Ústav fotoniky a elektroniky AV ČR, vvi (ÚFE – ÚRE)

Oddělení technologie optických vláken, Suchdol

www.ufe.cz (www.ufe.cz/~kasik)

Optické vláknové sensory a optická vlákna

Rozvoj vláknově-optických přístupů v biologii

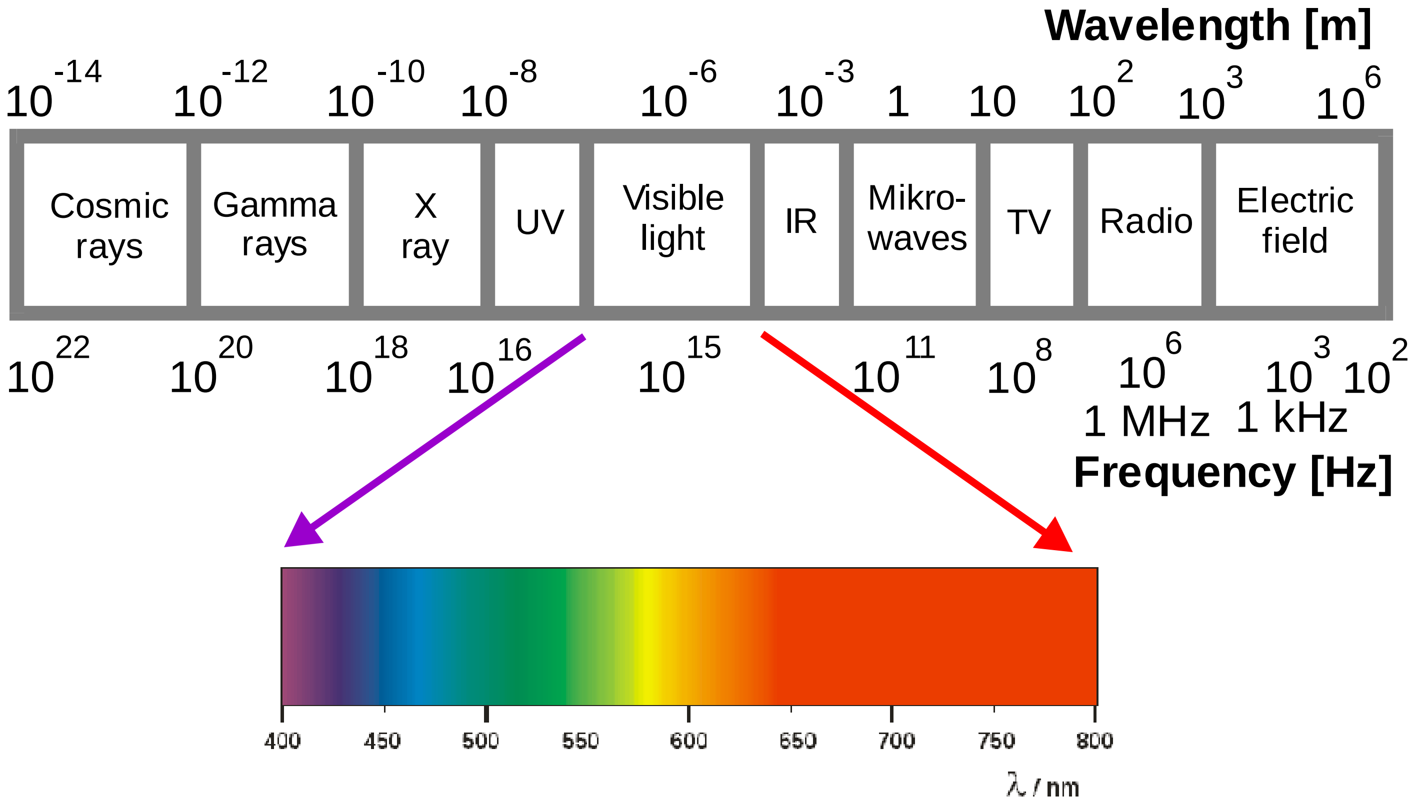
Outline

- 1. Introduction
- **2. Optical (fiber) sensors**
- **3. Optical elements – optical hardware**
  - Optical fiber processing & accessories
- **4. Optical fiber sensor consideration – example**
  - Micro point-sensor of pH – Remorost

1. Introduction

Optical fibers and optical fiber sensors

Fundaments of optical communication



$$\lambda = \frac{c}{\nu}$$

1964 – Towns & Basov & Prokhorov- LASER

1966 – C.Kao - Dielectric fiber waveguides (IEEE)

1974 – Bell At&T - commercial optical fiber- PCS

Fundaments of optical communication IREE (ÚFE-IPE)

1963 – Demonstration of ruby laser – IREE

70's – 1st hand-made optical fiber

1979 – Joint Lab of ***Silicates*** of Acad.Sci.& VSCHT

1981 - Demonstration of 1st PCS optical fiber [20 dB/km]

1987 – GI (SM) optical fiber technology=>SkloUnion

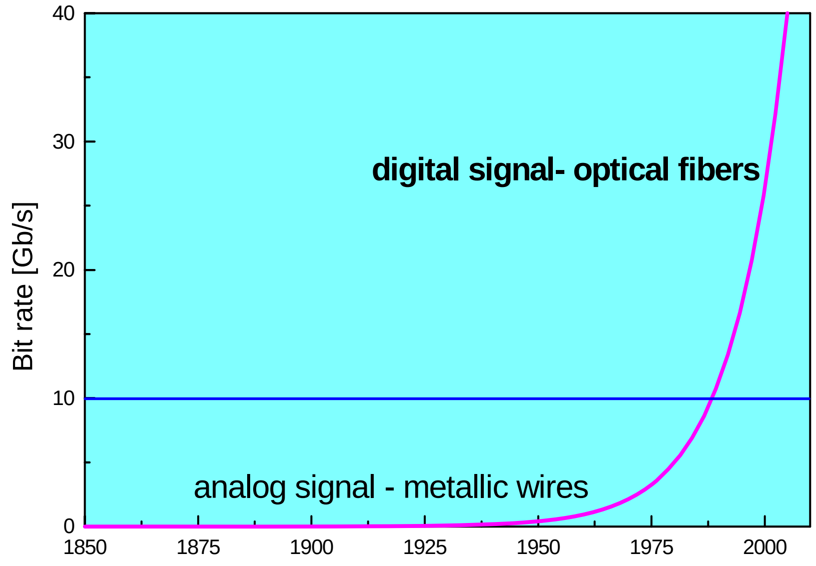
1988 - Inst. of Chemistry of Glass and Ceramic Materials

1993 – Lab of Optical Fibers again in IREE

2006 – Centrum LC ***Remorost*** – ÚEB-UK-...-ÚRE

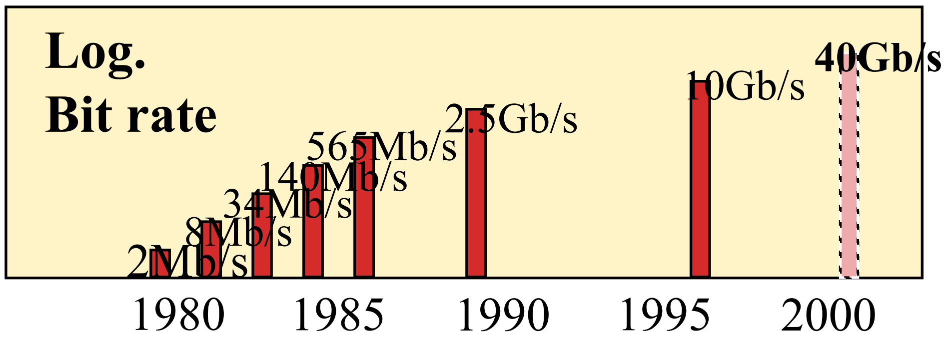
2007 – Inst. Photonics and Electronics AS CR, VVI

Optical Fibers for Telecommunications

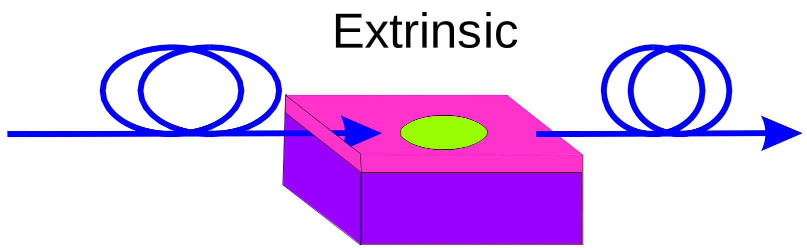


Requirements:

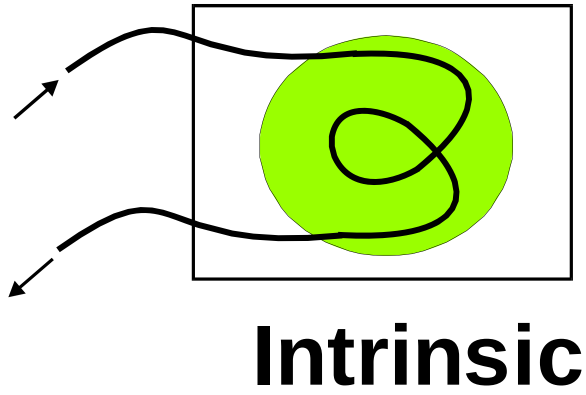
- Low attenuation
- Low dispersion
- Immunity to the surroundings (temperature, pressure, electromagnetic interference ...)
- Price



Conventional and specialty optical fibers



- **Conventional - telecommunications**
  - Low loss
  - **Immunity** to chemical/physical influences of their surroundings
  - Transmission of information



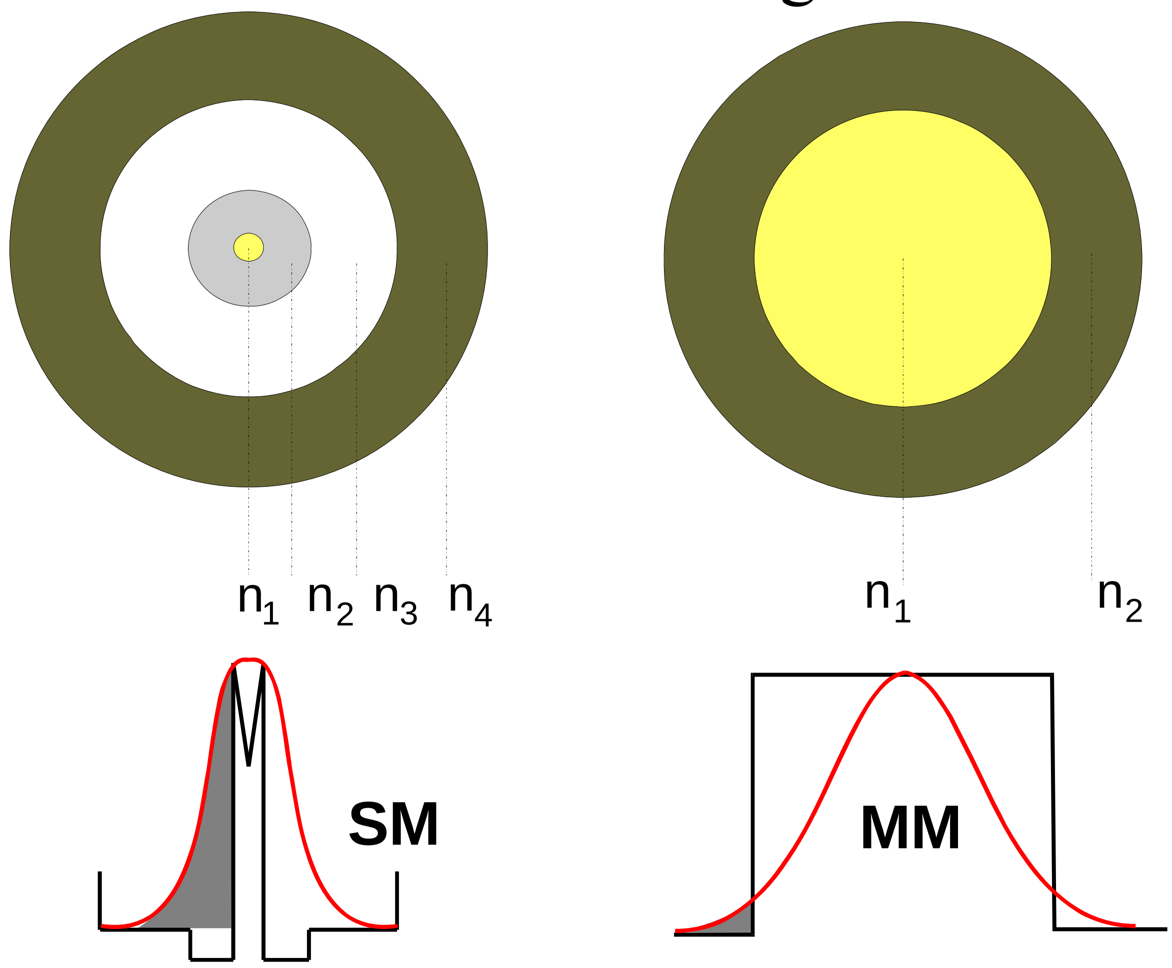
- **Specialty – e.g. sensing**
  - **Sensitivity to the surroundings changes (chemical, physical)**
  - 1978 : detection of uranium solution, Fr
  - Generation or processing of information

Goal:

