

Glass-Based Sub-Wavelength Photonic Structures

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INSTITUTE FOR PHOTONICS AND NANOTECHNOLOGIES (IFN)



lighting the way ahead

Dipartimento di Fisica Scuola di Dottorato Università di Perugia 09 aprile 2014

OUTLINE

- MOTIVATION OF GLASS PHOTONICS RESEARCH

- GLASS CERAMICS

 - § DOWN CONVERSION SYSTEMS

 - § SnO_2 NANOCRYSTALS AS SENSITIZERS

- PHOTONIC CRYSTALS

 - § OPALS

 - § 1D MICROCAVITIES

- SPHERICAL MICRORESONATORS

- CONCLUSIONS AND PERSPECTIVES

CNR DSFTM

Scienze fisiche e tecnologie della materia

CSMFO Lab. <http://www.tn.ifn.cnr.it/>



Consiglio Nazionale delle Ricerche
Istituto di Fotonica e Nanotecnologie - Unità di Trento



MOTIVATION OF GLASS PHOTONICS RESEARCH

MOTIVATION

Light control

Nano scale **confined structures**

- Glass-ceramic waveguides
- Photonic crystals (n-D, Fibers)
- Colloidal systems

Micro scale **confined structures**

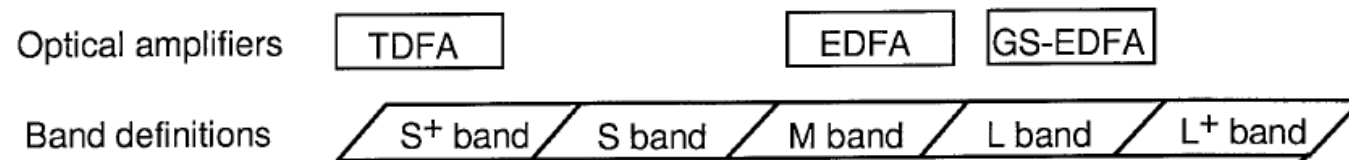
- Fibers and Waveguides
- **Resonators**
- Cavities

Novel materials, functionalizations, and geometries

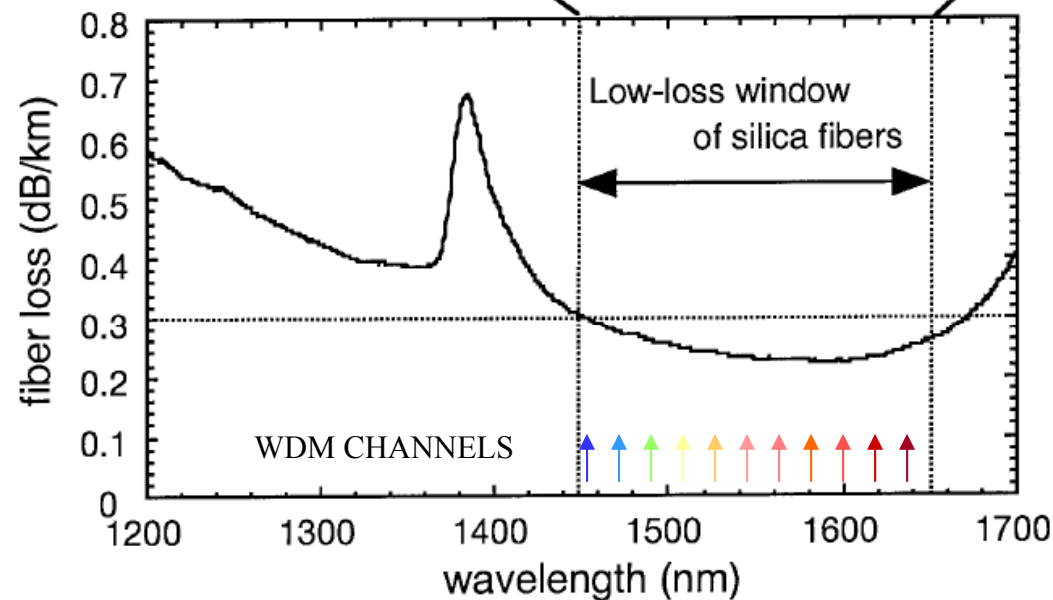
WE START FROM:

$$I_{th} = \frac{h \nu_p}{\sigma_p \tau}$$

AND.....



