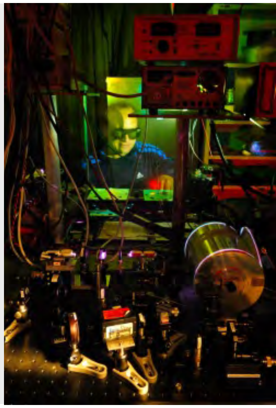
Capabilities of the Swiss granulated silica fiber research effort



presented by Valerio Romano[1,2]

[1]Institute of Applied Physics,University of Bern, Sidlerstrasse 5, 3012 Bern, Switzerland,
[2]ALPS Institute, Bern University of Applied Sciences, Pestalozzistrasse 20, 3400 Switzerland

in collaboration with APRI, GIST, 1 Oryong-dong, Buk-gu, Gwangju 500-712,Republic of Korea

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Berner Fachhochschule
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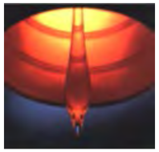

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SCHWEIZERISCHER NATIONALFONDS
ZUR FÖRDERUNG DER WISSENSCHAFTLICHEN FORSCHUNG

UNIVERSITÄT BERN

Content

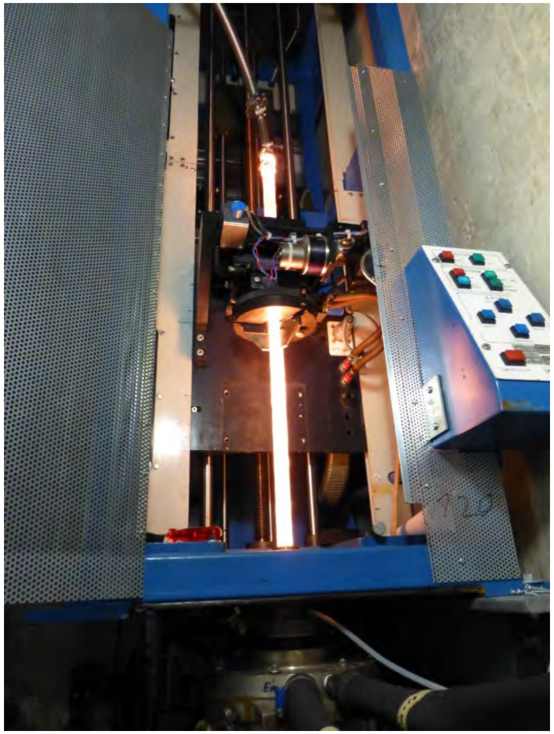
- 1) Team tasks and resources
- 2) Alternative method: granulated silica method and examples of drawn fibers
- 3) Improvement of homogeneity and loss properties by iterative milling/remelting
- 4) Homogeneization by Sol-Gel production of granulated silica
- 5) Summary and conclusions

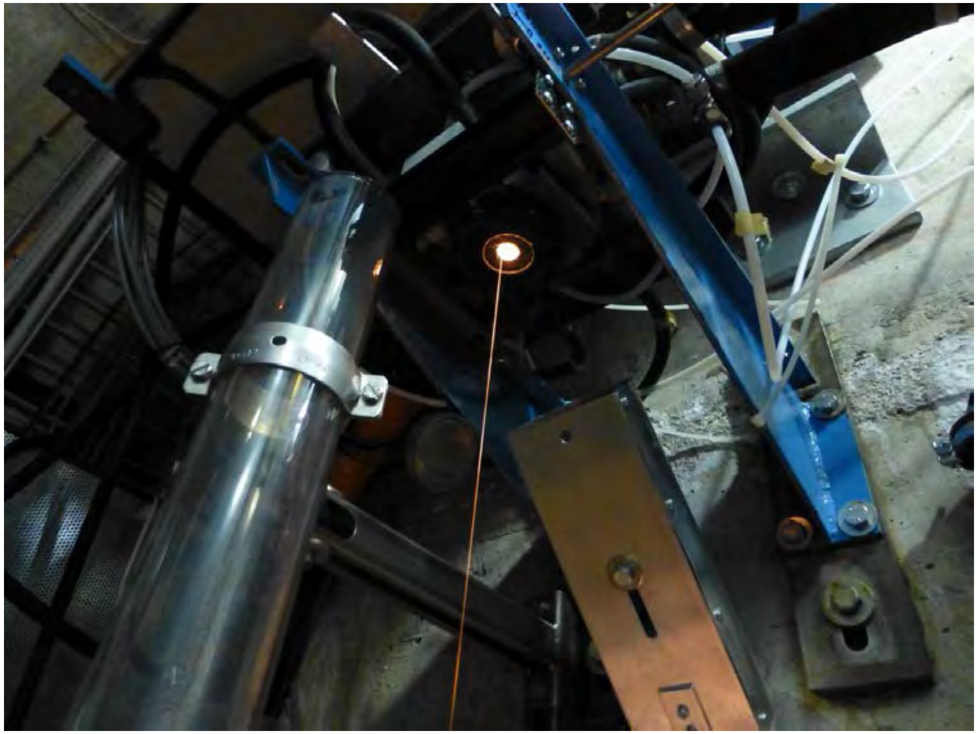
1) Team and tasks (IAP and BFH-ALPS)

- Fiber systems : M. Ryser, Ch. Bacher
- Microstructured fibers: J. Scheuner
- Fibers, fiber drawing: All
- Analysis: Ph. Raisin, A. El Sayed, H. Najafi
- Fiber handling: J. Boas
- Fiber Materials: S. Pilz, D. Kummer, (D. Etissa)
- Applications: A. Burn, B. Bessire, Ch. Heger

1 b) Equipment / other ressources

- 2 fully equipped optical labs
- Fiber drawing tower
  - Operated by both institutions
  - "Refurbished" this year jointly by IAP and BFHwith help
- Splicers, cleavers, recoaters in both labs
- Analysis equipment (in house or in collaboration with other institutions, e.g. EPFL)