- Increasing of sensitivity and selectivity, miniaturization, price

Optical fibers

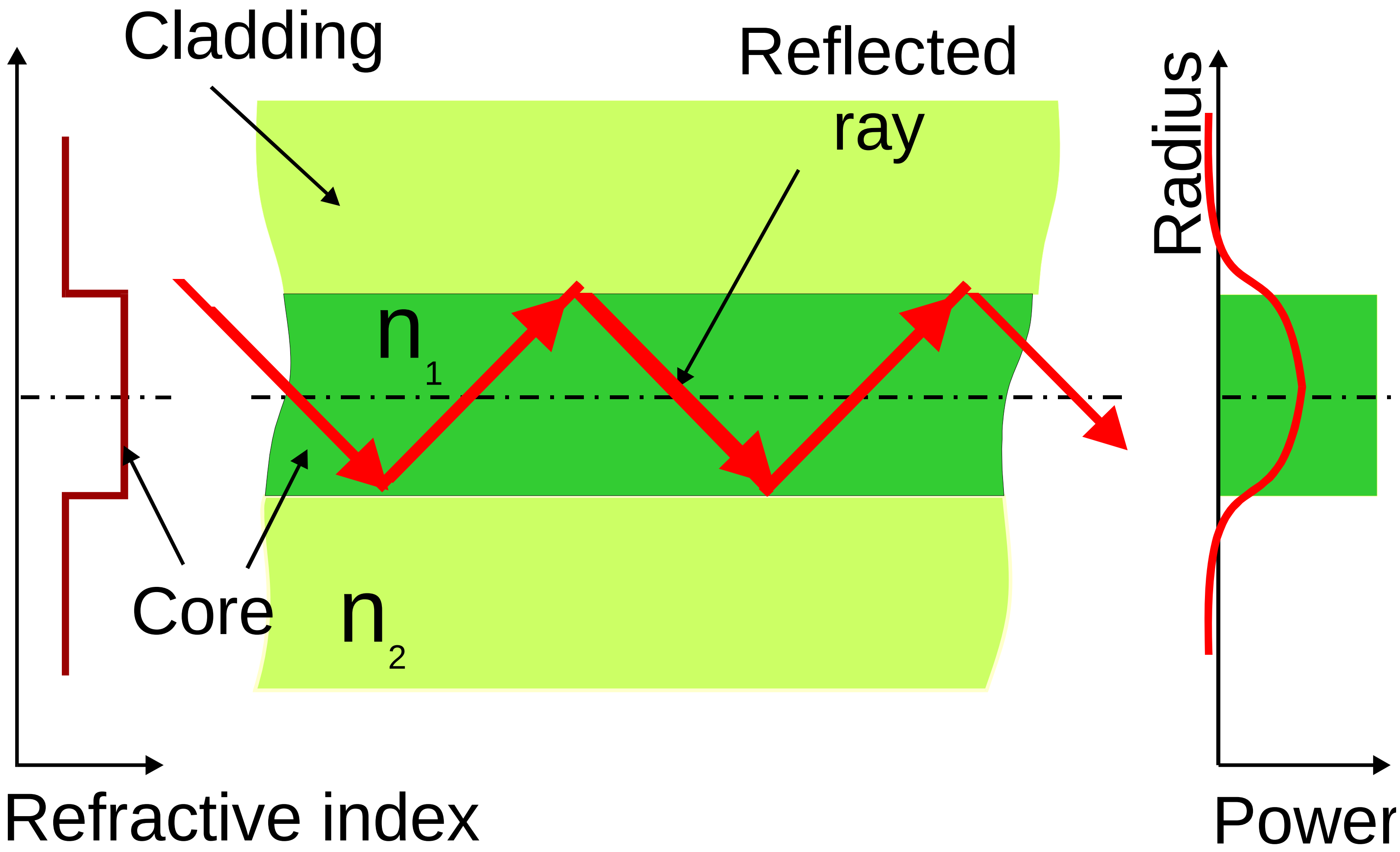
Dielectric **structures**, mostly of cylindrical symmetry,
diameter << length

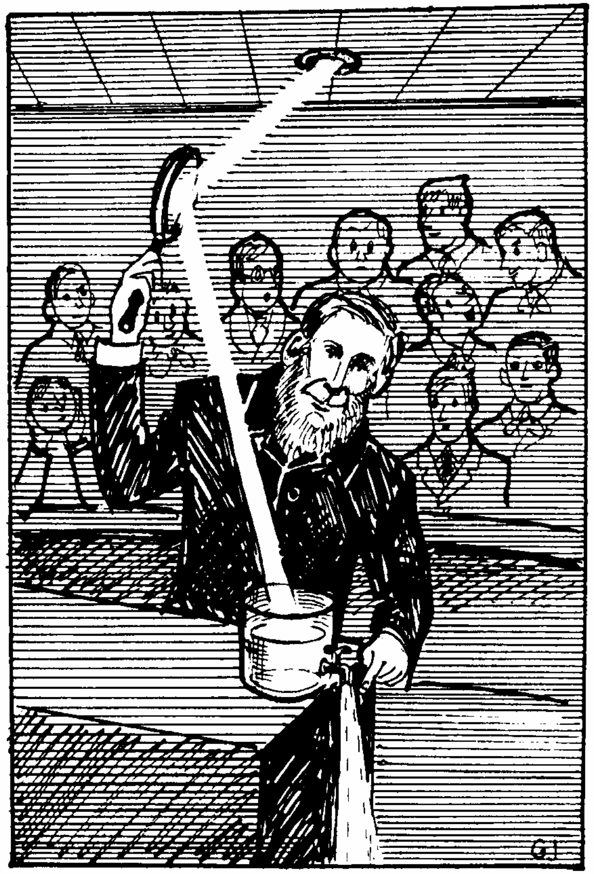


$n_{(core)} > n_{(cladding)}$

Principle of optical fiber performance - total reflection

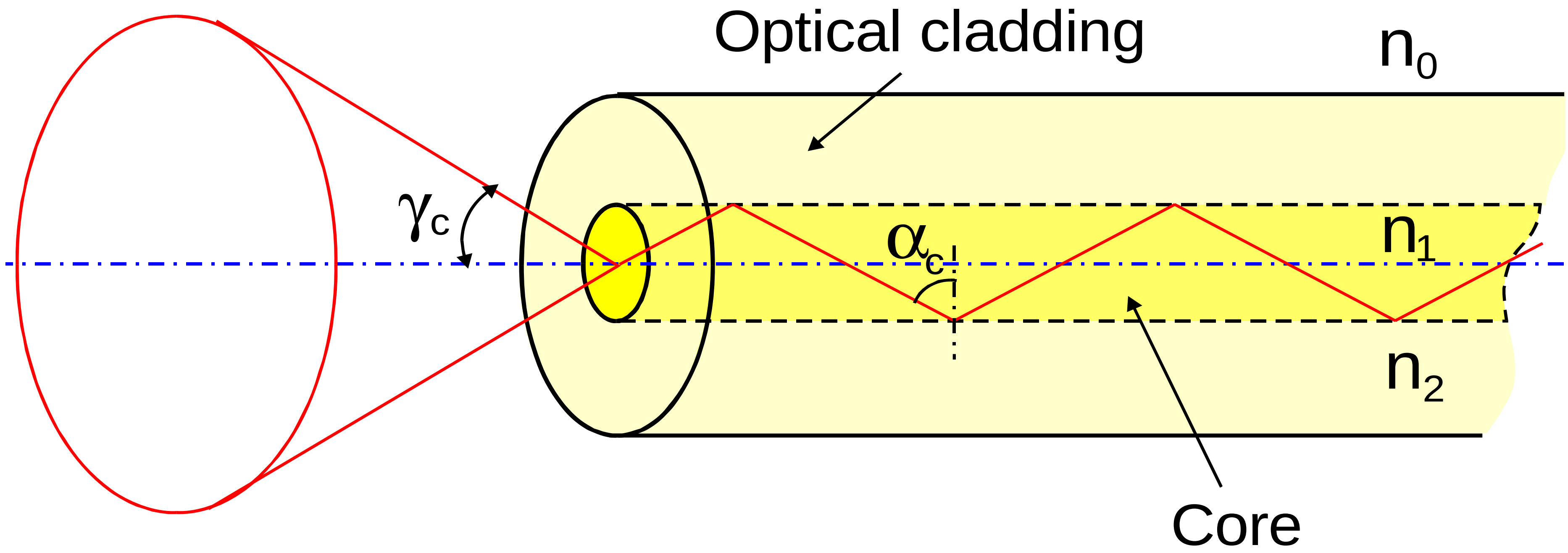
$$n_1 > n_2$$





Sir J. Tyndall

Numerical aperture



$$NA = n_0 \sin \gamma_c = \sqrt{n_1^2 - n_2^2}$$

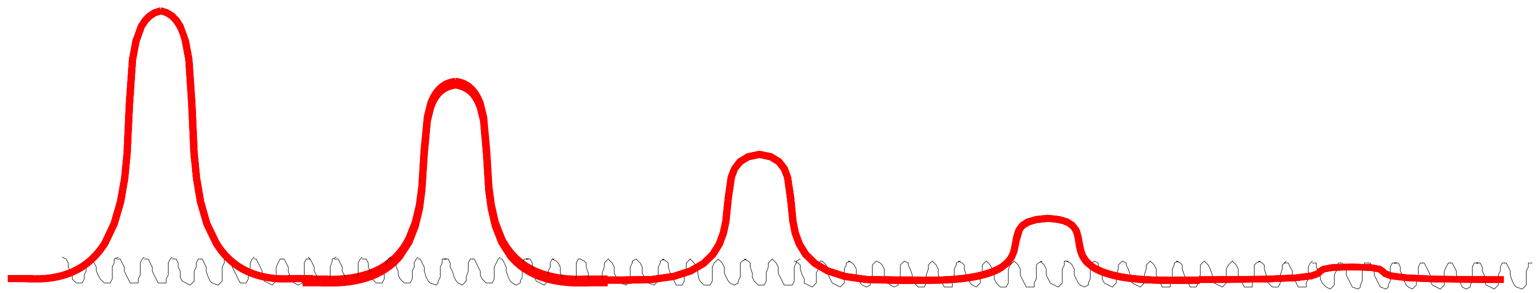
Typical values of NA: 0.1 – 0.5

Optical attenuation

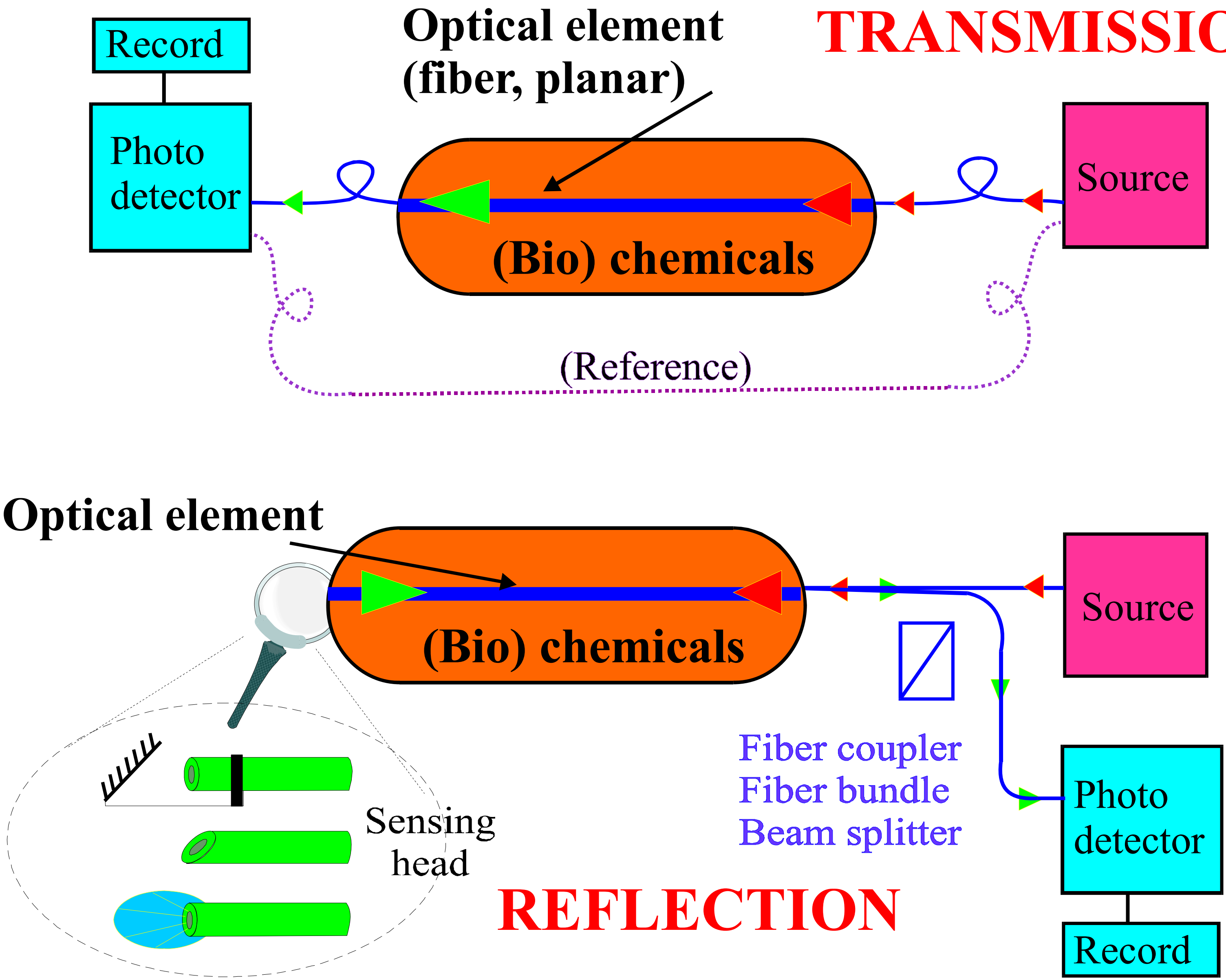
$$A = 10 \cdot \log \frac{P_0}{P} \quad [dB]$$

- Measure of optical losses
- Attenuation coefficient [dB/km]:

  represents total optical losses of fiber-optic waveguide (reflection, absorption, scattering)



Optical sensors – reversible operation



INSTRUMENTATION

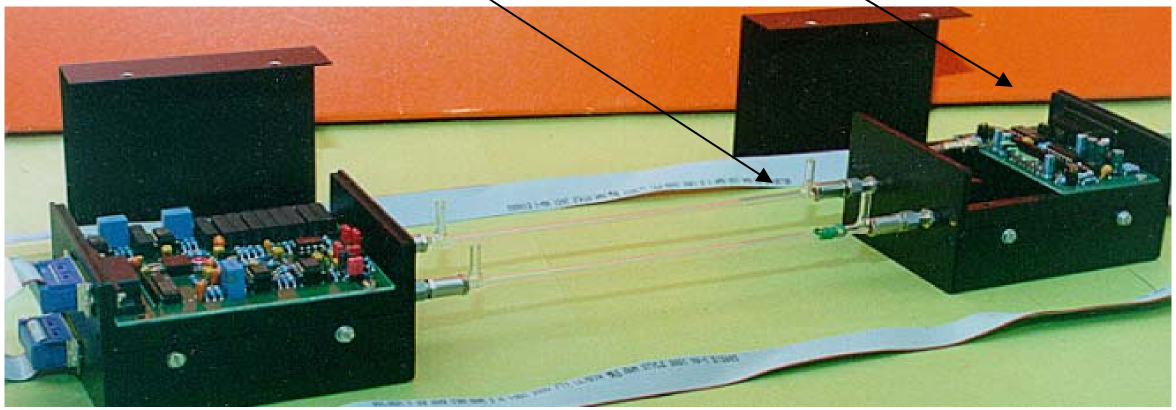
"Optical hardware"

