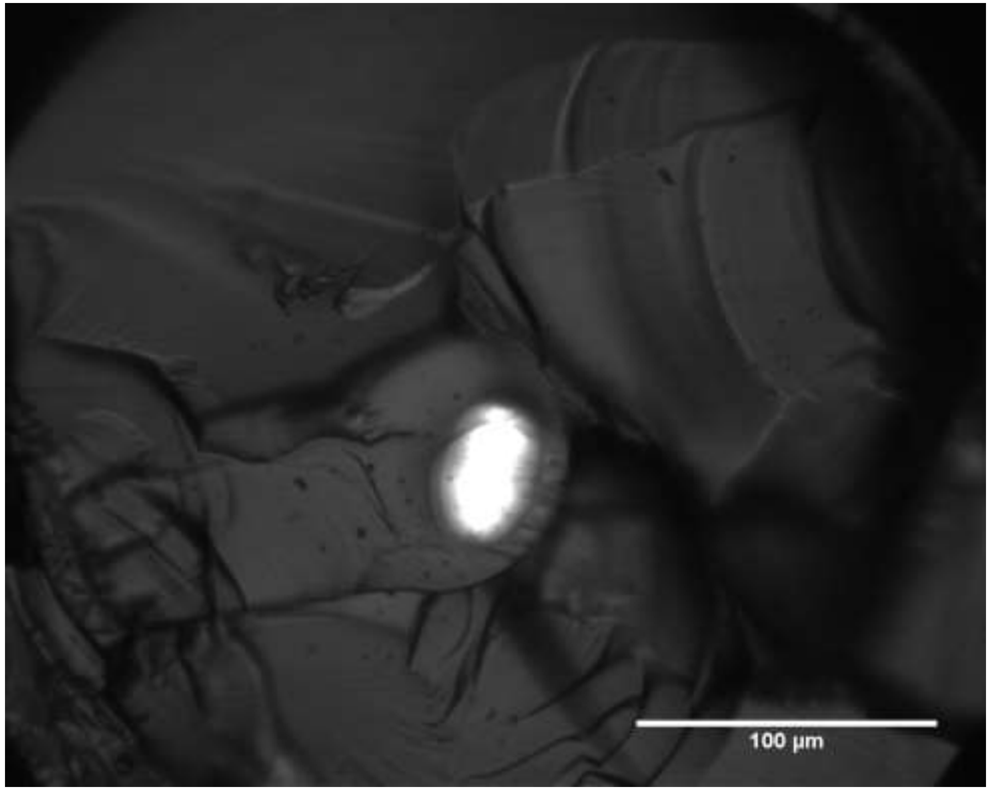
6. Elevated Alumina Fibres

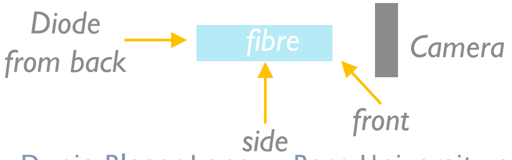
1) Crystal Sapphire core / SiO_2 cladding