25 THz



Low loss window
(< 0.3 dB/Km) is
200 nm wide

- More channels
- Largely-spaced channels
- Non-zero dispersion

Range of high Gain EDFAs 4 THz

GLASS CERAMICS AND ENERGY TRANSFER

Tb^{3+} and Yb^{3+} ions in sol-gel derived
 $\text{SiO}_2\text{-HfO}_2$ glass ceramic planar waveguides



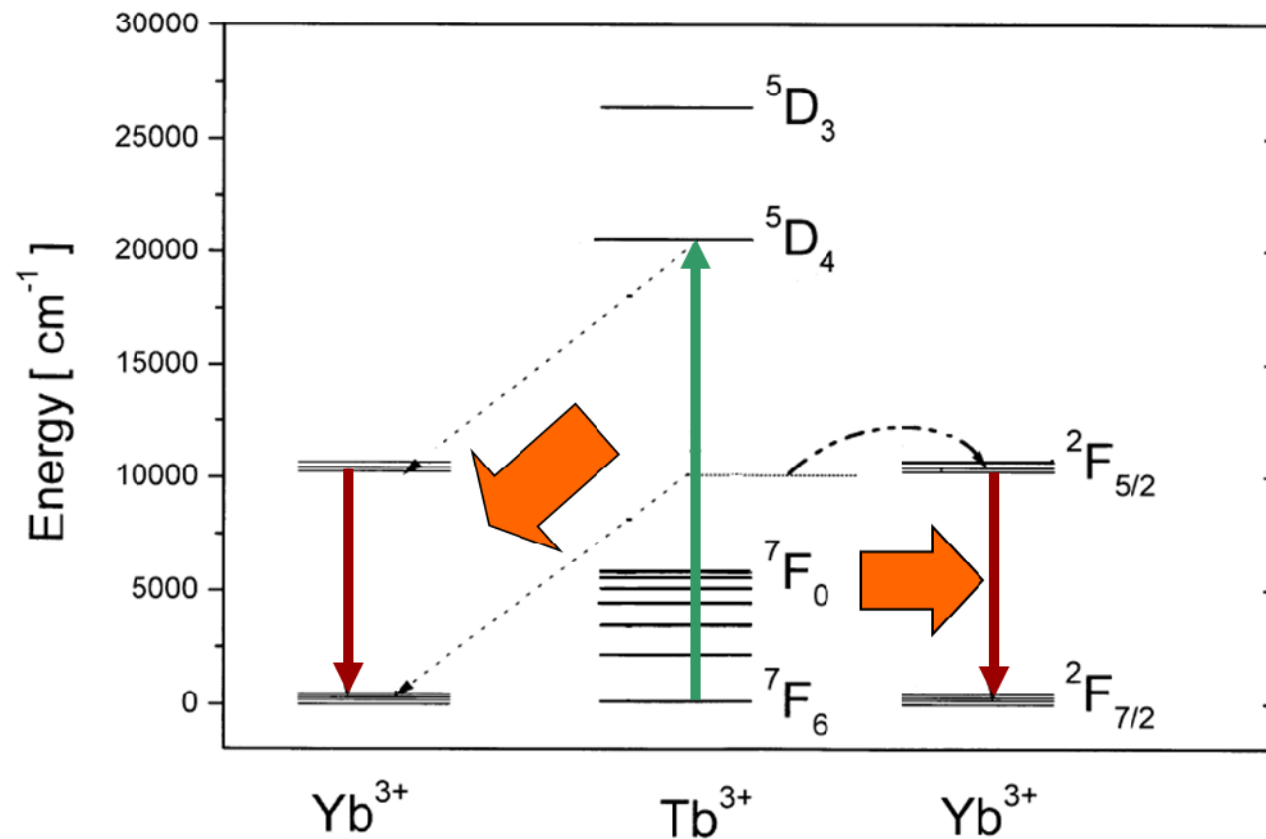
Newsroom

10.1117/2.1201105.003701

**Rare-earth doped materials
enhance silicon solar cell
efficiency**

Guillaume Alombert-Goget, Davor Ristic,
Alessandro Chiasera, Stefano Varas, Maurizio Ferrari,
Giancarlo C. Righini, Belto Dieudonne, and
Brigitte Boulard