- Source
- **OPTICAL ELEMENT**
  - **Planar**
  - **Optical fiber**
- Photo-detector

"Chemical software"

Sensing (receptor) layer-"coctails"

Change of optical properties in dependence on analyte, tailoring of the selectivity, sensitivity

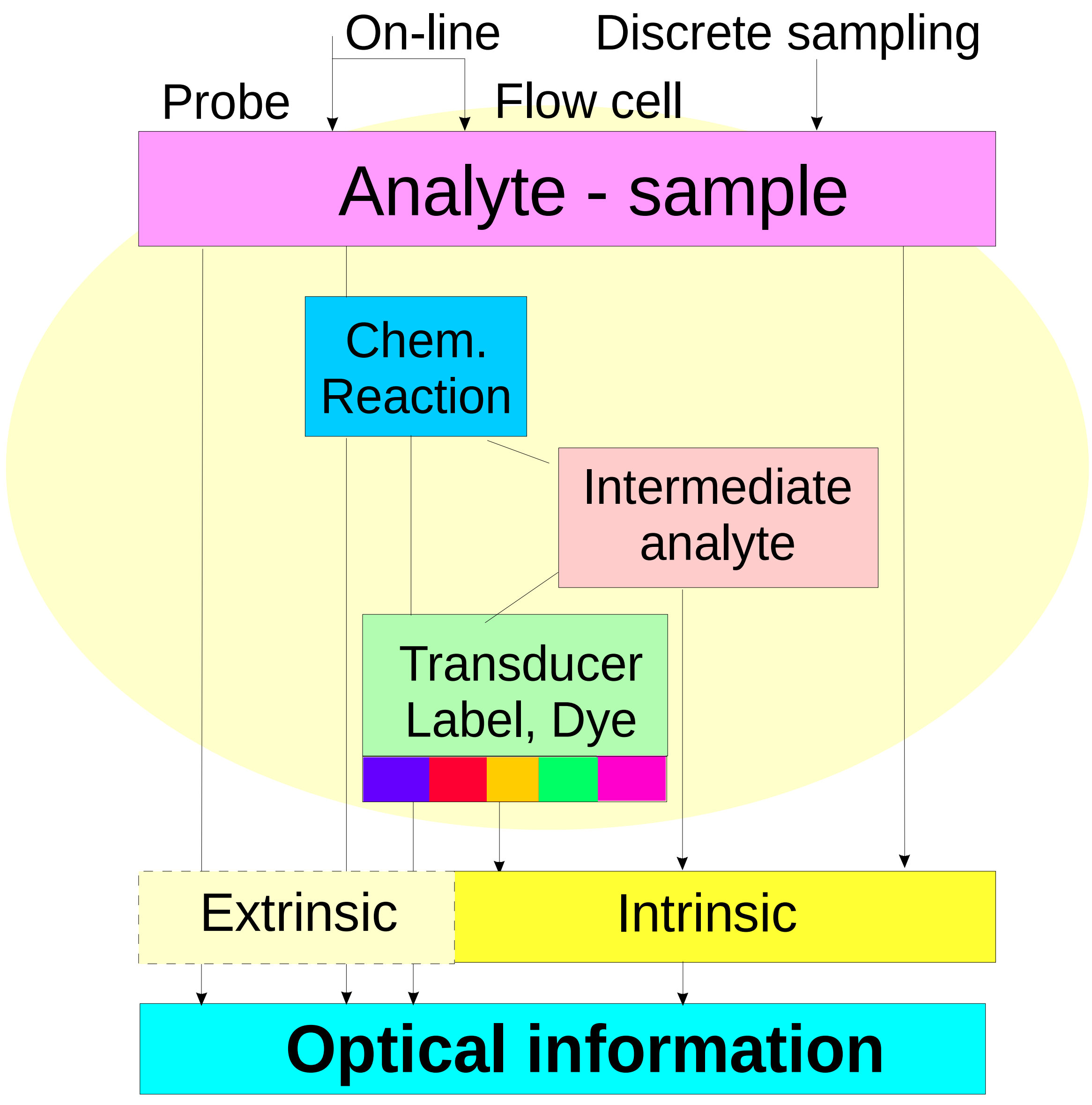


2. Optical (fiber) sensors

Optical detection methods

- Amplitude changes
  - Spectral-dependent variations of
    - absorption [Gopel...]
    - fluorescence [Orellana, Klimant, Wolfbeis, Lubbers ...] Konopásek, Lakowicz
    - luminescence [Blum, Gautier, Marquette ...]
  - refractive index changes [UFE...]
  - scattering
  - Time resolved changes
    - luminescence, fluorescence
  - Polarization changes
- Phase changes – interferometry [Gauglitz, Lambeck...]

Optical detection



- [Boisde – Harmer]

What can be optically detected

- Physical sensors
  - temperature, pressure, strain, current/voltage, vibration ...

- Chemical sensors
  - O_2
  - CO_2
  - NH_3
  - H_2
  - SO_2
  - NO_2
  - chlorinated HC
  - pH
  - cations – K^+, heavy metal ions
  - anions – chlorides, nitrates

- Bio-chemical sensors
  - alcohols
  - glucose
  - lactate
  - creatinine
  - esters
  - urea
  - glutamate
  - oxalate
  - phenols
  - sulfite
  - ascorbate
  - bilirubin
  - xantin
  - cholesterol

Commercially available sensors

GeoCenters	-	pH
FiberChem	decisionlinkinc.com	HC
Presens	presens.de	O2, pH
Yellow Springs	ysi.com	CO2
Soundek	otech.fi	oil
OceanOptics	oceanoptics.com	pH, O2
SMSI	s4ms.com	O2
Photosense	photosense.com	O2

- *"Fibers" = 2-mm fibers; "Tips" = 140 μm*

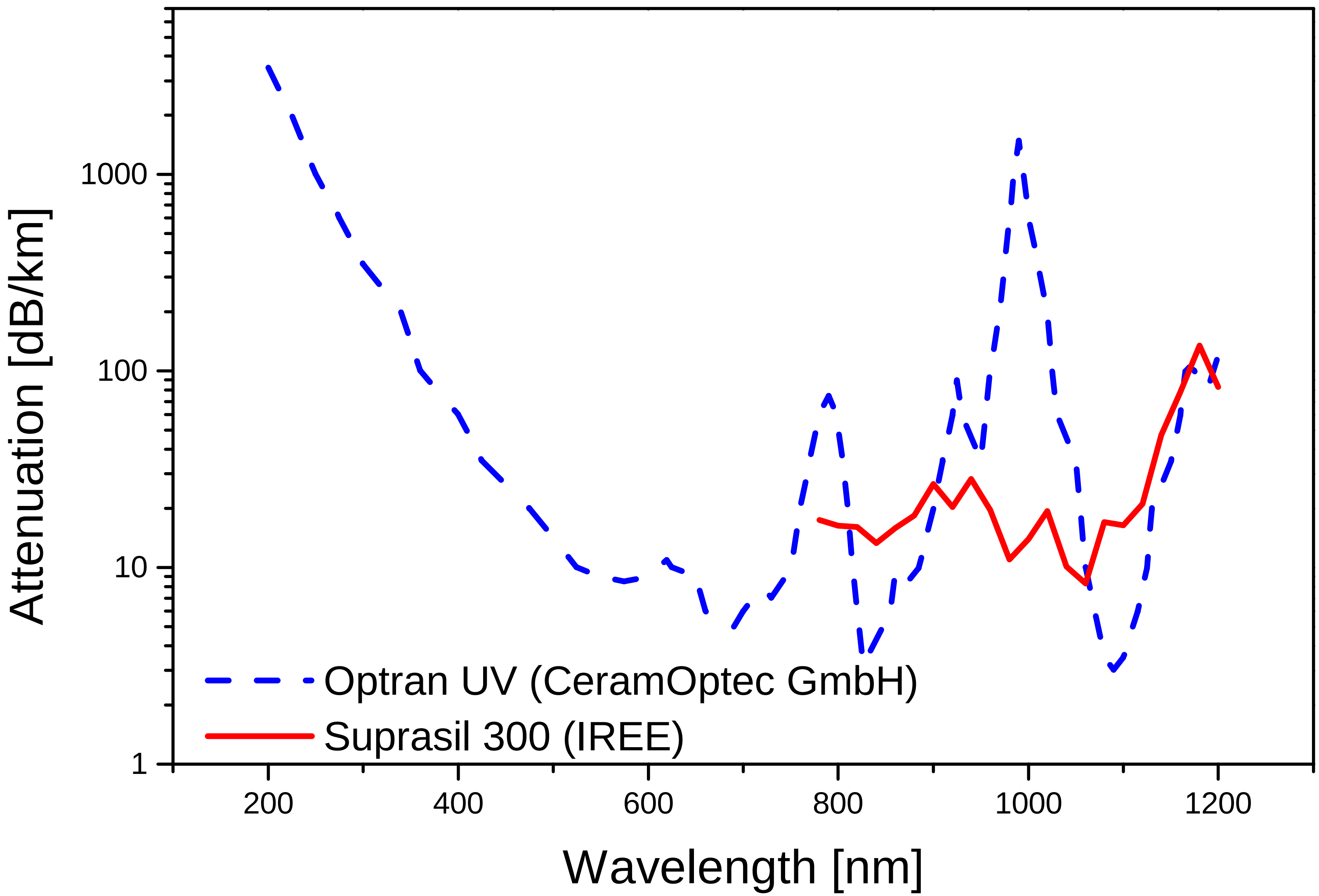
3. Optical elements – optical hardware

Requirements

- **Durability** to the analyte
  - glasses > polymers > crystals
- High **transparency** in a wide spectral range
  - important for distributed sensing
- Common **availability**
  - market, price

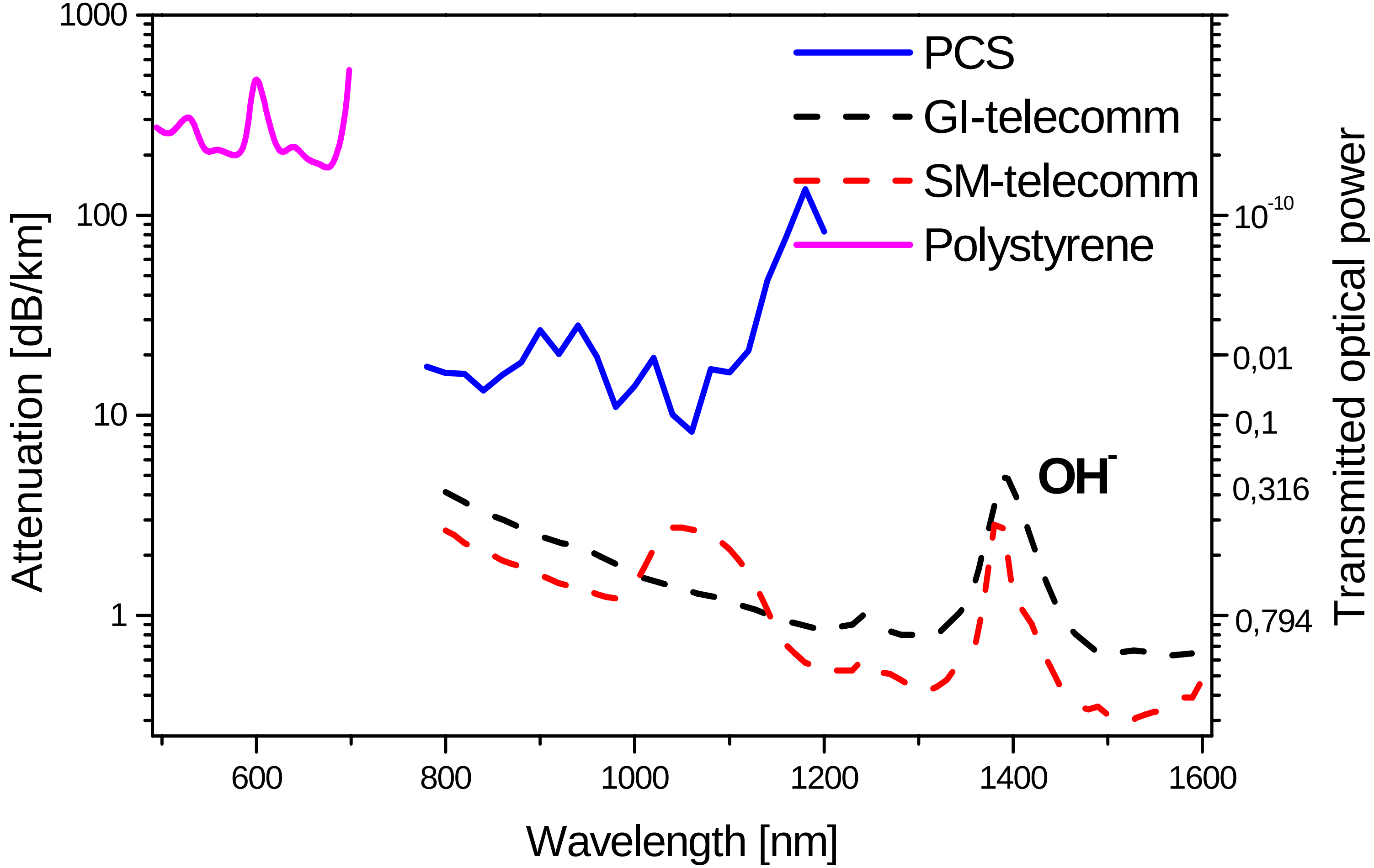
- => Choice of material, structure, coating of optical element