Drawing Temperature: 1990°C

1a) Fibre piece of ca. Ø 350 µm and 50 mm length







Fluorescence 915nm diode (1.51 V / 0.550 A) from back edge – no filtering

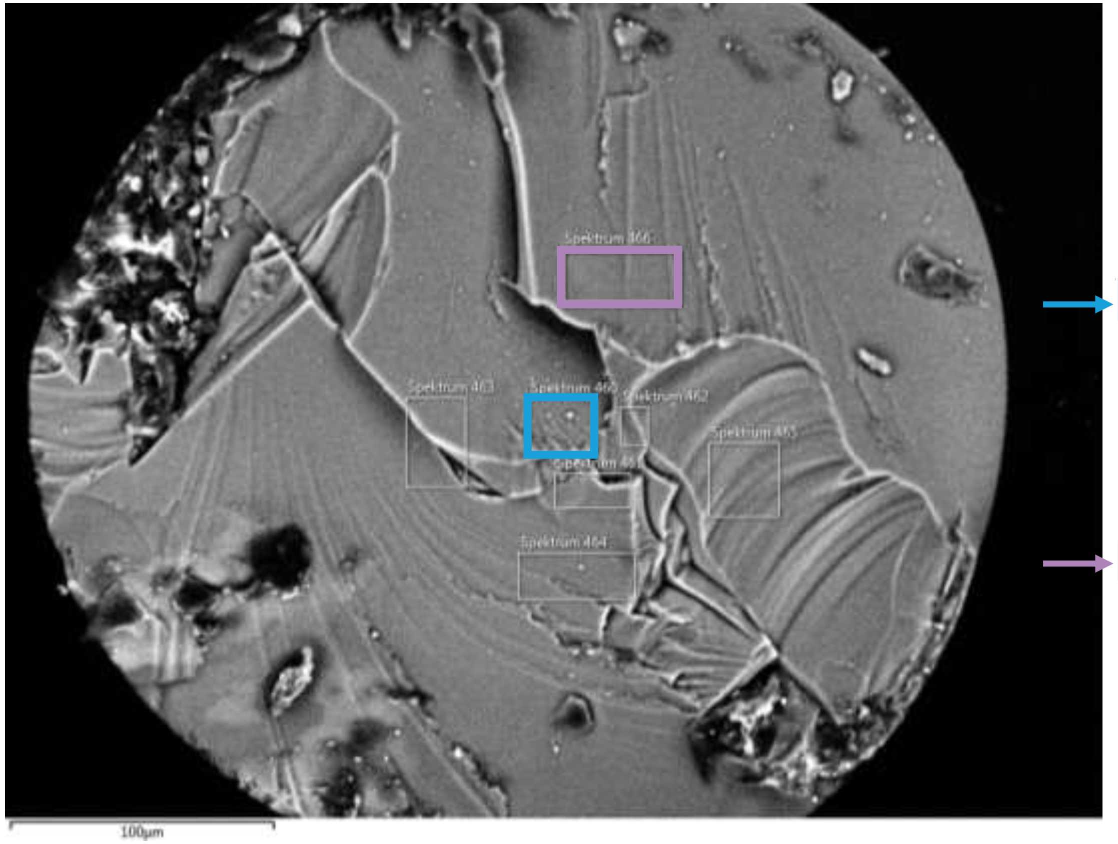
6. Elevated Alumina Fibres

EDX mapping

1) Crystal Sapphire core / SiO_2 cladding

Drawing Temperature: 1990°C

1a) Fibre piece of ca. Ø 350 µm and 50 mm length



Preform:
Passive core: **Al/Si**
Passive cladding: **Si**

Spectrum name	O	Al	Si	Total
Spektrum 460	49.02	23.55	27.44	100.00
Spektrum 461	48.69		51.31	100.00
Spektrum 462	54.04		45.96	100.00
Spektrum 463	55.57		44.43	100.00
Spektrum 464	44.46		55.54	100.00
Spektrum 465	49.59		50.41	100.00
Spektrum 466	57.03		42.97	100.00

EDX mapping of precursors in wt.%

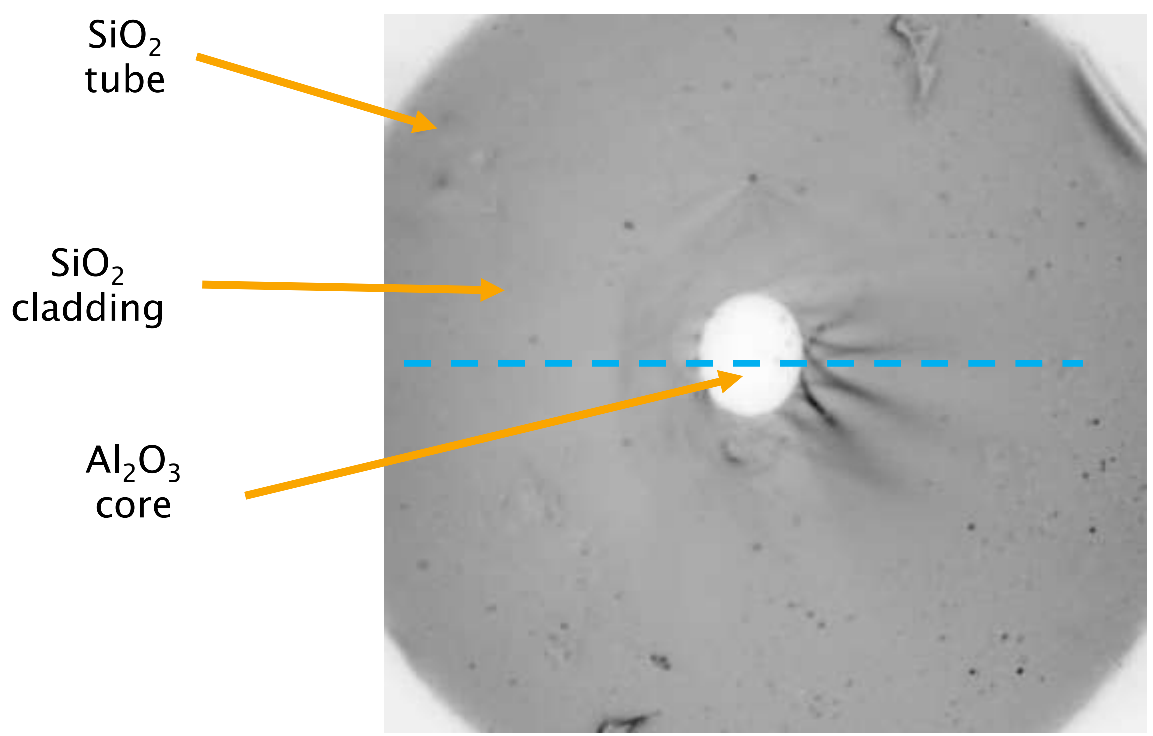
EDX proved the presence of elements within the areas, although not completely accurate for providing absolute values.