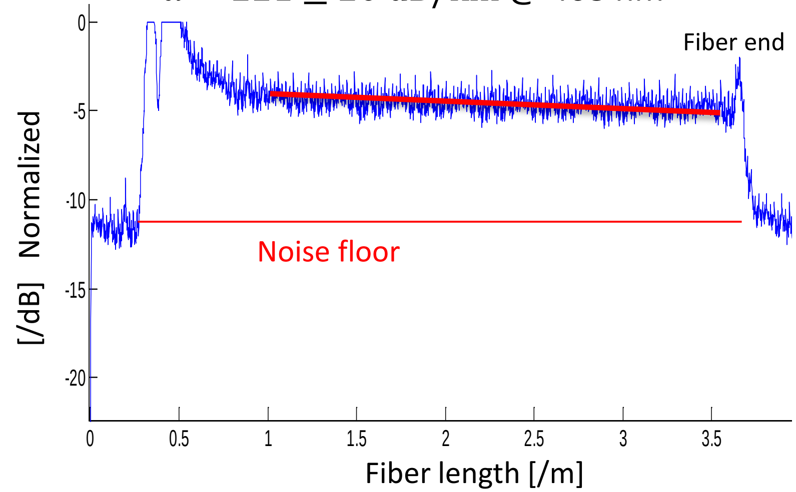


1c) Other resources: High spatial-resolution OTDR

- Picosecond pulsed laser diode @ 468 nm
  Pulse-width = 60 ps
- Single photon avalanche diode (SPAD)
  Times resolution = 50 ps
- Time correlated single photon counting module (TCSPC)

In-house Erbium doped fiber. 3 m length

$\alpha = 221 \pm 20$ dB/km @ 468 nm

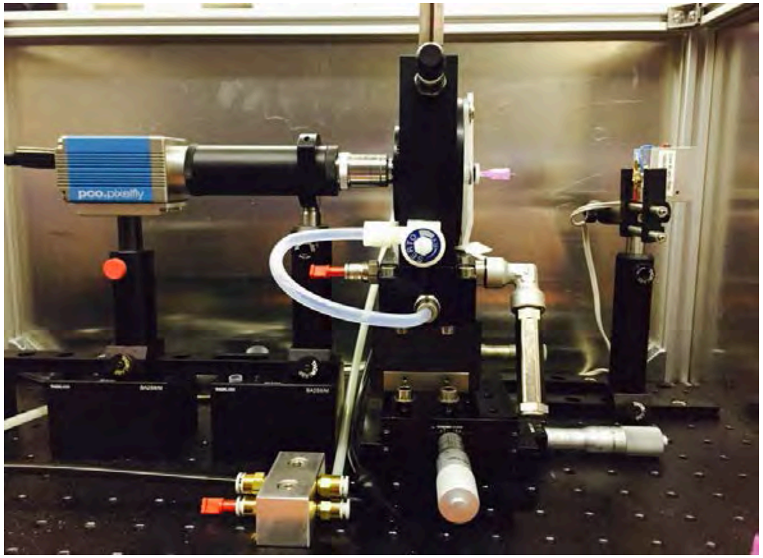


- Non-destructive technique
- Measurement of the attenuation coefficient of short fibers
- Measurement of losses at localized discontinuities such as, splices and connectors
- Spatial resolution down to 1 cm

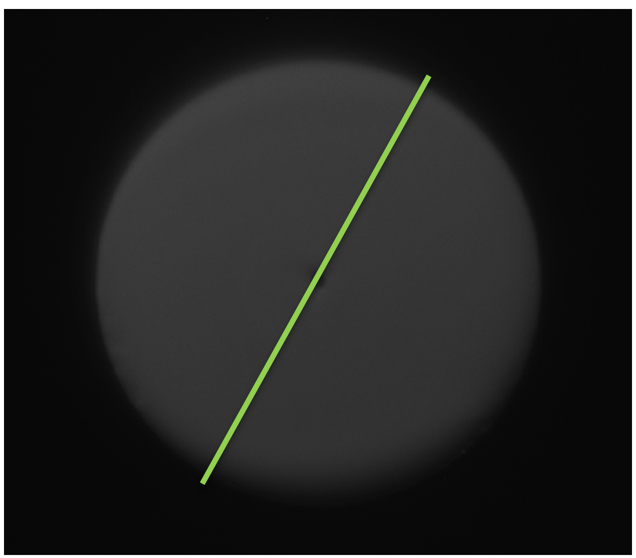
(El Sayed, Master Thesis, IAP 2014)

1d) Other resources: Imaging refractive index profiling

Based on the refractive near field technique (RNF)

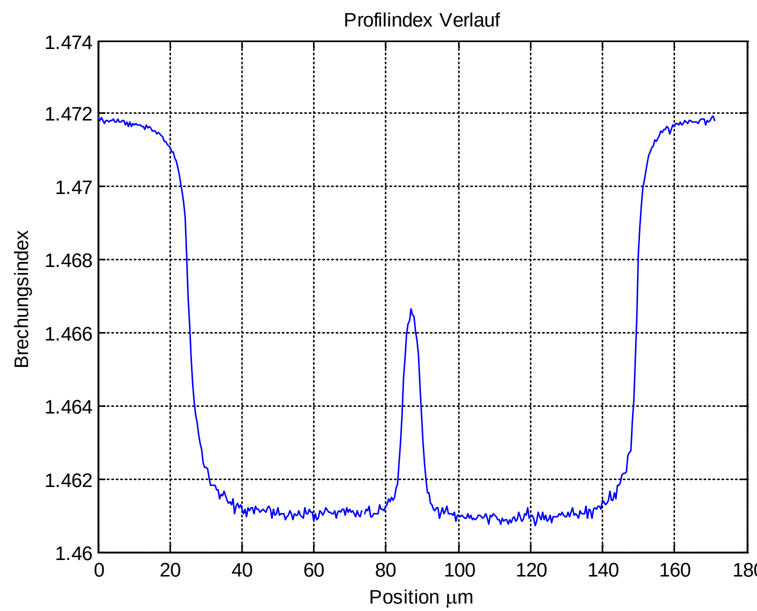


- 2-D refractive index profile
- No scanning is required
  - Robust
  - Fast measurements
  - Allows averaging
- Accuracy in the range of $< 10^{-3}$

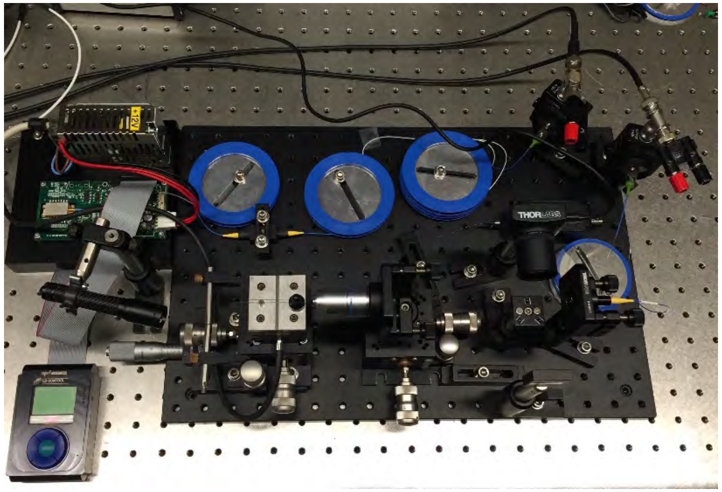


(El Sayed, BFH, 2015)