Optical fibers UV-transparent



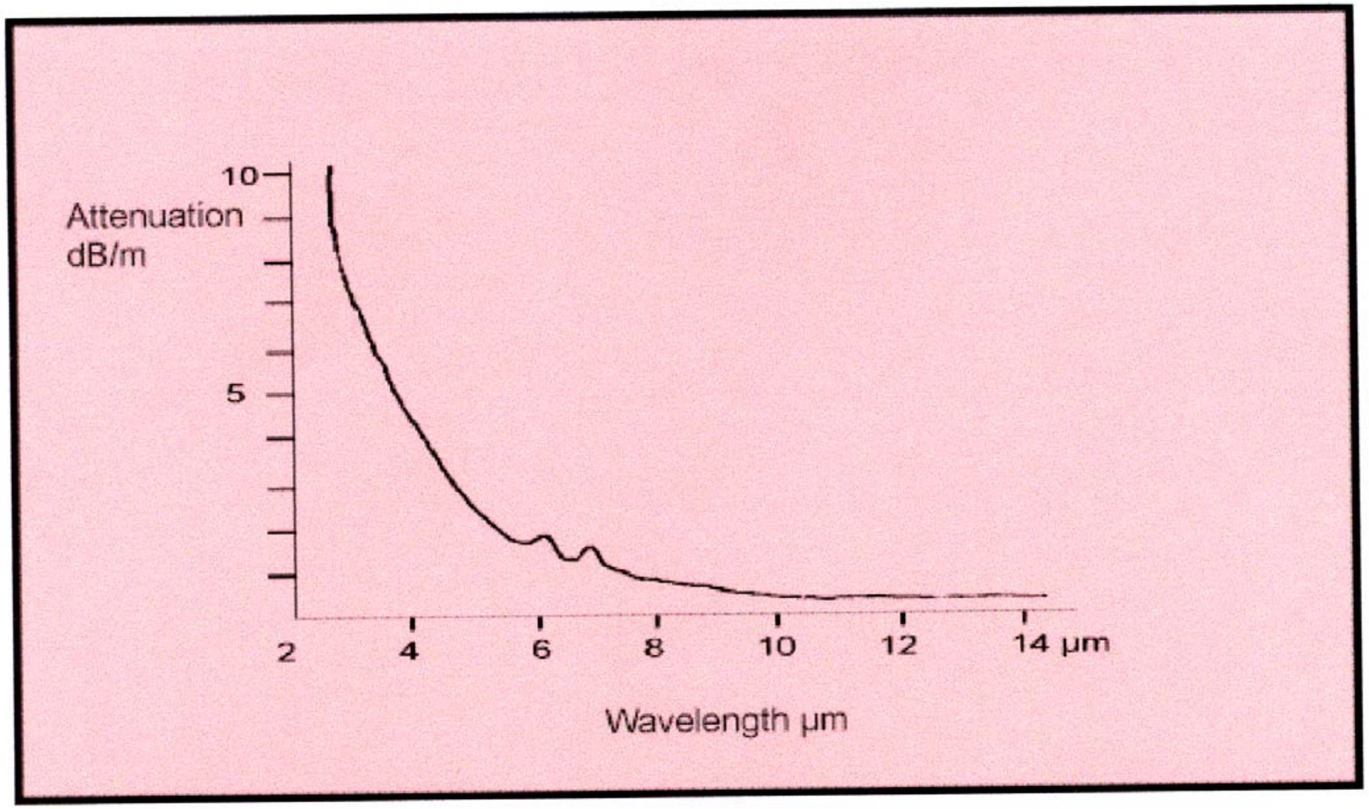
Silica-SUPRASIL $n_{200nm} = 1.55$ [ceramoptec.de, Ocean_Optics.com, UFE]

Optical fibers VIS+NIR-transparent



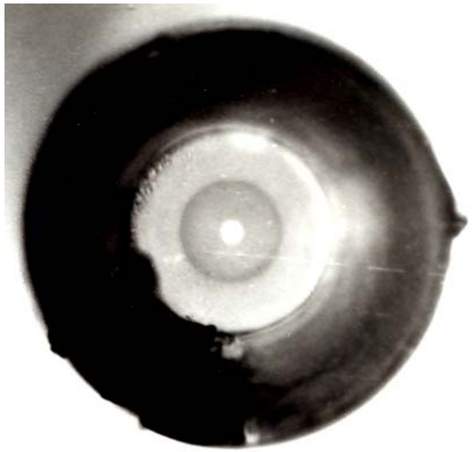
Silica n_{633} =1.457 [Corning, Lucent, Ocean Optics …]; doped silica n_{633} = 1.45-1.50 [Sumita, UFE…], silicate "glass" n_{588} =1.5-1.95 [Schott, UFE…], plastic n_{588} =1.5-1.6 [mitsubishi.com, luceat.it, unlimited-inc.com…]

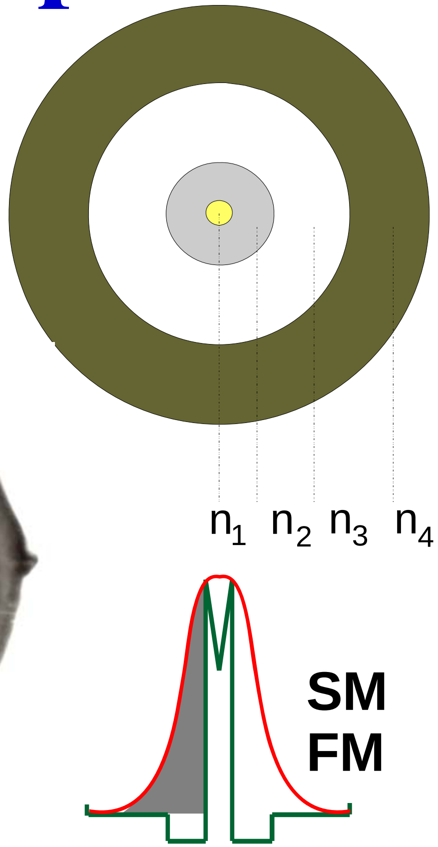
Optical fibers IR-transparent

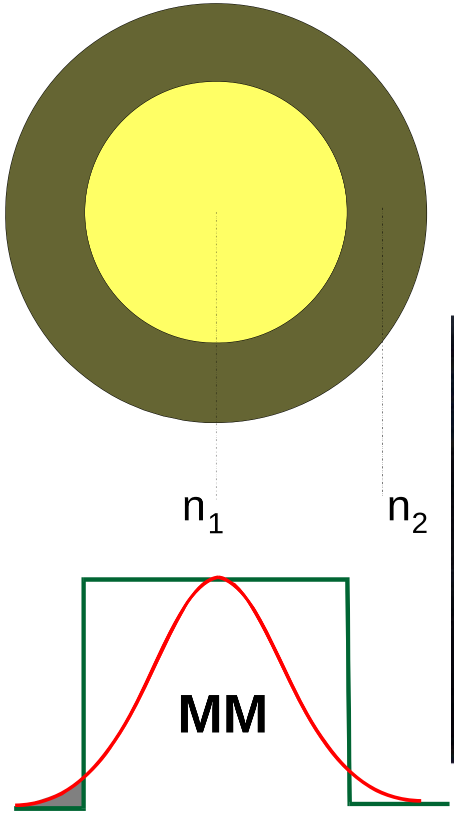


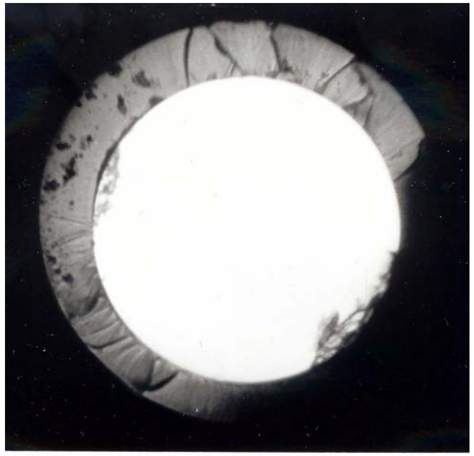
- fluoride glasses [irphotonics.com, univ-rennes1.fr …] (up to ~4 µm)
- sapphire [B.Mizaikoff, photran.com, fiberoptictechnology.net …] (up to ~4 µm)
- silver-halides $AgCl_xBr_{1-x}$ (up to 15 µm)
- chalcogenide (Se, As_2S_3, As_2Se_3…) and chalco-halide glasses [oxford-electronics.com, orc.soton.ac.uk …] (up to 20 µm)
- refractive indexes $_{2\text{-}20um}$ ~ 2 – 2.5 >> silicate glasses ; PROBLEMS

Optical fiber structures









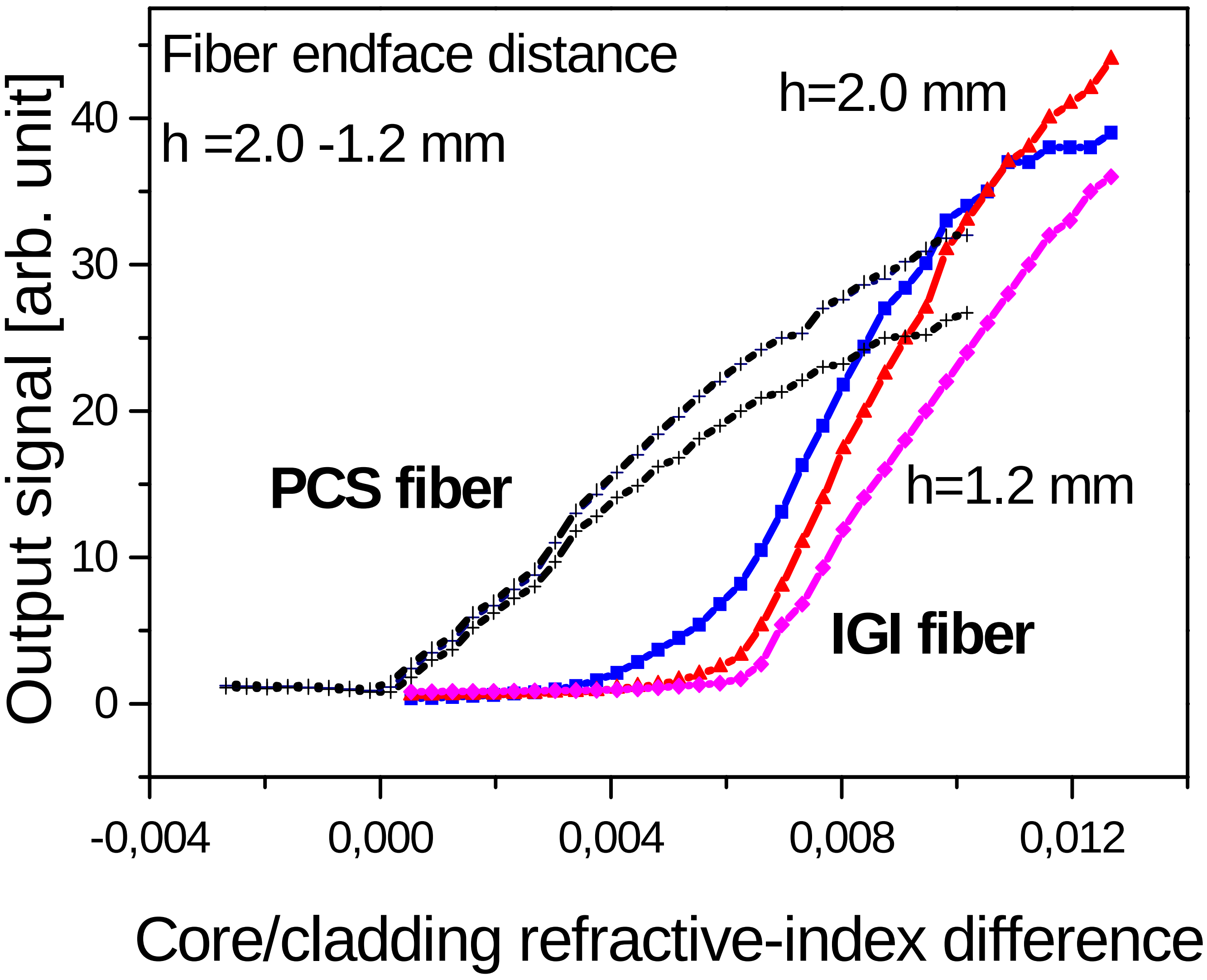
NA_{SM} (0.1-0.25) << NA_{MM} (0.2-0.5)

$Ø_{core}$SM-FM (2-15 µm) << $Ø_{core}$MM (50-1000 µm)

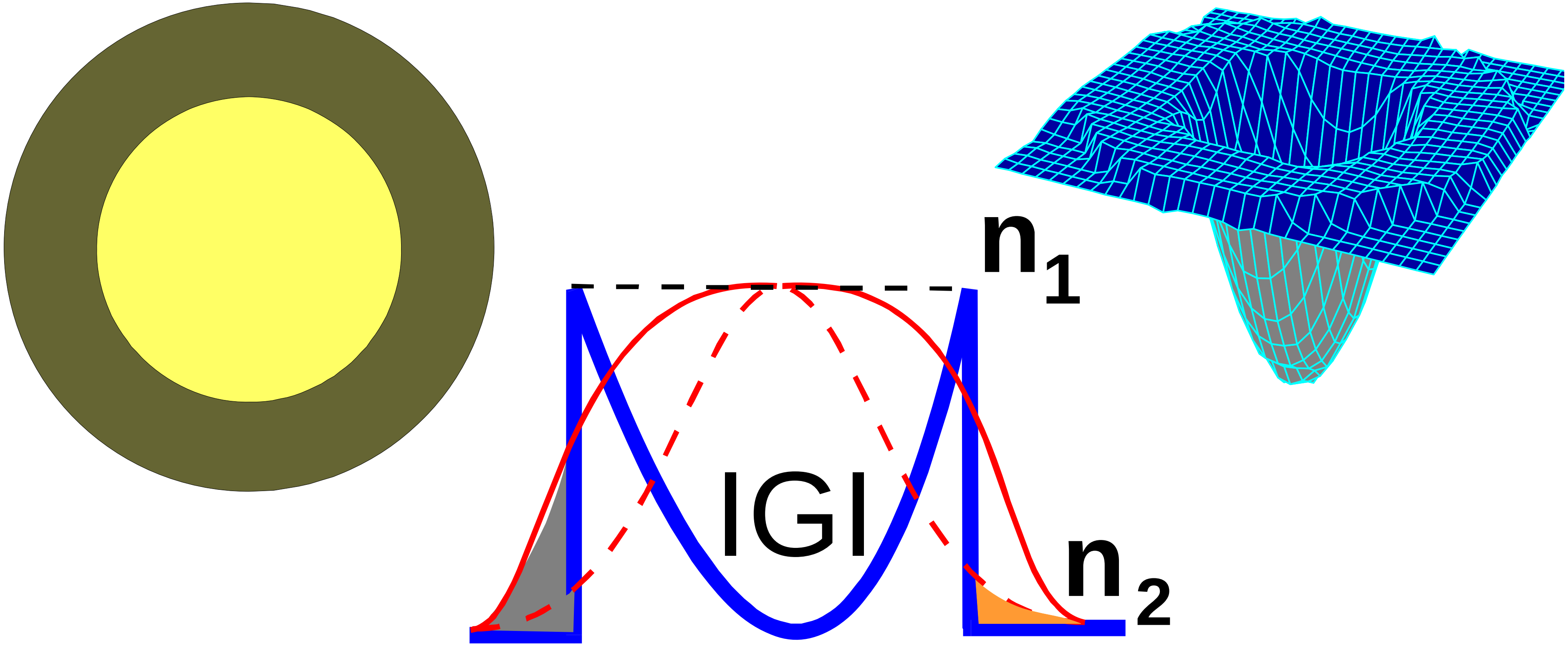
- Intensive evanescent field
- No access of the analyte to the core

- Polymer-clad-silica (**PCS**)
  Polymer-clad-glass (**PCG**)
  Plastic optical fiber (**POF**)
- Poor evanescent field !