Down conversion with $\text{Tb}^{3+}/\text{Yb}^{3+}$



One green photon @ 488nm $\longrightarrow$ Two red photon @ 980 nm

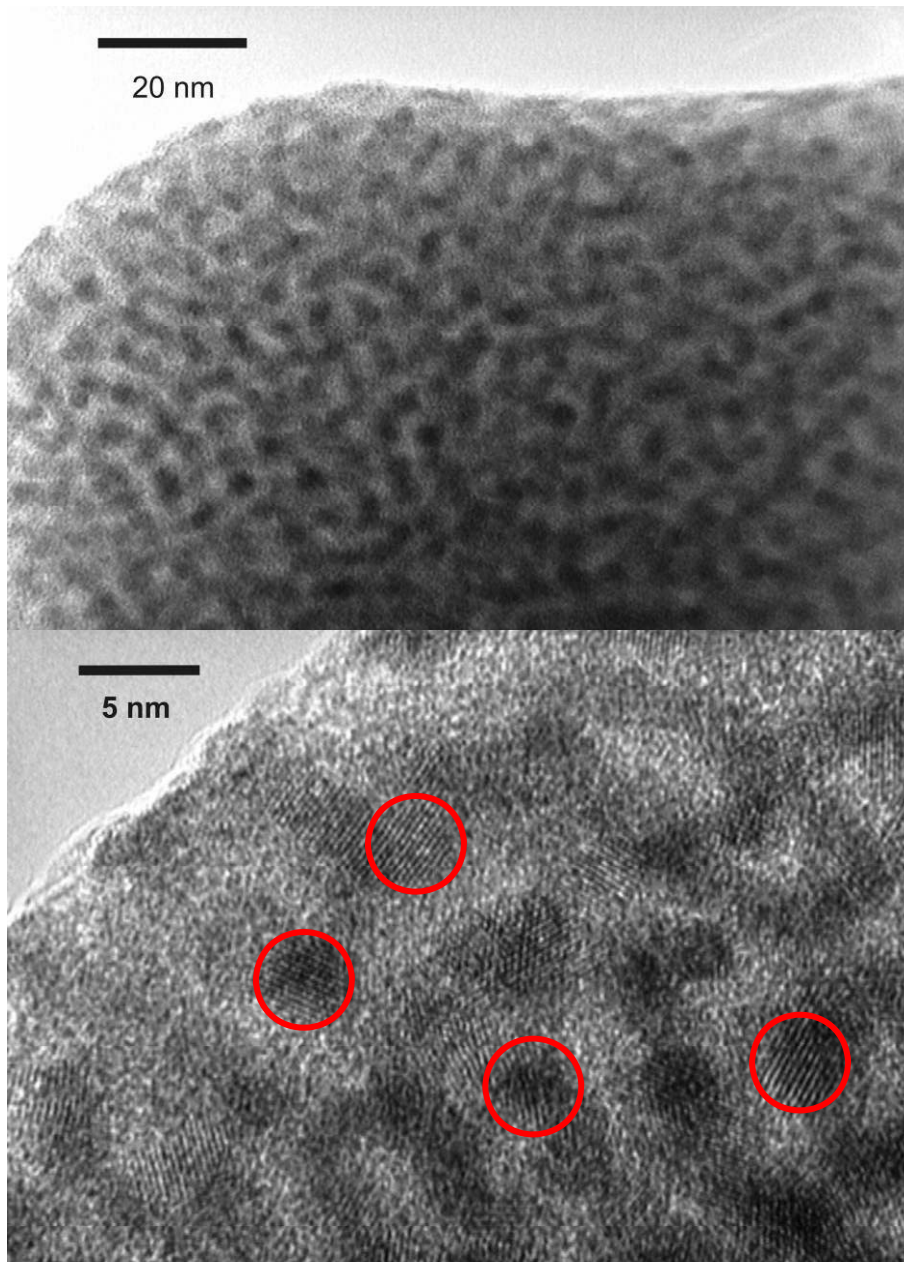
Down-converters Silica-Hafnia glass ceramics

- ❑ Low phonon energy ($\sim 700 \text{ cm}^{-1}$)
- ❑ Rare earth solubility
- ❑ Combine spectroscopic properties of the crystal with optical properties of the glass
- ❑ Determine the efficiency of the process
- ❑ Optimize the rare earth ions content

Prepared waveguides

Rare earth concentration, refractive index and thickness of the waveguides

Sample label	Terbium concentration in mol%	Ytterbium concentration in mol%	n@543.5 nm TE polarization [± 0.001]	n@632.8 nm TE polarization [± 0.001]	Layer thickness [$\pm 0.2\mu\text{m}$]
AR1	0.5	0	1.621	1.616	1.0
A1	0.5	1	1.626	1.621	1.0
A2	0.5	2	1.631	1.626	1.0
A3	0.5	3	1.633	1.628	1.1
BR1	0.2	0	1.623	1.617	1.1
B1	0.2	0.8	1.604	1.608	0.9
BR3	0.6	0	1.624	1.620	1.1
B3	0.6	2.4	1.630	1.625	1.2
BR5	1	0	1.626	1.621	1.2
B5	1	4	1.638	1.633	1.1