Δn = 0.0061 ± 0.0005

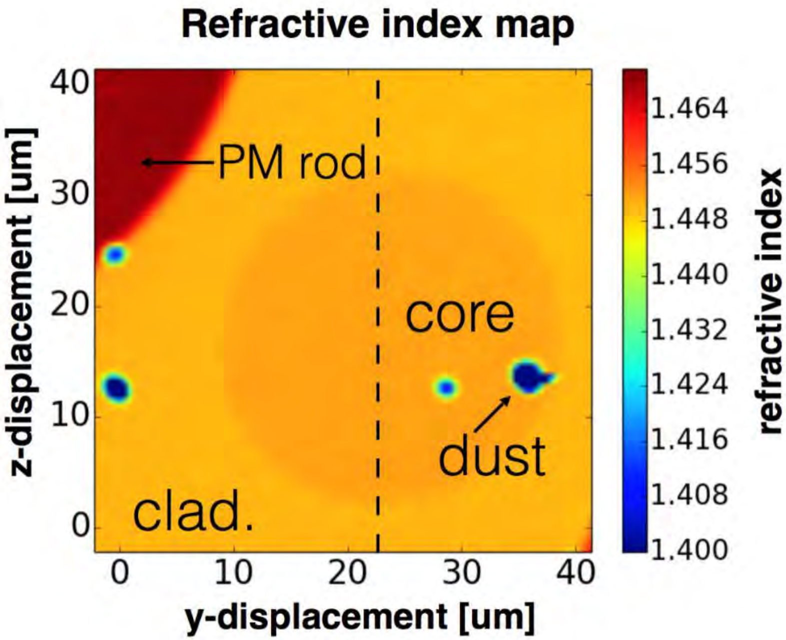


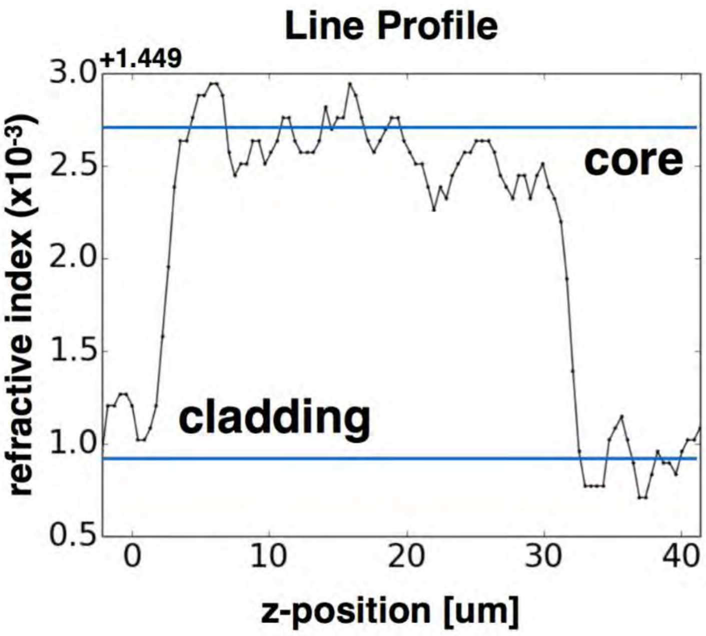
1e) Other resources: Scanning 2-D Refractive index measurement



Capabilities of refractive index setup

- Accuracy < 10^{-3}
- Precision limit: currently ~10^{-4}.
- Spatial resolution <2μm
- Minimal sample preparation is needed (cleave)
- confocal scanning technique: scan time typically 3-4h.



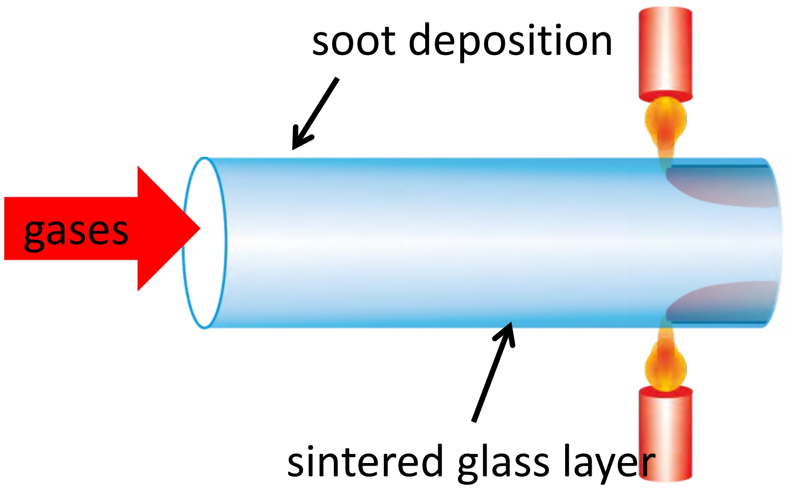


(Scheuner, Raisin, IAP 2013 ... 2015)

2.0 Limitations of standard preform production

Chemical Vapor Deposition methods
(MCVD, VAD, OVD, PCVD, IMCVD)

✓ highest quality, very low scattering losses (0.6dB/km@1100nm, 0.18dB/km@1550nm) for passive fibers (only Germanium co-dopin)



✗ not very versatile:
> difficult to fabricate large homogeneous cores
> Best suited for shapes with cylindrical symmetry
> relatively big technical effort/time consuming

> Standard production methods do not offer enough freedom with respect to dopants and microstructure at "affordable" cost and effort.
Example: trigonal fiber array produced by "creative" standard method.

