Welcome to Today's Webinar!

SOFT GLASS OPTICAL FIBRES: PROPERTIES, FABRICATION AND APPLICATIONS

12 May 2021 • 20:00 EDT (UTC -4:00)

OSA Fiber Modeling and Fabrication Technical Group

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About the Fiber Modelling and FabricationTechnical Group

Our technical group focuses on all aspects related to the Fiber Design, Modelling, Fabrication, and Applications of fibers.

Our mission is to connect the 900+ members of our community through technical events, webinars, networking events, and social media.

Our past activities have included:
Webinars, Campfire sessions, poster session at CLEO US, Special Talk at FIO US, and Networking event at NLO Hawaii.

Connect with our Technical Group

Join our online community to stay up to date on our group's activities. You also can share your ideas for technical group events or let us know if you're interested in presenting your research.

Ways to connect with us:

- Our website at https://www.osa.org/FF
- On LinkedIn at https://www.linkedin.com/groups/8302193/
- On Facebook at https://www.facebook.com/groups/OSAfibermodelingandfabrication/
- Email us at TGactivities@osa.org or contact group chair at deepakjain9060@gmail.com

Today's Speaker

Prof. Heike Ebendorff-Heidepriem

Deputy Director of the Institute for Photonics and Advanced Sensing, University of Adelaide, Australia

Short Bio:

- Ph.D. degree in chemistry from the University of Jena, Germany, in 1994. During 2001–2004, she was with the Optoelectronics Research Centre at the University of Southampton, U.K. Since 2005, she has been with the University of Adelaide, Australia.
- Research Interests: Novel optical glasses, specialty optical fibers, hybrid glasses and fibers, surface functionalization and sensing approaches.
- Director of the Adelaide University Optofab Hub of the Australian National Fabrication Facility (ANFF) and Senior Investigator of the ARC Centre of Excellence for Nanoscale BioPhotonics (CNBP).
- Recipient of Weyl International Glass Science Award and prestigious Marie-Curie Fellowship.

Soft Glass Fibres:
Properties, Fabrication, and Applications

Heike Ebendorff-Heidepriem

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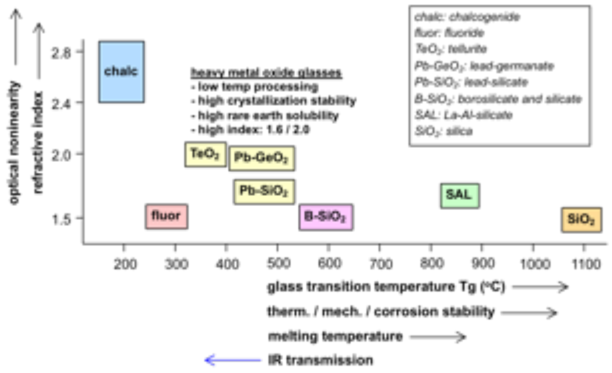
Institute for Photonics and Advanced Sensing (IPAS), University of Adelaide, Australia
Australian National Fabrication Facility (ANFF) – Optofab Adelaide

OSA Webinar, 13 May 2021

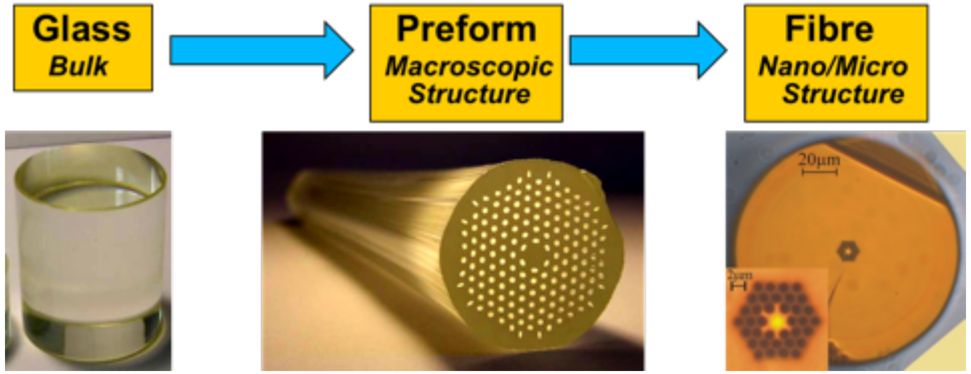
Outline

1. Glass types and properties

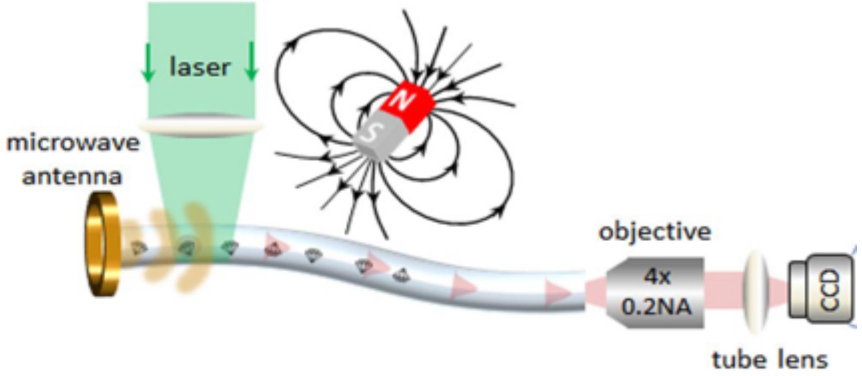


2. Fabrication technologies

- dehydration
- nanocrystal doping
- preform



3. Applications



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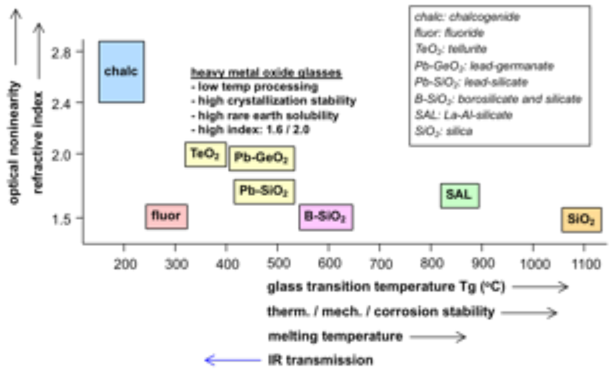

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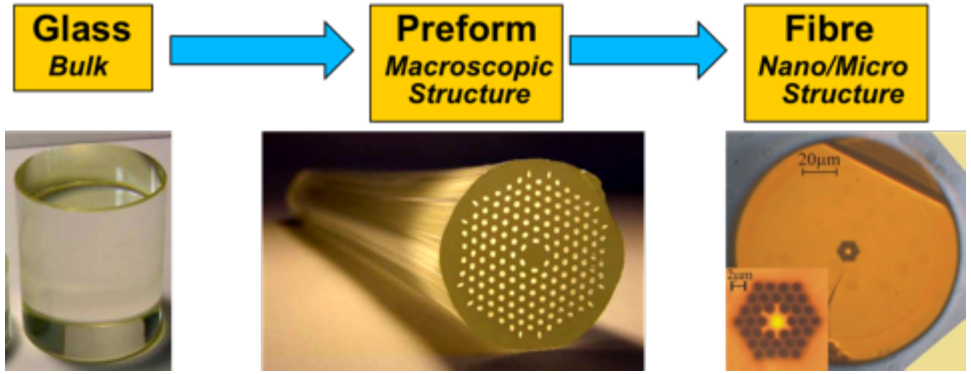
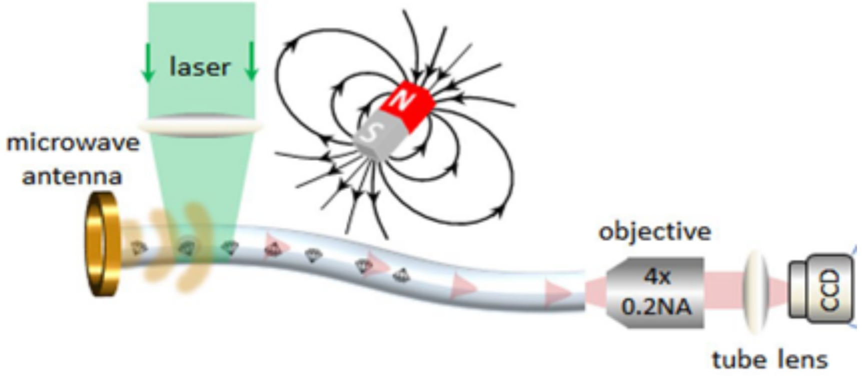
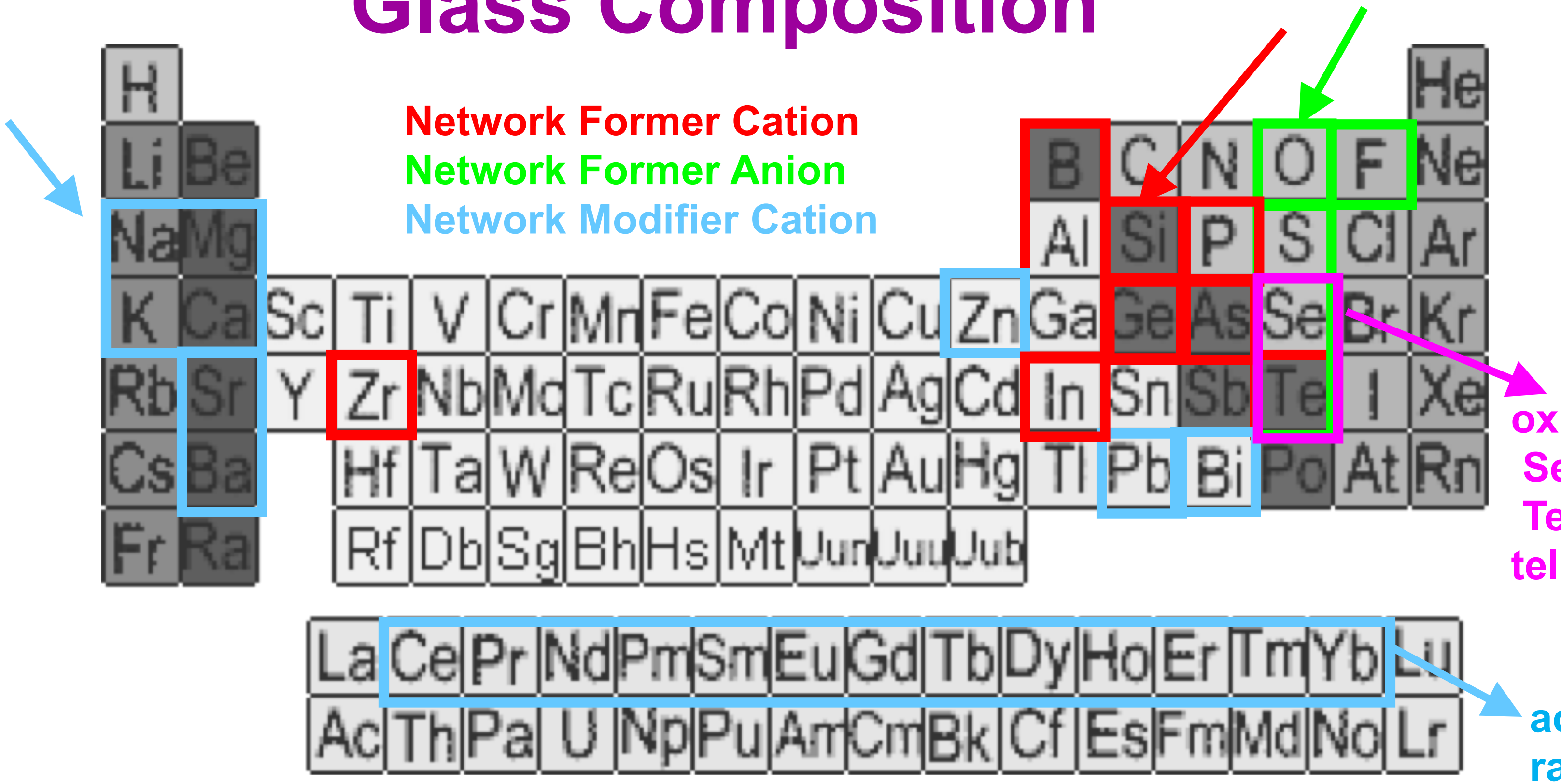


3. Applications



Glass Composition



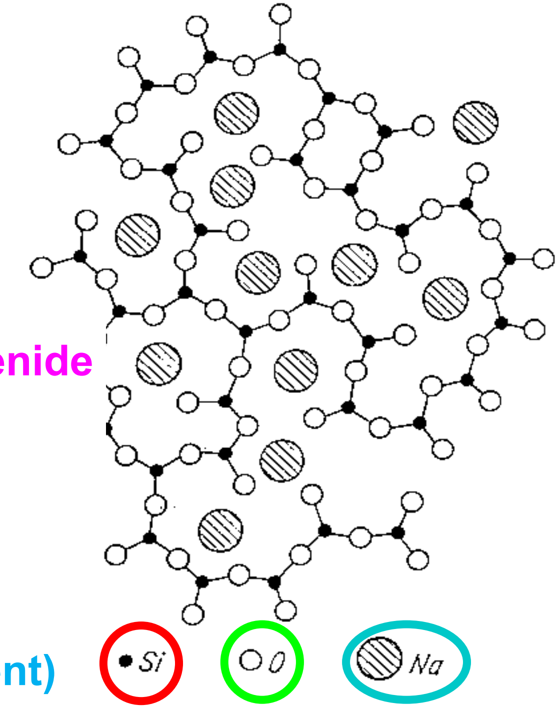
oxide vs chalcogenide

SeO_2 vs As_2Se_3

TeO_2 vs As_2Te_3

tellurite vs telluride

active (luminescent) rare earth ions



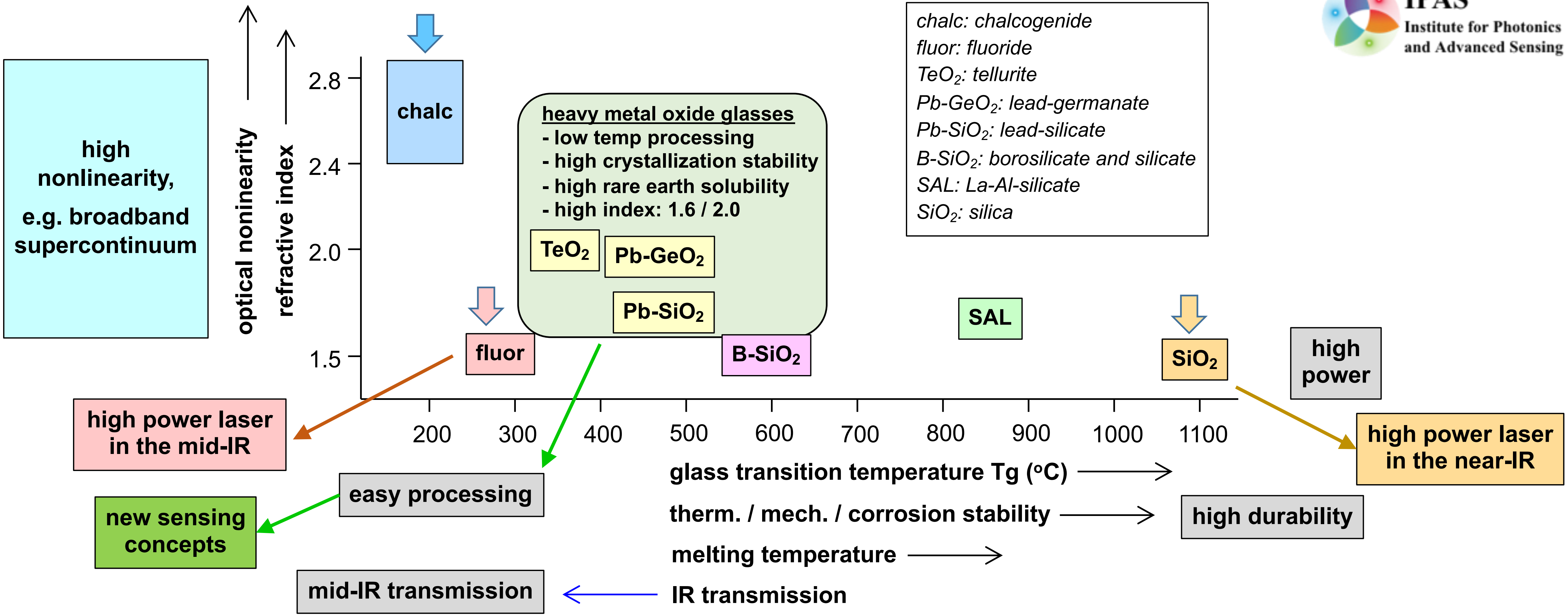
Silica (Quartz glass): SiO_2

- used for the majority of fibres
- superior thermal, mechanical and corrosion stability
- low refractive index
- transmission window (0.2-2.1μm)

Multicomponent Glasses

- oxide (B_2O_3, Al_2O_3, Ga_2O_3, SiO_2, GeO_2, P_2O_5, TeO_2)
- fluoride (ZrF_4, InF_3)
- chalcogenide (As_2S_3, As_2Se_3, GeS_2)
- soft glasses (heavy metal oxide, fluoride, chalcogenide)
- wide range of properties (index, transmission)

Glass Properties



H. Ebendorff-Heidepriem, ECOC, London, Sep2013

Outline

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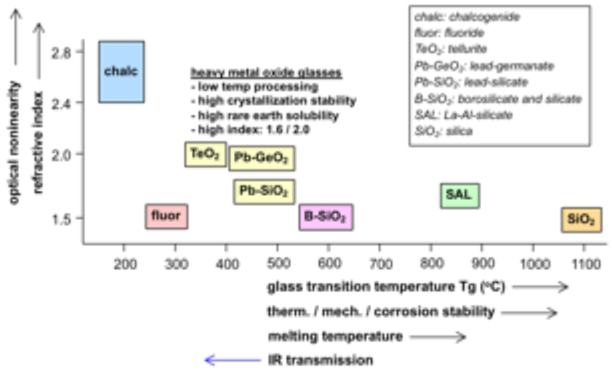