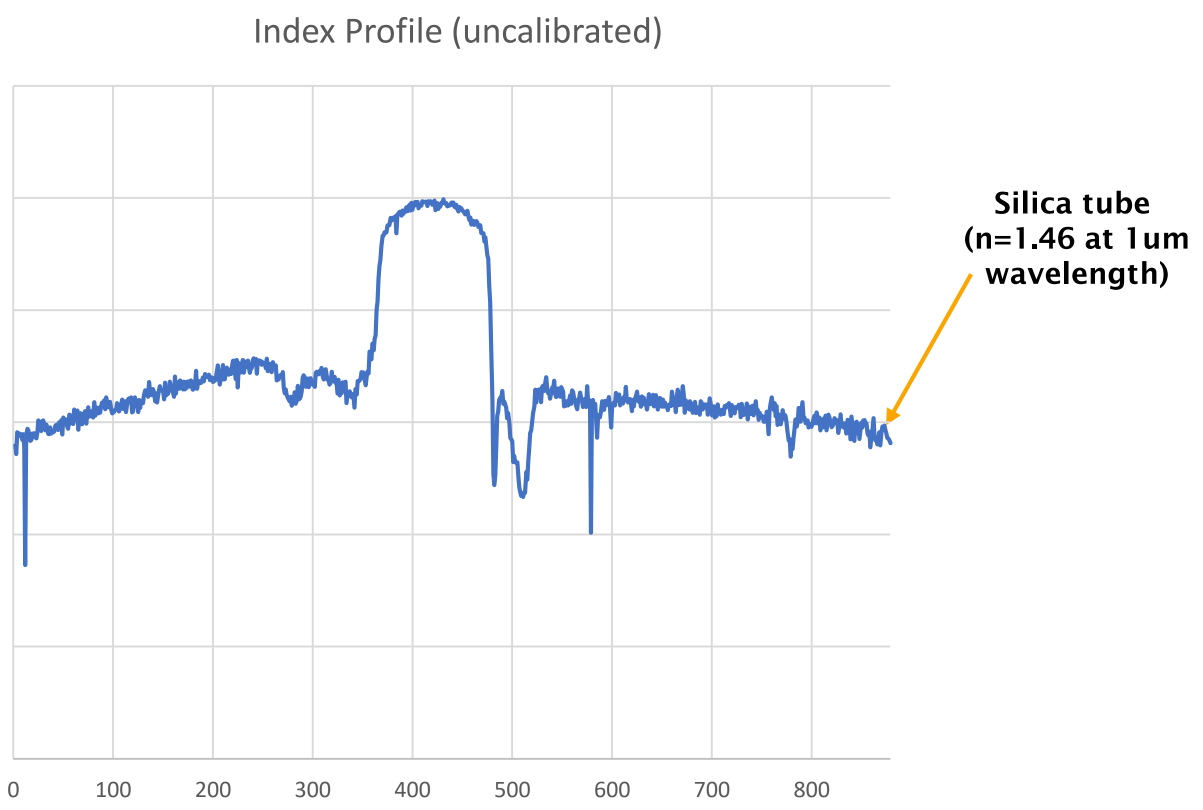
Refractive Index

1) Crystal Sapphire core / SiO_2 cladding

Drawing Temperature: 1990°C

1a) Fibre piece of ca. Ø 450 µm and 45 mm length



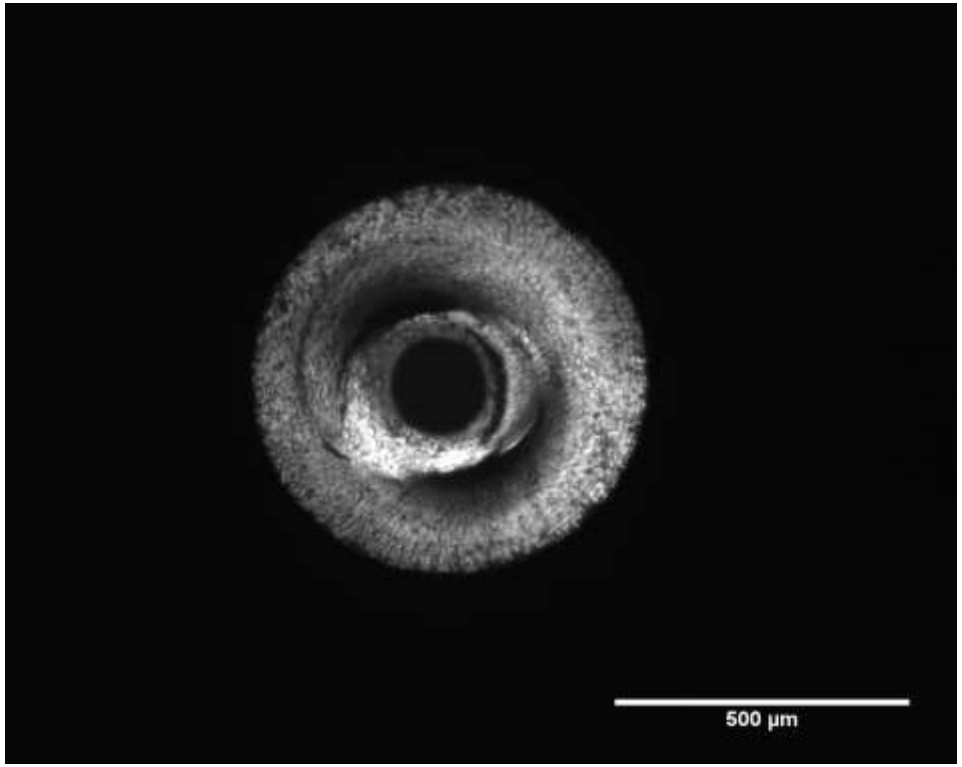


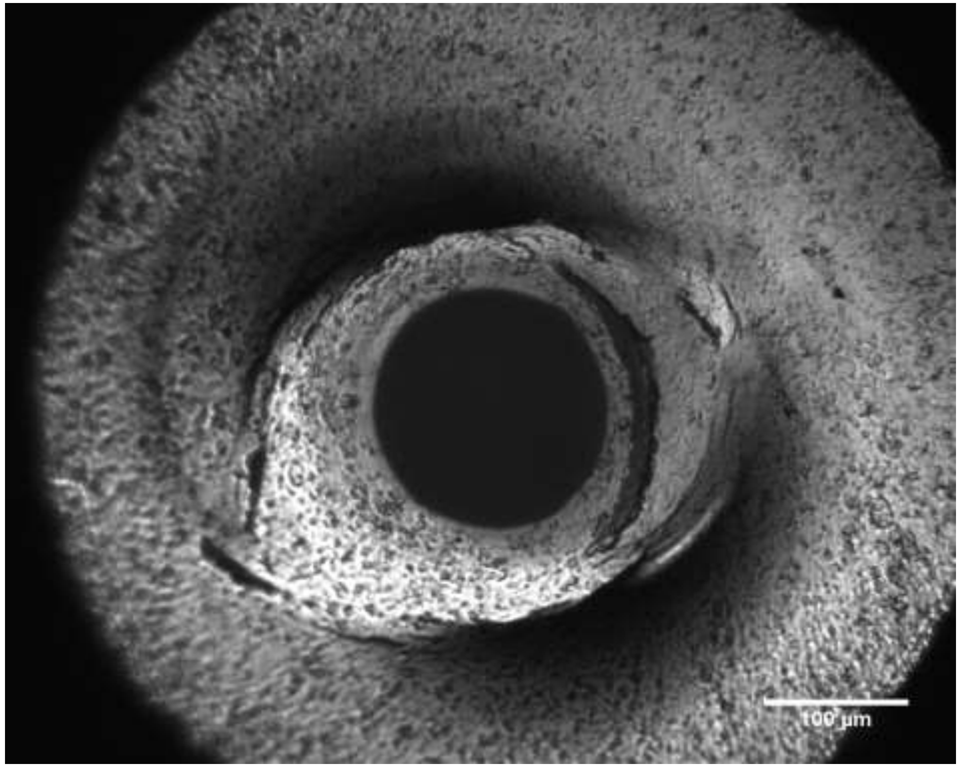
6. Elevated Alumina Fibres

1) Crystal Sapphire core / SiO_2 cladding

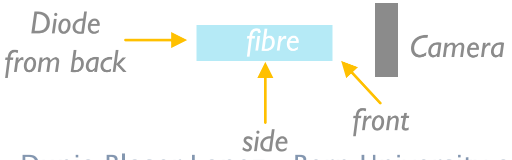
Drawing Temperature: 1990°C

1b) Fibre piece of ca. Ø 700 µm and 50 mm length





Fluorescence 915nm diode (1.51 V / 0.550 A) from back edge – no filtering