2.1 Alternative approach: which Fiber Materials?

> Stick to silica

- because of "good" fiber drawing properties

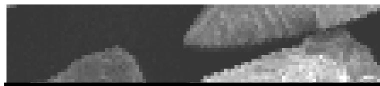
> Add same dopants / Co-dopants as in mCVD for:

- activation (Rare Earths, some transition metals e.g. Bi)
- increasing solubility of RE (Aluminum)
- mitigating photodarkening (Phosphorus)
- increasing refractive index (TiO_2)
- ……

2.2 Alternative Approach: Granulated silica method

- proof of principle

100µm

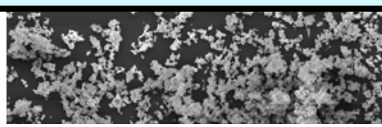


SiO_2

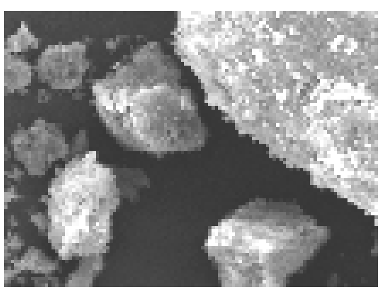
The melting points of Al_2O_3 and RE_2O_3 are between 2054 °C and 2435 °C.
At the drawing temperature of ~1850 °C no melting occurs!

→ soluble in SiO_2 ? -> YES

100µm

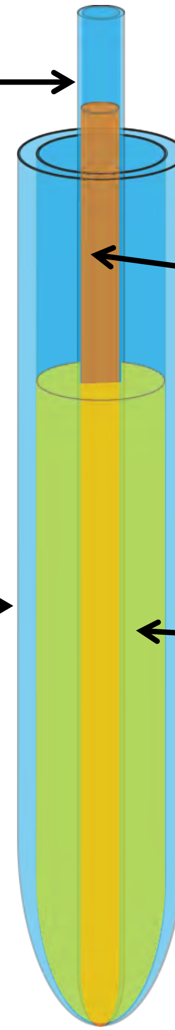


100µm



Al_2O_3
(improve solubility)

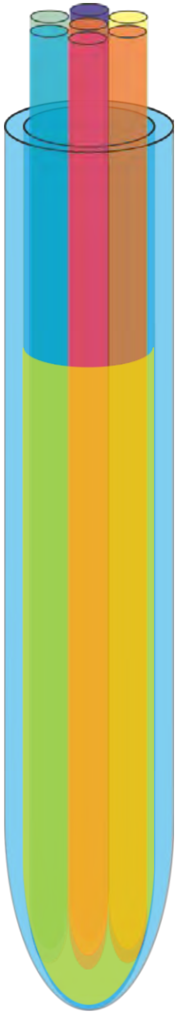
$+P_2O_3$
(reduce photodarkening)

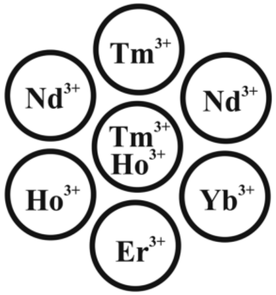


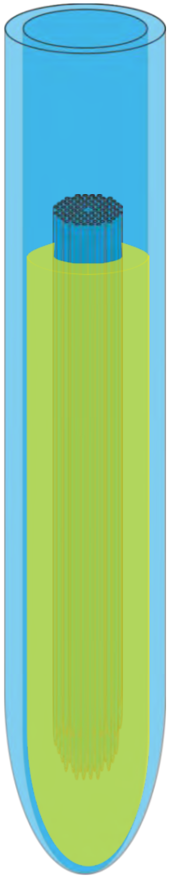
R. Renner-Erny, L. Di Labio, and W. Lüthy,
"A novel technique for active fibre production," *Optical Materials*, vol. 29, no. 8, pp. 919–922, Apr. 2007.

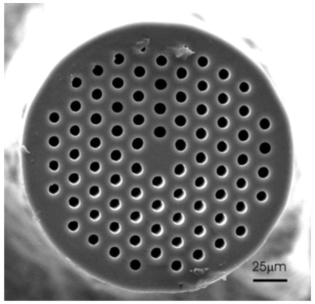
2.3 Special features by granulated silica-method