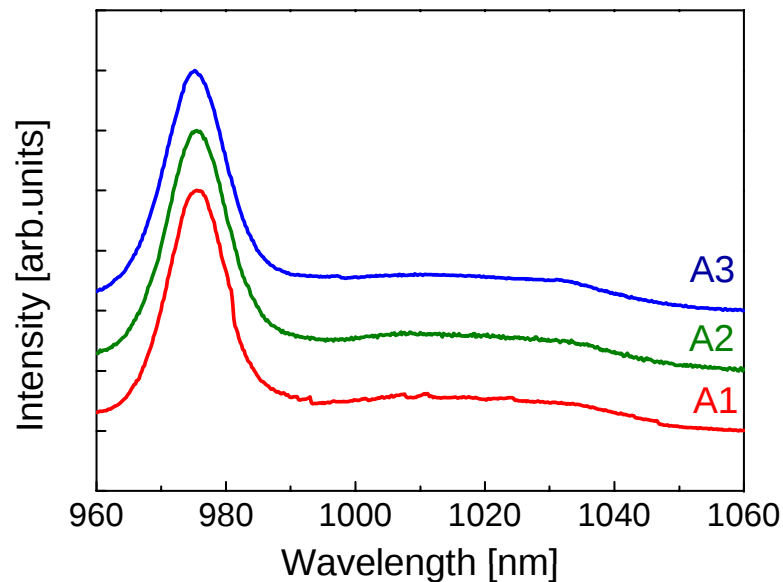
TEM images

- **Validation of the process:**
Incorporation of hafnia nanocrystals in the waveguide
- **Nanocrystals identified as monoclinic HfO_2**
- **Size of about 3-4 nm**

Photoluminescence spectra

Room temperature photoluminescence spectra of the $^2F_{5/2} \rightarrow ^2F_{7/2}$ transition of Yb^{3+} ions after excitation at 476 nm for the three samples of the first series. Each spectrum was normalized to the maximum of the luminescence intensity.

The emission of the Yb^{3+} ion after excitation at 476 nm is a indication of the presence of an **efficient energy transfer from Tb^{3+} to Yb^{3+}** .



Tb 0.5 mol%

A1 1 mol% Yb

A2 2 mol% Yb

A3 3 mol% Yb

However, it is not possible to *evaluate* the conversion efficiency only on the base of the photoluminescence spectra.

The TE_0 mode waveguiding excitation was used for luminescence measurements, by detecting the light coming out from the waveguide surface. Luminescence spectroscopy was performed using the 476 nm line of an Ar^+ ion laser as excitation source at 600 mW power.

Decay curves analysis

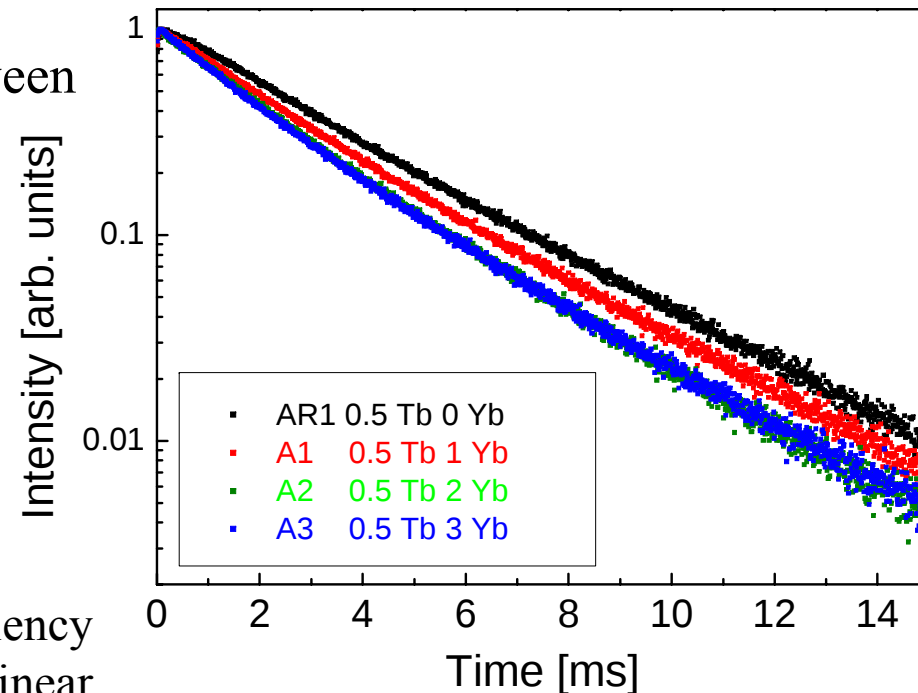
Energy transfer efficiency between Tb^{3+} and Yb^{3+} :

$$\eta_{\text{Tb-Yb}} = 1 - \frac{\int I_{\text{Tb-Yb}} dt}{\int I_{\text{Tb}} dt}$$