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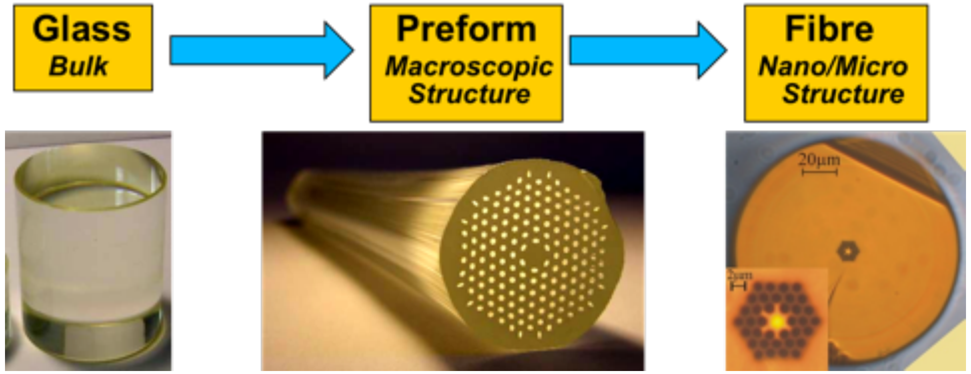
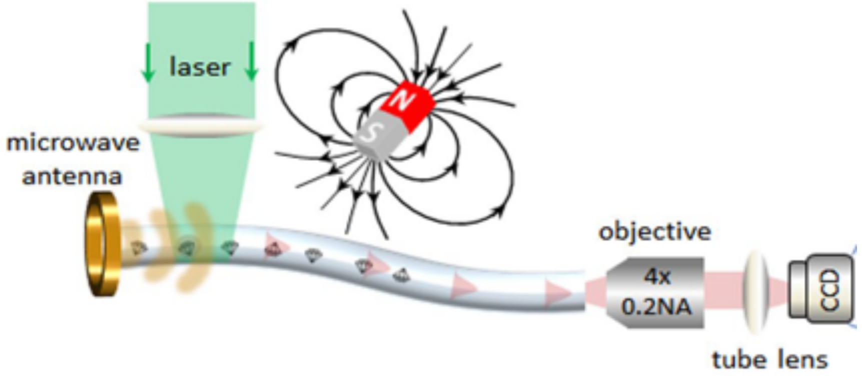


3. Applications



Glass Fabrication Techniques

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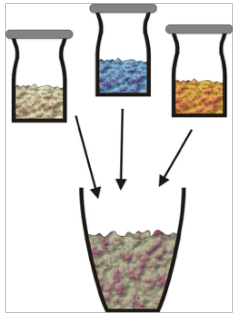
IPAS
Institute for Photonics and Advanced Sensing

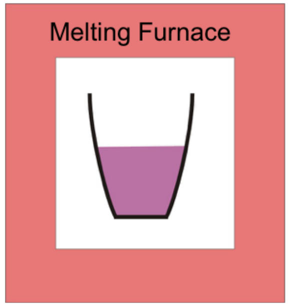
Technique	Application area	Features	Examples
melt-quenching	most widely used used at Optofab Adelaide	any shape and size	window panes, household and lab glass ware
chem/phys. vapour deposition	high purity applications	limited thickness	silica fibre preforms
nanoparticle sintering	high purity applications	some thickness limitation more flexibility than deposition	silica fibre preforms 3D printed items
sol-gel	low temperature processing unusual glass compositions	limited thickness → films	glass coating nanoparticle fabrication

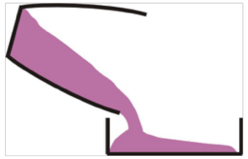
1. → 2. → 3. → 4.

1. **batching** = mixture of powdered crystalline raw materials
2. **heating** of raw materials within crucible → fusion of raw materials = liquid formation
3. **casting** into mould to form into a shape
4. **annealing** and slow cooling to room temp.

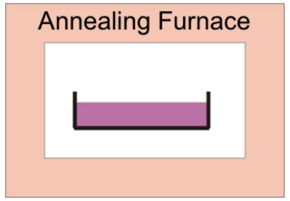


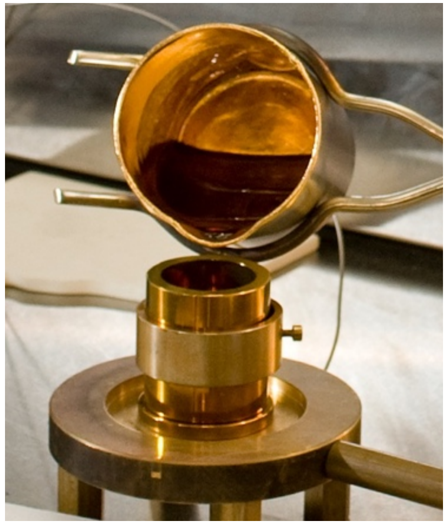
Crucible





Preheated Mould





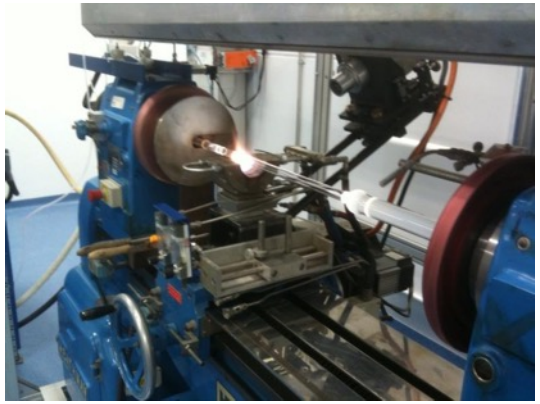
Fabrication → Loss, Properties

chemical/physical vapour deposition

- silica
- ultra-low loss: **0.0002dB/m** at 1.5µm **(close to theoretical loss)**

BUT only few compounds available (Si, Ge, B, P)

➔ **limitation in glass composition → properties**

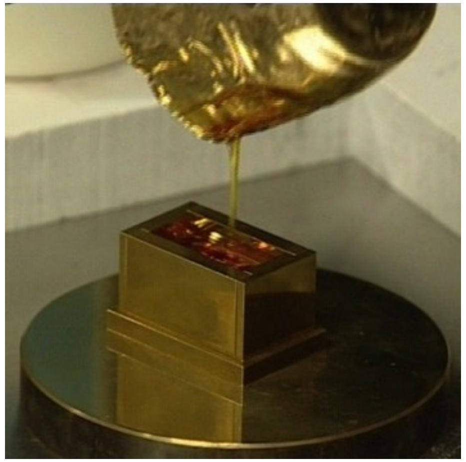


melt-quench technique

- multicomponent glasses
- higher losses: **0.1-10 dB/m**, depending on solid raw material purity **(4-7 order of magnitude higher than theoretical loss)**
- lower losses only via in-house purification

BUT raw materials can be easily mixed in any proportion

➔ **huge range of glass compositions → property tailoring**



Glass Melting

ambient atmosphere

- easy, cheap → ideal for testing new ideas:
  - when OH content in glass does not matter, e.g. applications in the visible
  - air atmosphere (N_2/O_2) sufficient for redox state, e.g. oxide glasses without HMOs

controlled atmosphere

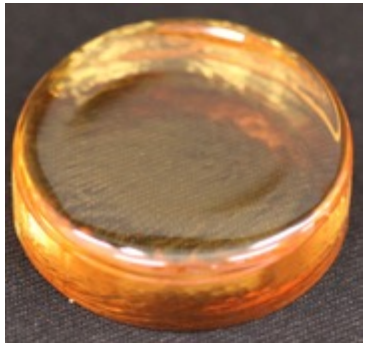
e.g. furnace in or attached to glovebox

- dry atmosphere → low OH content in glass
- reducing or oxidising conditions
  → control of redox state of polyvalent ions

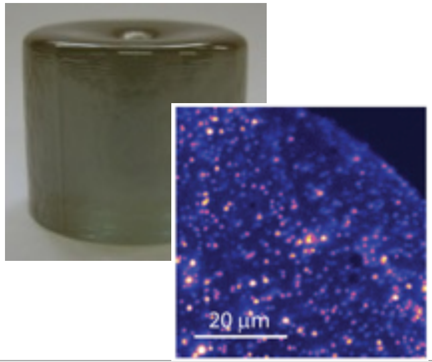
<u>**glass types melted in glovebox at IPAS:**</u>

- **tellurite**: $TeO_2 – ZnO – Na_2O$
- **germanate**: $GeO_2 – PbO – Ga_2O_3 – Na_2O$
- **ZBLAN**: $ZrF_4 – BaF_2 – LaF_3 – AlF_3$ - NaF
- **InF_3** glass: $InF_3 – ZnF_2 – SrF_2 – BaF_2$
- **FP** glass: P_2O_5 – MO – $MF_2 – AlF_3$ (M=Ca, Sr, Ba)

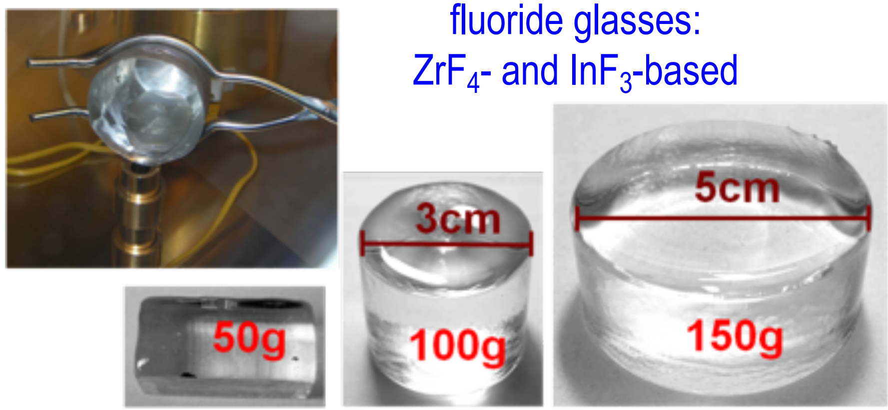
Ho^{3+} doped germanate glass



nanodiamond doped tellurite glass



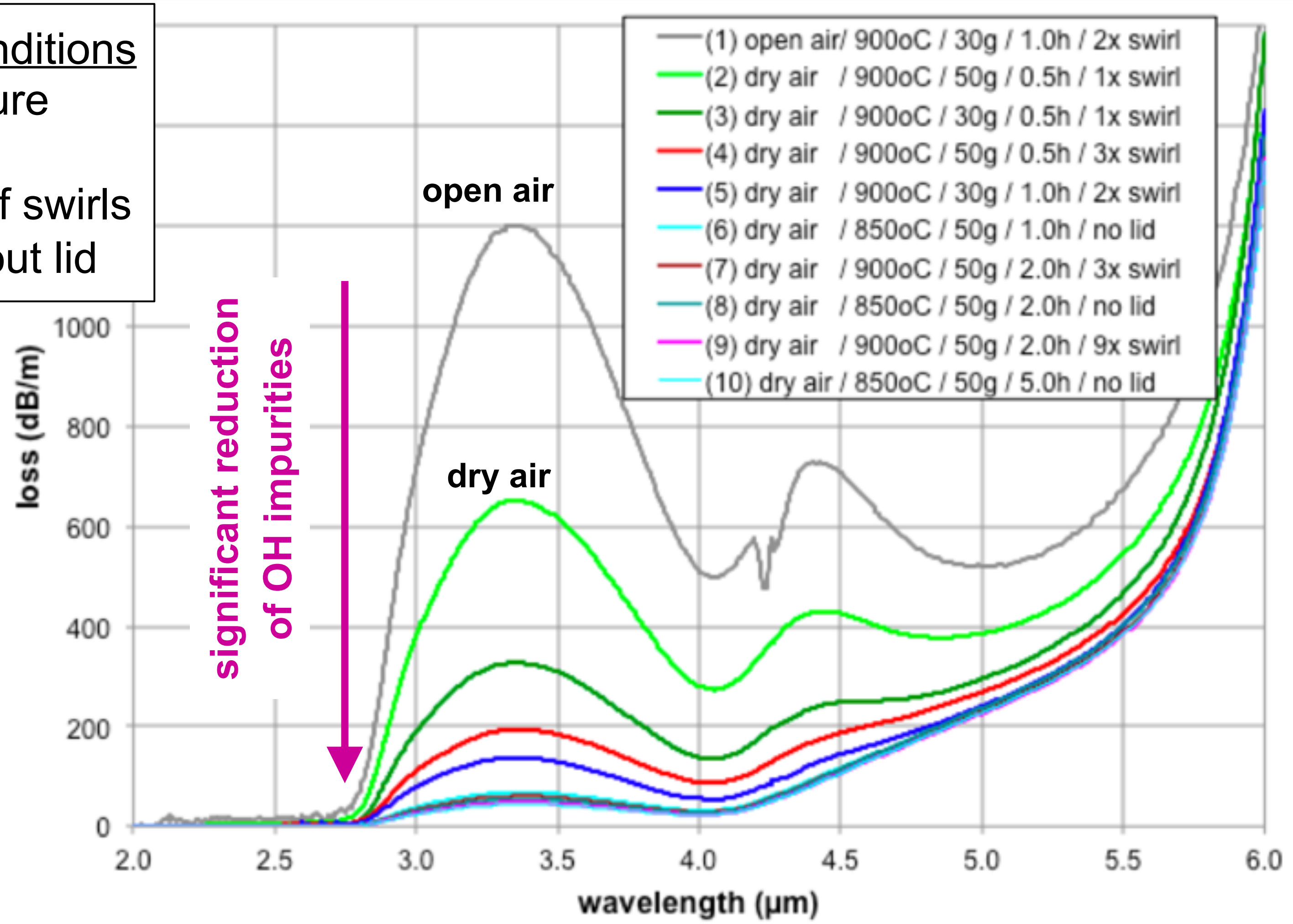
fluoride glasses: ZrF_4- and InF_3-based



Tellurite Glass Dehydration

Impact of melting conditions on OH peak

<u>melting conditions</u>
- temperature
- time
- number of swirls
- with/without lid



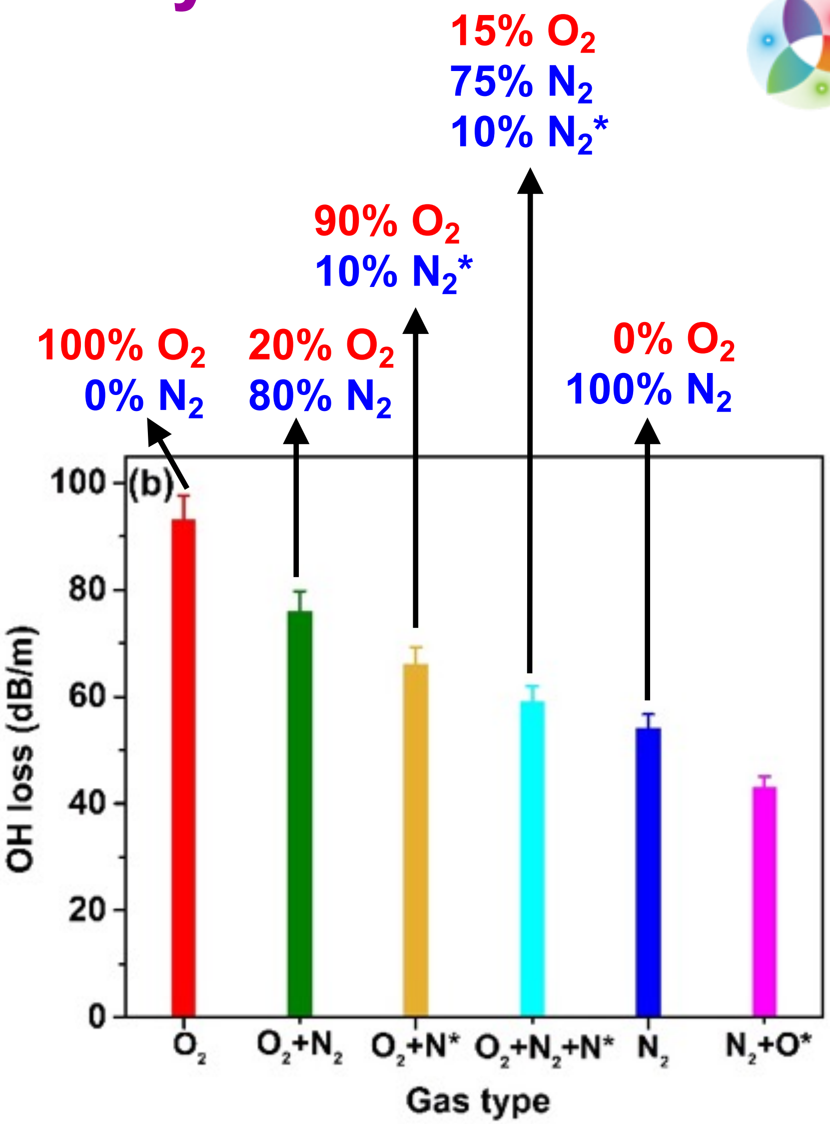
H. Ebendorff-Heidepriem, et al., Opt. Mater. Express 2, 432 (2012)

Review of tellurite glasses purification issues for mid-IR optical fiber applications

F. Desevedavy, et al., J. Am. Cer. Soc. 103, 4017 (2020)

Lead-Germanate Glass: Gas Type and Amount → Dehydration

Gas type		Gas amount
O_2,N_2, Ar:	dry	1.6 ; 4 ; 10 ; 16 L/min
N_2*:	ultra-dry	~10 vol%
O_2*:	super-dry	4 mol% Na_2O as $NaNO_3$



Pengfei Wang, et al., "Development of low-loss lead-germanate glass for mid-infrared fiber optics", Part I and II, J. Am. Cer. Soc. 104, 833 and 860 (2021)