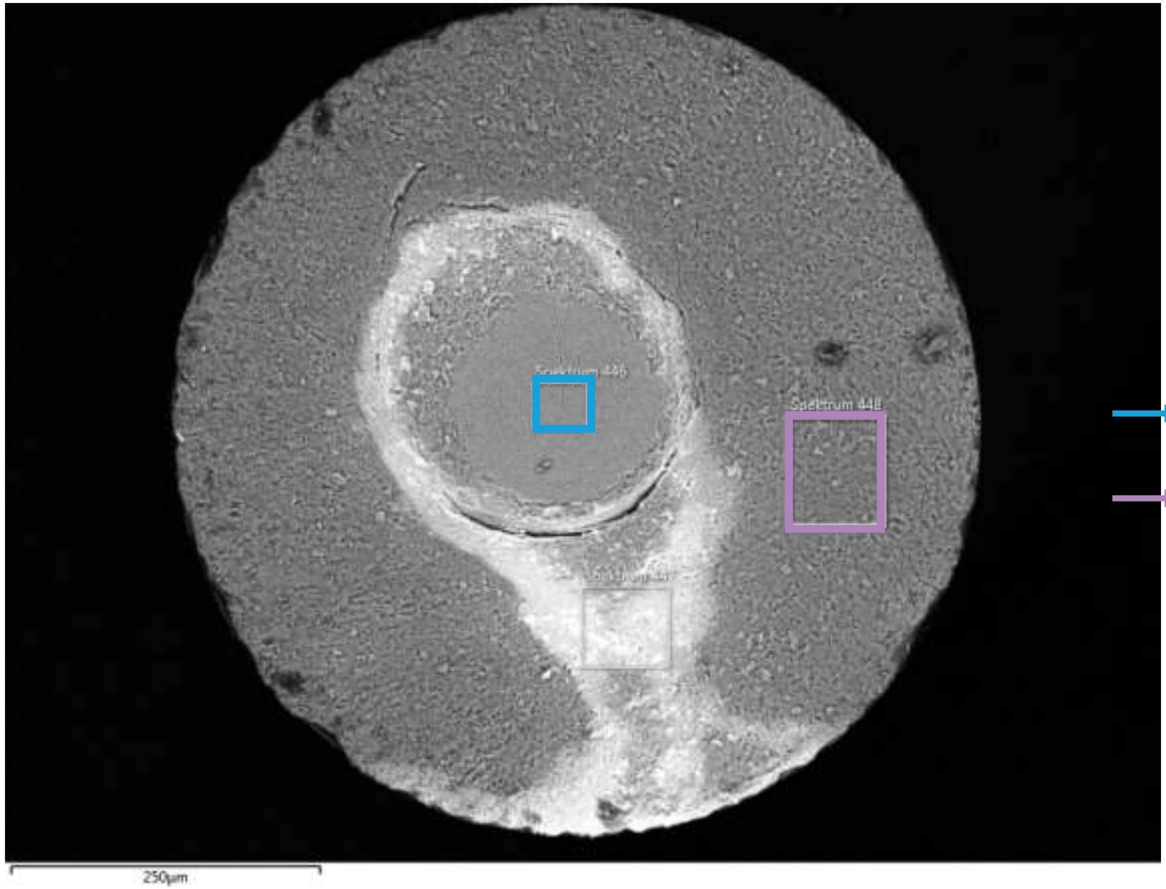
6. Elevated Alumina Fibres

EDX mapping

1) Crystal Sapphire core / SiO_2 cladding

Drawing Temperature: 1990°C

1b) Fibre piece of ca. Ø 700 µm and 50 mm length



Preform:
Passive core: **Al/Si**
Passive cladding: **Si**

Spectrum name	O	Al	Si	Total
Spektrum 446	47.18	40.38	12.44	100.00
Spektrum 447	63.21		36.79	100.00
Spektrum 448	51.48		48.52	100.00

EDX mapping of precursors in wt.%

EDX proved the presence of elements within the areas, although not completely accurate for providing absolute values.