The relation between the transfer efficiency and the **effective quantum efficiency** is linear and is defined as:

$$\eta_{\text{EQE}} = \eta_{\text{Tb-r}}(1 - \eta_{\text{Tb-Yb}}) + 2\eta_{\text{Tb-Yb}}$$

where the quantum efficiency for Tb^{3+} ions, $\eta_{\text{Tb-r}}$ is set equal to 1.



Decay curves of the luminescence from the $^5\text{D}_4$ metastable state of Tb^{3+} ions for the first samples series under excitation at 355 nm.

Tb³⁺/ Yb³⁺ down conversion efficiency

Transfer efficiency and effective quantum efficiency as a function of Yb³⁺ molar concentration for A samples where Tb³⁺ content is fixed at 0.5 mol%

Composition (Yb concentration in mol%)	1%	2%	3%
Estimated transfer efficiency (in glass ceramic)	14%	24%	25%
Estimated effective quantum efficiency (in glass ceramic)	114%	124%	125%
Estimated transfer efficiency (in glass)	2%	4%	6%
Estimated effective quantum efficiency (in glass)	102%	104%	106%

For compositions with Tb³⁺ content kept constant at 0.5 mol% and increasing Yb³⁺ molar concentration, we observed that:

- the Tb-Yb energy transfer efficiency increases with the increase of the molar ratio Yb/Tb;
- the energy transfer efficiency does not exceed 24-25%.

SnO₂-SiO₂ Glass-Ceramic Waveguides

Heat Treatment at
900, 1000, 1100 °C for 30'- 5h

Samples:

92 SiO₂ - 8 SnO₂ : 1 mol% Eu³⁺ _HT at 800, 900, 1000, 1100°C for 30'.

84 SiO₂ - 16 SnO₂ : 1 mol% Eu³⁺ _HT at 800, 900, 1000, 1100°C for 30'.

75 SiO₂ - 25 SnO₂ : 1 mol% Eu³⁺ _HT at 800, 900, 1000, 1100°C for 30', 1100 °C for 1h and 5h.

S.N.B. Bhaktha, F. Beclin, M. Bouazaoui, B. Capoen, A. Chiasera, M. Ferrari, C. Kinowski, G.C. Righini, O. Robbe, S. Turrell

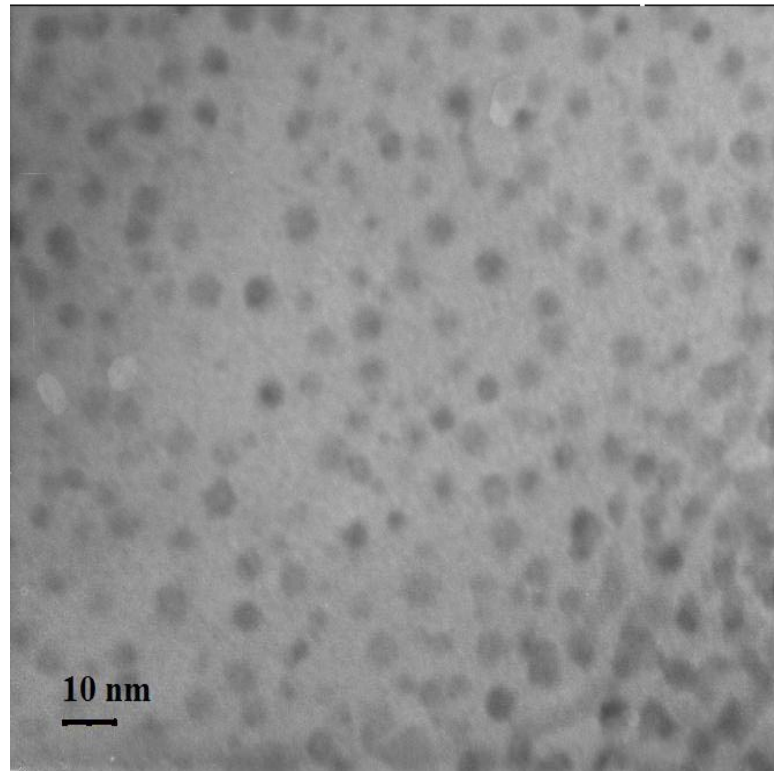
“Enhanced fluorescence from Eu³⁺ in low-loss silica glass-ceramic waveguides with high SnO₂ content”
Applied Physics Letters 93 (2008) pp. 211904-1/3.

