POLITECNICO DI TORINO

# Specialty phosphate fibres for advanced lasing and sensing applications

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Light

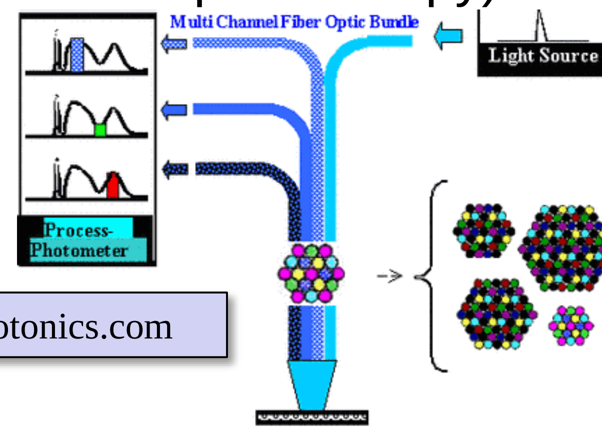
# Overview

- ✓Optical fibres in medicine
- ✓Bioresorbable optical fibres
- ✓Fiber Bragg Gratings in bioresorbable fibres
- ✓Conclusions

# Optical fibres for biomedicine

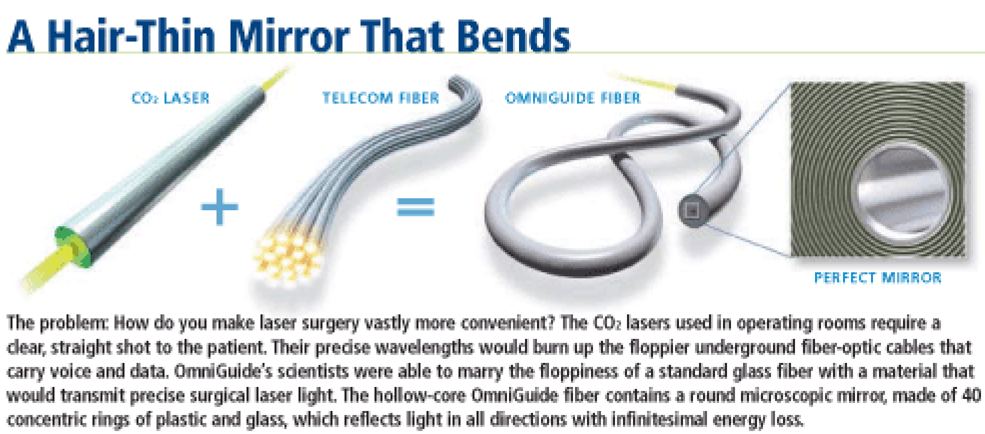
Optical fibres are employed for numerous bio- and medical applications such as:

- diagnosis (imaging, signal detection, fluorescence spectroscopy)
- therapy (laser delivery and surgery)
- real-time sensing




www.artphotonics.com

www.fiso.com

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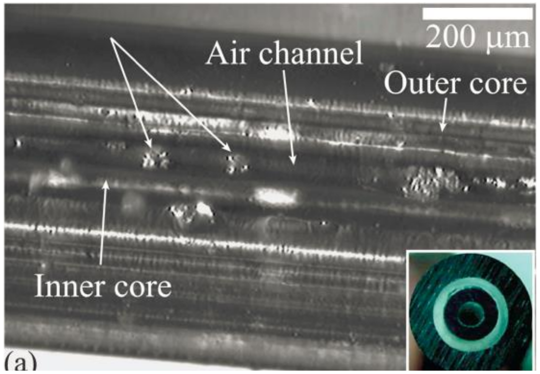
# Bio-resorbable optical fibres

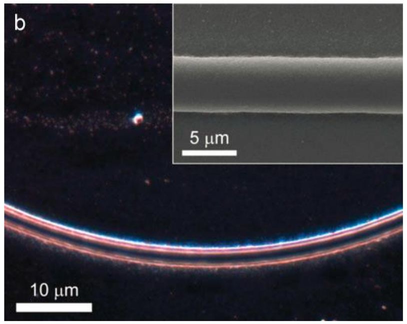
- So far development of optical fibres for biomedicine focussed on bio-compatibility issues
- Recent research efforts focussed on **bio-resorbable** optical fibres, which…
  - can be destroyed by the human body without leaving any harmful residual
  - can be inserted during interventions and left in place to monitor the healing process or detect inflammation or abnormal response
  - can be used to track the regeneration of tissues in implants with no burden
  - are of interest for interstitial photodynamic and photothermal therapies with in-situ irradiation

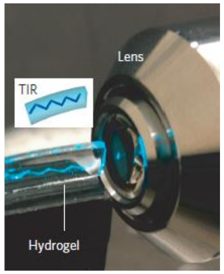
# Bio-resorbable materials

- Bio-degradable waveguides and fibres have been developed mainly for sensing applications
- Materials investigated so far:
  - Cellulose butyrate and hydroxypropyl cellulose *[Dupuis et al., Opt. Lett. **2007**, 32, 109]*




  - Agarose - gelatin – agarose *[Manocchi et al., Biotechnol. Bioeng. **2009**, 103, 725]*
  - Silk *[Parker et al., Adv. Mat. **2009**, 21, 1]*




  - Polyethylene glycol-based hydrogel *[M. Choi et al., Nat. Photonics, **2013**, 7, 987]*

# Phosphate glass in Photonics and in Biomedicine

**Phosphate glasses** are promising materials in the fields of Photonics and Biomedicine:

- High solubility in aqueous media
- Good thermo-mechanical properties
- Low non-linear refractive index and low attenuation loss
- Transparency in UV-Vis/NIR region (300 to 2600 nm)
- Mature manufacturing technology

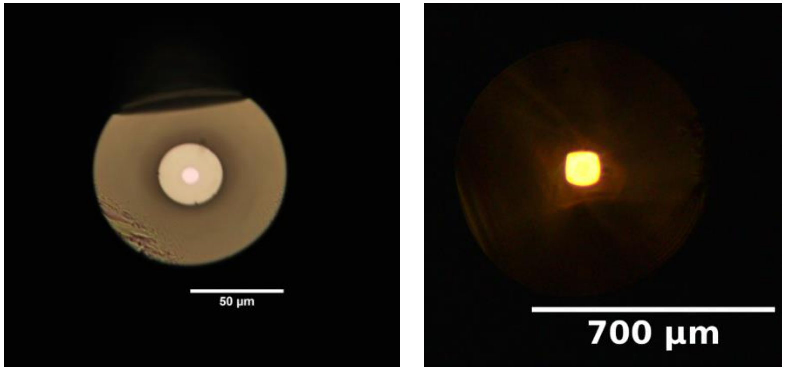
M. Yamane, Y. Ashara: Glasses for Photonics, Cambridge Univ. Press, 2000