6. Elevated Alumina Fibres

6.2 Conception of *Green compacts*

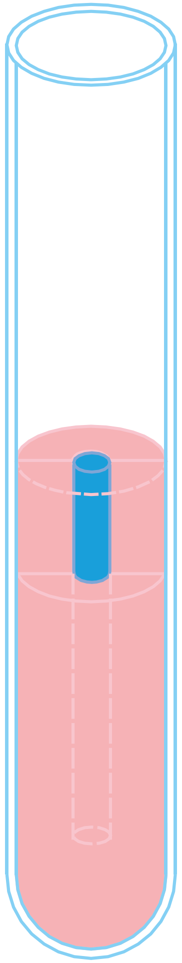
Further improvement

- **Green compacts**, where the granulated powder:
  - separated by **grain size**,
  - **fine powder mixing** (dopants addition),
  - **heat treatment**

- Enduring **hardness and compactness** of the powder, **minimizing** material **diffusion** due to the different densities during drawing a fibre.

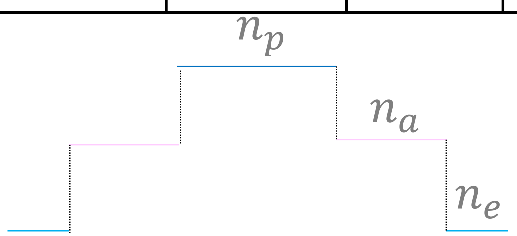
6. Elevated Alumina Fibres

6.3 Sapphire core rod and Yb3at.% cladding – marker



	active or passive	composition	base material	condition	Diameter *mm*	Length *mm*	region	refractive index
	Active	Yb/Si = 3/97at.%	Oxides	Sintered parts (Green compact - heat treated) gaps filled with loose powder	16	130	Cladding	n_a
	Passive	Sapphire	Sapphire	Rod	2	100	Core	n_p
	Passive	Si = 100at.%	Silica tube	Envelope (tube)	18/21	-	-	n_e

Drawing Temperature: 2010°C



Index Profile

6. Elevated Alumina Fibres

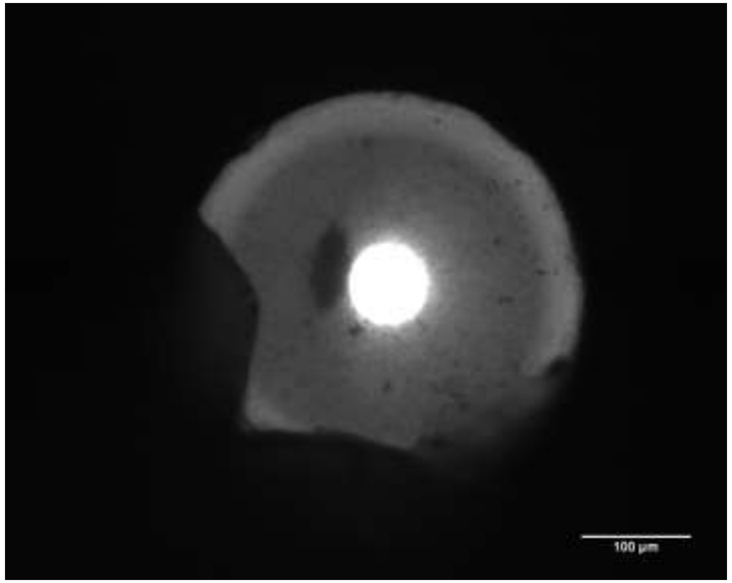
2) Crystal Sapphire core / Yb3at.% cladding

Drawing Temperature: 2010°C (temperature on outer wall of heating)

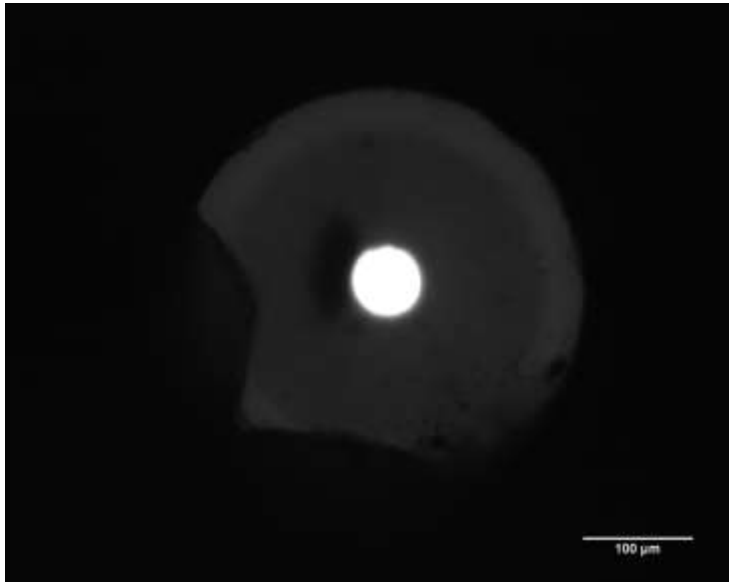
2a) Fibre piece of ca. Ø 350 µm and 45 mm length