Lead-Germanate Glass: Gas Type and Amount → Dehydration

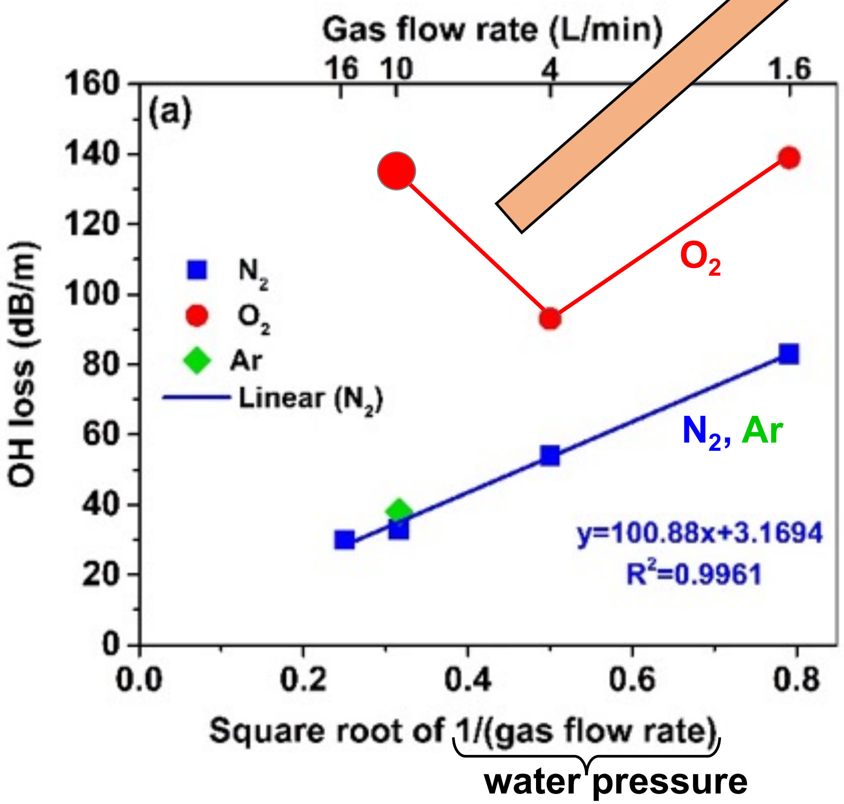
Gas type		**Gas amount**
O_2,N_2, Ar:	dry	1.6 ; 4 ; 10 ; 16 L/min
N_2*:	ultra-dry	~10 vol%
O_2*:	super-dry	4 mol% Na_2O as $NaNO_3$

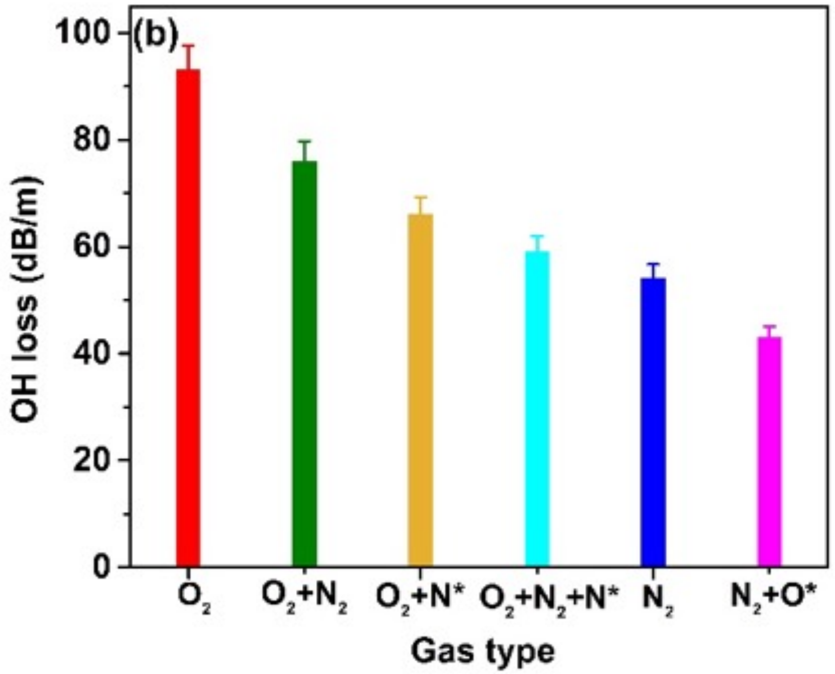
N_2, Ar: physically dissolved

O_2: chemically dissolved

➔ facilitates H_2O dissolution:

$$Pb^{2+} + 0.5\ O_2 + H_2O \rightarrow Pb^{4+} + 2\ OH^-$$





Pengfei Wang, et al., "Development of low-loss lead-germanate glass for mid-infrared fiber optics", Part I and II, J. Am. Cer. Soc. 104, 833 and 860 (2021)

Challenges of doping nanocrystals (NCs) into glass melt

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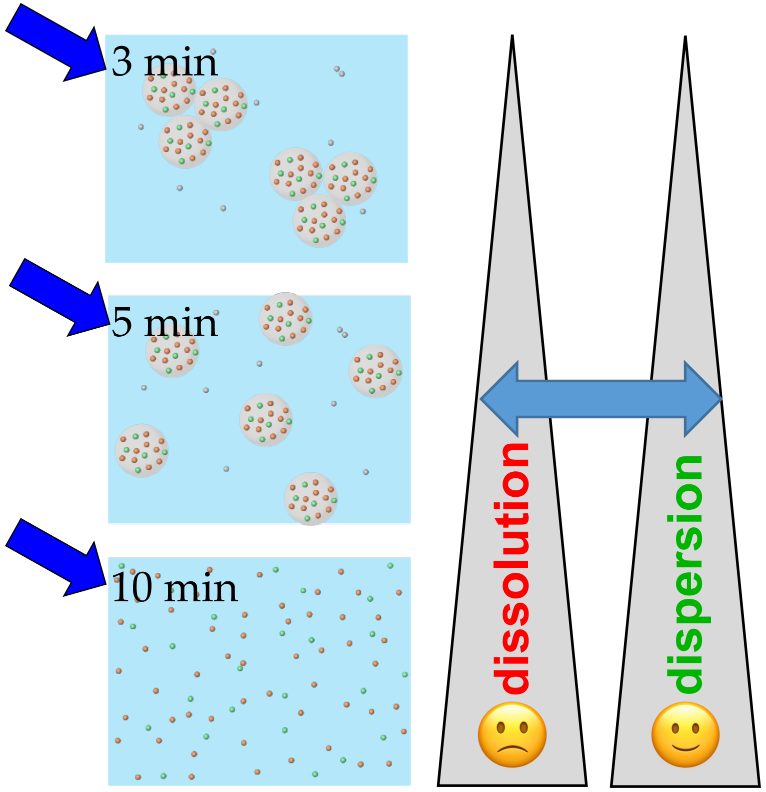
Balancing NC dispersion and dissolution through doping temperature and time



at 577°C

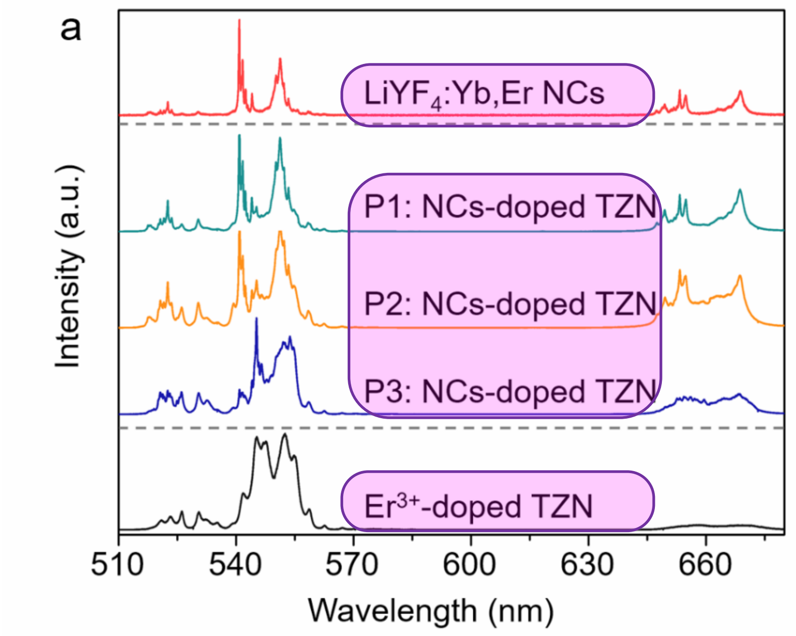
$LiYF_4$:Yb,Er in TZN tellurite glass

dissolution

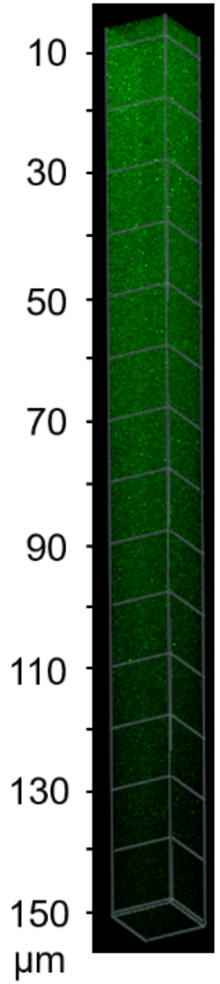
comparison of luminescence of hypersensitive Er^{3+} in NC powder and in glass

➔ NC doped glass shows features of both

➔ NC size reduced from 70nm to 50nm



J. Zhao, et al., Adv. Opt. Mater. 4, 1507 (2016)



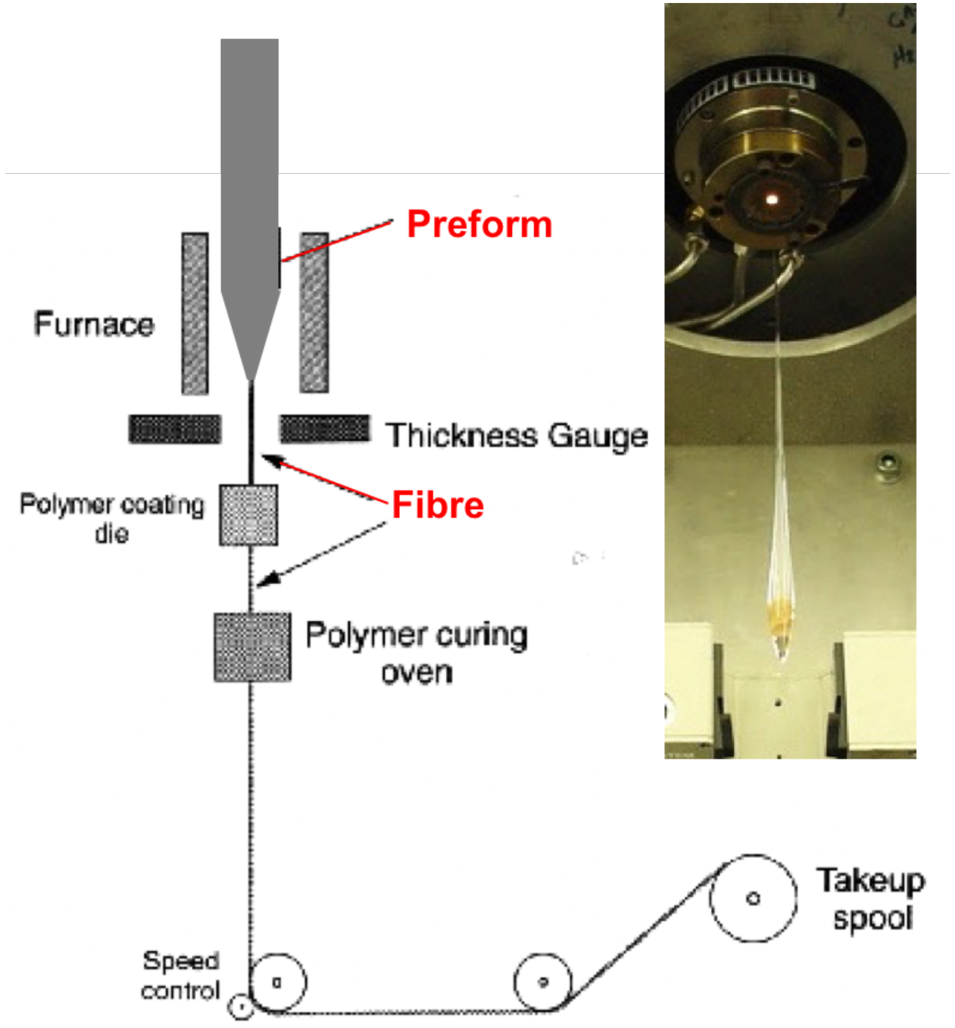
dispersion

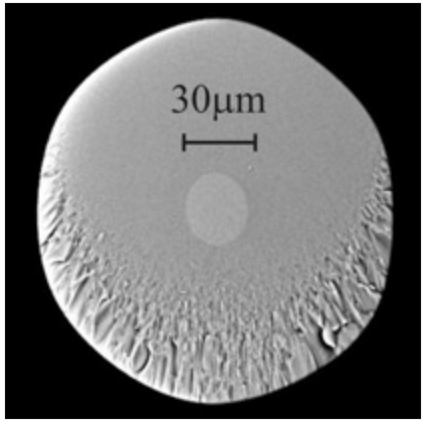
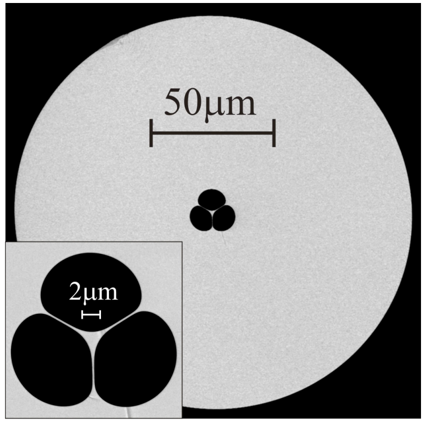
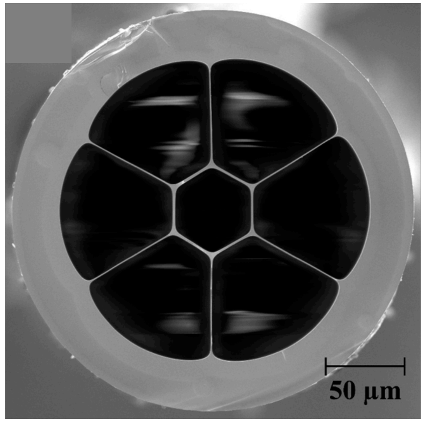
3D confocal upconversion microscopy

➔ large volume imaged

Fibre Types

Drawing



solid core step-index	solid core microstructured	hollow core microstructured
		

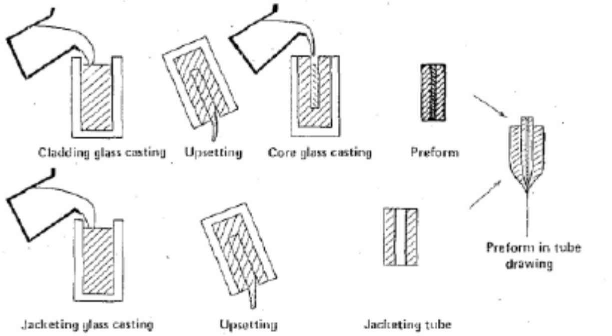
1. Preform Fabrication (10-20mm diameter) *macroscopic*
2. Drawing of Preform into Fibre (120-300μm) *microscopic*

Preform Fabrication Techniques

- **casting** (step-index, microstructured fibres)
- **stacking** (microstructured fibres)
- **drilling** (microstructured fibres)
- **extrusion** (microstructured fibres)
- **3D printing** (microstructured fibres)

Casting

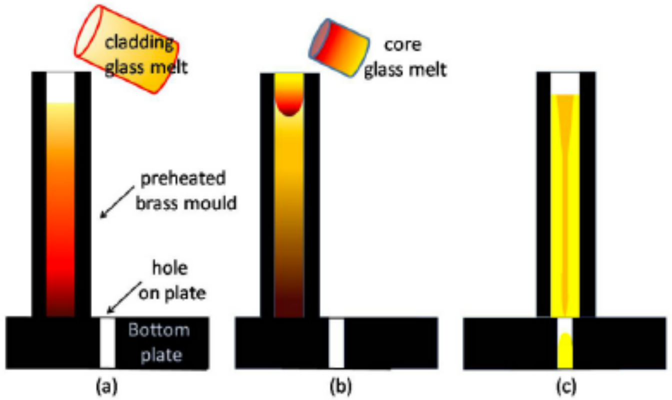
core/clad preform
rotational casting
fluoride; tellurite