C. Spiegelberg *et al.*, *J. Lightwave Technol.* **2004**, 22, 57

H. Gao *et al.*, *J. Controlled Release* **2004**, 96, 21

# Phosphate glass in Photonics and in Biomedicine

## In Photonics

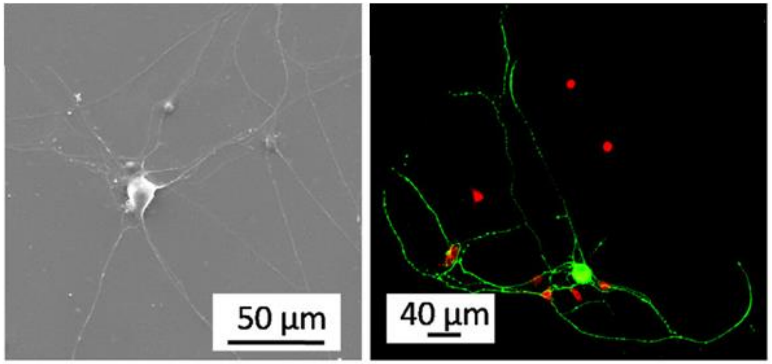



- Laser generation
- Laser amplification
- Special fiber configurations

*N. G. Boetti et al., J. Opt. (2015) 17: 065705*

*D. Pugliese et al., J. Alloys Compd. (2016) 657: 678-683*

## As a Biomaterial




- Bone scaffolds
- Soft tissue engineering
- Axonal growth direction

*C. Vitale-Brovarone et al., Acta Biomater. (2012) 8: 1125-1136*

*G. Novajra et al., Mater. Sci. Eng. C (2016) 67: 570-580*

# Aims and applications

## *CONCEPT*

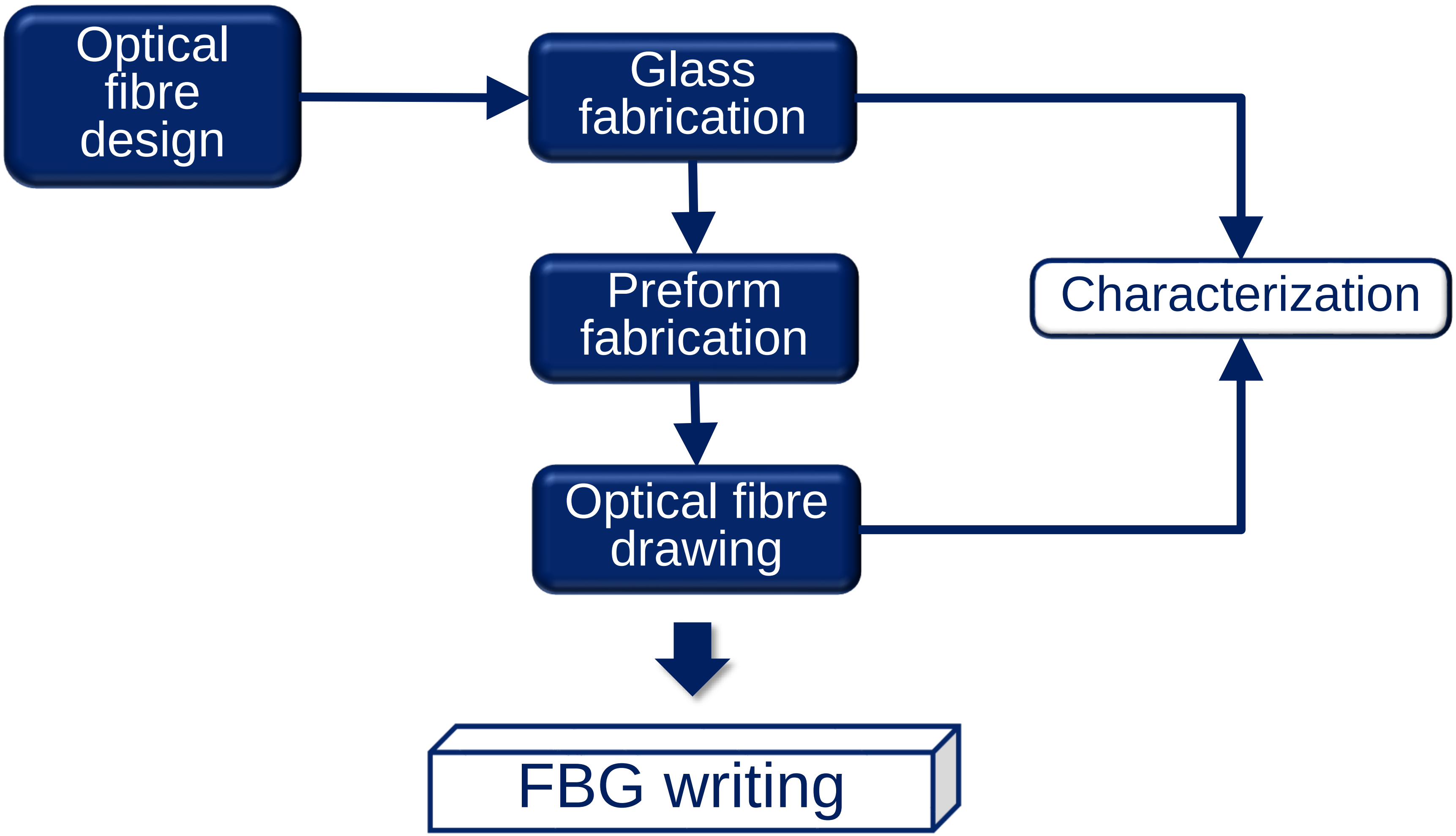
Combine in a single material optical and biomedical functionalities

## *AIM*

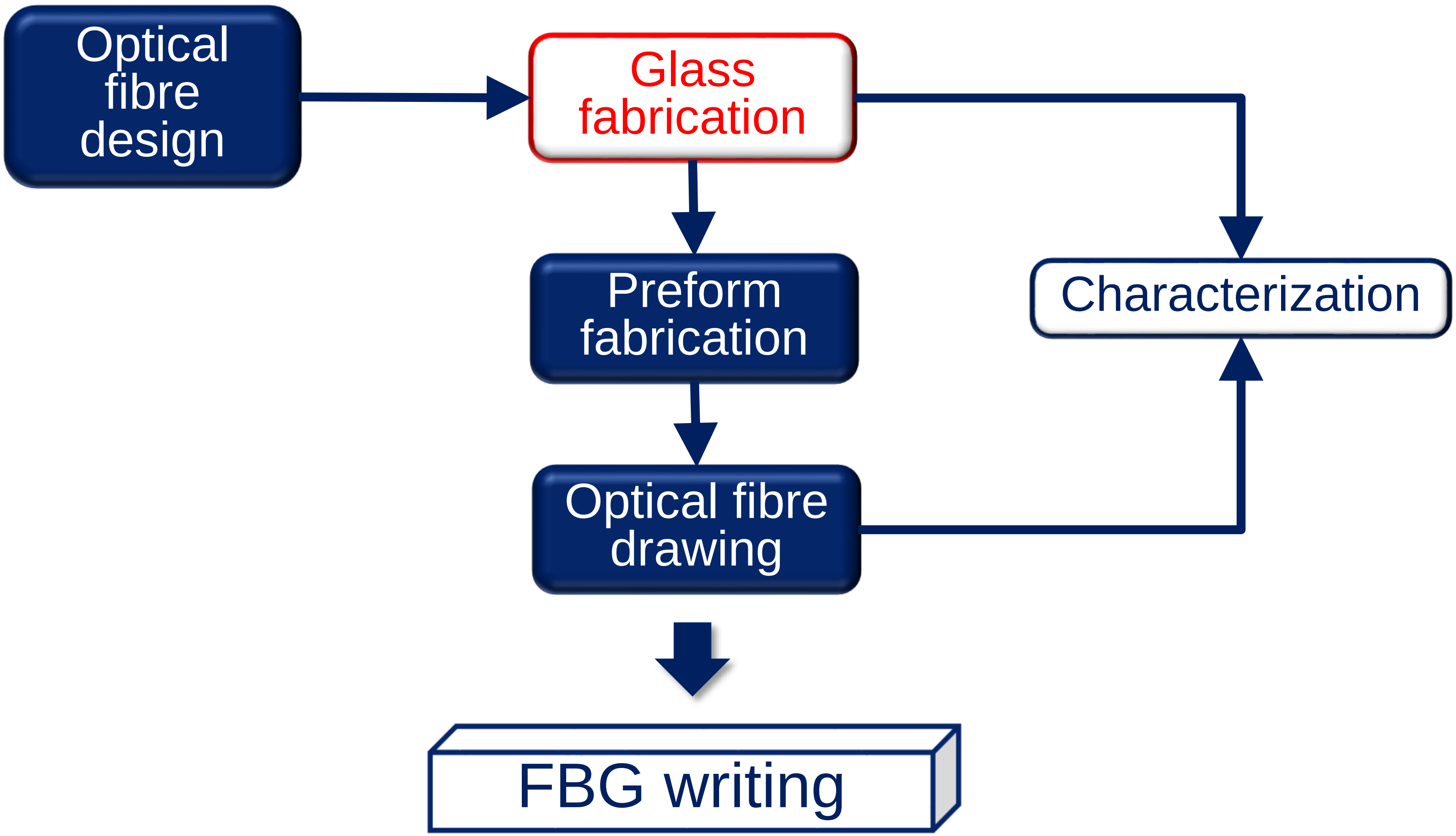
Develop phosphate optical fibres with the following properties:

- Optically transparent (extended to UV) and biocompatible
- Soluble in biological fluids (bio-resorbable)
- Suitable for fiber drawing
- Photonsensitive $\rightarrow$ Fibre Bragg Grating (FBG) fabrication