T.T. Van Tran, T. Si Bui, S. Turrell, B. Capoen, P. Roussel, M. Bouazaoui, M. Ferrari, O. Cristini, and C. Kinowski

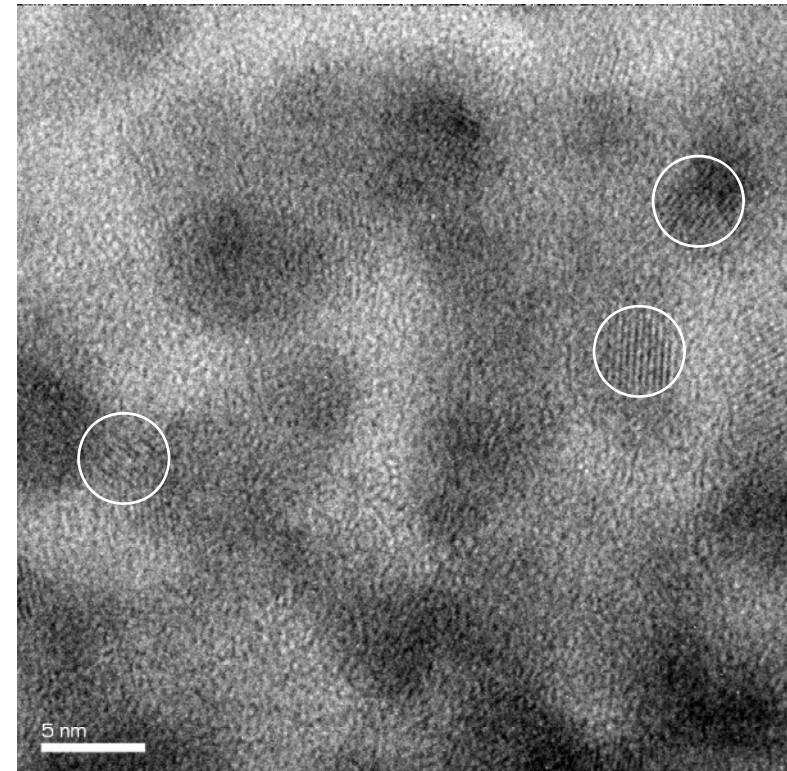
“Controlled SnO₂ nanocrystal growth in SiO₂-SnO₂ glass-ceramic monoliths”
Journal of Raman Spectroscopy 43 (2012) pp. 869-875.