Y. Ohishi, J. Lightwave Tech. 2, 593 (1984)
A. Mori, J. Cer. Soc. Jpn. 116, 1040 (2008)

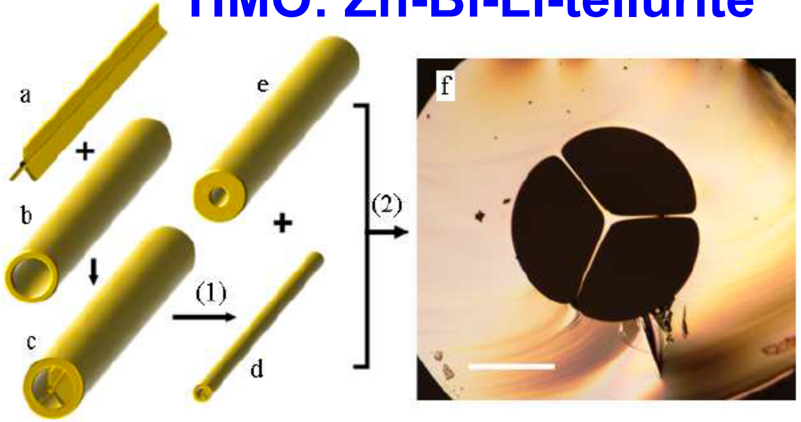
- directly step-index preform via rotational or built-in
- rods and tubes for preform assemblies;
  - rod-in-tube for core/clad prefo
  - stacking for microstructured fibres
  - special-shaped rod for microstructured fibres

core/clad preform
built-in casting
HMO: Zn-Ba-tellurite



H. Shi, Opt. Mat. Expr. 6, 3967 (2016)

microstructured preform
casting and assembly
HMO: Zn-Bi-Li-tellurite

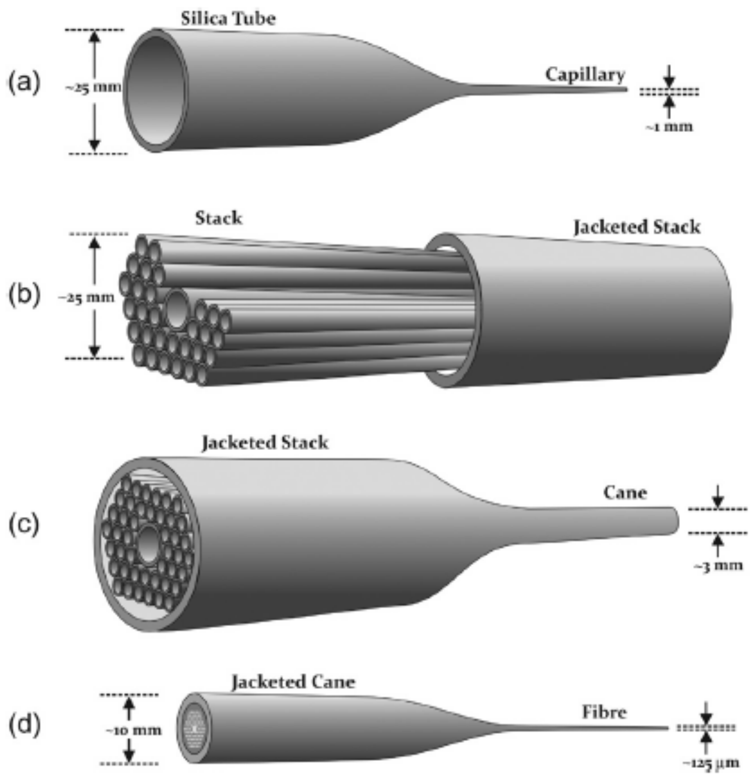


M. Liao, Opt. Expr. 18, 9089 (2010)

challenges:

- surface treatment (smooth, no contamination)
- low viscosity
- limited structures

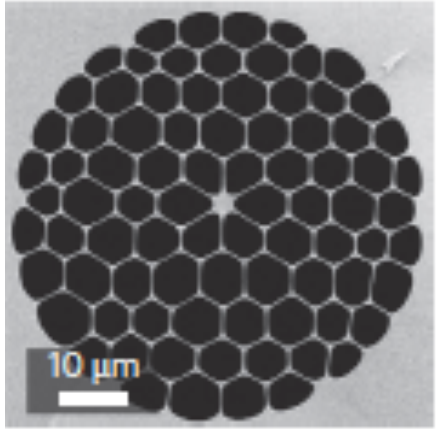
Stacking



Y. Wang, High Power Laser Science and Engineering 1, 17 (2013)

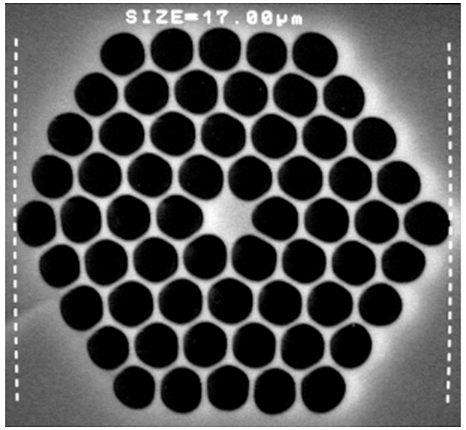
- established method for silica
- advantage: very complex structure, many large holes
- few soft glass fibres demonstrated
- challenges:
  - labour-intensive, skilled operator
  - scratching of soft glass capillaries → loss
  - additional step of making capillaries (cf. extrusion, drilling)

ZBLAN fluoride



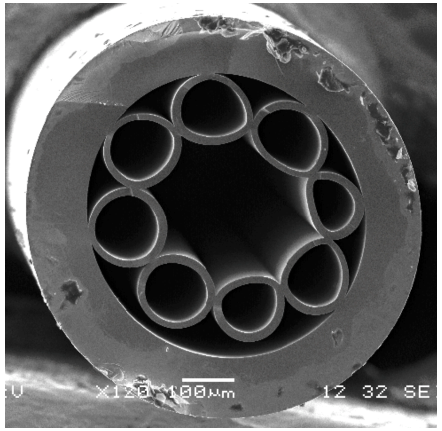
X. Jiang, Nat. Photon. 19, 133 (2015)

Pb-Bi-Ga-silicate



R. Stepien, Opt. Mater. 35, 1587 (2013)

As-Se-Te chalcogenide



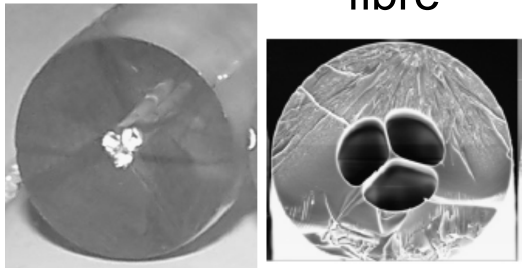
A. Kosolapov, Opt. Express 19, 25723 (2011)

Drilling

- well suited for silica (robust, fire-polishes well during drawing)
- few examples for soft glass (fragile, less degree of fire-polishing)

tellurite: TeO_2-ZnO-Na_2O

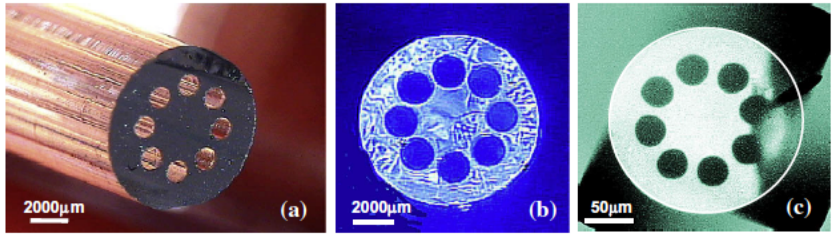
preform fibre



I. Savelii, Opt. Mater. 33, 1661 (2011)

fluoride: AlF_3-ZrF_4-YF_3-MF_2-NaF

preform fibre



P. McNamara, J. Non-Cryst. Sol. 355, 1461 (2009)

<u>loss at 2.1µm</u>
material: 0.1dB/m
MOF: 1.9dB/m

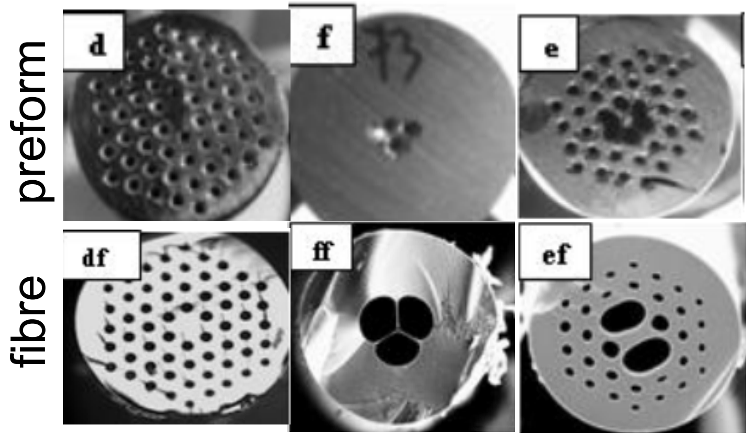
advantages:

- high geometric precision (shape, position)

challenges:

- limited hole size and shape, and preform length
- rough surface

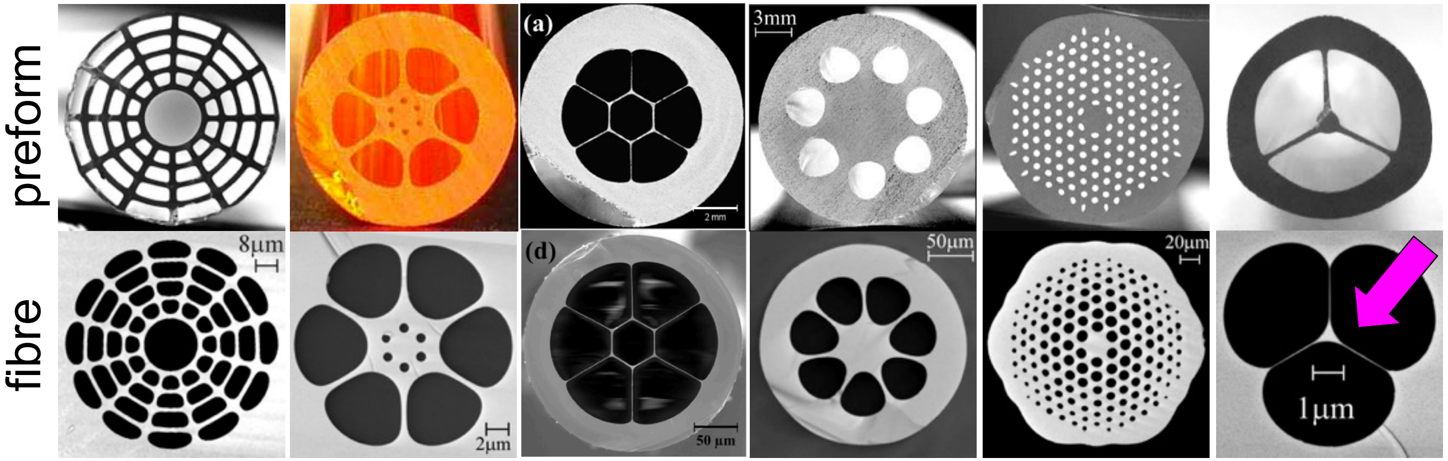
chalcogenide: As_2O_3

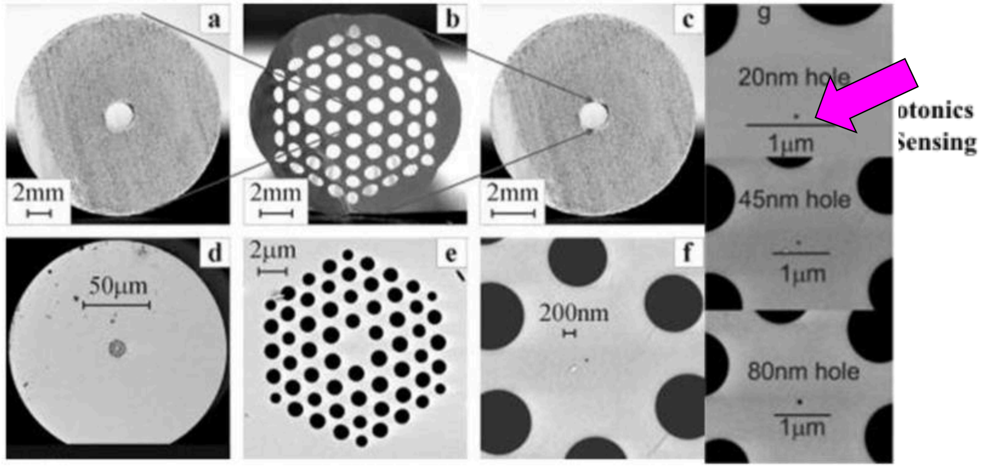


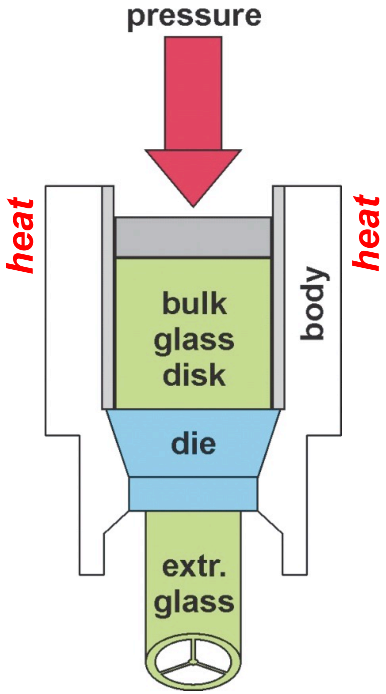
M. El-Amraoui, Opt. Expr. 18, 26655 (2010)

<u>loss at 1.5µm:</u>
material: 0.1dB/m
MOF: 0.35-0.7dB/m

Extrusion – air/glass structures at IPAS







advantages

- broad range of polymers and glasses (100-1000°C)
- almost unlimited range of structures
- small features (0.2mm holes, webs)
- large aspect ratios (20/0.5)
- fire-polishing during extrusion
- use of 3D printed dies
  → new die designs → new preform structures

- flexibilities in tubes for rod-in-tube preform assemblies
  → nanoscale cores and holes

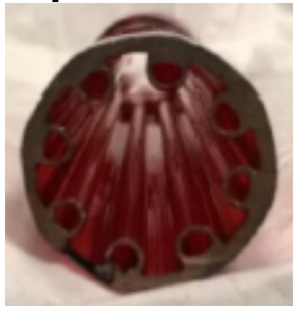
challenge

- die→ preform deformations

C. Kalnins, et al., J. Appl. Glass Science 10, 172 (2019)
H. Ebendorff-Heidepriem, et al., Opt. Mater. Express 4, 1494 (2014)
H. Ebendorff-Heidepriem, et al., Opt. Mater. Express 2, 304 (2012)
H. Ebendorff-Heidepriem, et al., Opt. Express 15, 15086 (2007)
H. Ebendorff-Heidepriem, Opt. Letters 33, 2861 (2008)
Y. Ruan, Opt. Express 18, 26018 (2008)
W. Zhang, Opt. Express 19, 21135 (2011)
G. Tsiminis, Opt. Express 24, 5911 (2016)

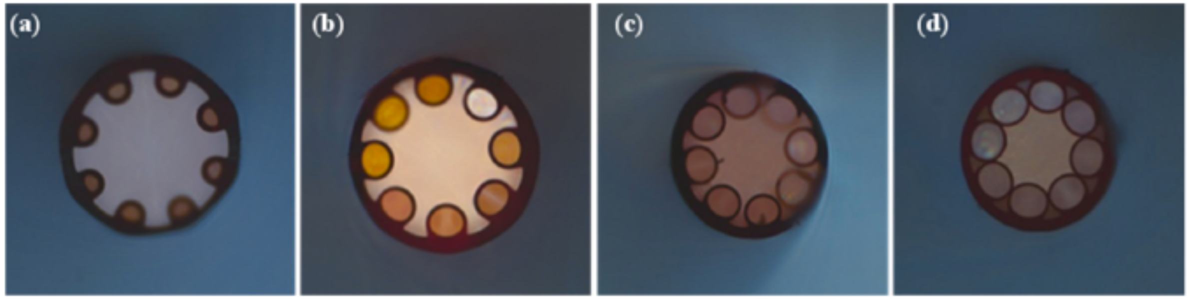
Extrusion – air/glass structures of other groups

preform



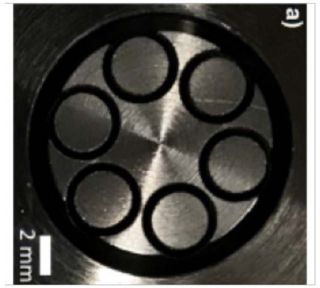
fibre

0 mbar | 35 mbar | 40 mbar | 45 mbar



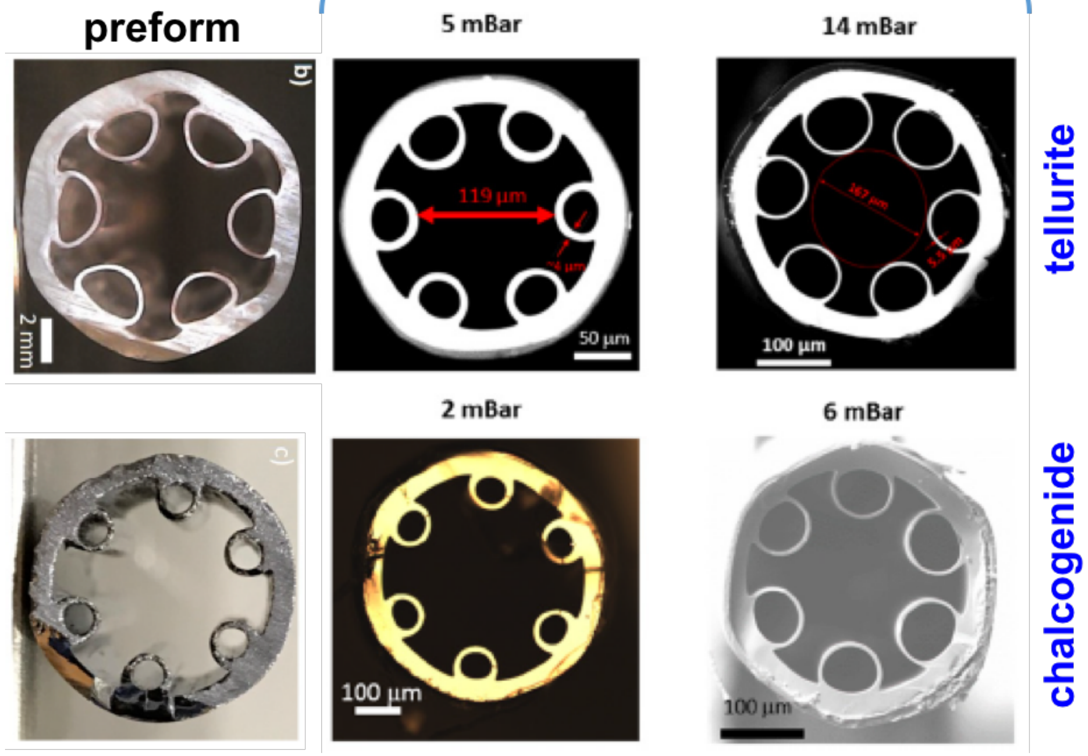
R. Gattass, Opt. Express 24, 25697 (2016)