Fiber (PCS/IGI) sensitivity

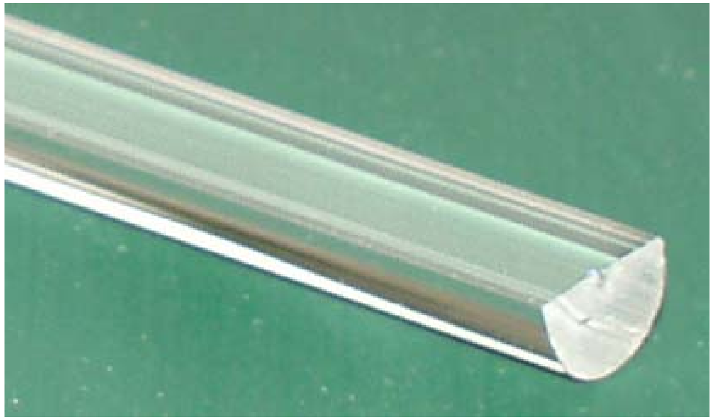


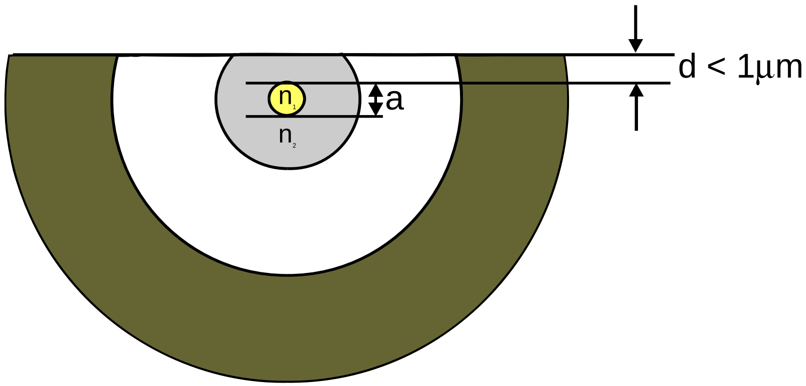
Enhancement of MM structures for evanescent-wave detection

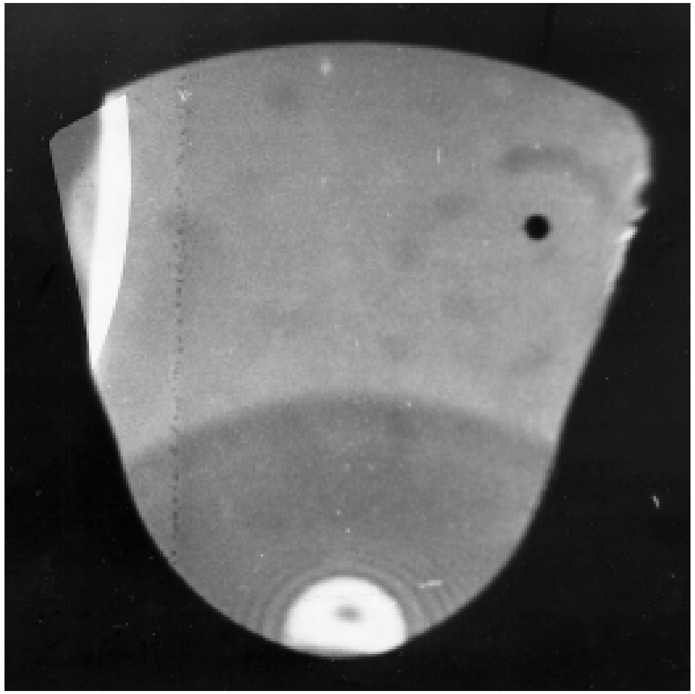


Inverted Graded-Index (**IGI**) structures; evanescent wave 0.5% => 5-7%, depending on conditions of excitation/detection (selective) [M.Chomat, V.Matejec]

D-shaped fibers, S-fibers



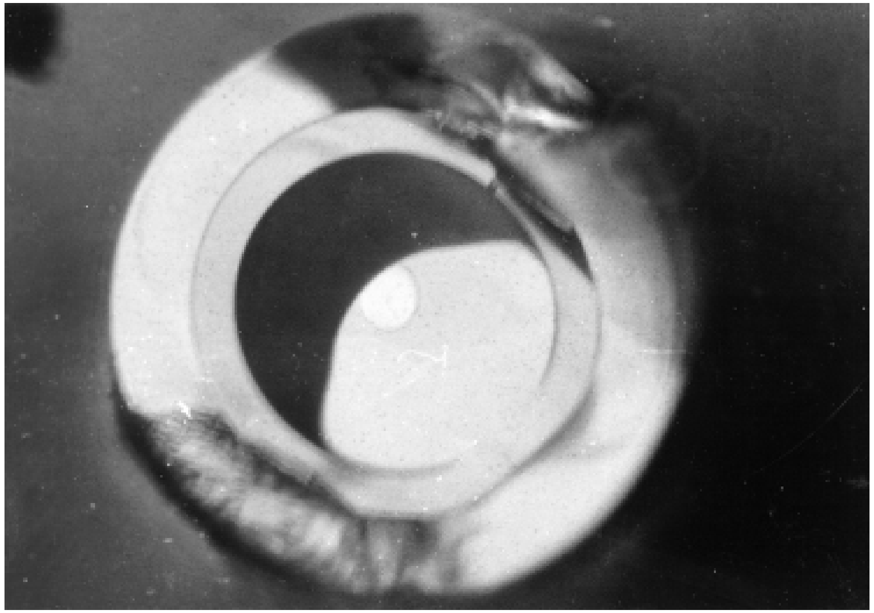


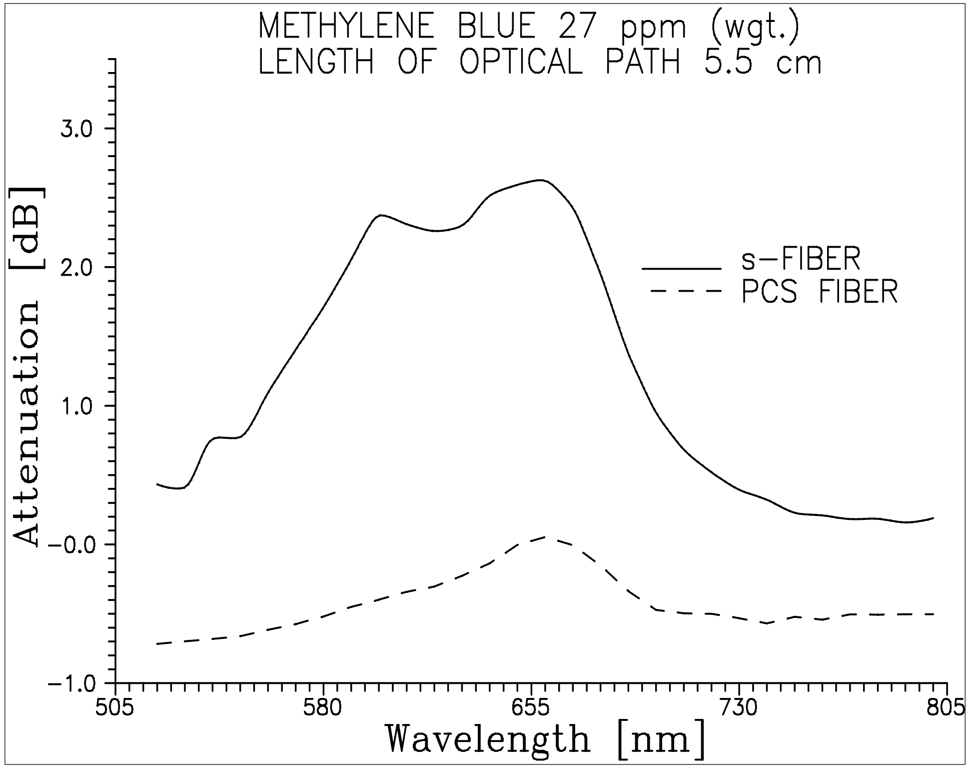


"S" = sectorial

- Non-circularly symmetric, robust, size of the detection site
  [G.Stewart, M.Chomat, V.Matejec]

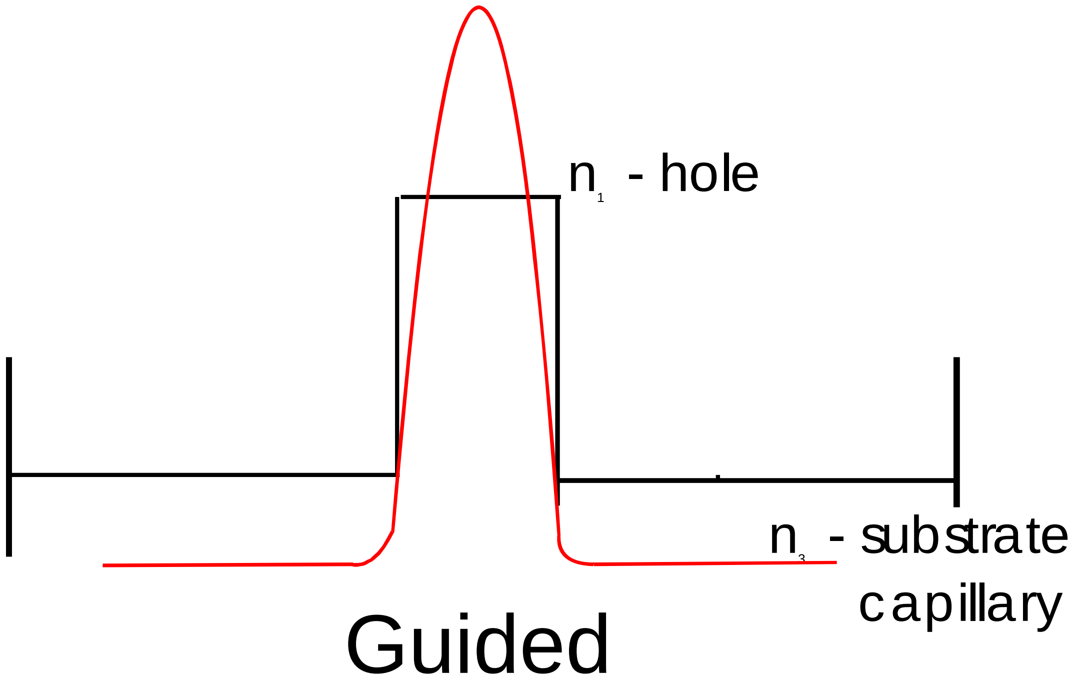
S-fibers, Capillary S-fibers

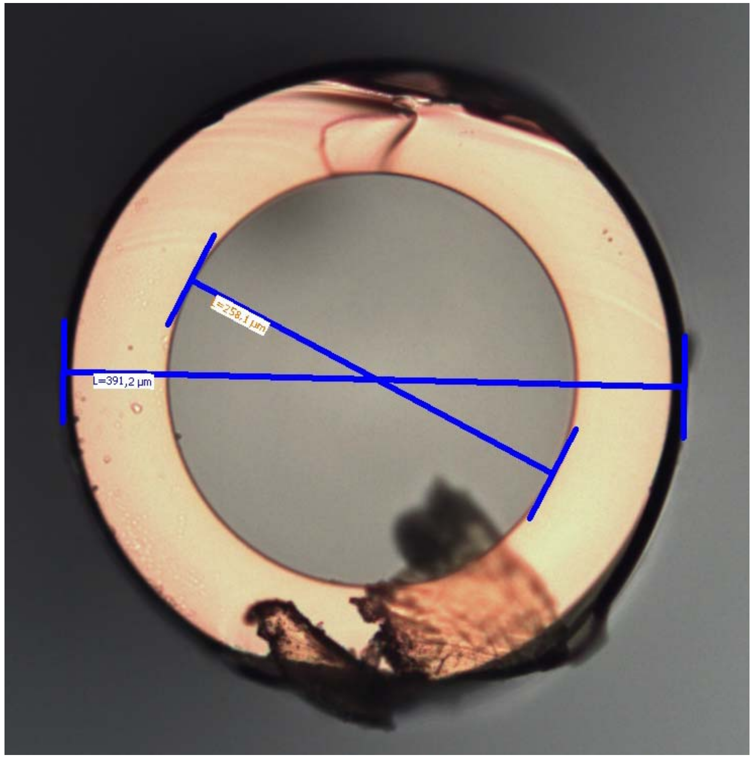




- Symmetric, compatible with silica fibers [V.Matejec]