long pass filter: FELH0950

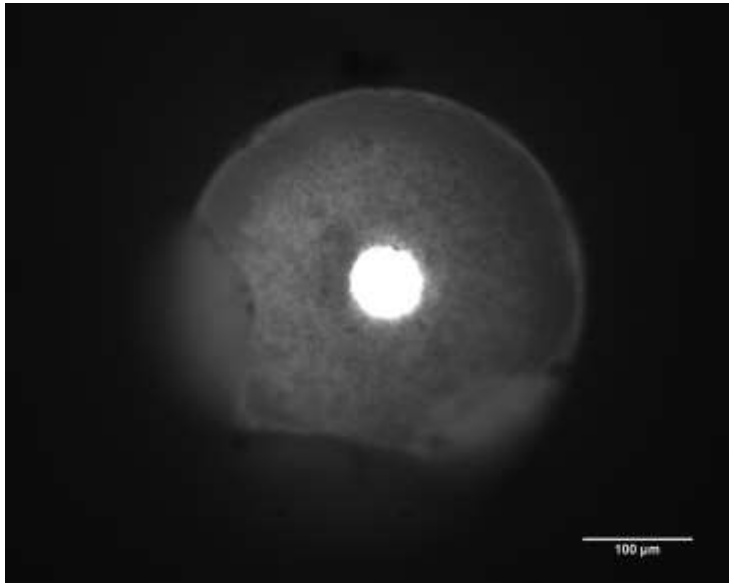
back

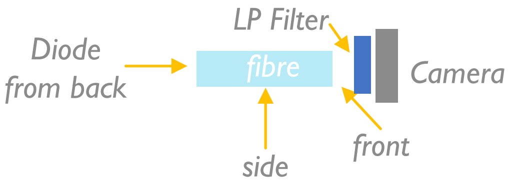


side



front





915nm diode (1.85 V) from back edge + FELH0950

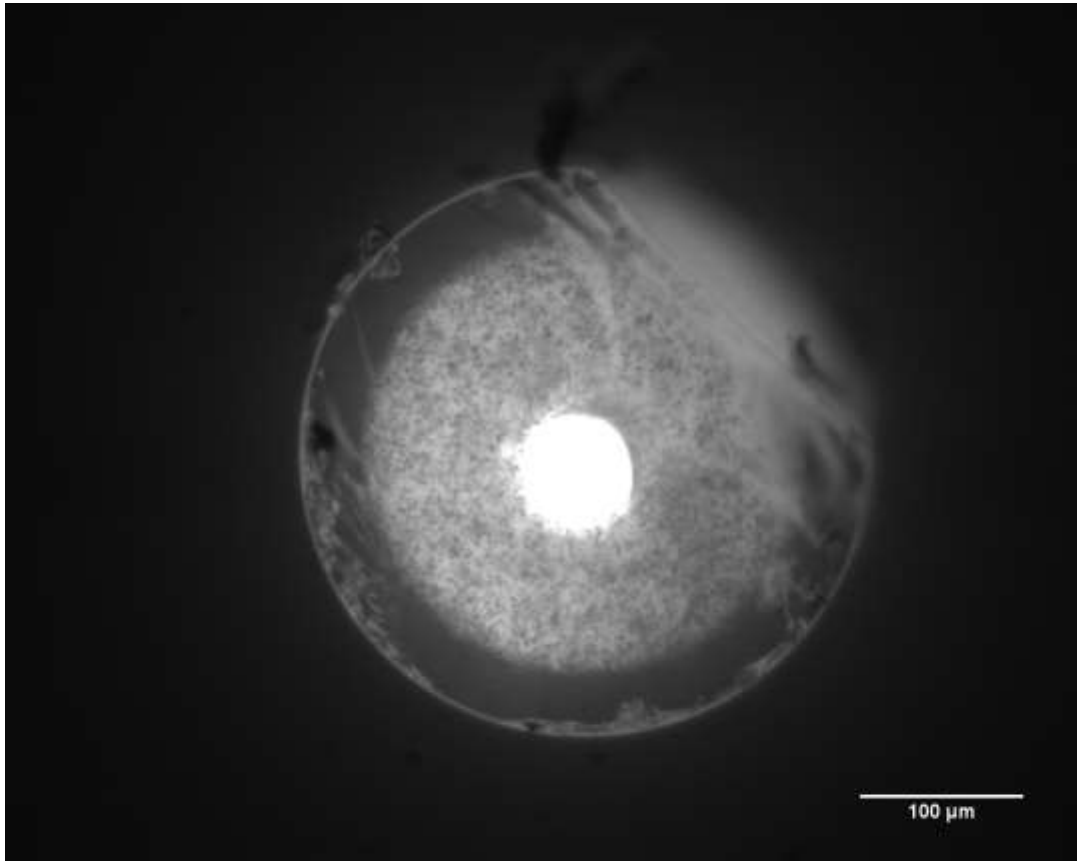
6. Elevated Alumina Fibres

2) Alumina core / Yb3at.% cladding

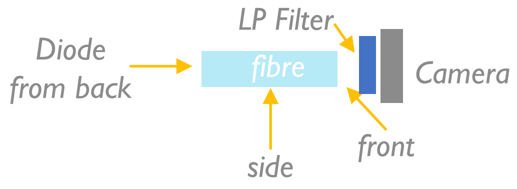
Drawing Temperature: 2010°C (temperature on outer wall of heating)