die



preform

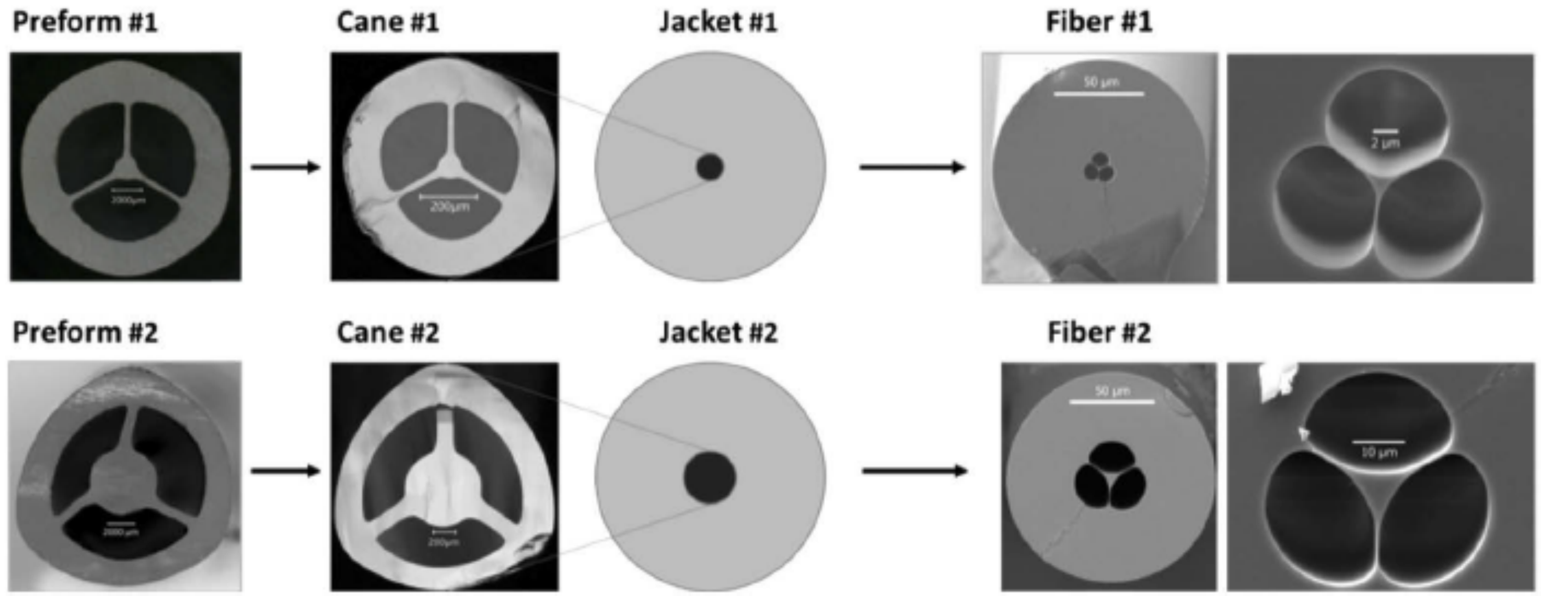
fibre



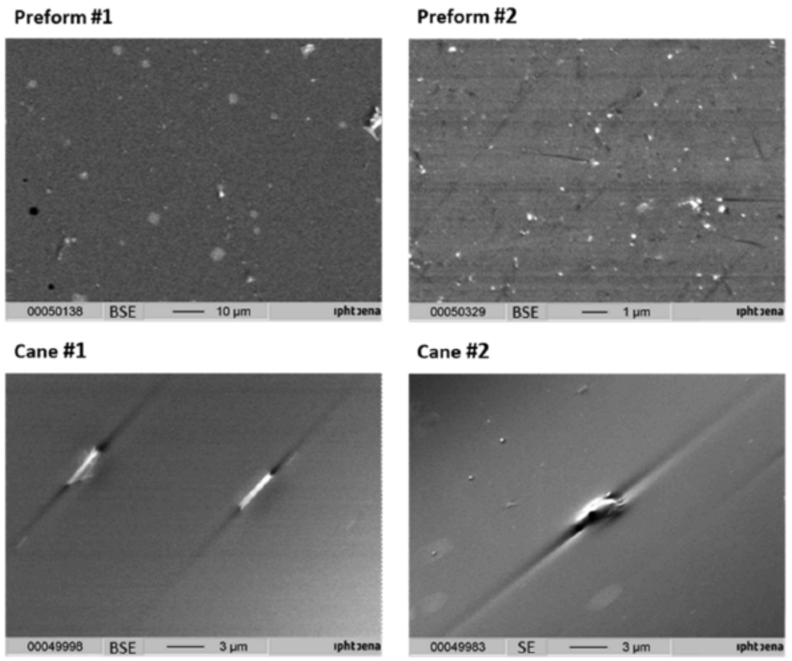
A. Ventura, Opt. Express 28, 16542 (2020)
J. Hayashi, ICTON conference (2020)

Extrusion – high temperature

- SiO_2-Al_2O_3-La_2O_3 glass
- high temperatures: T_g=860°C, **extrusion=1000°C**
- Nicrofer as die material
- ambient atmosphere as for HMO glasses
- suspended core as model geometry to study core surface



surface crystallization !!



<u>loss at 1.2µm</u>
material: 2-5 dB/m
MOF: 50-90 dB/m

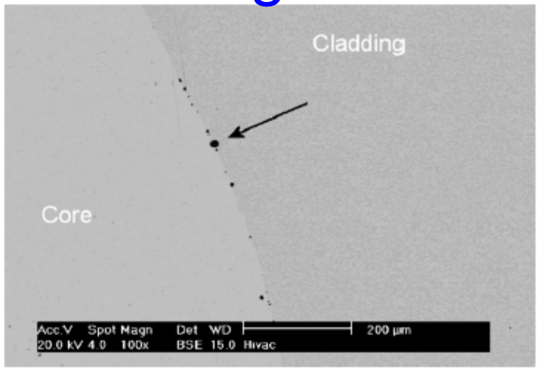
D. Litzkendorf, Opt. Mat. Express 11, 142 (2021)

Extrusion – multi-glass structures

horizontally stacked billets

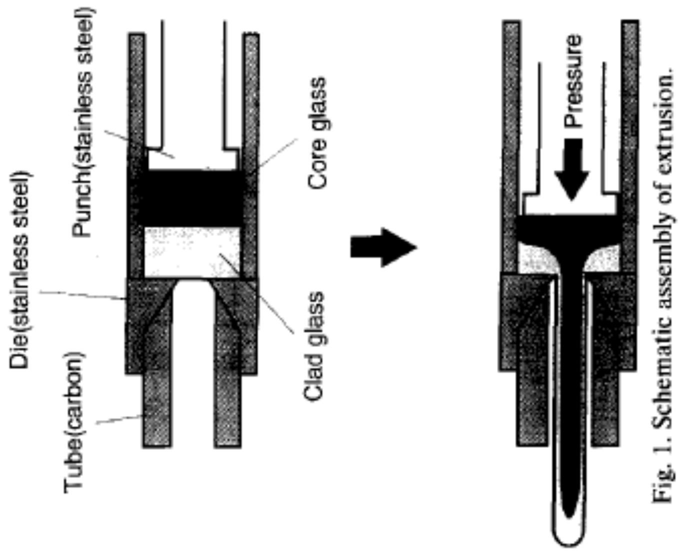
- large core/clad ratio
- limited length of consistent core/clad ratio
- interface quality depends on billet endface quality

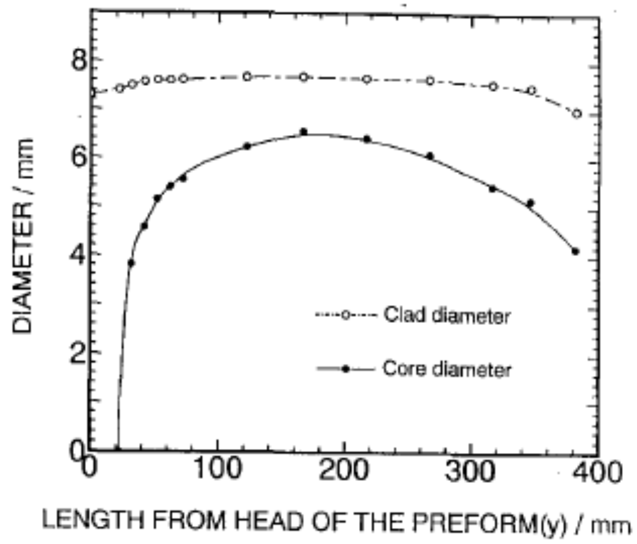
chalcogenide

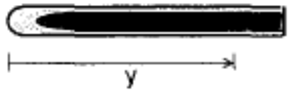


S. Savage, J. Non-Cryst. Sol. 354, 3418 (2008)

core/clad preform: fluoride



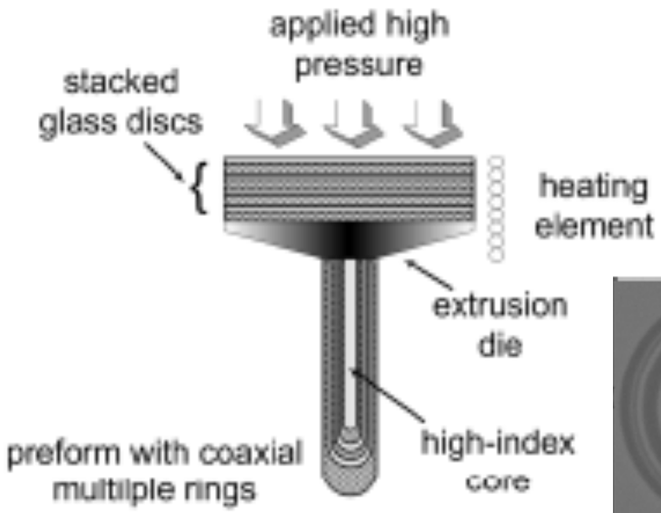




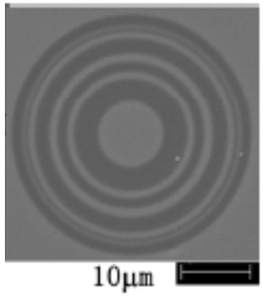
core/clad fibre: 0.01dB/m at 2.5µm

K. Itoh, J. Non-Cryst. Sol. 167, 122 (1994)

1D MOF preform: lead-silicate



fibre

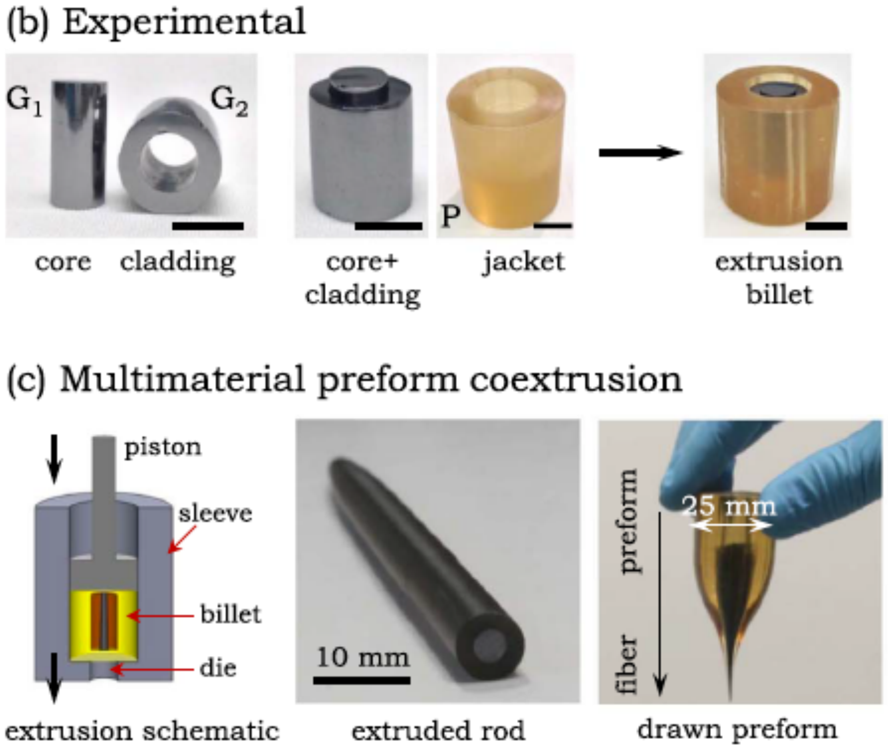


X. Feng, Appl. Phys. Lett. 87, 081110 (2005)

Extrusion – multi-glass structures
concentrically assembled billets

chalcogenide

core/clad/jacket preform



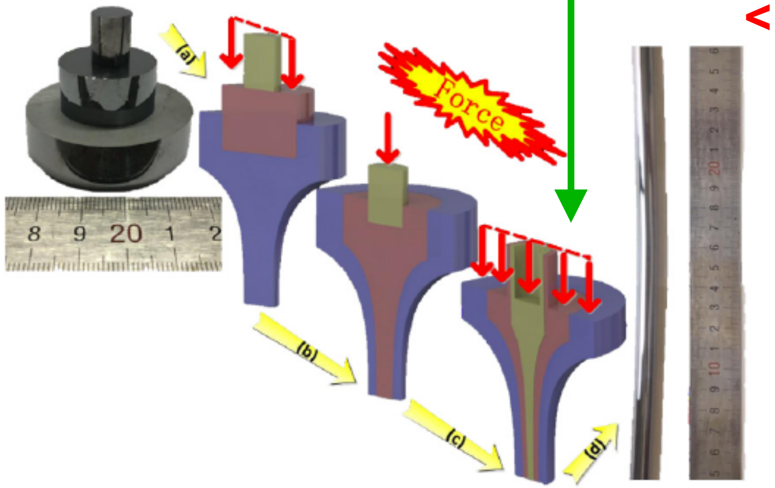
G. Tao, Opt. Letters 39, 4009 (2014)

core/clad/clad preform

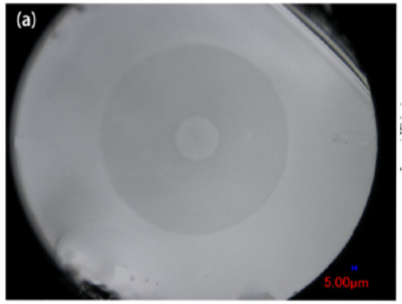
peel-of extrusion

→ removal of oxidized surface layer

<1dB/m at 7.4-9.7μm



fibre



K. Jiao, Opt. Express 27, 2036 (2019)

Preform Fabrication Techniques: 3D printing

method and results

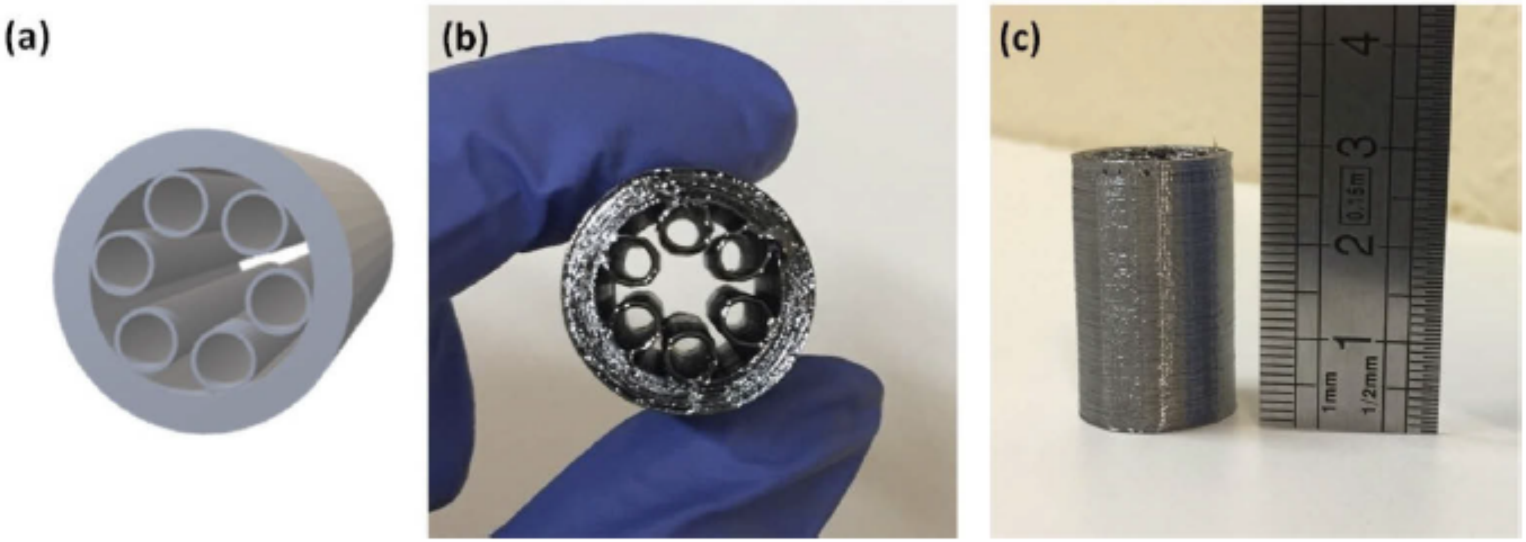
- Te-As-Se glass
- filament: 3mm diameter rods of 0.5m length
- adapted commercial polymer printer
- glass Tg – 137°C, nozzle – 300°C, build plate – 140°C
- loss: before printing 8dB/m, after printing 28dB/m