- rapid preform/fiber prototyping



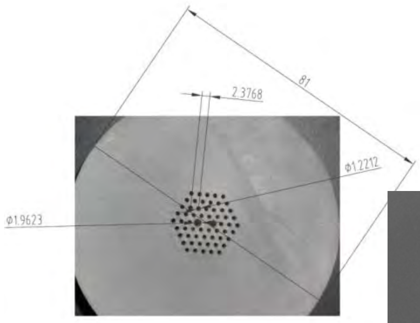


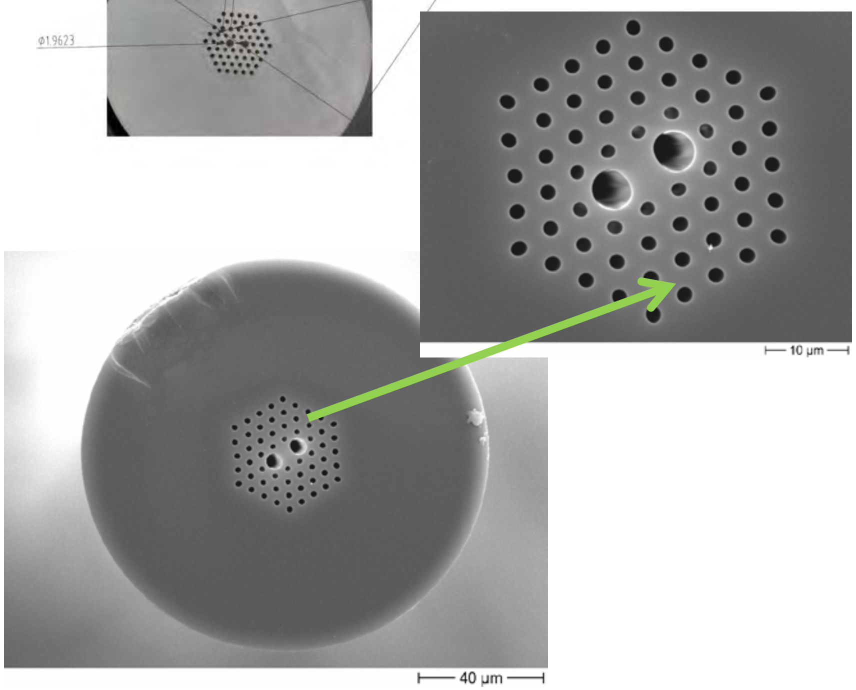


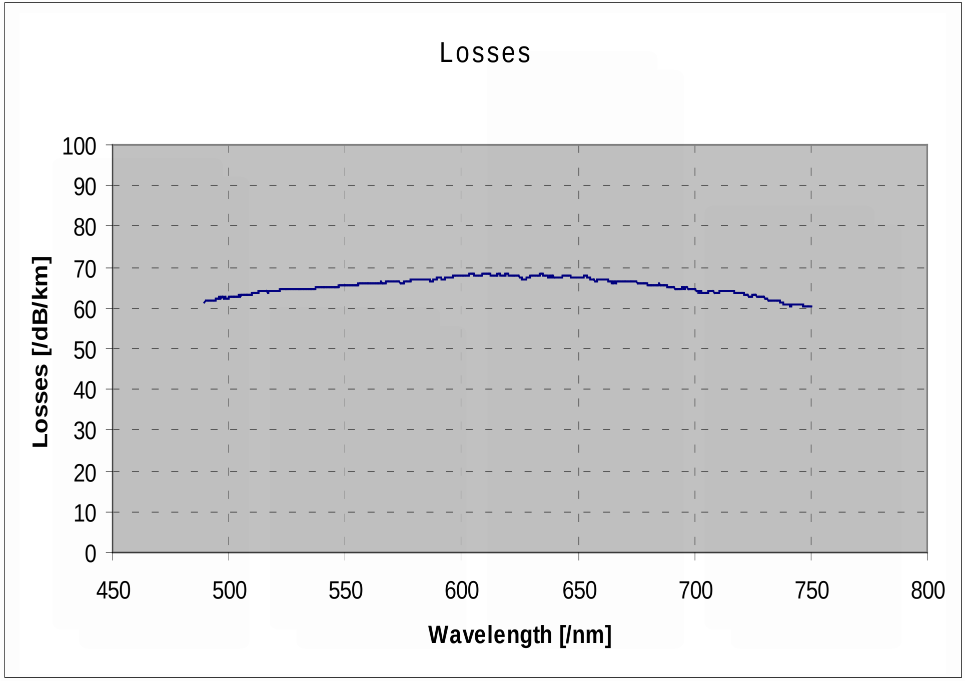


2.4 Example:Fibers: Polarization maintaining PCF

- **Fiber diam.: 125.0 µm**
- **Core: ~4 µm x 6 µm**
- **Hole diameter: 1.5 µm**





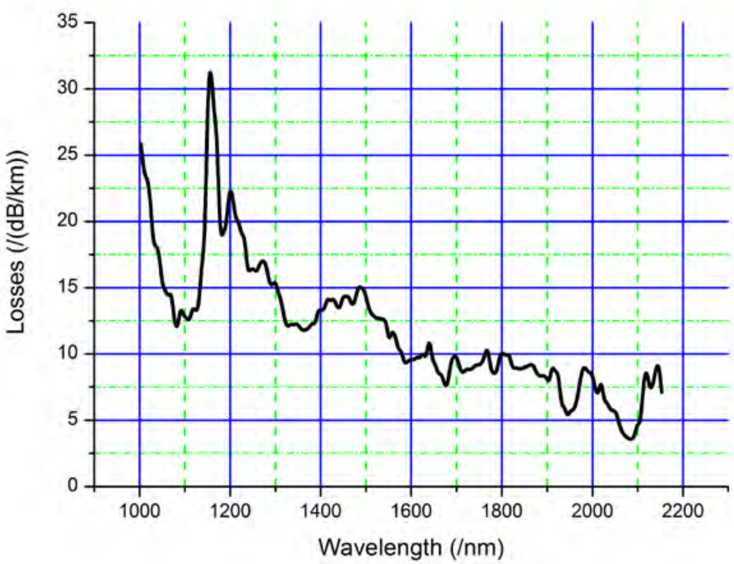


- **Flat spectrum in vis.**
- **Polarization maintaining**
- **(almost) single mode in complete range**

2.5 Example: Large core PCF with low OH-core

Fiber diameter:	**170µm**
Core diameter (d):	**21 µm**
Hole diameter:	**7 µm**
Pitch:	**16 µm**
d/pitch:	**0.46**

Attenuation [/dB/km]