TEM

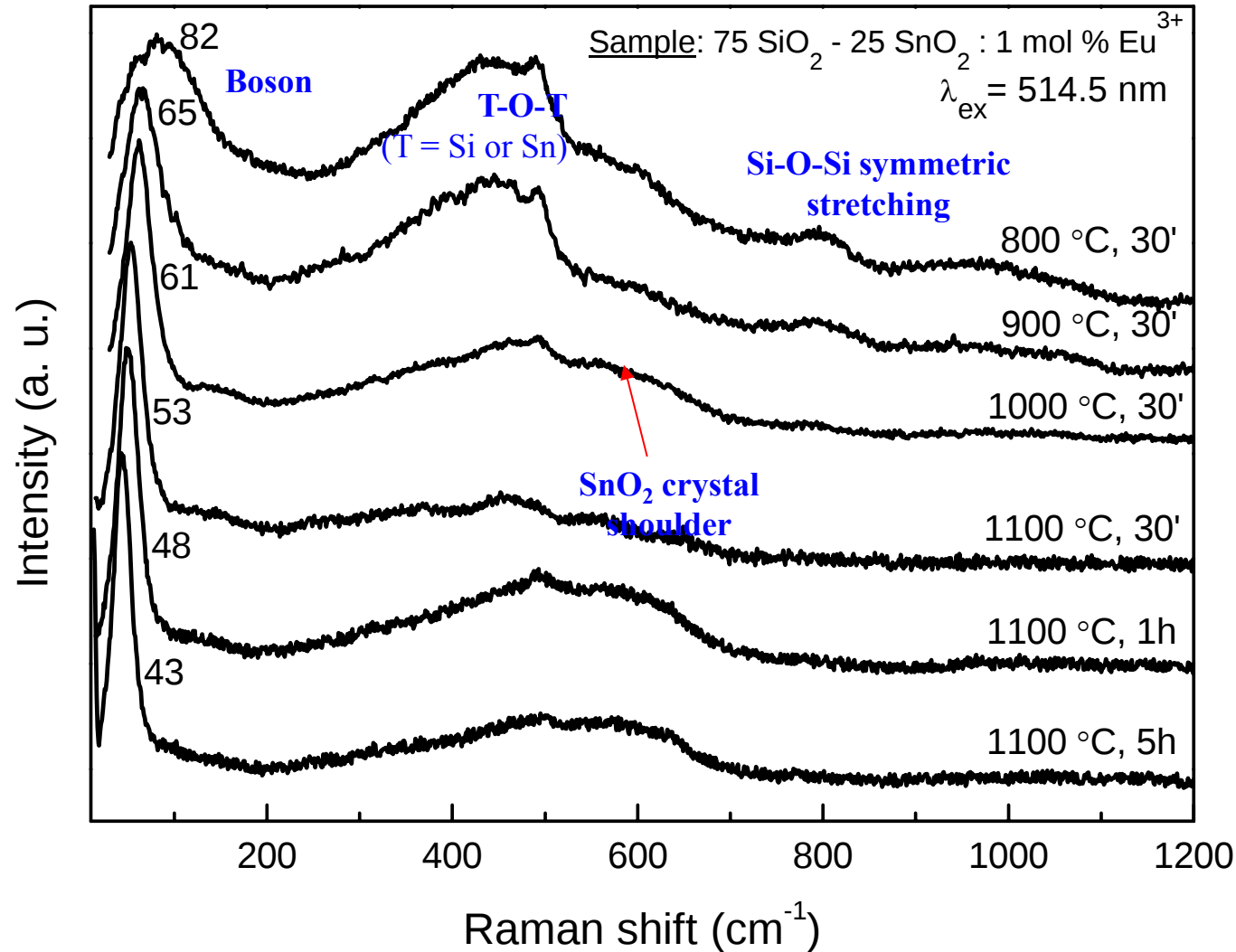
Homogeneous distribution



About 4 nm NCs



Raman spectra



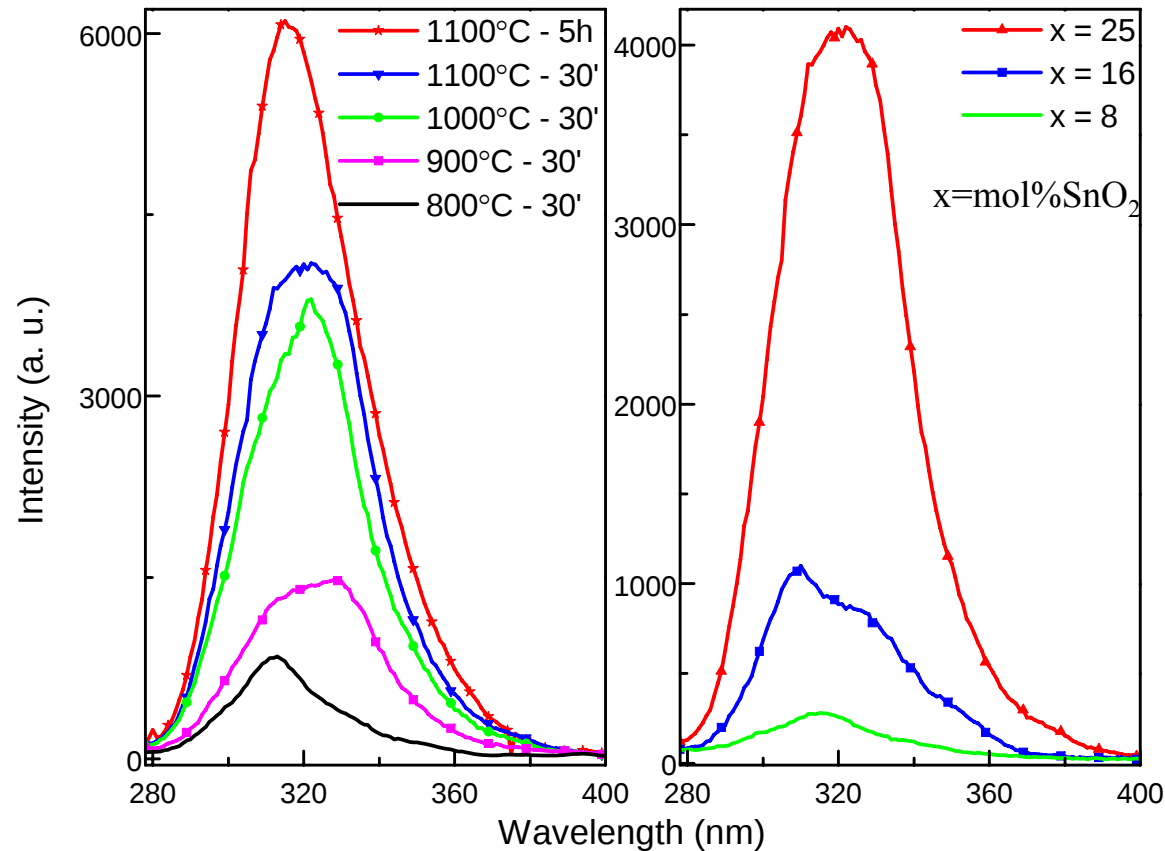
$$\omega = S_{l,n} \frac{v}{Lc}$$

ω = wavenumber
 v = sound velocity
 L = particle size
 c = speed of light

- Crystal-peak observed in the low-frequency region.
- Increasing shoulder centered around 550 cm⁻¹ is attributed to the SnO₂ crystals.