Fiber capillaries & hollow fibers



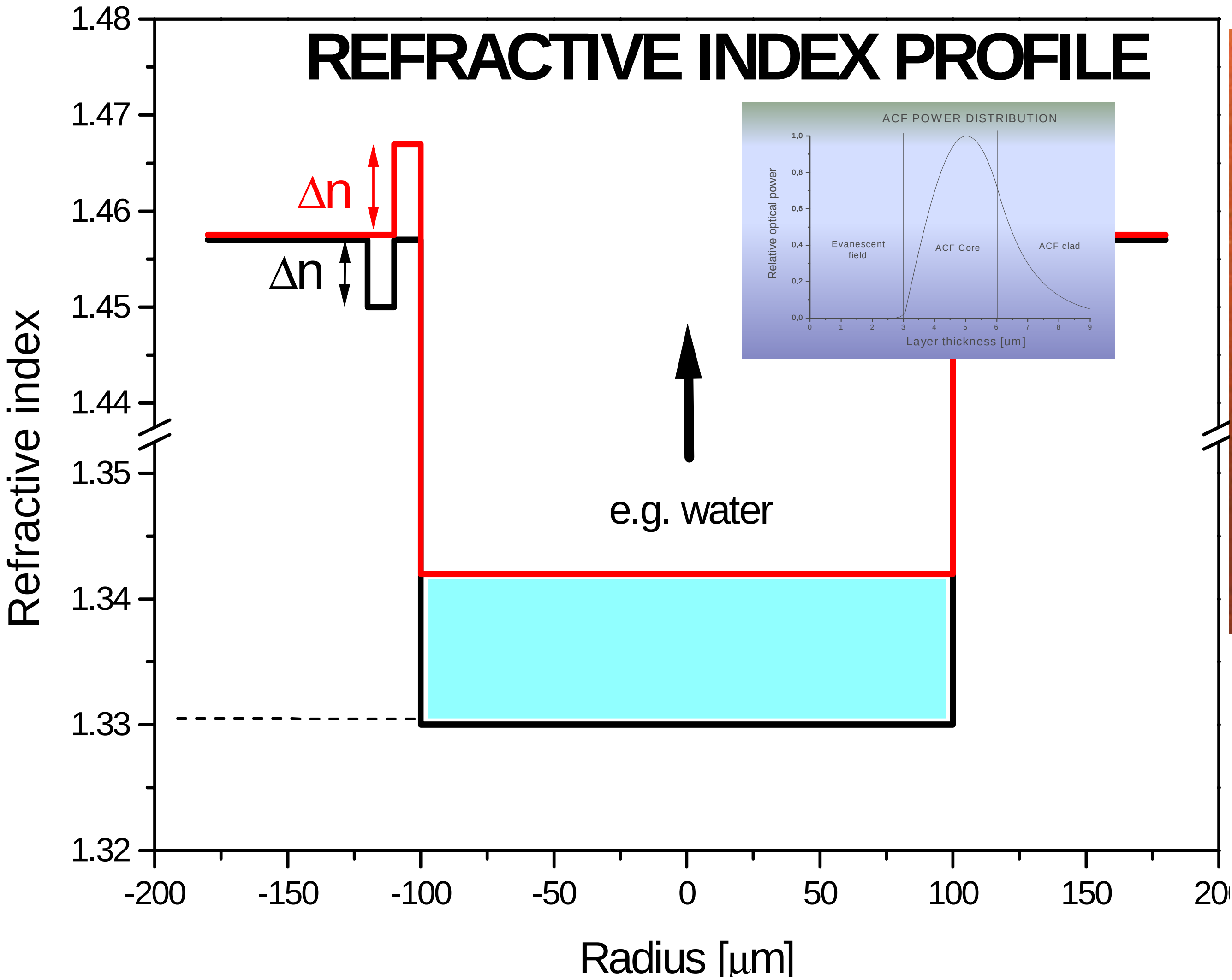


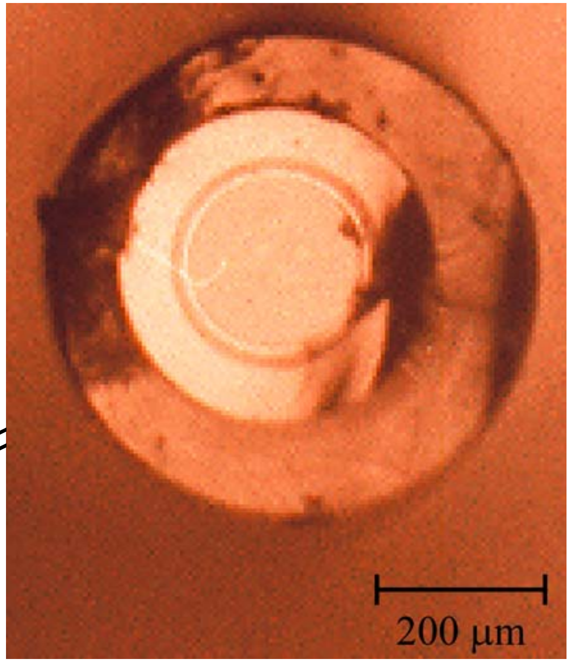
Liquid-core fiber capillary,

- Silica fiber capillary 365/360/160 µm
- [polymicro.com, verrillon.com, UFE …]

good transparency of analyte, $\mathbf{n_{analyte} > n_{substrate}}$

SM (FM) Annual Core Fiber capillaries





ACF-capillary

380/210/200 µm

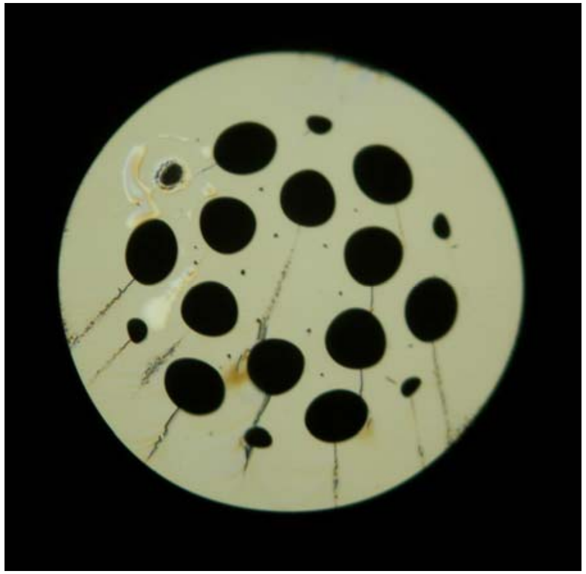
[UFE]

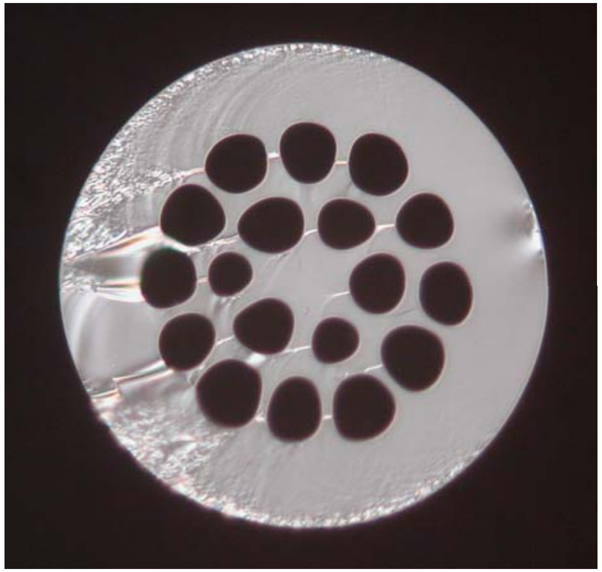
$$\mathbf{n_{analyte} < n_{substrate}}$$

- Usual dimensions : 365 / 250 µm; wall thickness ~ 50 µm

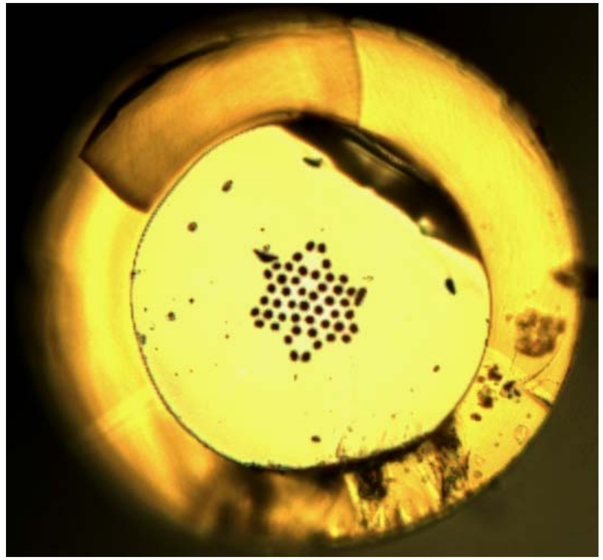
Photonic crystal fibers (PCF)

- Index-guidance





- Bandgap-guidance



- P.Russel, A.Bjarklev; [crystal-fibre.com, UFE]
- intensive evanescent field
- micro => NANO

Optical fiber coatings

- **Conventional**
  - **Mechanical protection**
- **Special**
  - **Immobilising** of opto-chemical transducers (receptors)
  - **Tailoring of access of analyte** to the detection site (porosity, thickness, phobicity) – membranes

Optical fiber coatings

Conventional (primary) coatings

- Polysiloxane (n_D = 1,41), soft [PCS]
- UV-acrylate (n_D = 1,65), hard
- PTF– polytereftalate (n_D>1,46), hard
- PI – polyimide (n_D>1,46), hard
- PTFE – teflon (n_D = 1,29)
- thickness 4 μm (hard) – 100 μm (soft)
- **on-line coating** (fiber drawing)

Special coatings – sol-gel / polymers

- thickness ~10^2 nm (!) – several μm (~ lambda)
- **additionally coating** (dip-coating)

Optical Fiber Processing & Accessories

- decladding
- cutting
- joining – splicing & connectoring
- grinding & bevelling
- bundling
- tapering

Optical fiber decladding

- mechanically – stripping tool (pliers)
- chemically - leaching
  - trichloethylene (acrylates)
  - HF acid (siloxanes)
  - exposition – seconds-minute

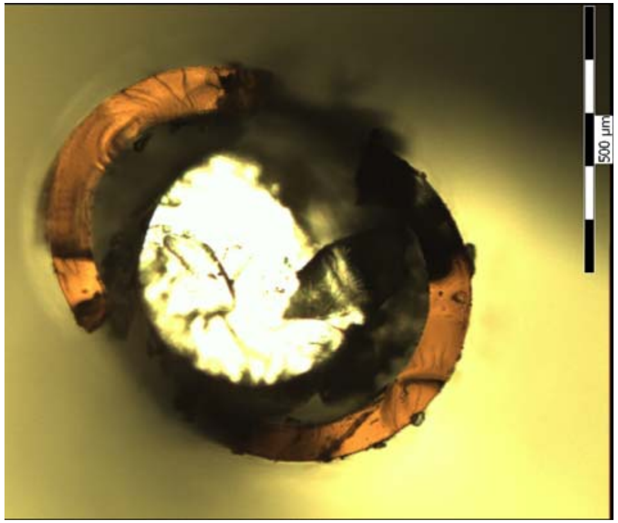
Optical fibers to be handled with care = “No drop on floor” [NPL, UK]

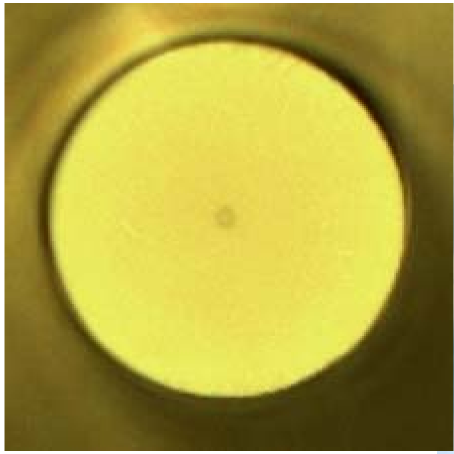
Fiber cutting

- primitively :

  scissors, knife, razor blade (suitable only for POF)

- more primitively: fire



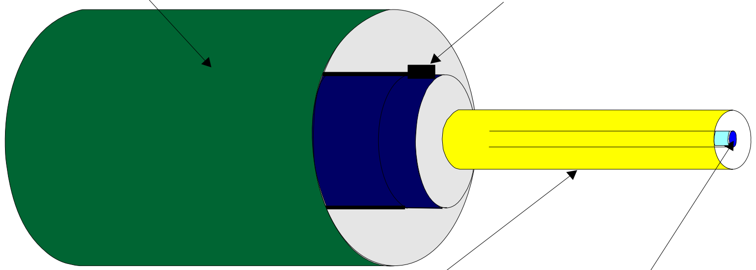


- correctly :
  - **fiber cleaver** FK11 (York Tech, UK, Ericsson, S) [UFE]

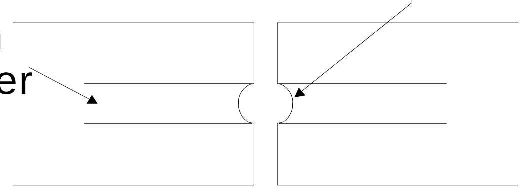
Optical fiber joining : splicing/connectors

- Splicing device (Fujikura, Ericsson) [UFE], silica-based fibers only; losses ~0.15 dB

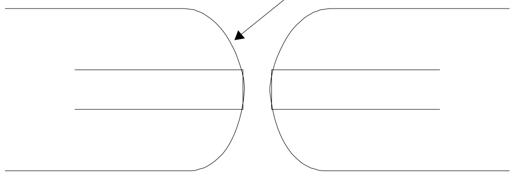
Optical fiber joining : connectoring



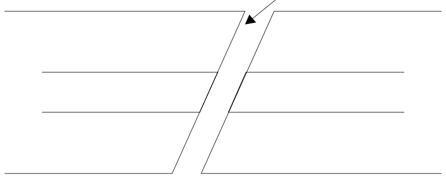
Fiber Connection (FC)



Physical Contact (PC)



Angled Fiber Connection (APC)