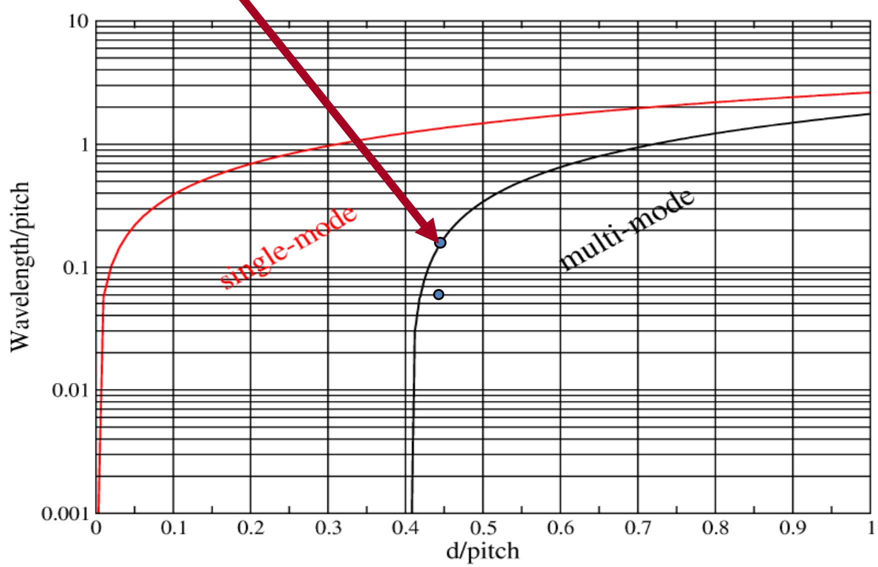
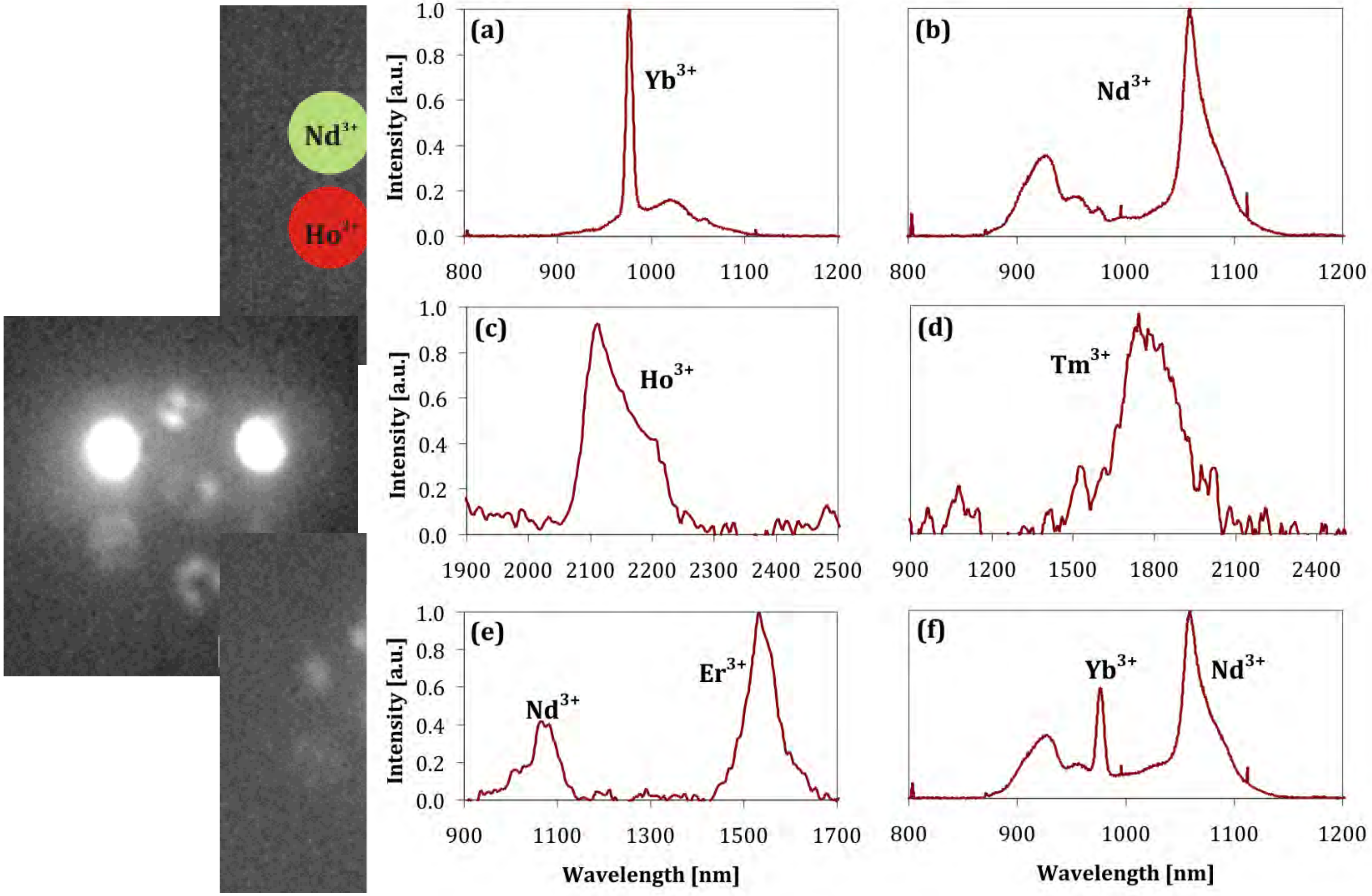


Figure 6: Cut-off phase-diagram

Fiber drawn at Silitec SA, Boudry

2.6 Example: 7-core fiber

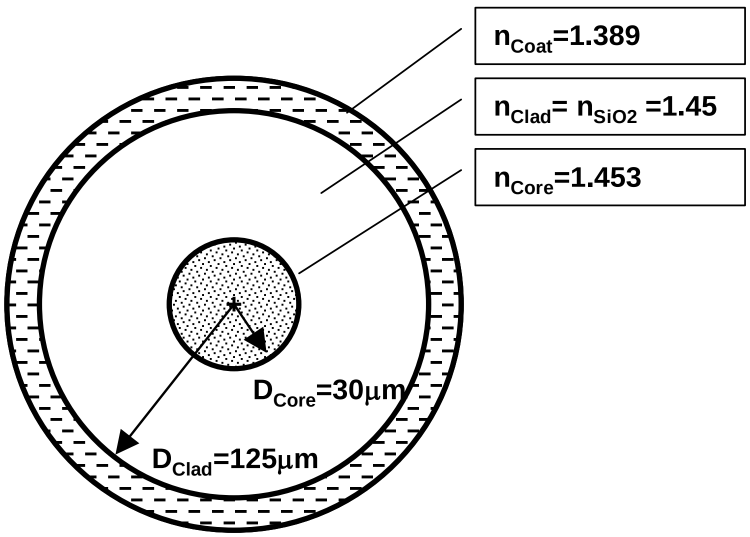


Di Labio, L., Lüthy, W., Romano, V., Sandoz, F., & Feurer, T. (2008).
Broadband emission from a multicore fiber fabricated with granulated oxides.*Applied optics*, *47*(10), 1581-1584.

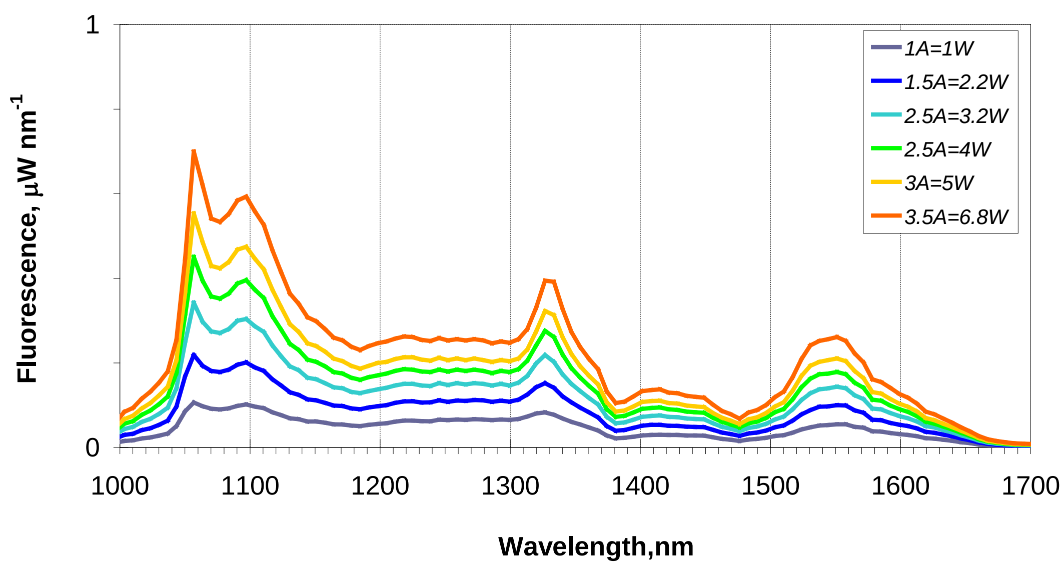
2.8) Example: Superbroadband double clad fluorescence source