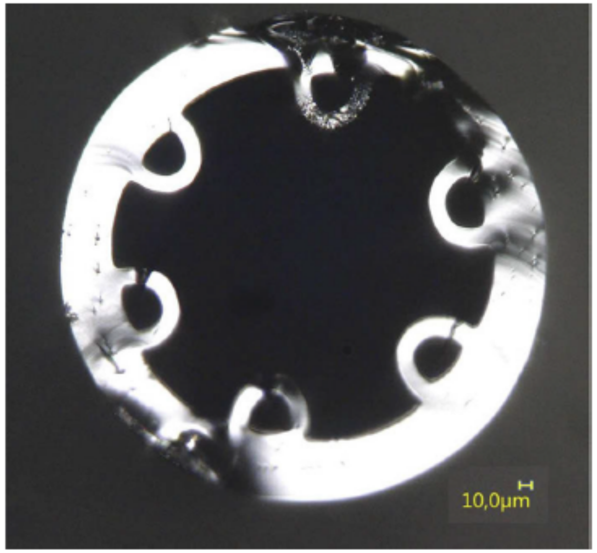
challenge:

- porosity due to incomplete fusion of the layers → loss

opportunity:

- structures that cannot be made in any other way



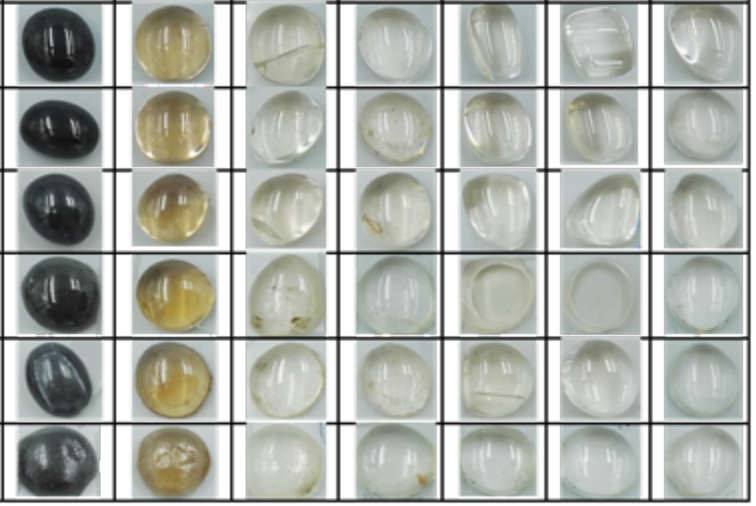


J. Carcreff, et al., Opt. Express 11, 198 (2020)

Lead-Germanate Glass: Extrusion and Drawing

0% O_2 / 100% N_2 — 20% O_2 / 80% N_2 — 100-90% O_2 / 0-10% N_2

Heat treatment conditions as for extrusion and drawing		Melting atmosphere and conditions						
		S0-A2-N4	S0-A2-ON4	S0-A2-O4	P5-B2-O4	N10-A3-O4	P5-B2-O4-N*2-O*	N10-C2-O4-N*1
		None	20% O_2	100% O_2	100% O_2	100% O_2	90% O_2	90% O_2
Temperature and time	Atmosphere	100% N_2	80% N_2	None	None	None	~10% N_2^*	~10% N_2^*
		None	None	None	5% $PbCl_2$	10% NaCl	5% $PbCl_2$	10% NaCl
465°C (6 h)	100%O_2							
	80%N_2+20%O_2							
	100%N_2							
518°C (1 h)	100%O_2							
	80% N_2+20%O_2							
	100%N_2							



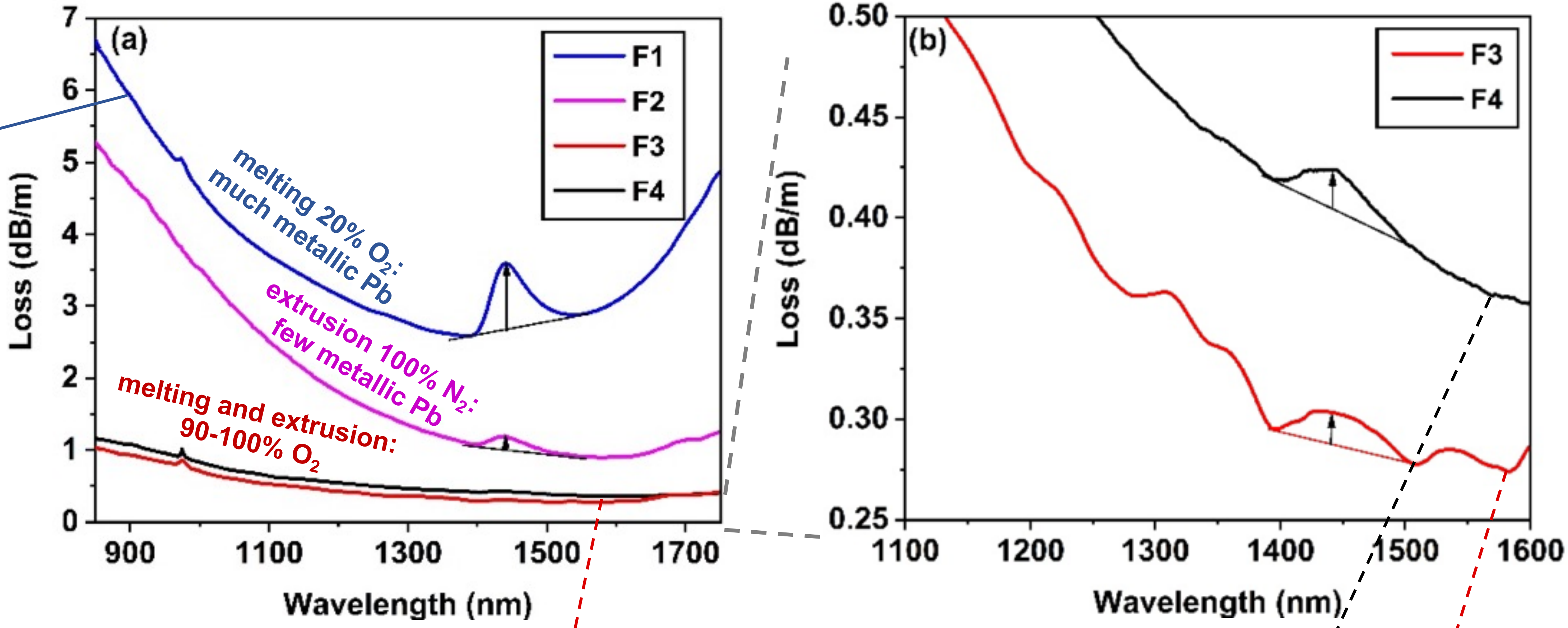
20% O_2 / 80% N_2 — 90% O_2 / 10% N_2*

O_2 — high amount (>50%) **during melting** necessary to avoid metallic Pb nanoparticles

Pengfei Wang, et al., "Development of low-loss lead-germanate glass for mid-infrared fiber optics", Part II, J. Am. Cer. Soc. 104, 860 (2021)

Lead-Germanate Glass: Fibre Loss - minimum

all fibres drawin in 90% N_2, 10% O_2



0.3 dB/m – lowest minimum loss for lead-germanate glass

preform postanneal in ambient → surface crystallization during drawing

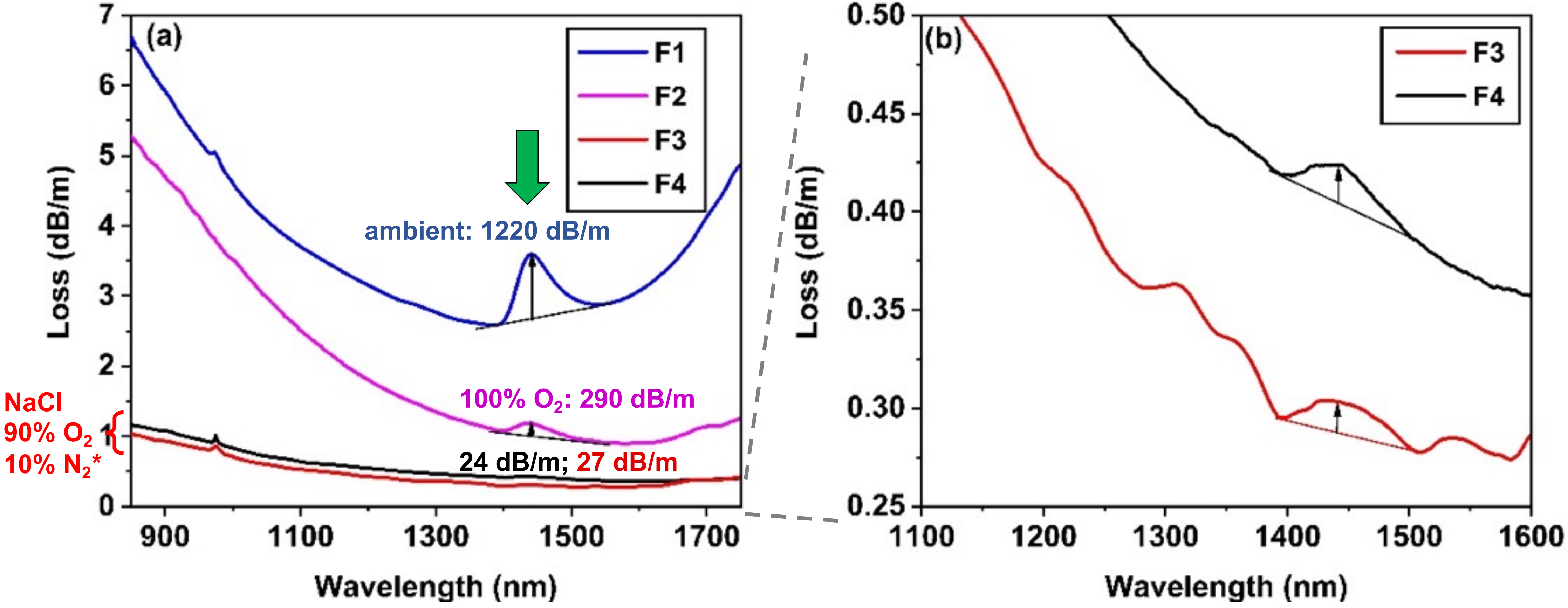
preform postanneal in 100% N_2 → smooth fibre surface

P. Wang, et al., "Development of low-loss lead-germanate glass for mid-infrared fiber optics", Part II, J. Am. Cer. Soc. 104, 860 (2021)

Lead-Germanate Glass: Fibre Loss - OH

THE UNIVERSITY of ADELAIDE

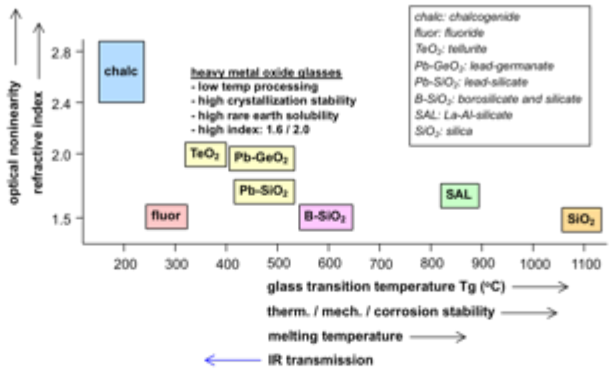
melting atmosphere: OH loss at 3.1μm



- melting atmosphere is most important:
  - O_2 rich atmosphere to avoid metallic Pb
  - ultradry gas via recirculation
- extrusion: O_2 atmosphere is recommended
- drawing: N_2 rich atmosphere to avoid surface oxidation

P. Wang, et al., "Development of low-loss lead-germanate glass for mid-infrared fiber optics", Part II, J. Am. Cer. Soc. 104, 860 (2021)

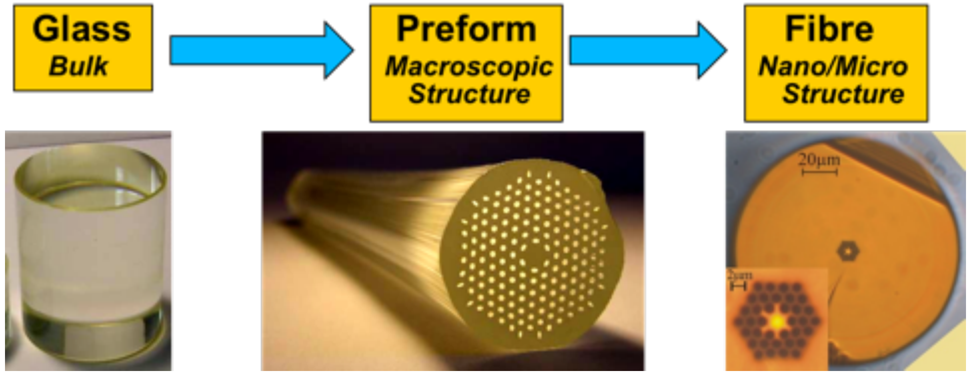
Outline



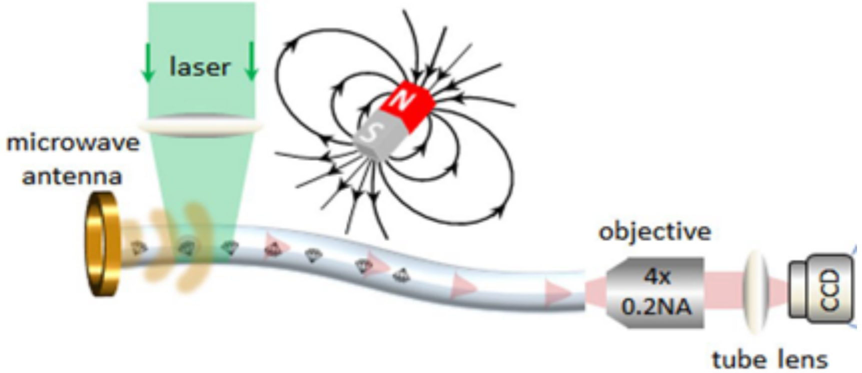
1. Glass types and properties

2. Fabrication technologies

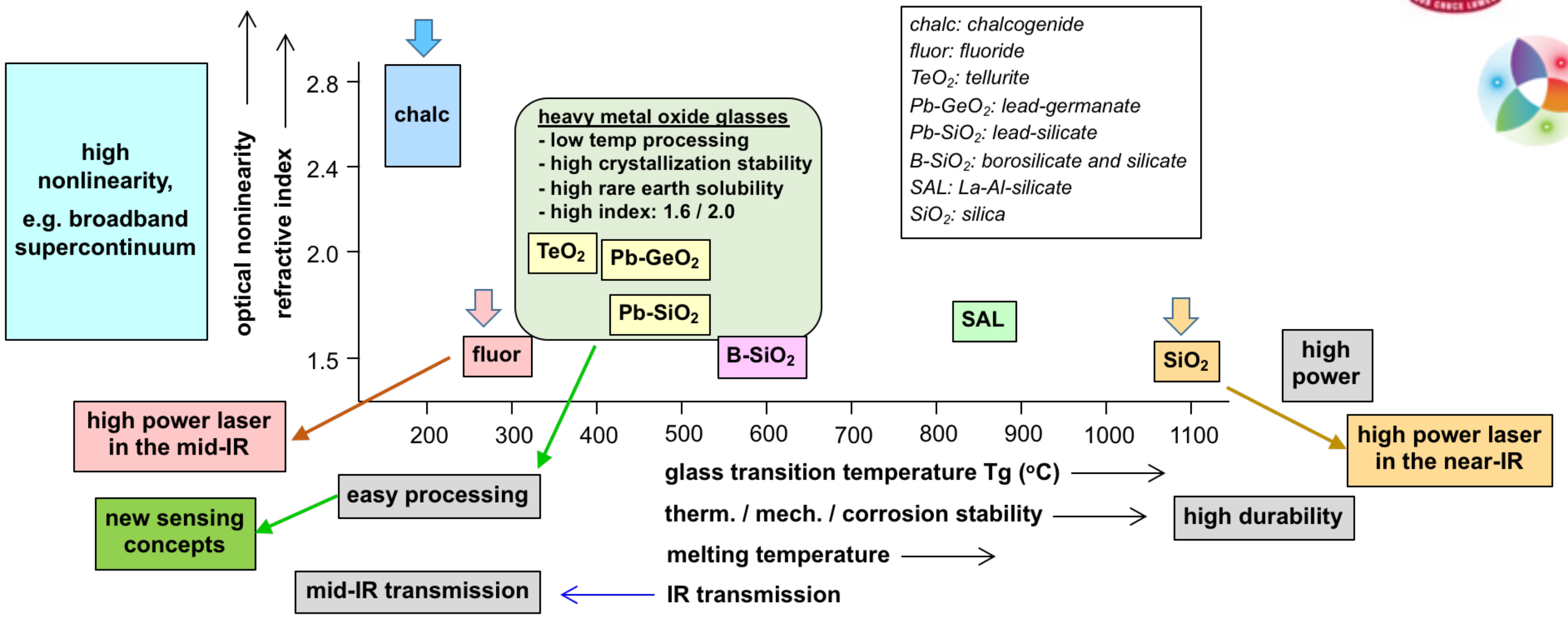
- dehydration
- nanocrystal doping
- preform