# Research activity



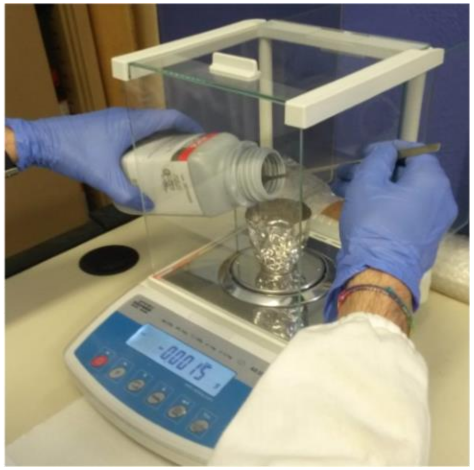
# Research activity



# Glass fabrication

## Glass components

$P_2O_5 – CaO – Na_2O – SiO_2 – MgO$

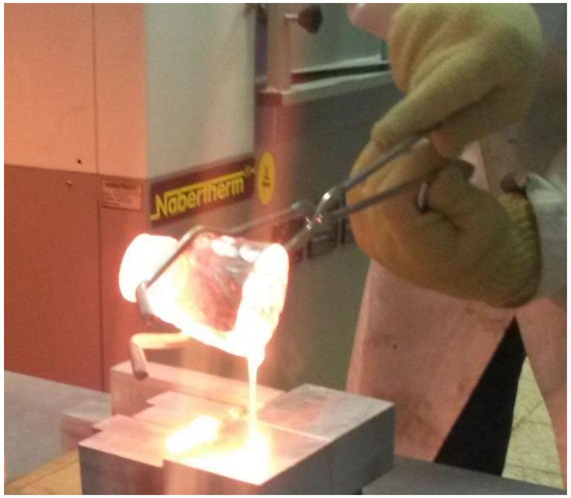


### Weighing

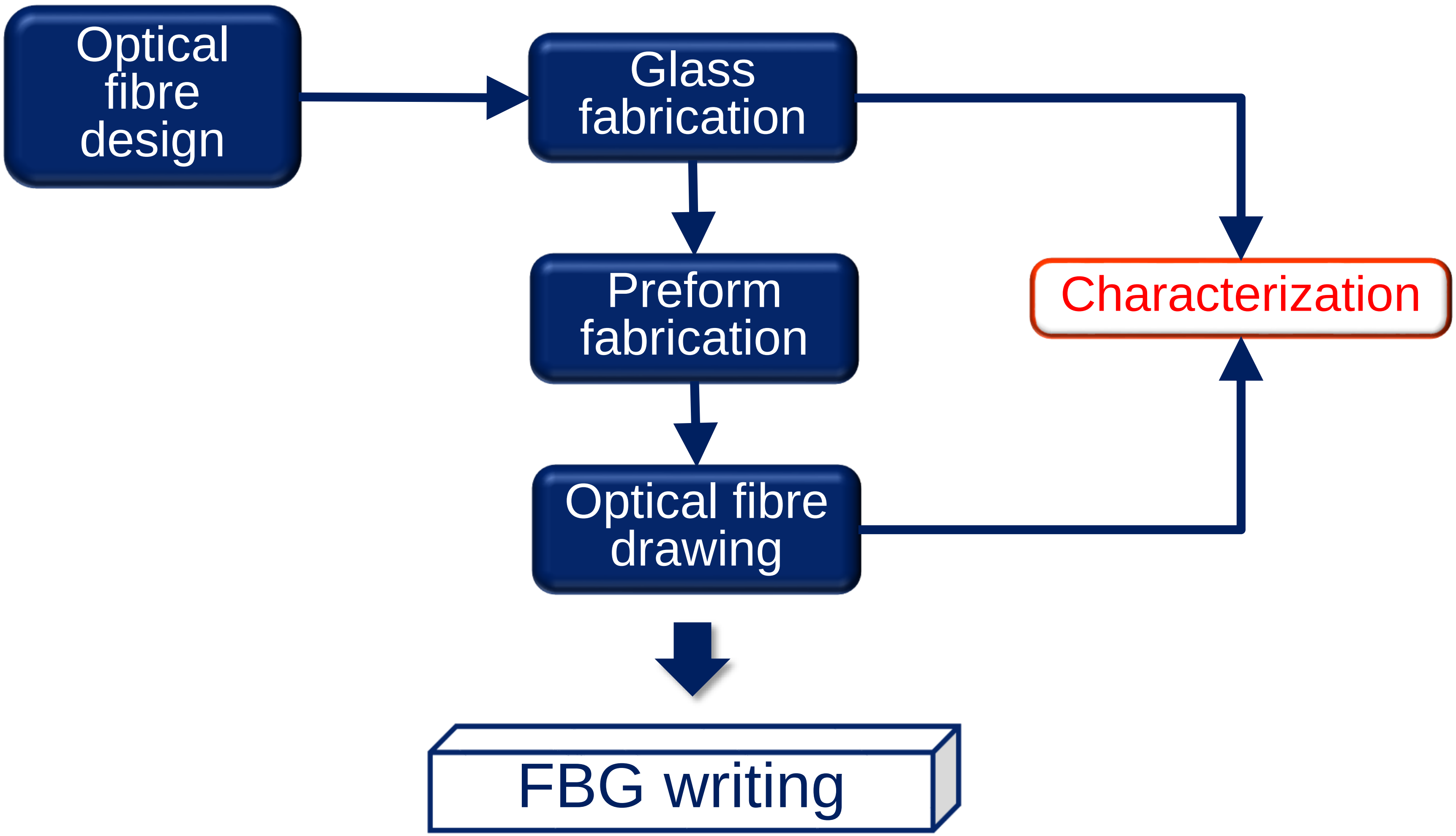
- High purity chemical precursors
- Weight in glove box

### Melting under controlled atmosphere

- Pt crucible
- Temperatures 1150 to 1250 °C
- Flow of dried air (< 3 ppm $H_2O$)
- Cast into metal mould
- Annealing at $T_g$°C for 3 hours