2b) Fibre piece of ca. Ø 350 µm and **820 mm** length

long pass filter: FELH0950



915nm diode (1.85 V) from back edge



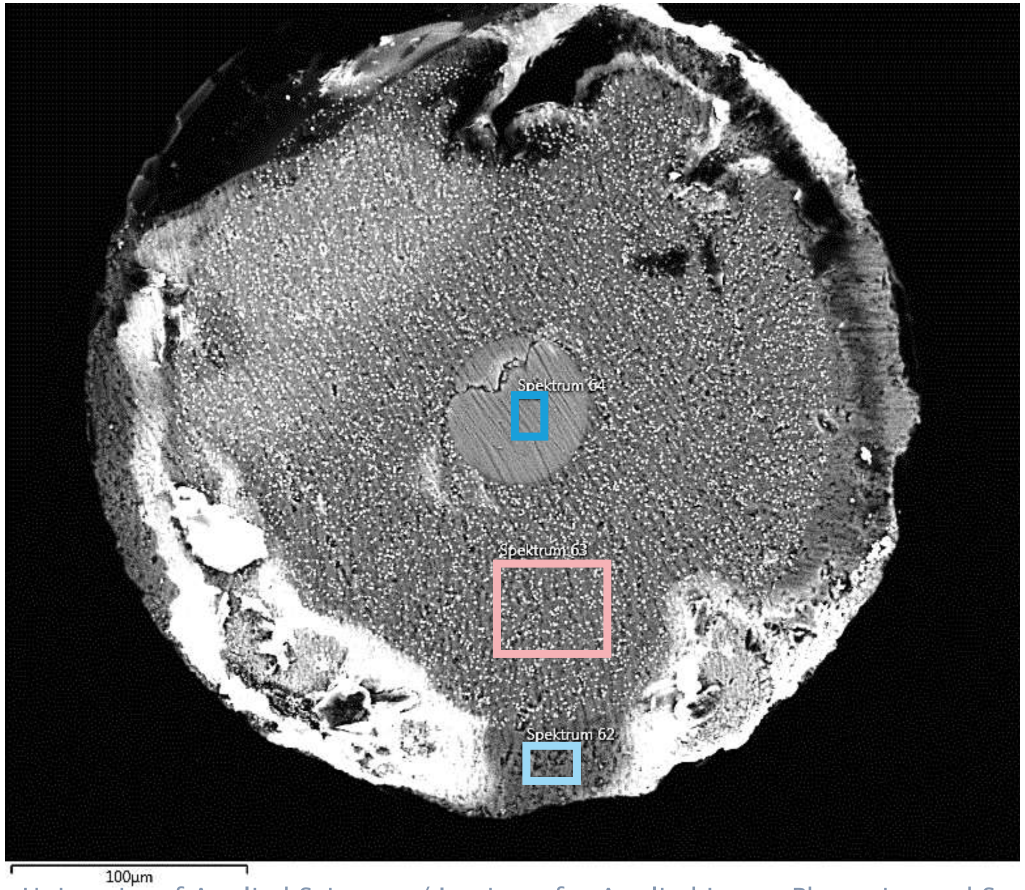
6. Elevated Alumina Fibres

EDX mapping

2) Crystal Sapphire core / Yb3at.% cladding

Drawing Temperature: 2010°C (temperature on outer wall of heating)

2a) Fibre piece of ca. Ø 350 µm and 45 mm length



Preform:
Passive core: **Al/Si**
Active cladding: Yb/**Si**
Passive envelope: **Si**

Spectrum number	Al at.%	Si at.%	Yb at.%	Total at.%
Spectrum 62		100.00		100.00
Spectrum 63		98.28	1.72	100.00
Spectrum 64	71.95	27.15	0.90	100.00

EDX mapping of precursors in wt.%

EDX proved the presence of elements within the areas, although not completely accurate for providing absolute values.