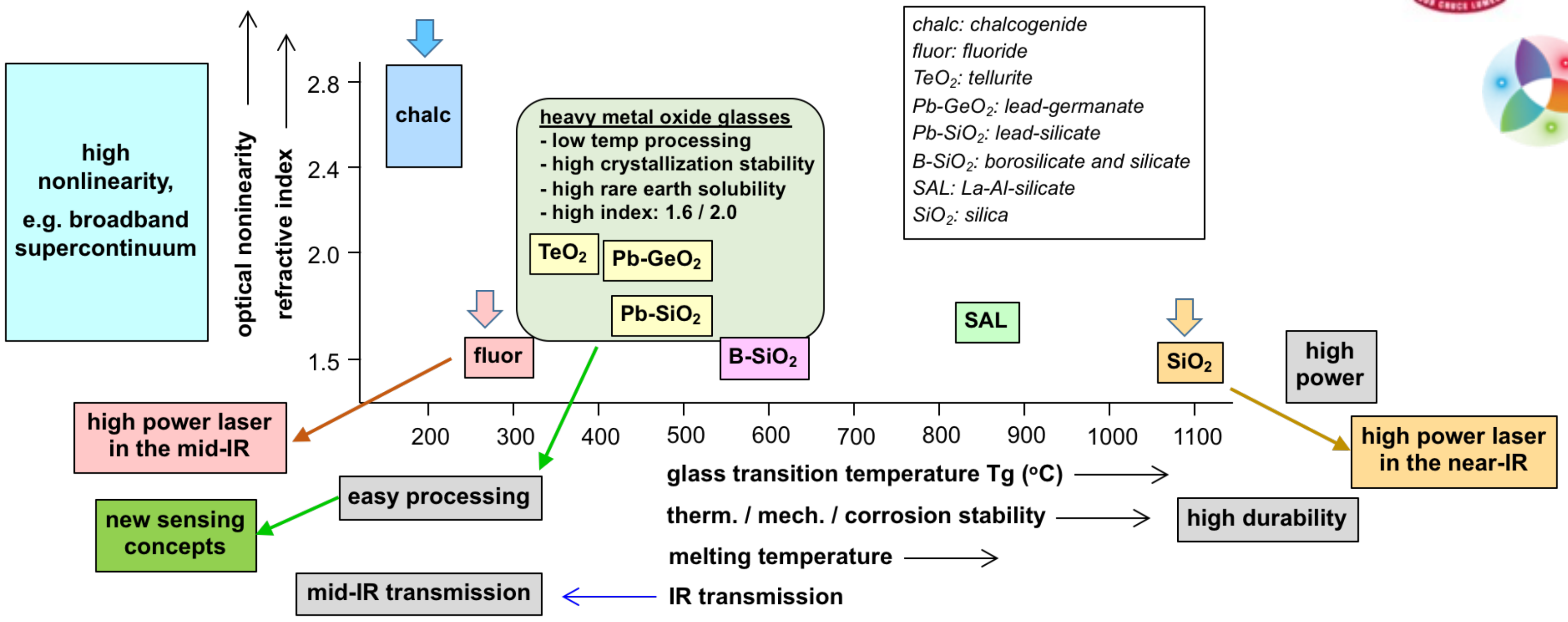
3. Applications



Application areas



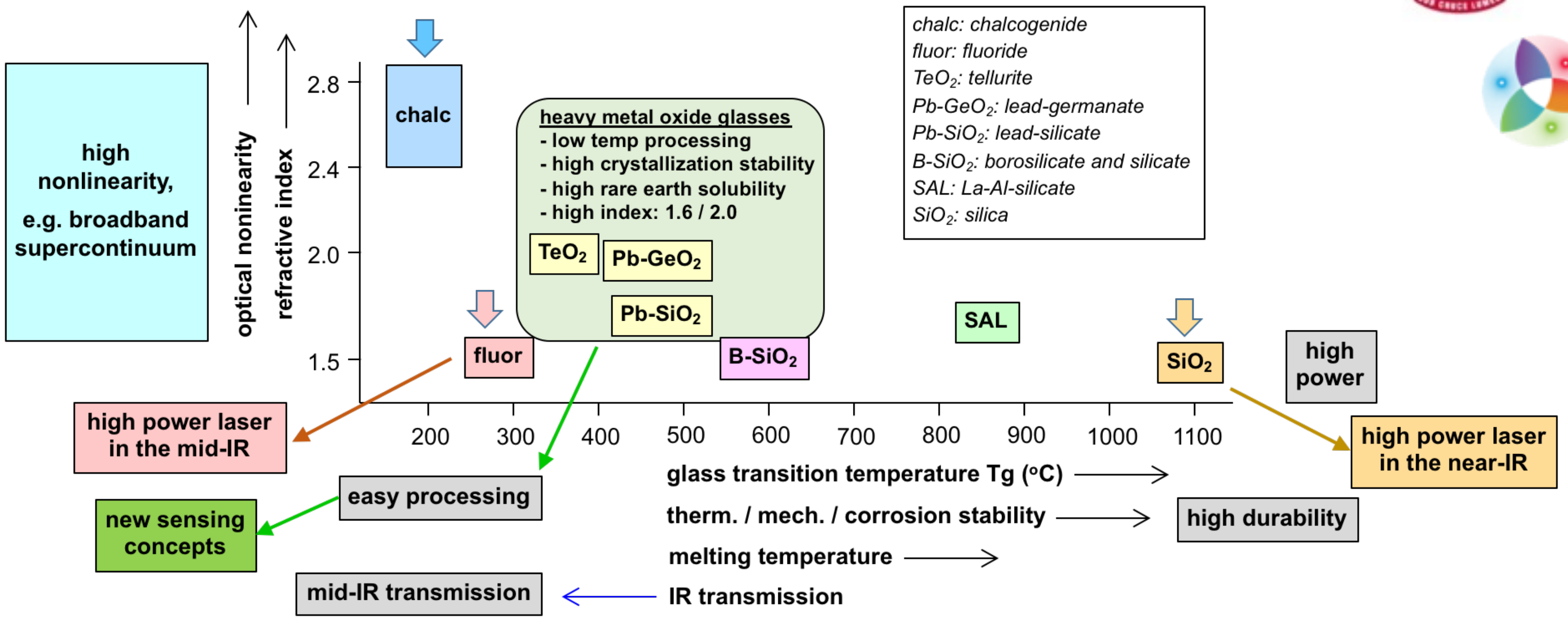
Application areas



supercontinuum generation

- demonstrated for huge range of soft glass fibres
- index variation, fibre structure → dispersion control
- transmission window beyond silica → UV, mid-IR

Application areas



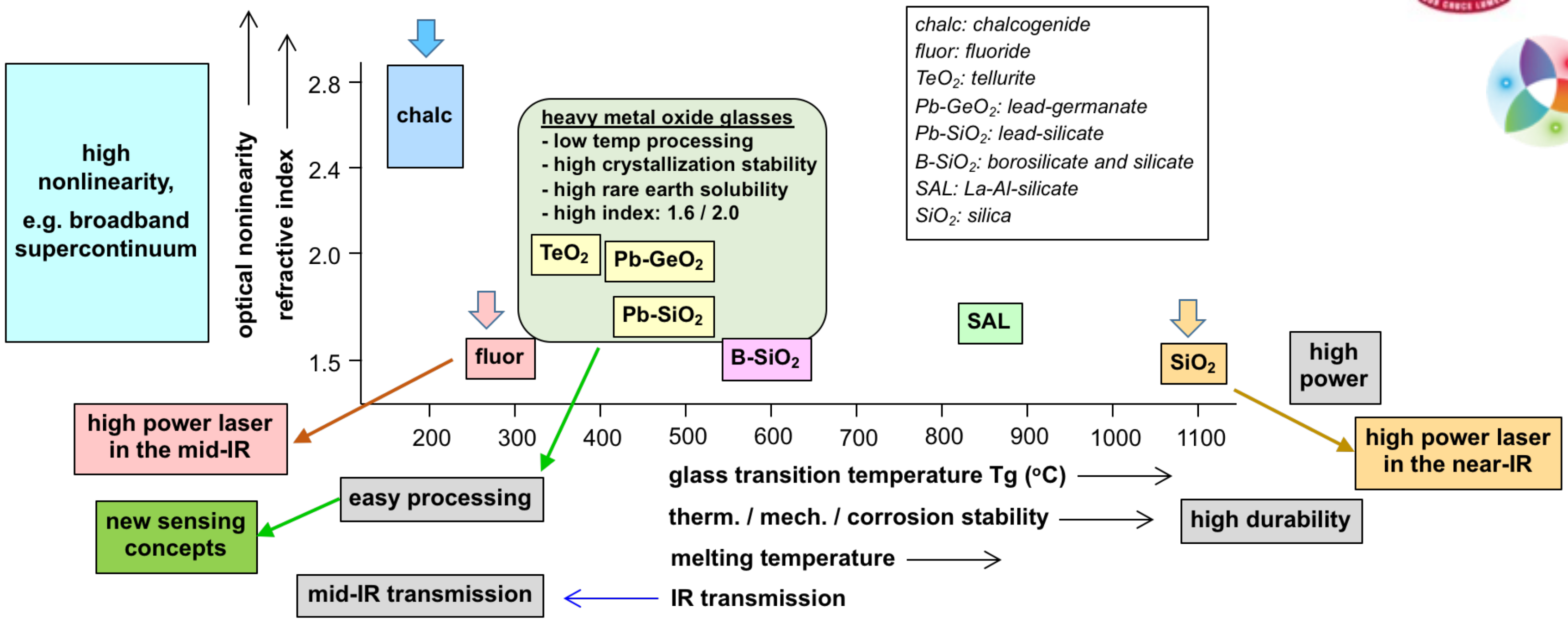
mid-IR fibre lasers

- dominated by fluoride step-index fibres (commercially available)
- high power, compact, robust: germanate step-index fibres
- other examples: tellurite, nanocrystal-in-glass, …
- chalcogenide: challenging

mid-IR high power delivery

- recent trend: hollow core fibres (chalcogenide, tellurite)

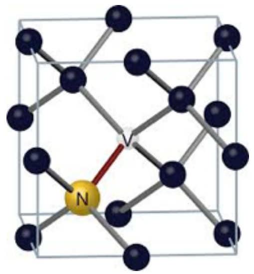
Application areas



new sensing and imaging concepts
- easy processing
- nanocrystal doping → new properties

Examples:
- magnetic field sensing
- in-vivo temperature sensing
- prototyping

Magnetic Field Sensitive Fibre: First Results



by embedding NV:Diamond in fibre

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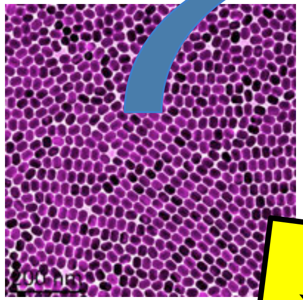
IPAS Institute for Photonics and Advanced Sensing

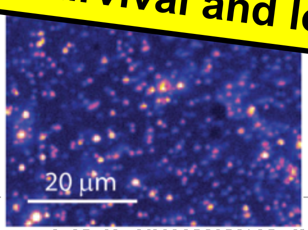
Tellurite Glass:

- visible transmission → NV excitation and emission
- low temperature (600-900°C) → diamond survival

NV:diamond nanoparticles + glass melt = NV:diamond embedded in glass

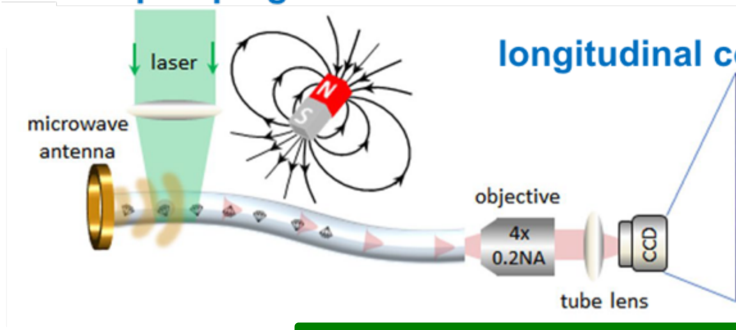


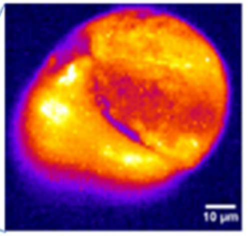
5 year optimization
➔ balance of diamond survival and low fibre loss



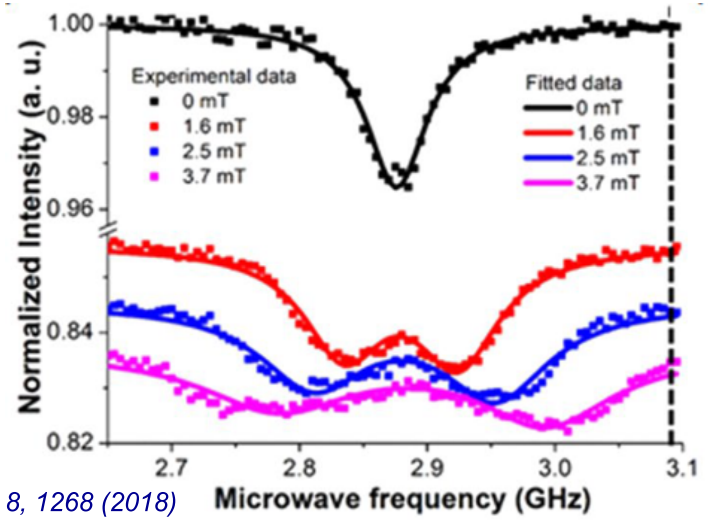
rod extrusion & fibre drawing

magnetic sensitive fibre





sensitivity 11 µT/√Hz

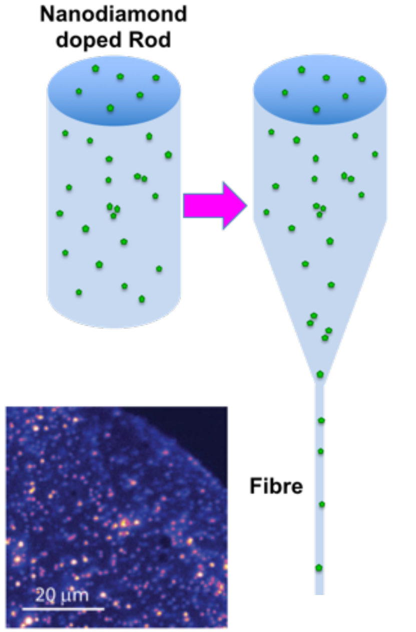


M. Henderson, et al., Advanced Mater. 23, 2806 (2011)
H. Ebendorff-H., et al., Opt. Mater. Express 4, 2608 (2014)
Y. Ruan, et al., Opt. Mater. Express 5, 73 (2015)

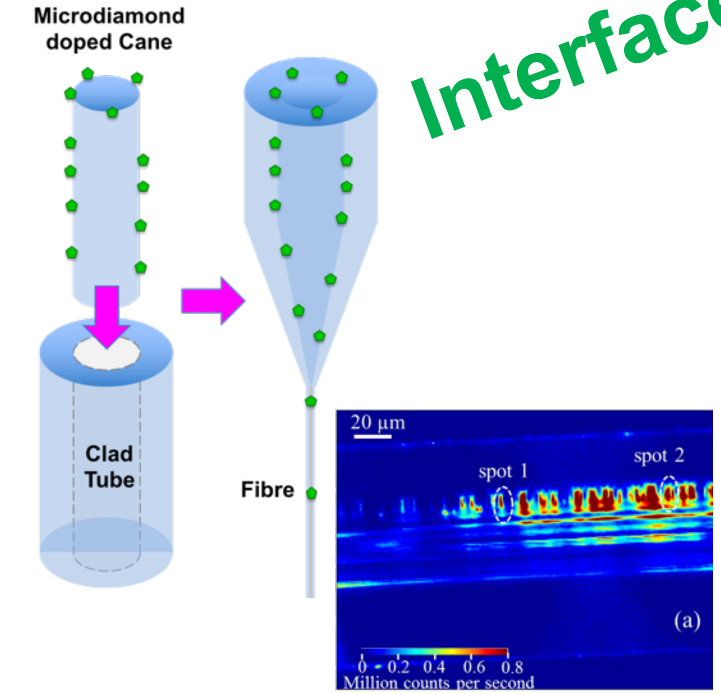
Y. Ruan, et al., Sci. Rep. 8, 1268 (2018)

Magnetic Field Sensitive Fibre: Enhanced Sensitivity

Nano-diamond in **Volume** of **Tellurite** → **Micro**-diamond at **Interface** of **Silicate**

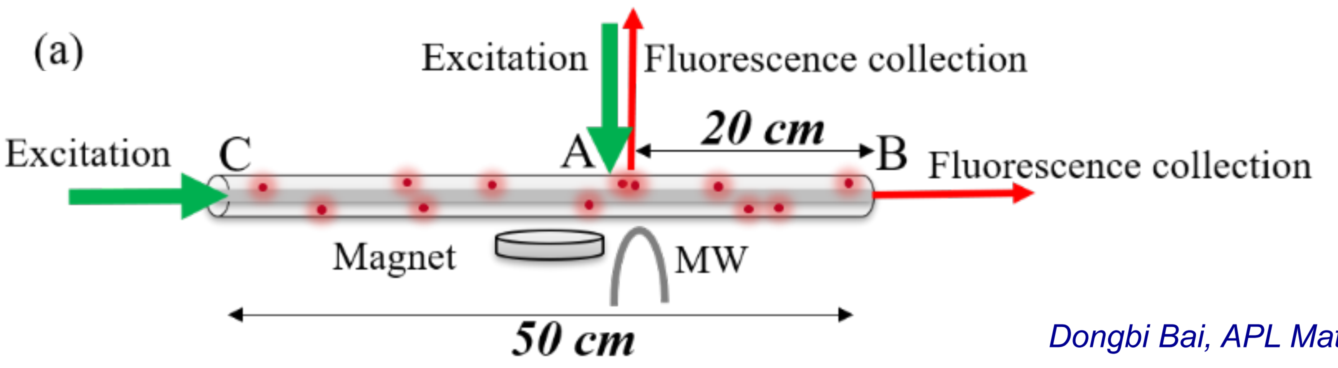


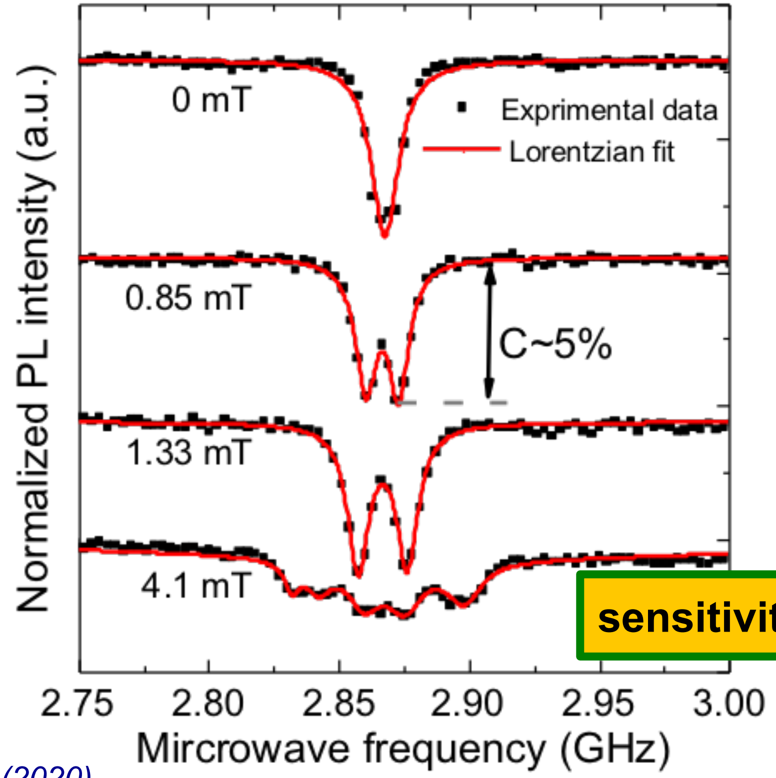
Y. Ruan, Sci. Rep. 8, 1268 (2018)