Dopant Concentration [relative to atoms of silicon]	
Al_2O_3	3.0%
Bismuth	0.4%
Erbium	0.04%
Neodymium	0.05%

Features:
- Double Clad for higher pumping
- Addition of Bismuth to fill „dip“ between 1100 nm and 1330nm



Length of DCF = 16.6m, pumped at 808nm



High background losses: up to 5dB / m expected

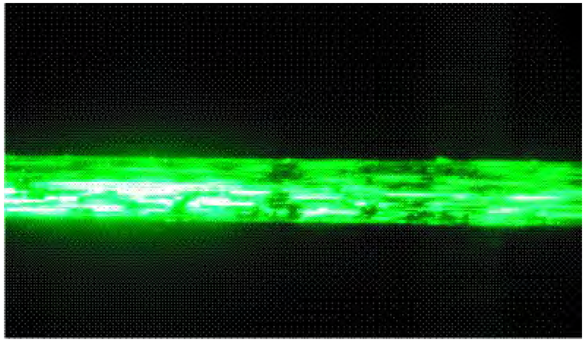
Pilz, S., Etissa, D., Barbosa, C., & Romano, V. , *INFRARED BROADBAND SOURCE FROM 1000NM TO 1700NM, BASED ON AN ERBIUM, NEODYMIUM AND BISMUTH DOPED DOUBLE-CLAD FIBER.* Proceedings of the ALT’12, DOI: 10.12684/alt.1.73

3.0) Granulated silica method: improving homogeneity

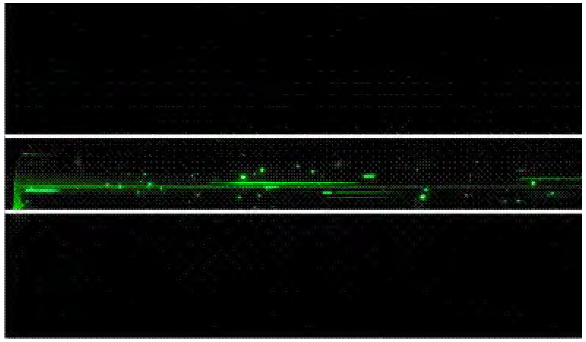
Probing with Erbium

***Green fluorescence from** the* Er^{3+} $^4S_{3/2} \rightarrow {}^4I_{15/3}$ *transition is excited by* ***energy transfer up-conversion (ETU)** pumping with a diode laser operated at 975 nm.*

Er^{3+}



Multi-mode fibre produced ***without melting and milling*** *(strongly over-exposed)*



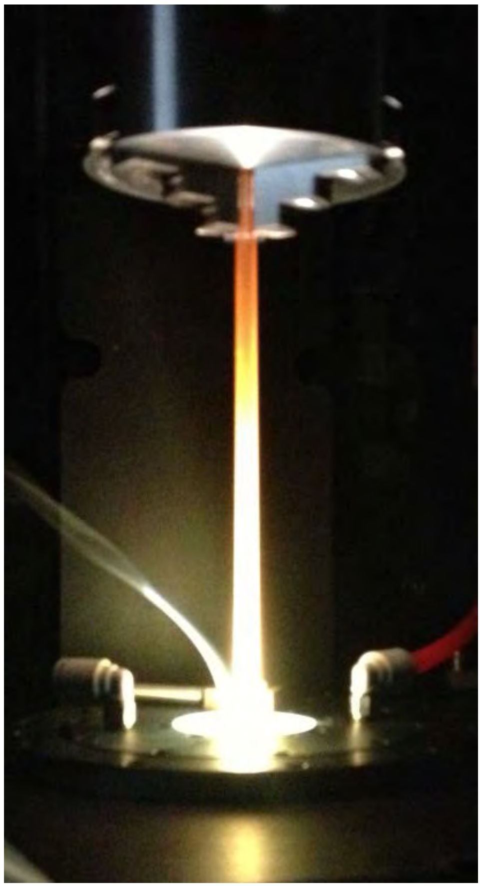
Multi-mode fibre produced ***with melting and milling applied twice**.* *The white lines indicate the position of the fibre.*

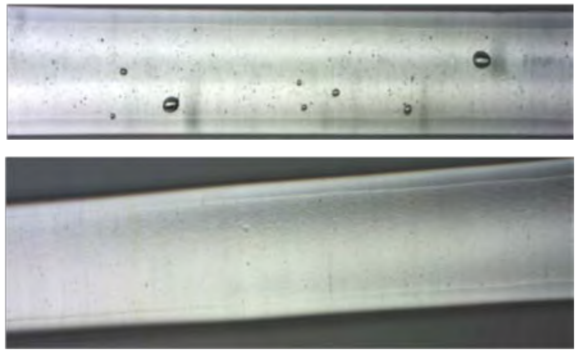
→ solution to increase homogeneity: Sol-Gel material
Result: piecewise 0.35dB/m @ 633nm

3.1) Further improvement

Laser-based travelling small zone vitrification

Significant improvement in losses



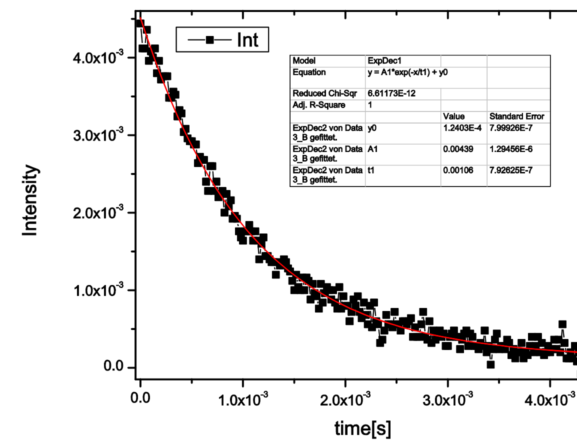


Core preform rods: 1mm diam.