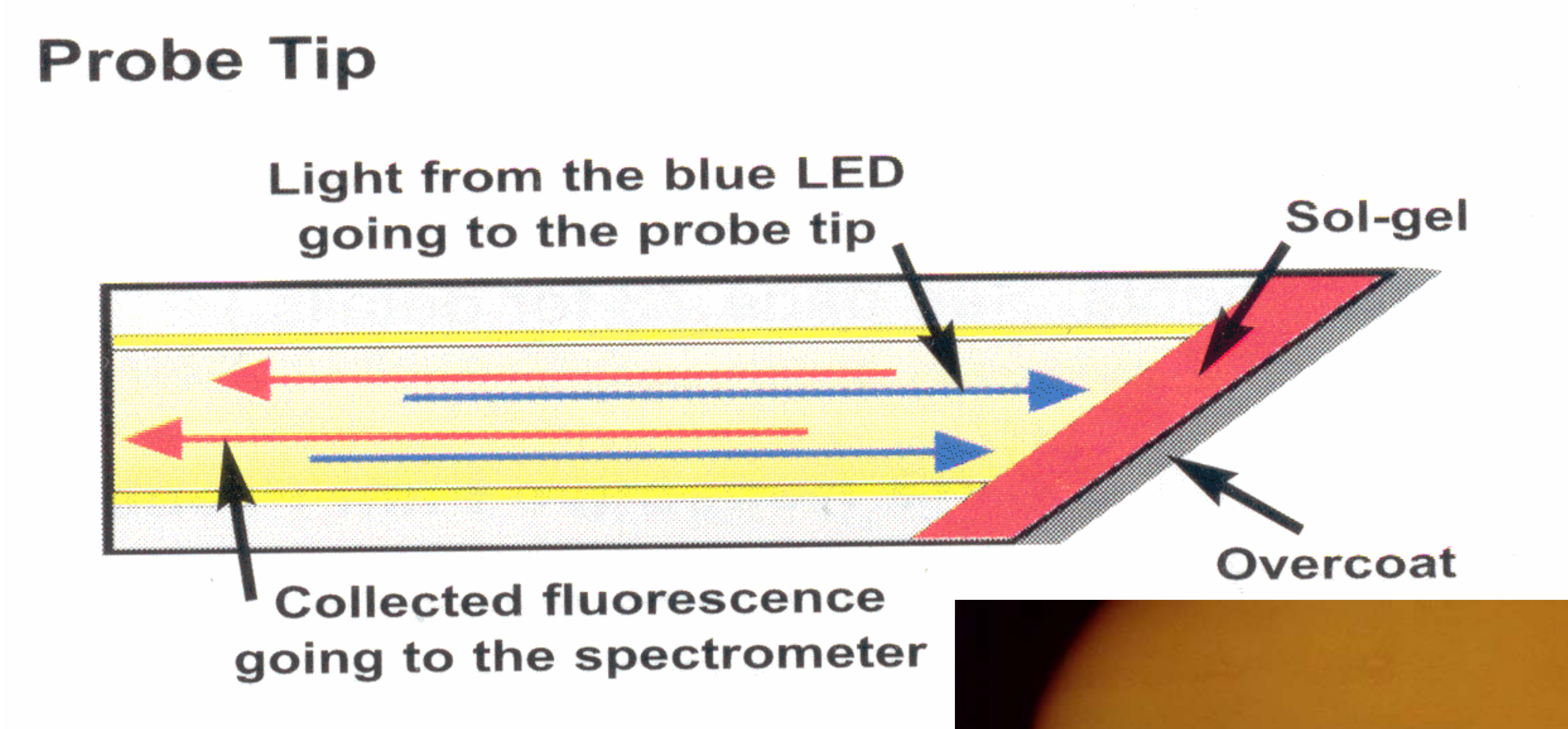


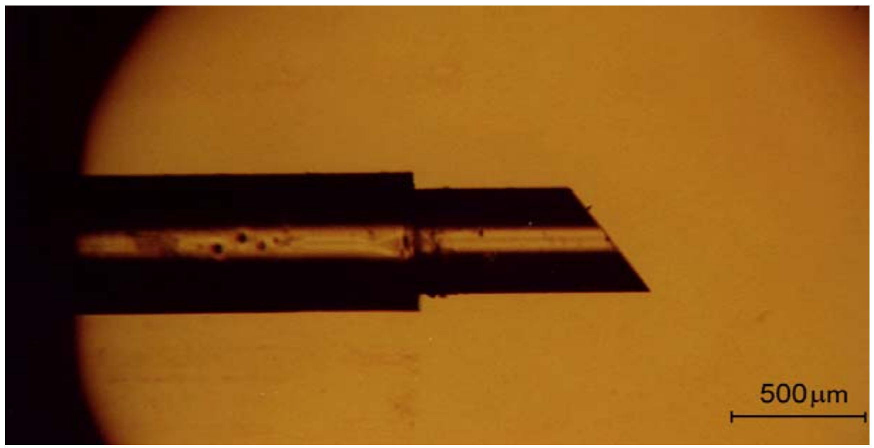
- FC, HMS-10, PC, D4, SMA, SC, DIN ST …; losses ~0.25 dB

Optical fiber grinding, bevelling

- Diamond tools [Logitech…] or lapping films [3M…]; HOLDERS !

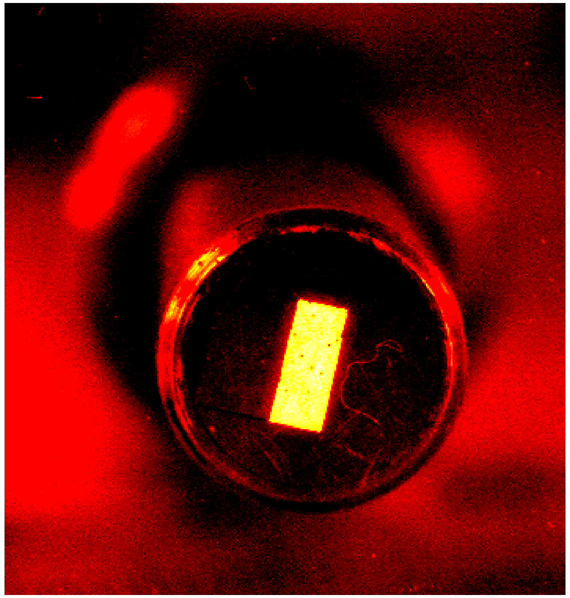
Optical fiber grinding, beveling

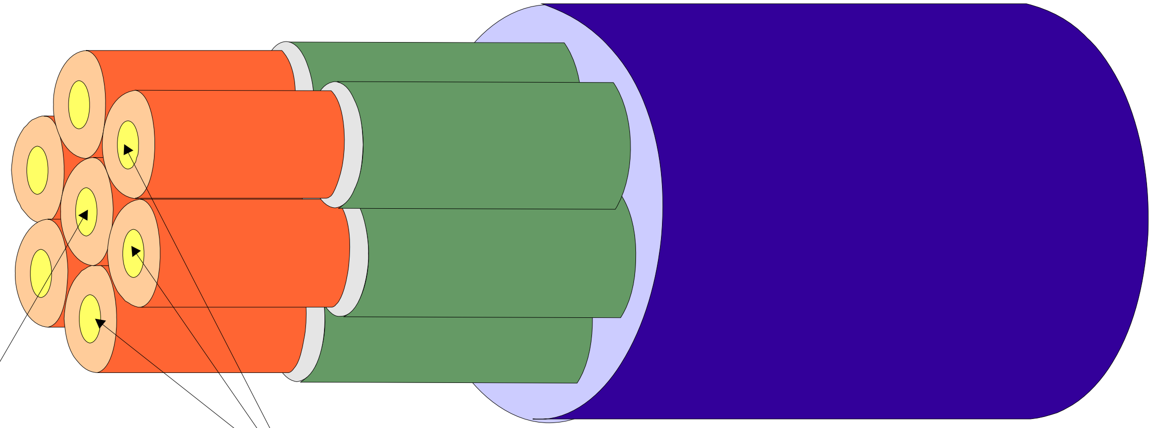




- coupling out unwanted exciting signal (or autofluorescence)
- transmission probes ...

Fiber bundles

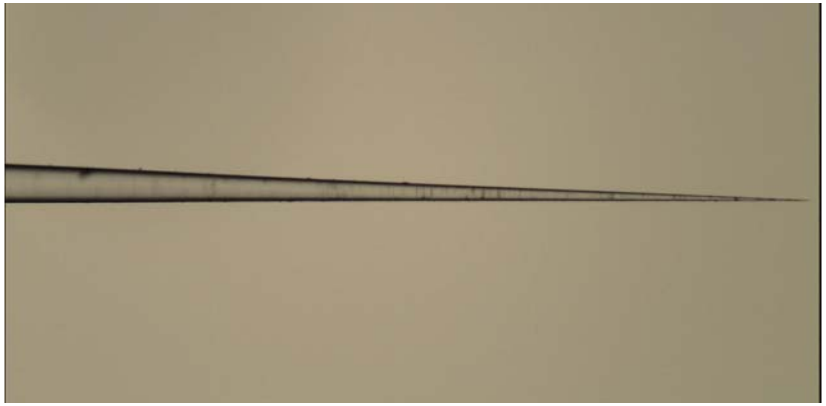


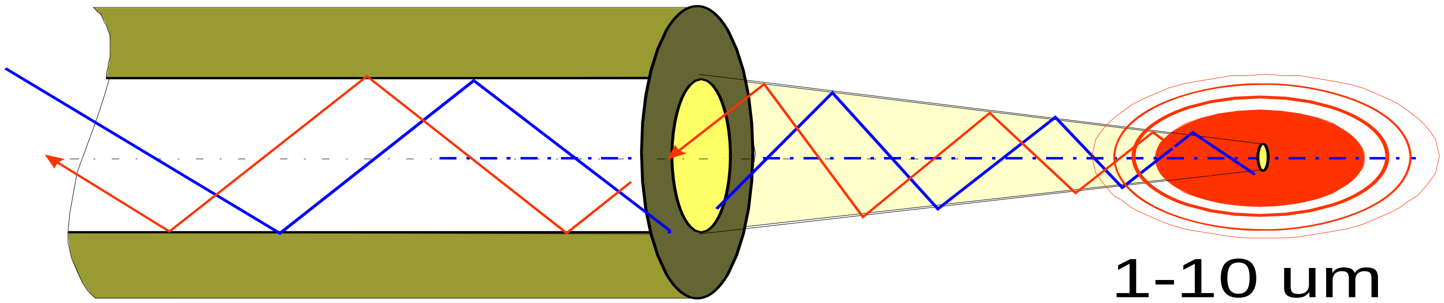


- **Reflection sensing arrangement; signal enhancement** [O.Wolfbeis]
- Imaging [UFE]
- Multianalyte analysis [D.Walt], larger area monitoring

Optical fiber tapering

1. Small area monitoring ~ µm^2

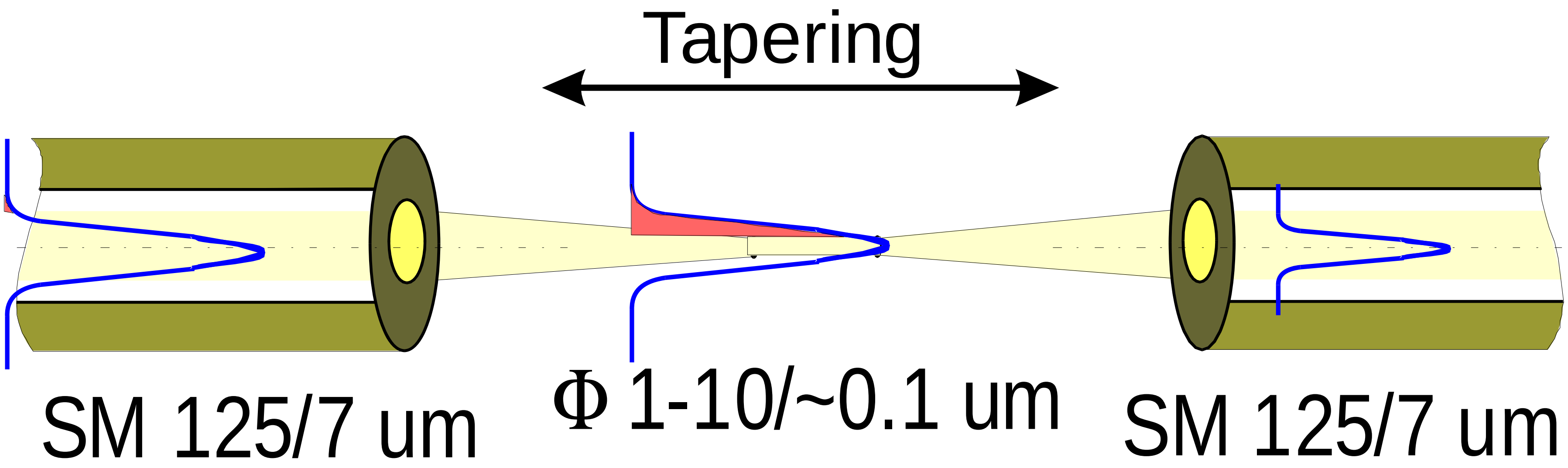




- [B.D.Gupta, C.D.Singh, F.Ligler, D.Kopelmann, F.Baldini]
- Preparation : slow withdrawing from acidic HF-containing solution (tips) + protective coating

Optical fiber tapering

- 2. Multipoint monitoring

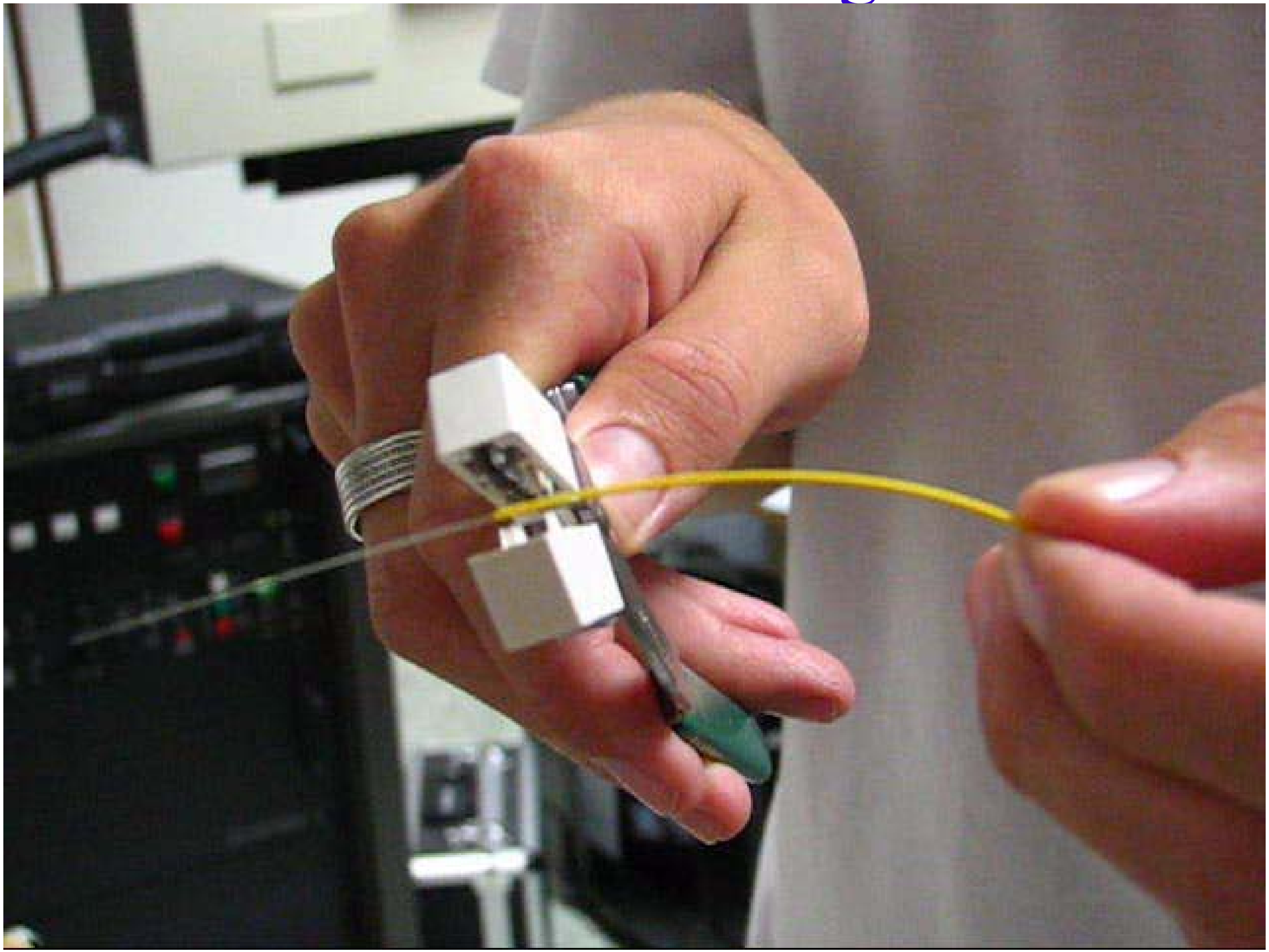


Optical losses : SM ~ 0.1 dB

- Preparation : flame frocessing + protective coating

Optical fiber tapering

Fiber decladding



Fiber cutting

Instrumentation consideration

Requirements :

- sensitivity - LOD
- selectivity
- reproducibility
- dynamics - time response
- reliability-stability ...