Interface Doping

20x higher sensitivity !
due to **Micro**-diamond and **Localisation**



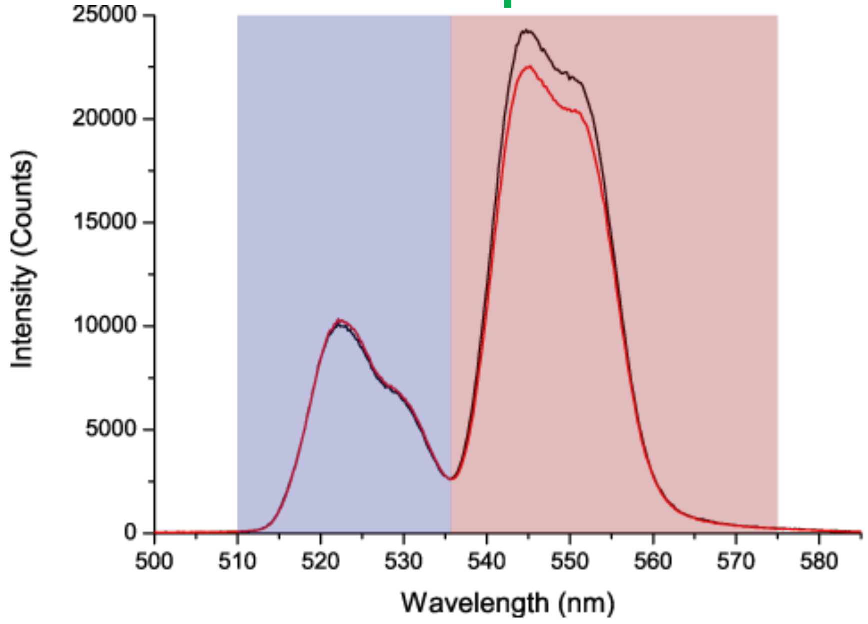


sensitivity 0.65 µT/√Hz

Dongbi Bai, APL Materials (2020)

in-vivo Temperature Sensor (1)

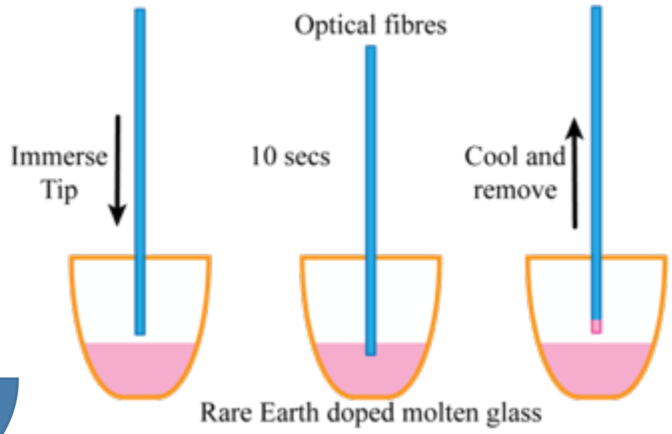
Temperature-sensitive Er^{3+} fluorescence excited via upconversion



Glass Requirements

- upconversion emission
  - → transmission Vis-NIR
  - → low phonon energy
  - → high Yb:Er concentration
- silica fibre dip coating
  - → low melt temperature

Dip coating



E. Schartner, et al., Sensors 14, 21693 (2014)

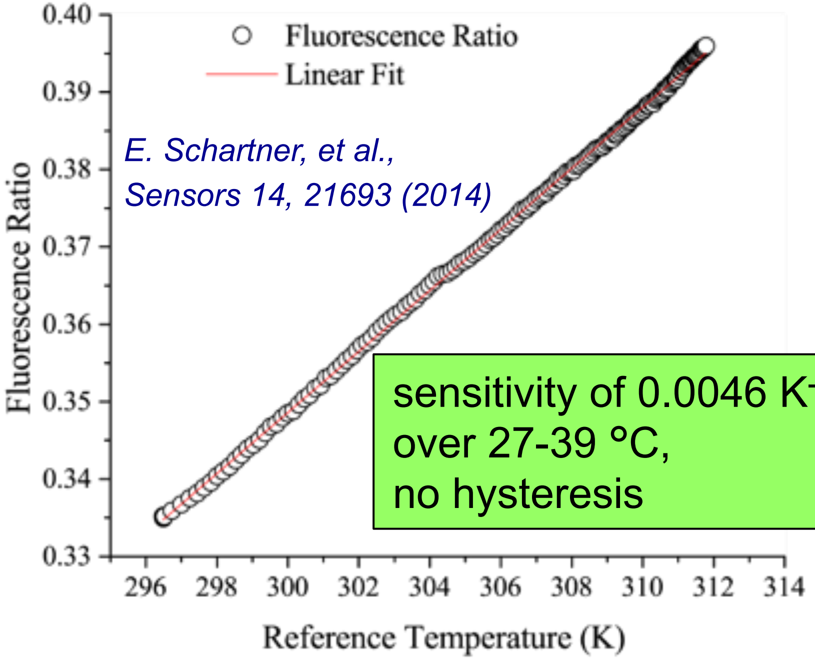
Tellurite glass: $TeO_2 – ZnO – Na_2O – La_2O_3$

- transmission 350nm – 4 μm
- high rare earth solubility
- low melting temperature of 700-900°C

M. Oermann, et al., Opt. Express 17, 15578 (2009)

in-vivo Temperature Sensor (2)

Calibration



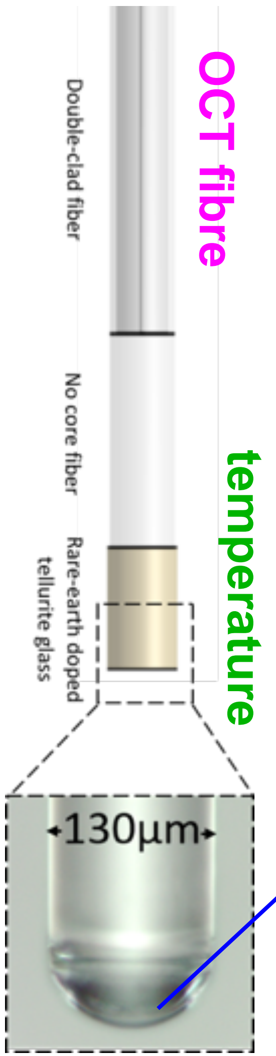
E. Schartner, et al., Sensors 14, 21693 (2014)

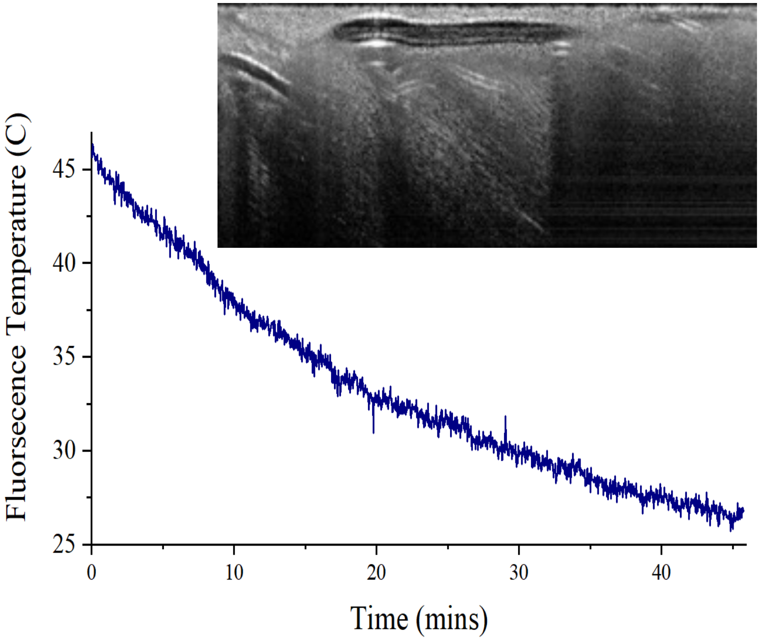
sensitivity of 0.0046 K^{-1}
over 27-39 °C,
no hysteresis

Brain Temperature

S. Musolino, et al., Biomed. Opt. Express 7, 3069 (2016)

Brain imaging + sensing





high index of tellurite
→ enhanced backreflection
➔ enhanced OCT signal

Jiawen Li, et al., Optics Letters (2018)

New Sensing and Imaging Fibre Concepts using lead-silicate glass

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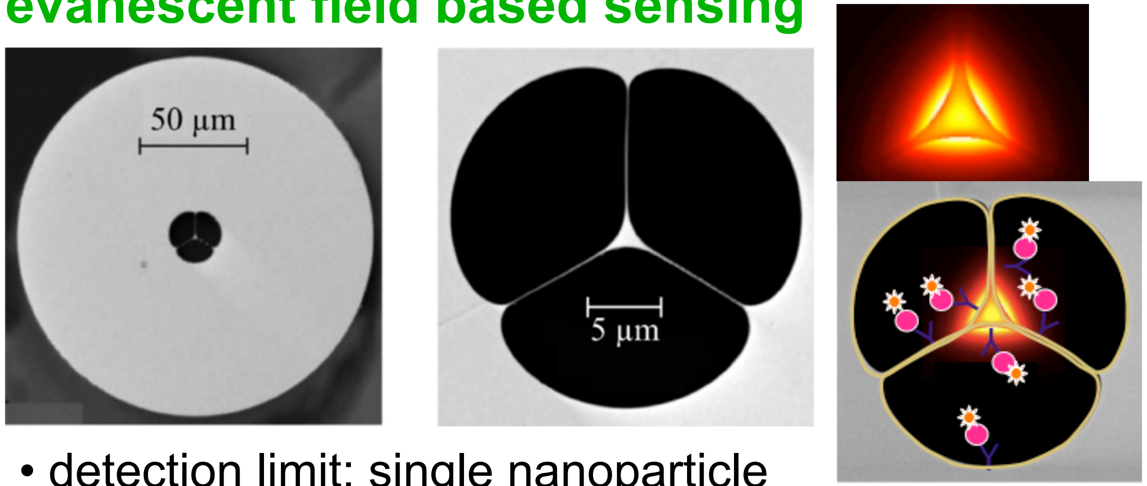
IPAS Institute for Photonics and Advanced Sensing

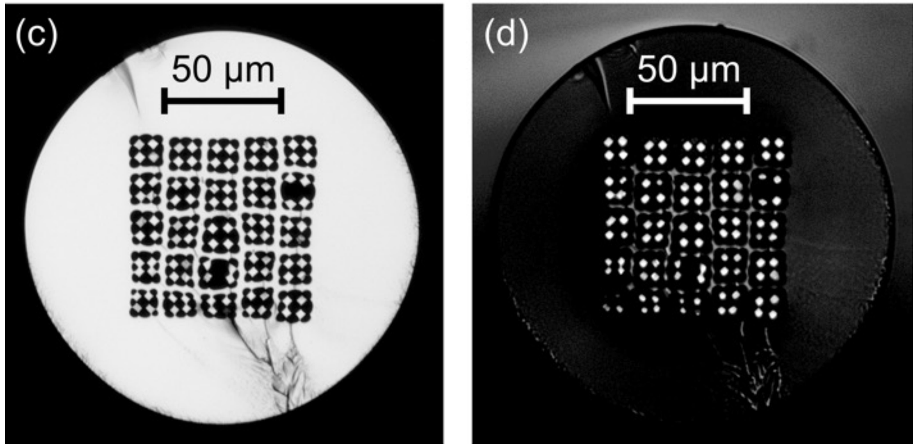
evanescent field based sensing



- detection limit: single nanoparticle
- selectivity and specificity via surface functionalisation

J. Zhao, et al., Nature Nanotech. 8, 729 (2013)
S. Warren-Smith, et al., Langmuir 27, 11 (2011)
E. Schartner, et al., Nanoscale 4, 7448 (2012)
Y. Ruan, et al., Opt. Express 16, 18514 (2008)

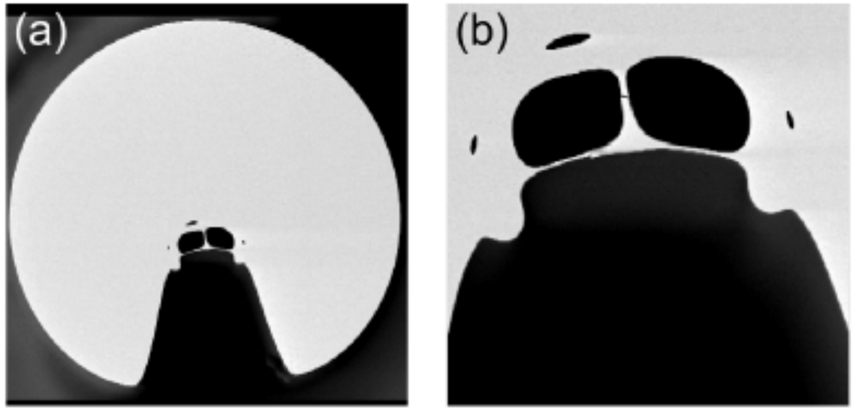
multi-core imaging



- 0.72µm core-core
- 50% glass fill factor
- 2500 pixels in 125 µm OD fibre possible

S. Warren-Smith, et al., Optics Express 26, 33604 (2018)

exposed core fibre concept



- easy access to the core
- distributed sensing

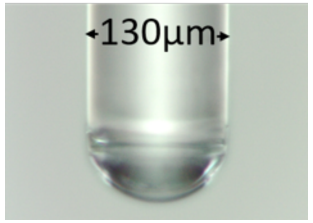
S. Warren-Smith, et al., Optics Express 17, 18533 (2009)

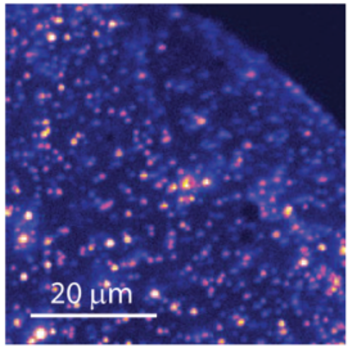
Conclusions

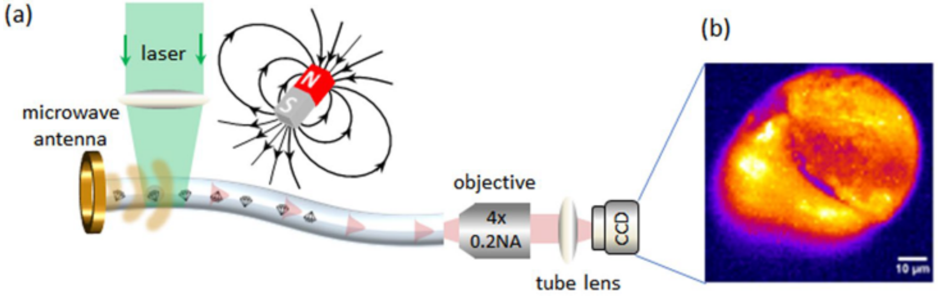
Soft Glass Highlights

- large variety in composition
  → properties (refractive index, transmission window)
- range of preform fabrication techniques

➔ range of fibre structures → fibre property tailoring



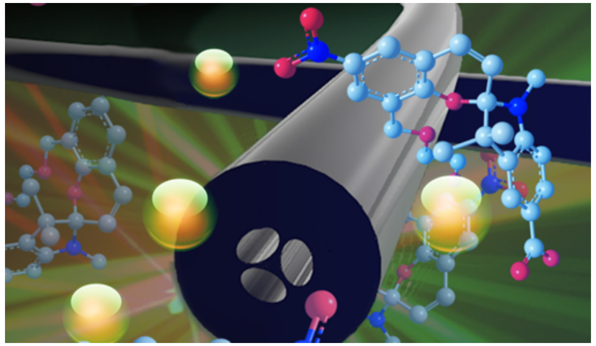


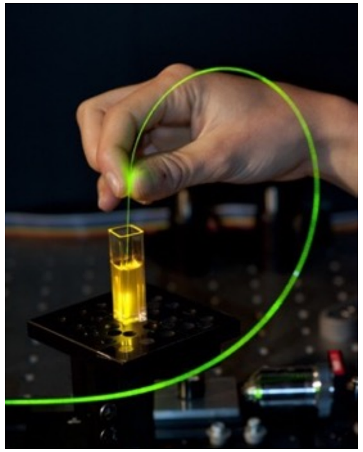


Real-world applications

challenges: - high purity → low loss

- reproducible loss and structure





Rapid Prototyping of New Fibre Concepts

Acknowledgment

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many colleagues, collaborators, students, technicians, …
in Australia and the world

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