# Research activity

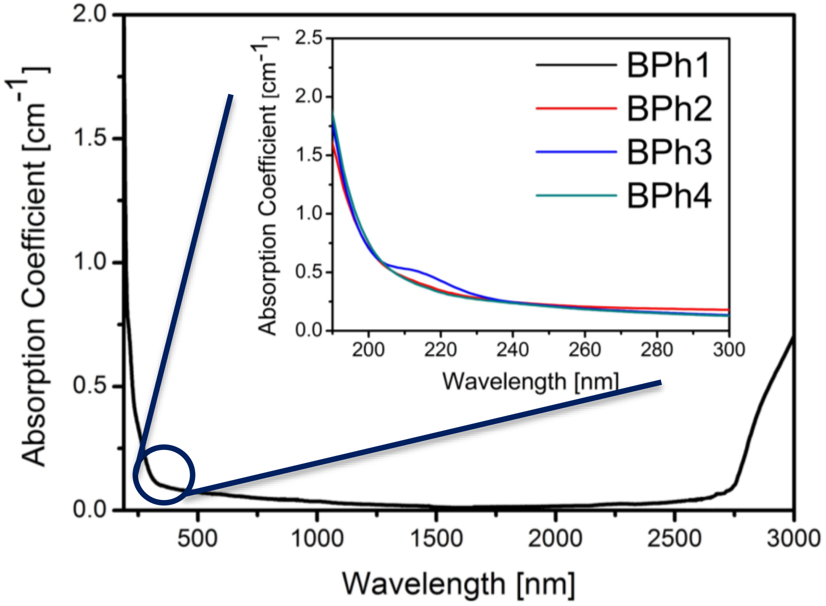


# Glass thermal characterization

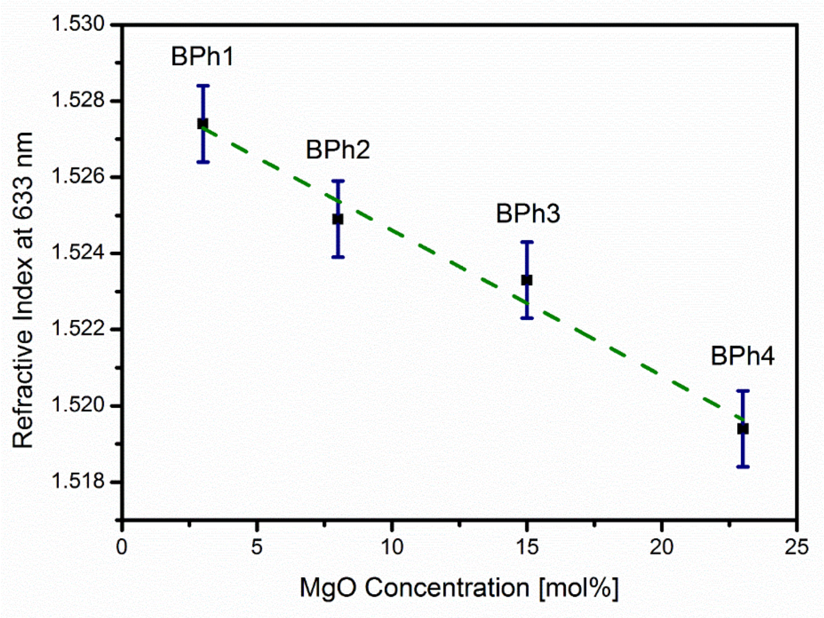
| Glass name | MgO (mol%) | $T_g$ ± 3 (°C) | $T_x$ ± 3 (°C) | $\Delta T = T_x - T_g$ ± 6 (°C) | CTE ± 0.1 ($10^{-6}$ $K^{-1}$) | $T_s$ ± 3 (°C) | $\rho$ ± 0.005 ($g/cm^3$) |
|---|---|---|---|---|---|---|---|
| **BPh1** | 3 | 435 | 658 | 223 | 12.6 | 467 | 2.606 |
| **BPh2** | 8 | 435 | 628 | 193 | 12.2 | 461 | 2.600 |
| **BPh3** | 15 | 442 | 632 | 190 | 12.0 | 471 | 2.598 |
| **BPh4** | 23 | 444 | 625 | 181 | 12.2 | 477 | 2.589 |

- Glass stability parameter $\Delta T \sim 200$ °C → suitable for fiber drawing
- Increase of MgO in substitution of CaO:
  - to tune the refractive index for core/cladding fabrication
  - has little effect in changing the density of the glasses
  - decrease of thermal stability and limited increase of $T_g$

# Optial characterization




## Refractive index

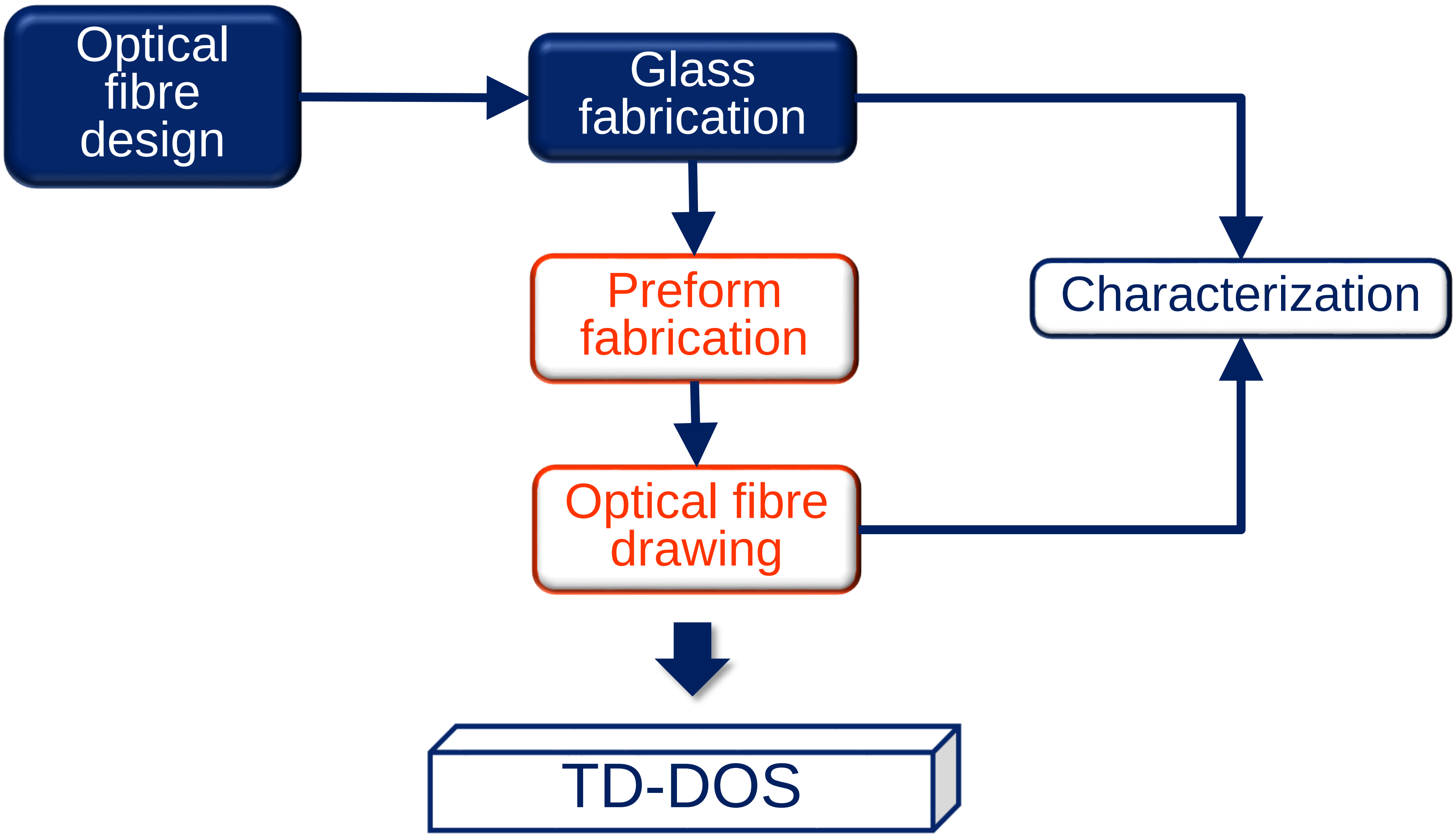



- UV edge at around 240 nm
- 1 mm sample thickness

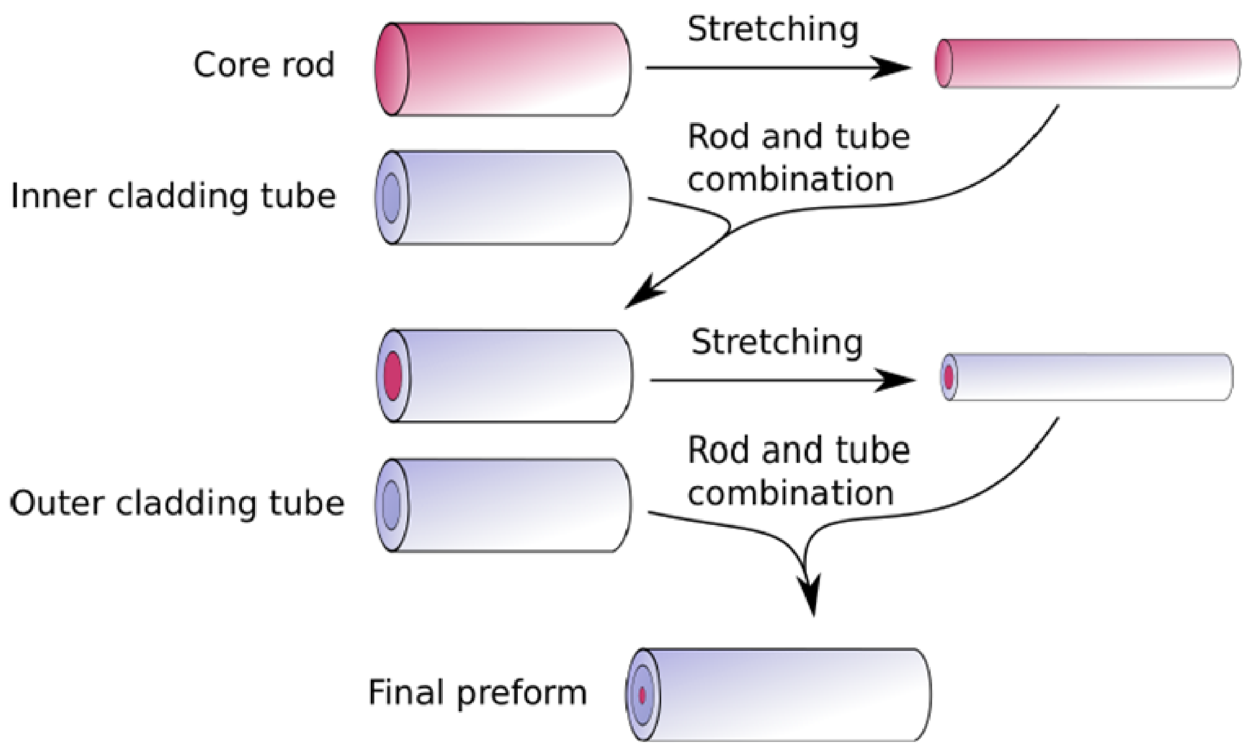
| Glass name | NA @633 nm |
|---|---|
| BPh1/BPh2 | 0.08 |
| BPh1/BPh3 | 0.11 |
| BPh1/BPh4 | 0.15 |