- **Method**

(optical, ultrasonic, electronics...)

- **Optical hw** & chemical sw
  - 1. **Planar / fiber-optic**
  - **2. Spectral range**
  - **3. Structure OF**
  - **4. Coatings & transducers**

- Expected utilization (market)
- Price

- **Feasible and easy ??***

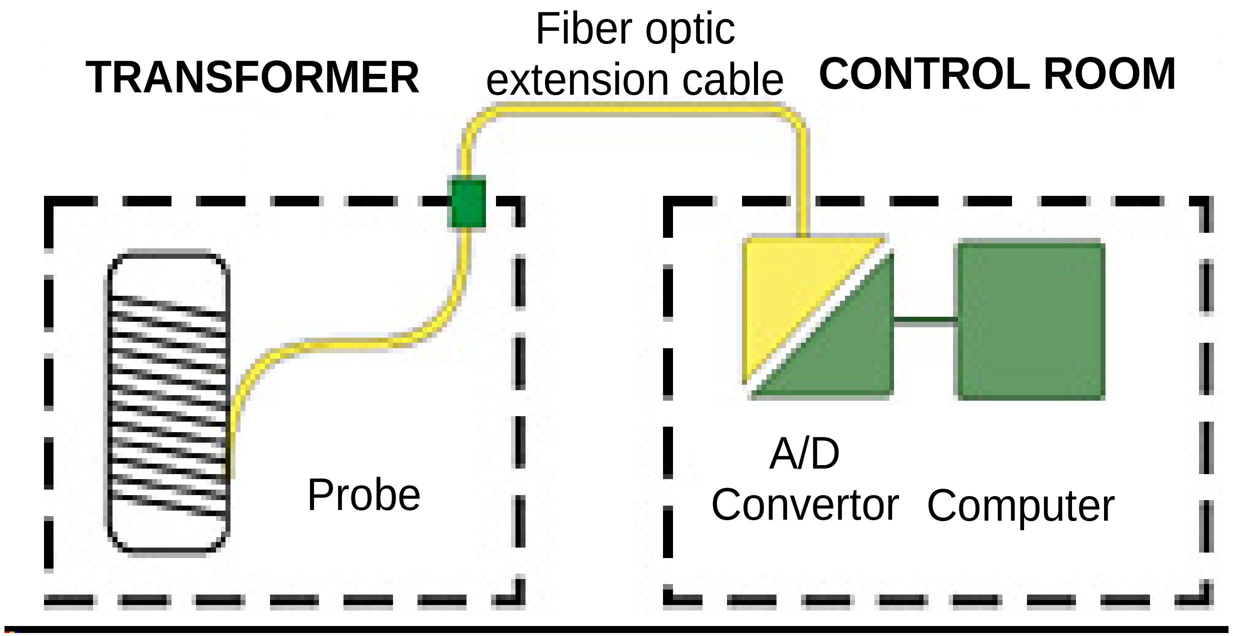
* Parkinson : The more complicated system, the higher probability of its failure.

*Murphy : What can go wrong, it will.

Indispensability of optical fibers

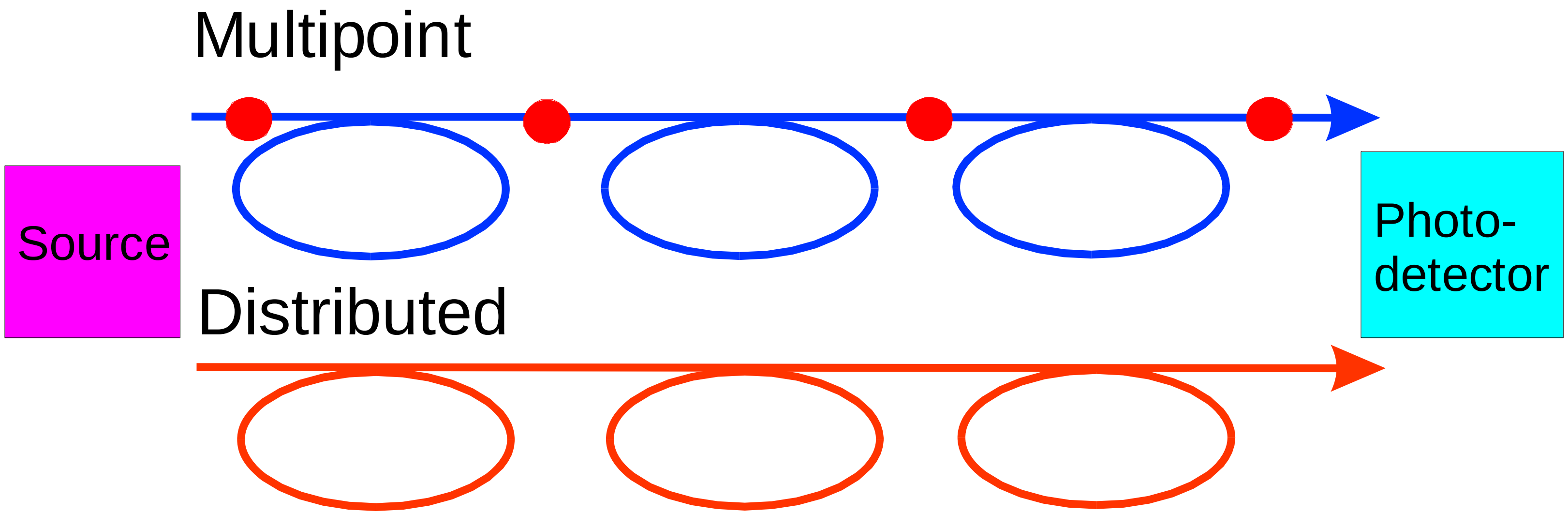
- **Medicine, small points** [F.Baldini, O.Wolfbeis–oxygen sensor in blood]
- **High-voltage areas**

  [www.FISO.com]



- **Explosives**, easily **flammable** or corrosive substances detection

- **SUITABILITY : distributed or multipoint** sensing [Wolfbeis, Birck, McCraith, Chomat -detection of gasoline leakage]



- *~0.5 \$/1m POF < ~5 \$/1 m PCS_{200} < ~ 50 \$/ 1"-silica substrate*
- *75% of production of laser sources are compatible fiber lasers*

4. Optical fiber sensor consideration - example

Micro point-sensor of pH

Remorost

Micro point-sensor of pH - Remorost

Requirements :

- DETECTION OF AUXIN FLOW
- selectivity : via pH
- pH range : <5.5 ; 7>
- time response : seconds
- stability : day(s) - recoverable probe
- SPATIAL RESOLUTION: 1-4 μm

- **Method**

Electrochemical : Diamond; 30 μm

Optical : OceanOptics; 30 μm

[Kopelmann] : oxygen ~1 μm

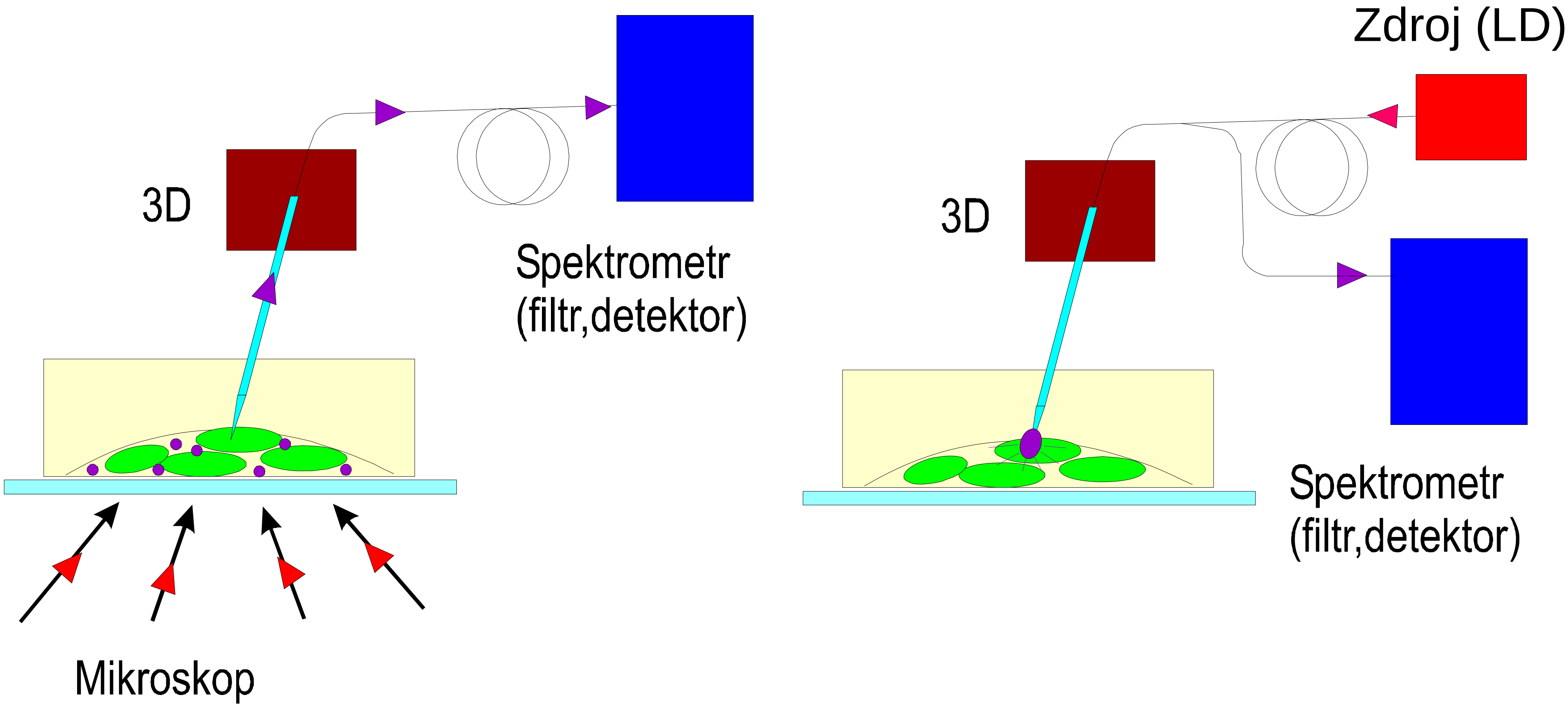
Optical hw & chemical sw

- 1. F**iber-optic; taper**
- **2. Spectral range; visible**
- **3. Structure OF; SM**
- **4. Transducer : fluorescence**

- Expected utilization (market): unique - scientific
- Price : not important

- **Feasible and easy ??***

Navrhované přístupy



Komponenty řešení

- 1. Návrh metody : fluorescenční měření intenzit (poměru)
- 2. Volba převodníku pro pH 5.5-7 : fluorescein, karboxy-fluorescein, DHPDS
- 3. Návrh bodové sondy : taper (adiabatický) s pokryvem polymeru pro zajištění pevnosti + experimentální příprava
- 4. Imobilizace převodníku : do tenké vrstvy TEOSu připravené metodou sol-gel; na čelo taperu cca 1-3 μm.
- 5. Volba excitačního zdroje v závislosti na převodníku : modrá LD (405 nm)
- 6. Návrh vlnovodné struktury : SM – cut off 410 nm.

Komponenty řešení - II

- 7. Návrh optického uspořádání (navazovací kolimační optika, coupler, pulzní režim excitace ...) + nástroje pro penetraci skrz buněčnou stěnu.
- 8. In vitro testy jednotlivých uspořádání. Citlivost (intenzita signálu) ? 3D-optomechanika ??

Acknowledgement

References

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- **K.T.V.Grattan, B.T.Meggitt:** : Optical Fiber Sensor Technology, Vol.4, Kluwer, 1999
- **O.S.Wolfbeis et.al. :** Optical sensors, 2004
- **F.Baldini et.al :** Optical chemical sensors, Springer 2006

- **S.E.Miller**, A.G. Chynoweth : “Optical Fibre Telecommunications”, Acad. Press., London,1979

Preparation of Optical Fibers Excursion - UFE

1) **Physical design** of structure

   Material choice

   SCIENCE

2) **Preparation of** original glass material - **preform**

   Ultra-pure **TECHNOLOGIES** !!!

   CVD – MCVD [(**M**odified) **Ch**emical **V**apour **D**eposition]

   (melting in crucible - history)

3) **Fibre drawing** from preform

   (drawing from crucible - history)