«Piecewise» 035 dB/m @633nm
Standard vitrification (2012)

0.2 ...0.3 dB/m @633nm (Sept. 2013)
laser small zone vitrification

High fluorescence lifetime :τ > 1 ms



Quality: OK
Commercially exploitable: No (small production yield)

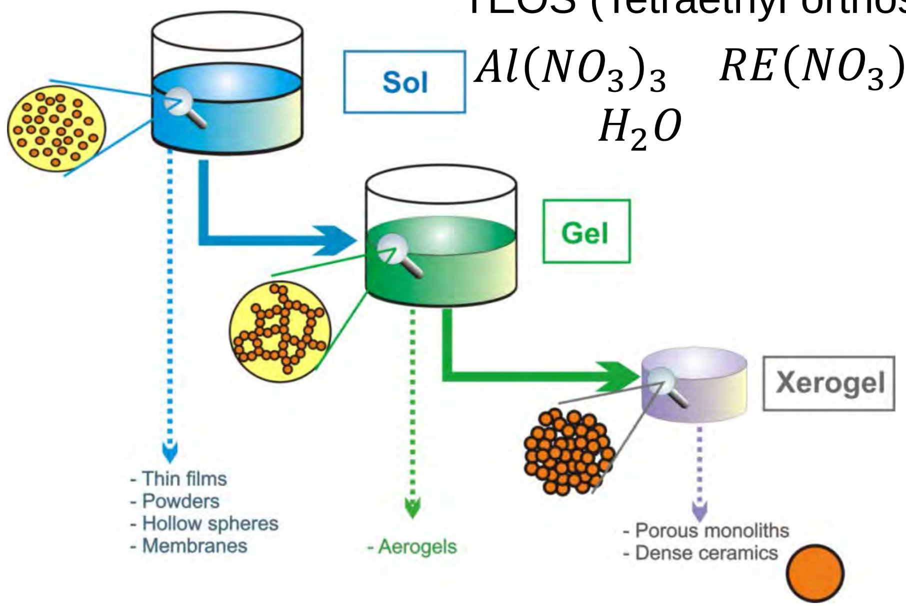
4.1) Improved homogeneity by Sol-Gel

- Sol-gel and granulated silica method

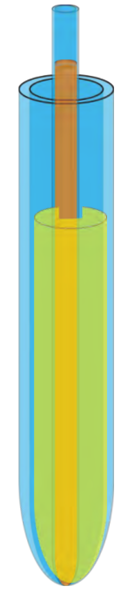
TEOS (Tetraethyl orthosilicate)

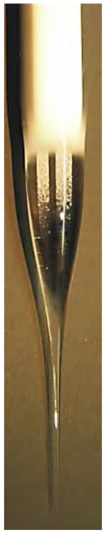
$Al(NO_3)_3$ $RE(NO_3)_3$

H_2O



produce granulate: all grains are homogeneously doped!



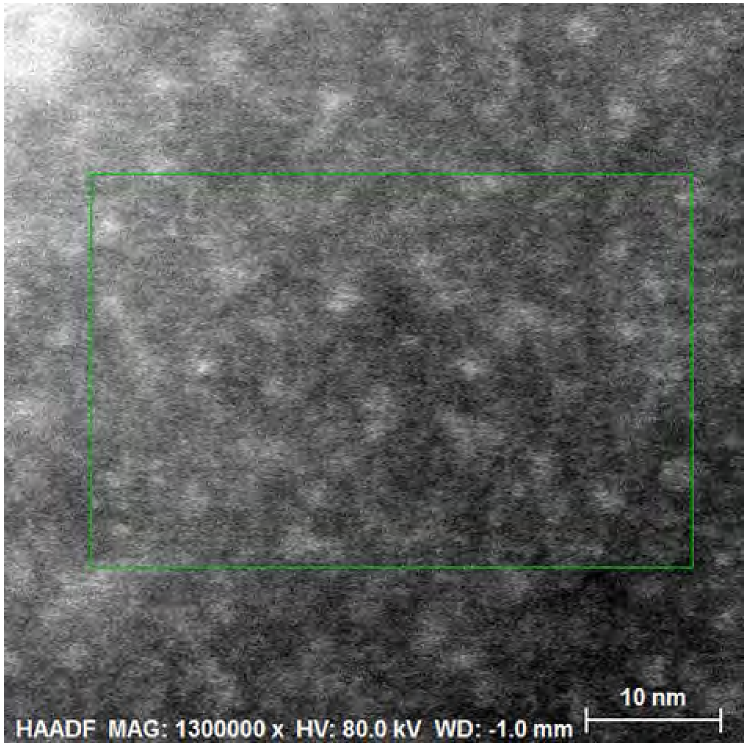


- ✓ rapid prototyping and manufacturing
- ✓ very cost-effective
- ✗ possible scattering losses
- ✗ ~~inhomogeneous distributed dopants~~

- ✓ 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
- ✗ wet chemical process → OH groups (large SiOH absorption @ 950nm, 1240nm, 1390nm)

4.2 HRTEM

Titan Themis (EPFL)



High resolution TEM analysis of Yb-Al-P - doped samples show that the distribution of Ytterbium is homogeneous down to the nanometer scale.

(Najafi, BFH 2015, in collaboration with EPFL)

5.1) Summary of losses of powder in tube fibers

Method	Who	(Losses @ 633nm) /(dB/m)	(Losses@1100nm) /(dB/m)	Remarks
Granulated Silica, unvitrified	IAP	***1 … 5***	***0.1 … 0.5***	
Granulated Silica, not remelted, vitrified	IAP	***0.8***	***0.08***	Much better for undoped material
Sol-Gel granulated silica, remelted, vitrified regularly	IAP	***0.35***	***0.03***	Some bubbles (fiber piecewise good)
Powder in tube, stack and draw	FORC, RAS	***1***	***0.1***	
Granulated Silica, remelted, vitrified at APRI	IAP / APRI	***0.2***	***0.02***	Only piecewise good
Fine powder	XLIM/IAP	***1 … 0.1***	***0.1 … 0.01***	Evacuation difficult
Sol-Gel granulated silica, no vitrification	BFH/IAP/ ReseaChem	***< 0.2***	***< 0.02***	Process close to industrial maturity

"Under construction": > try last step with vitrification / commercialize preforms, pref. materials and develop fibers;
> decrease OH and other impurities for multi-kW applications

5.2 Summary

Optical fiber fabrication with the granulated silica method

- high dopant concentrations possible (typical 0.1-2 at.%, up to 10 at. % without quenching
- improvement of homogeneity: by evacuation , vitrification , iterative milling and melting
- large core sizes possible
- allows almost arbitrary arrangement of core/cladding

Sol-gel combined with granulated silica method, regular vitrification

- further improvement of homogeneity
- Sol-gel allows to produce (already) homogeneously doped grains as starting material

***Latest results from ongoing-work (doped sol-gel grains and laser vitrification)**:*

- < 0.2 dB/m @633nm
- no crystalline silica in the fiber
- Virtually no bubbles
- Doped material homogeneous down to the nanometer scale