E. Ceci-Ginistrelli *et al.*, *Opt. Mater. Express* **2016**, 6, 2040

# Research activity



# Preform fabrication

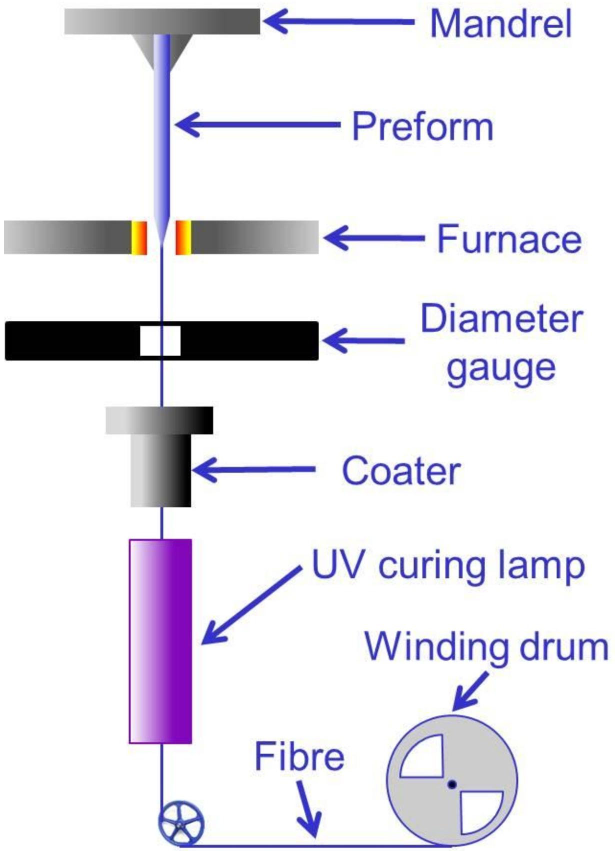



Tubes by rotational casting:

- O.D. from 12 to 20 mm
- I.D. from 4 to 8 mm (± 5% from end to end)
- Roughness < 10 nm (commercial silica tubes)

# Fibre drawing

## Fibre drawing tower (the only R&D drawing tower in Italy!)




**Main features & performances**

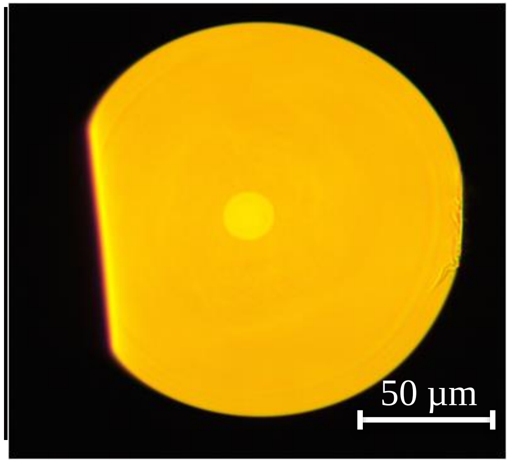
- Drawing Temperatures up to 1000 °C
- Typical drawing speed: 4 to 50 m/min
- Diameter fluctuations: ± 3 %

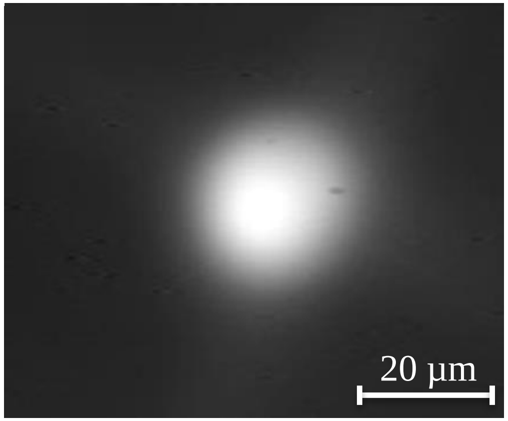
# Characterization of the optical fibres

## Single-mode fiber / Multi-mode fiber

| | |
|---|---|
| Core glass | BPh1 / BPh1 |
| Cladding glass | BPh2 / BPh4 |
| Core diameter | 15 µm / 45 µm |
| Fiber diameter | 120 µm / 125 µm |

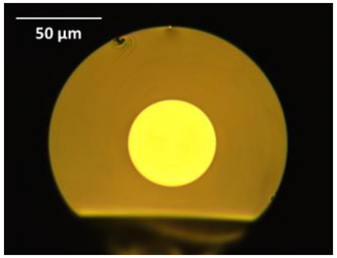
| | |
|---|---|
| Loss at 1300 nm | 2 dB/m / 6 dB/m |
| Loss at 633 nm | 14 dB/m / 5 dB/m |
| Numerical Aperture | 0.08 / 0.15 |