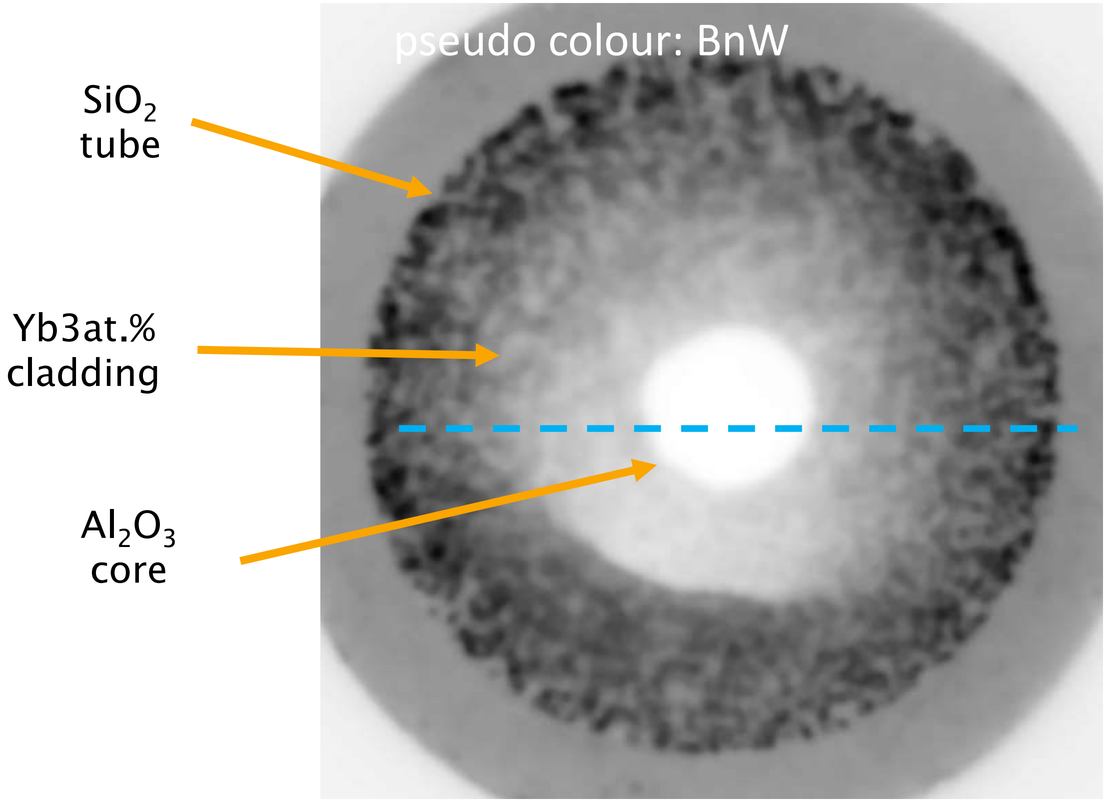
6. Elevated Alumina Fibres

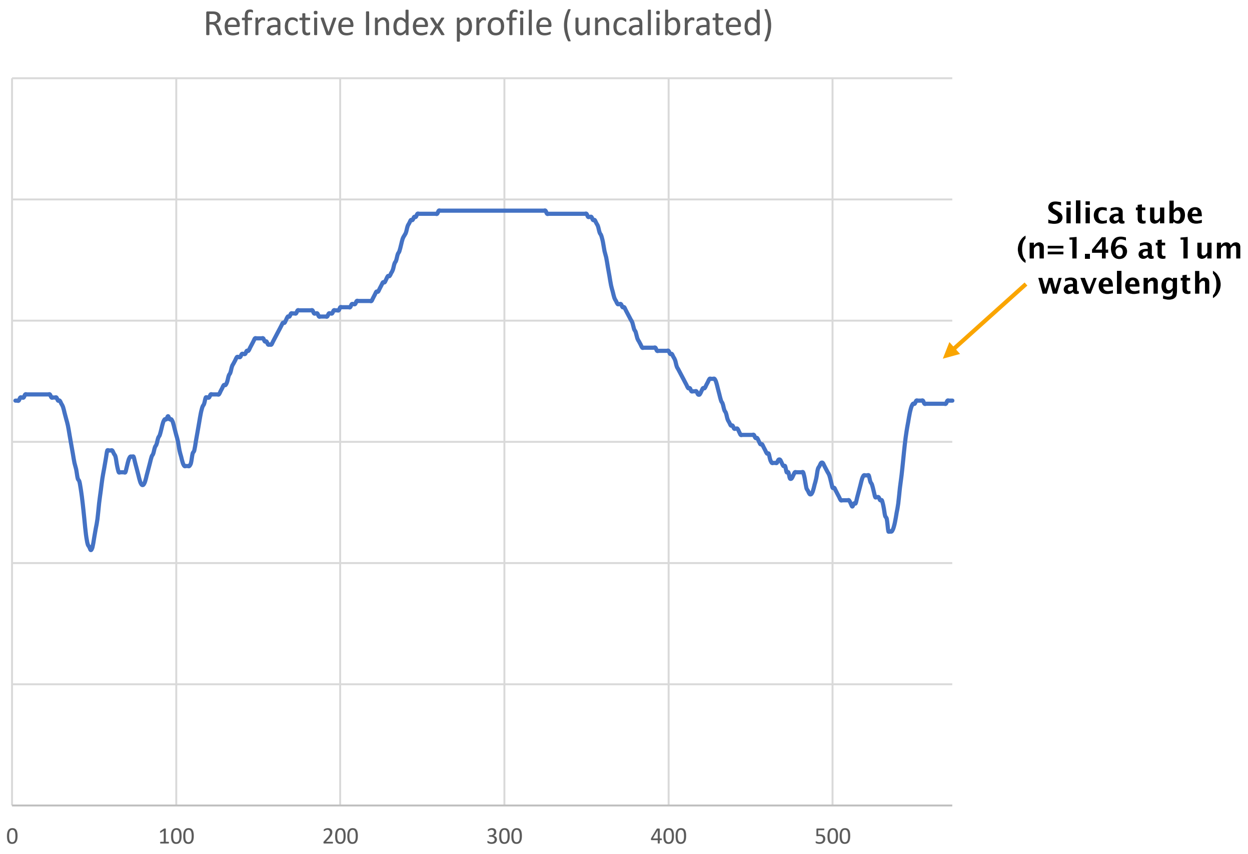
Refractive Index

2) Crystal Sapphire core / Yb3at.% cladding

Drawing Temperature: 2010°C

2b) Fibre piece of ca. Ø 350 µm and 45 mm length





7. Outlook

- Future steps of the research **targets two points**:
  1. **Fibre development:**
     - Fibre structure with high Alumina content as cladding (e.g. 10% $SiO2$)
     - Study and characterization of multiple high alumina cores, in order to understand the light behaviour and scalation of power delivery.

  2. **Testing:** fibre in a real medical infrared laser set-up, in order to confirm the implementation in the targeted applications.

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