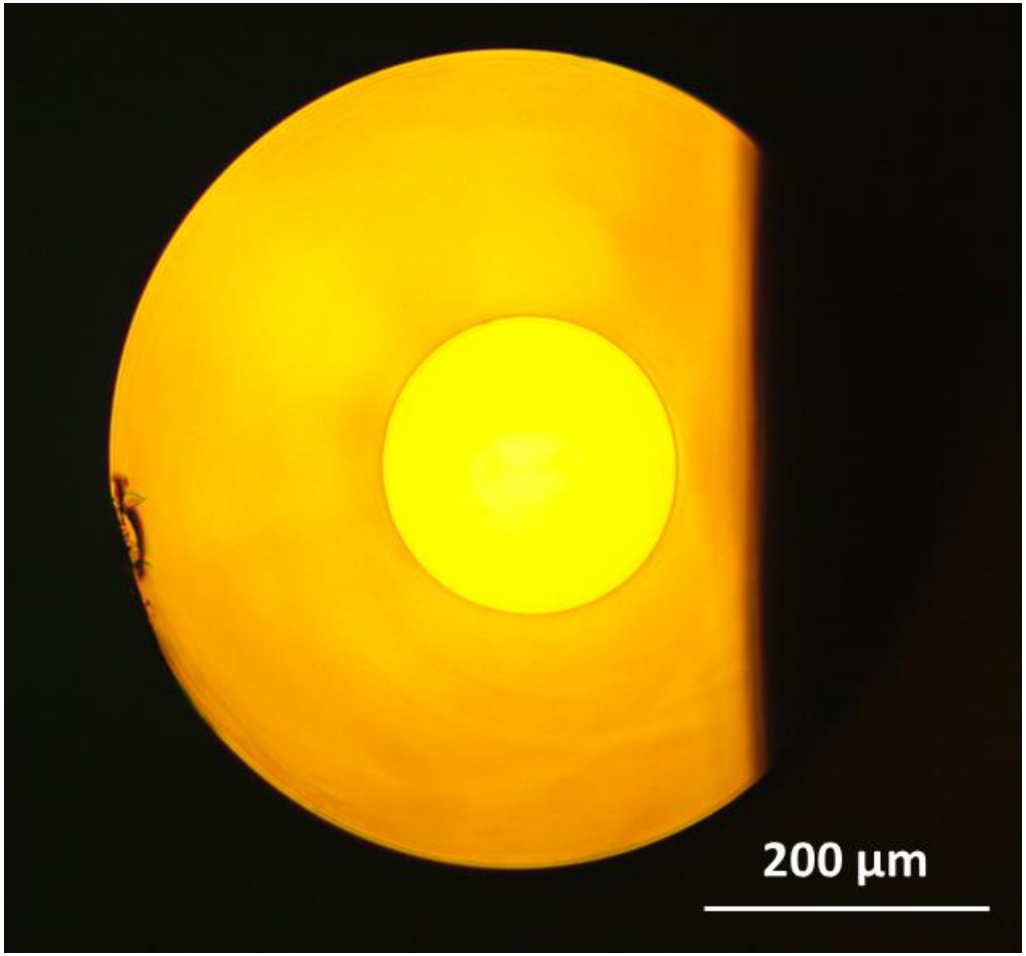

E. Ceci-Ginistrelli *et al.*, *Opt. Mater. Express* **2016**, 6, 2040

# Bioresorbable fibre dimensions




Core 50 µm

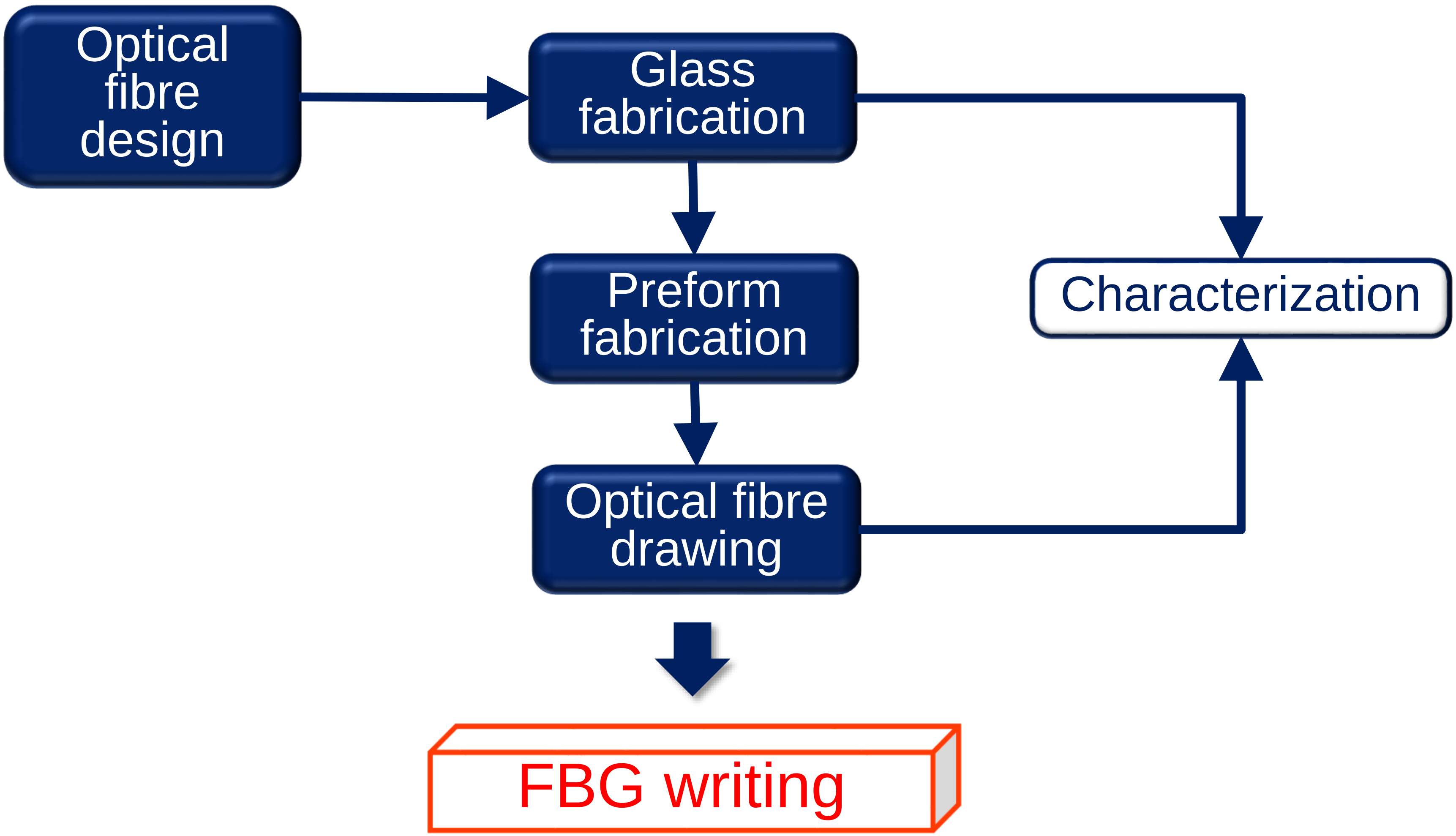
Cladding 150 µm




Core 200 µm

Cladding 600 µm

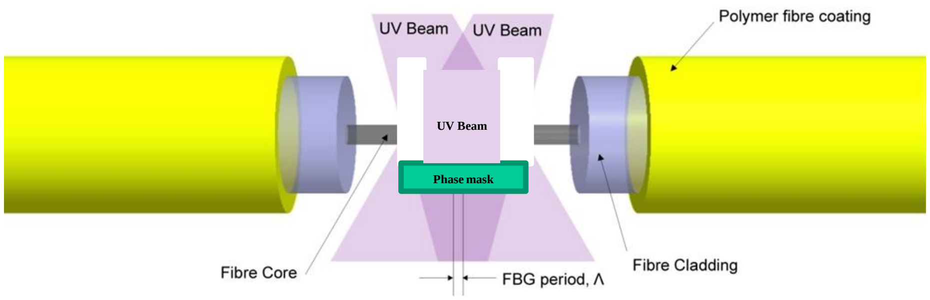
# Research activity

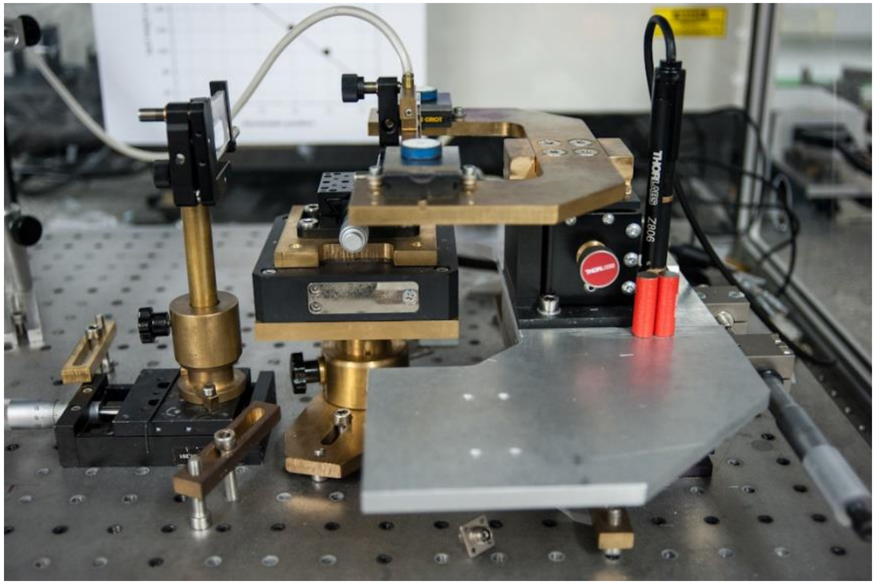



Work carried out during STSM1

# Bragg grating inscription set up

- Standard phase mask set up
- 193 nm, 10 ns excimer laser
- 1064.7 nm period mask
- 108 mJ/cm$^2$, 10 -20 Hz
- 5 mm long FBG
- Phosphate fiber spliced to SMF

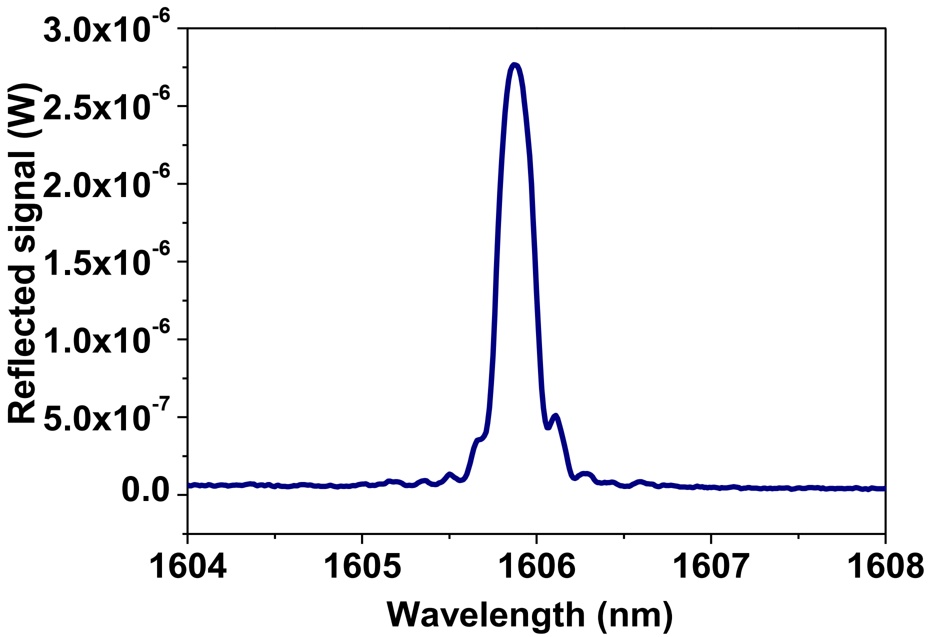


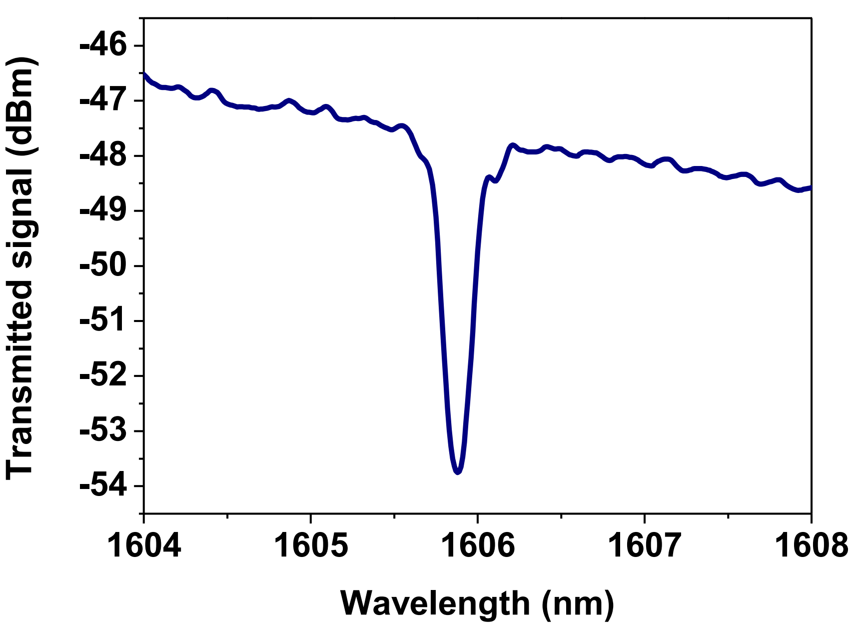
*M. Konstantaki et al., Proc. ICTON 2016*

# FBG inscription

- 108 mJ/cm$^2$ pulse energy density,
- 100 min inscription, accumulated energy density ~7 kJ/cm$^2$
- Well defined symmetric 6 dB FBG




Reflected signal

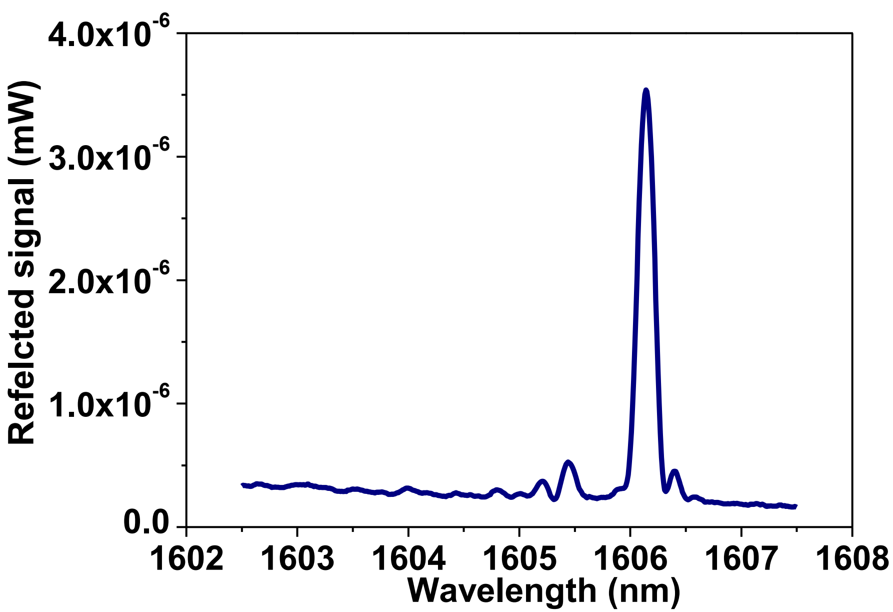



Transmitted signal

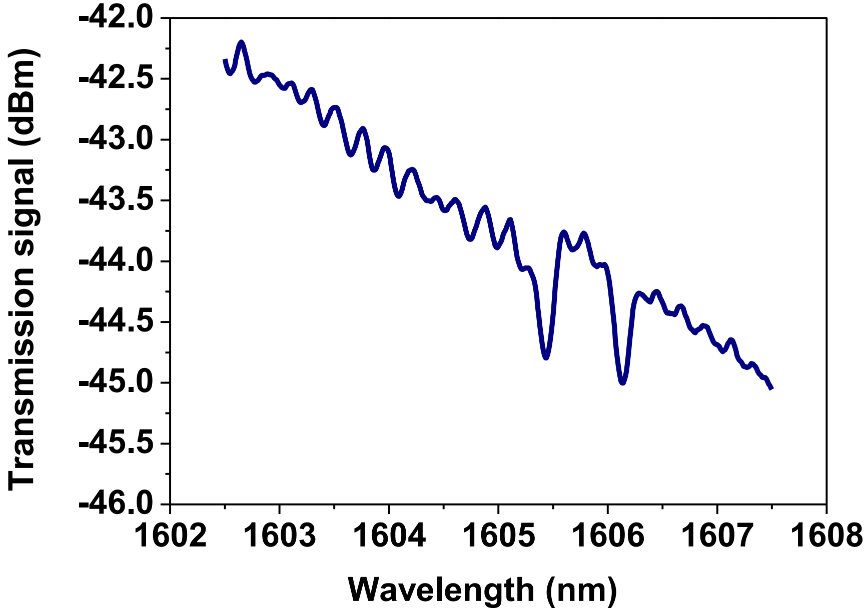
*M. Konstantaki et al., Proc. ICTON 2016*

# Tilted FBGs inscription

- Inscription conditions same as with standard FBG
- Phase mask tilted with 1º tilt angle with respect to the fiber axis



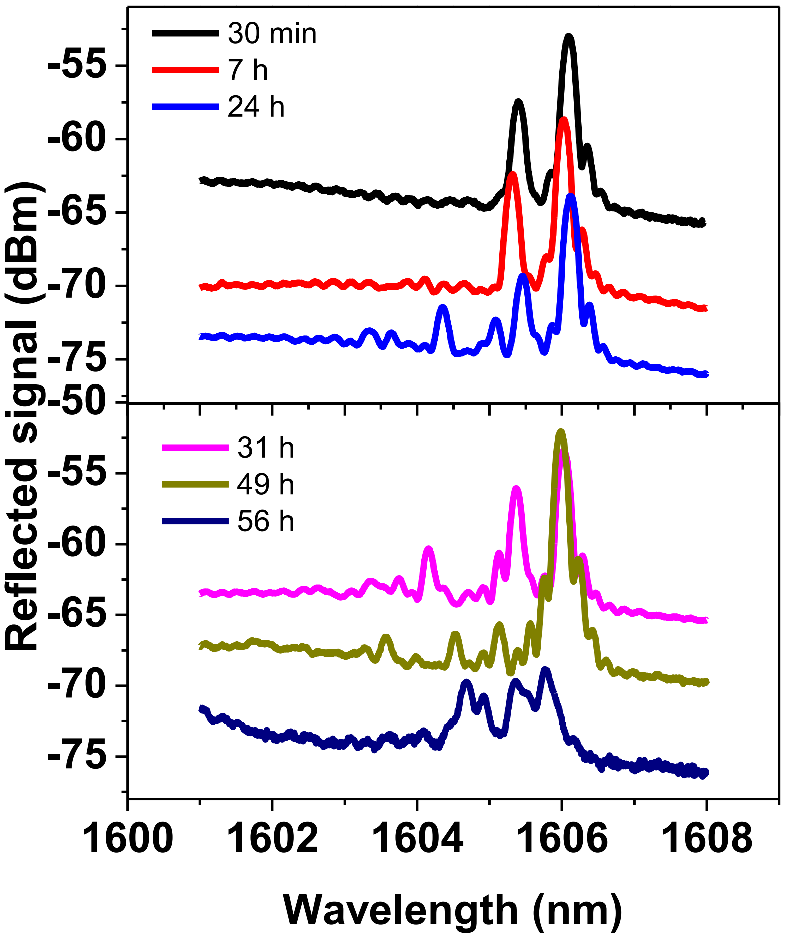

Reflected signal

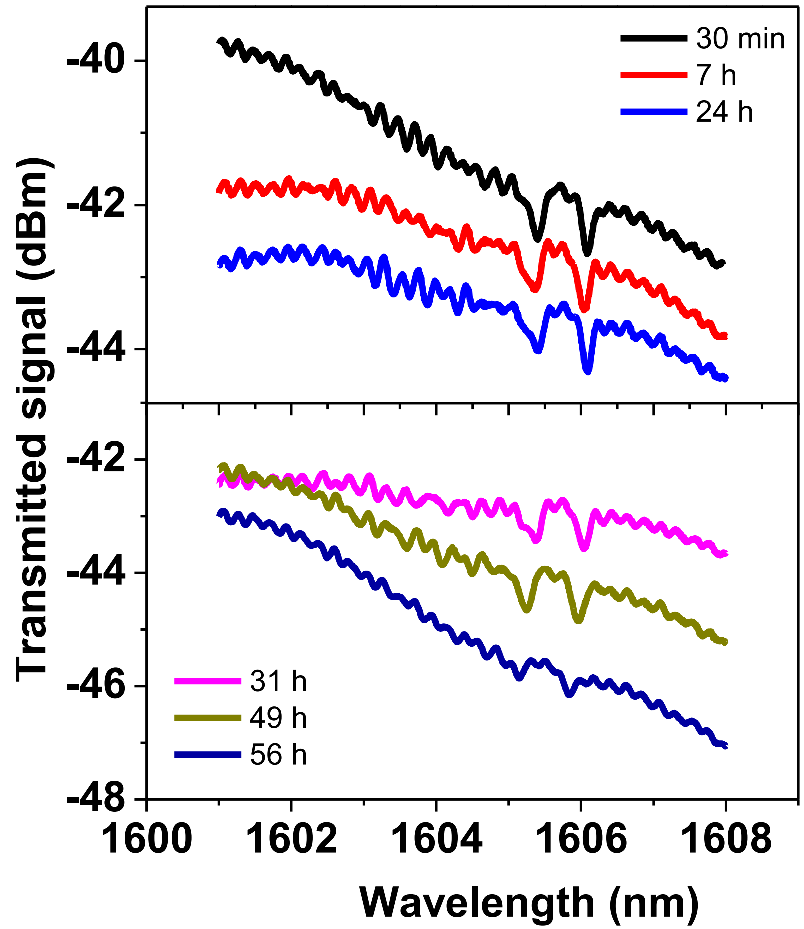



Transmitted signal

*M. Konstantaki et al., Proc. ICTON 2016*

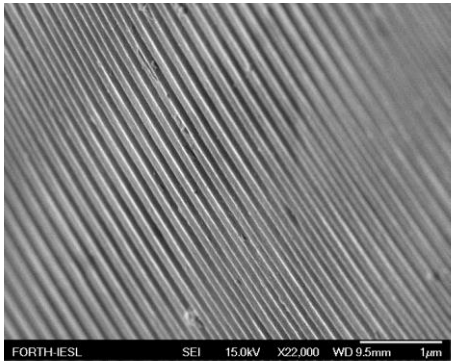
# FBG spectral evolution during dissolution in PBS



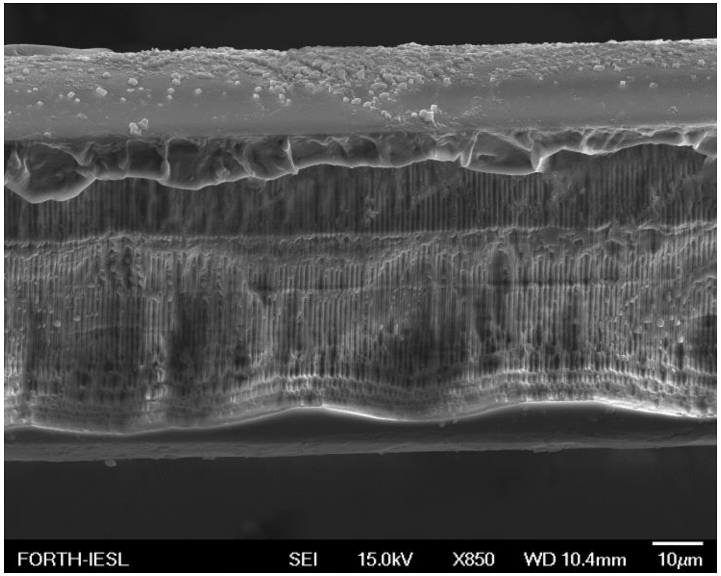




*M. Konstantaki et al., Proc. ICTON 2016*

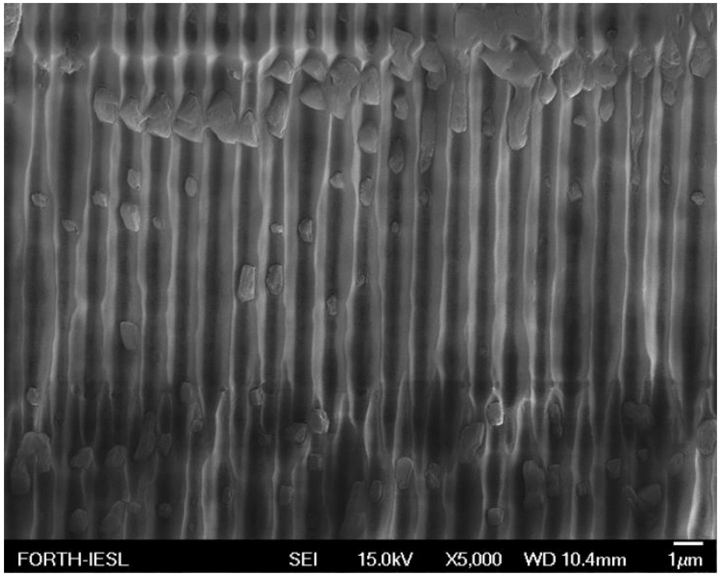
# UV irradiated phosphate fiber dissolution in PBS




Fiber surface in contact with UV beam **8 h in PBS**




**3 days in PBS**




*M. Konstantaki et al., Proc. ICTON 2016*

# Conclusions and future developments

- Novel optical bioresorbable glasses were fabricated and characterized
- The glasses were successfully drawn into fibres:
  - single glass solid
  - single glass (hollow)
  - core/cladding with low loss (comparable to s-o-t-a phosphate optical fibres)
- Successful FBG writing using laser @193 nm using phase mask:
  - Uniform FBGs
  - Tilted FBGs
  - Test of FBG dissolution in PBS

# Ongoing work and future developments

- Demonstration of diffuse optics experiments using bioresorbable fibres (in collaboration with A. Pifferi et al., Politecnico di Milano)
- Towards 1.5 $\mu$m laser source for biomedicine:
  - Yb/Er doped bioresorable glass fabrication and characterization
  - 1.5 $\mu$m bulk and fibre laser demonstration (in collaboration with S. Taccheo @Swansea University, UK)
  - Integration of FBGs and active fiber for DFB lasers

# *Optical Materials Express* Feature Announcement

## “Multimaterial and Multifunctional Optical Fibers”

- **Submission Opens:** 1 December 2016
- **Submission Deadline:** 15 January 2017

This special issue will highlight the most recent results in the field of multimaterial optical fibers, including new materials, new processes, and new functionalities

Contributions on applications of novel or existing multimaterial fibers are also welcome, especially highlighting the opportunities offered by multifunctionality in optical fibers, or the advantages of fiber configuration with respect to current technologies

# Thank you for your attention!