Excitation spectra

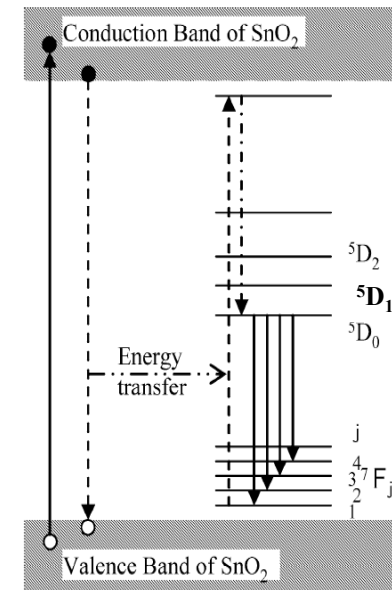
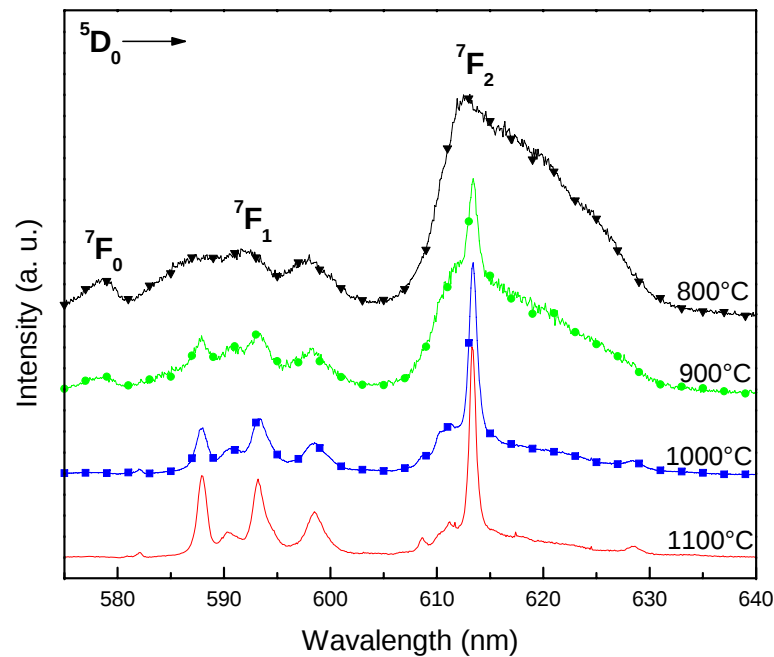
Room temperature excitation spectra of $^5D_0 \rightarrow ^7F_2$ emission at 613 nm



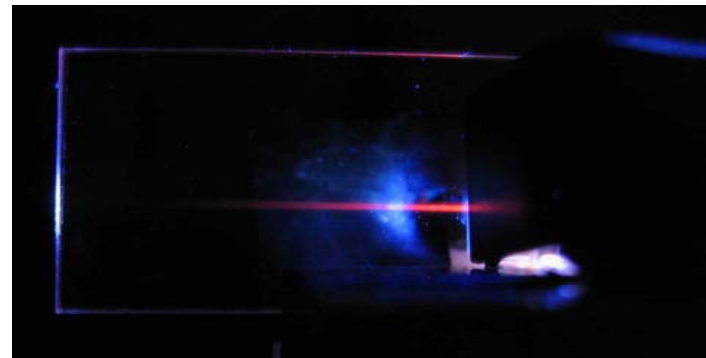
The broad and strong band observed at 310 nm (4.0 eV) corresponds to the SnO₂ band-gap energy.

PL spectra

$$\lambda_{\text{ex}} = 351 \text{ nm}$$



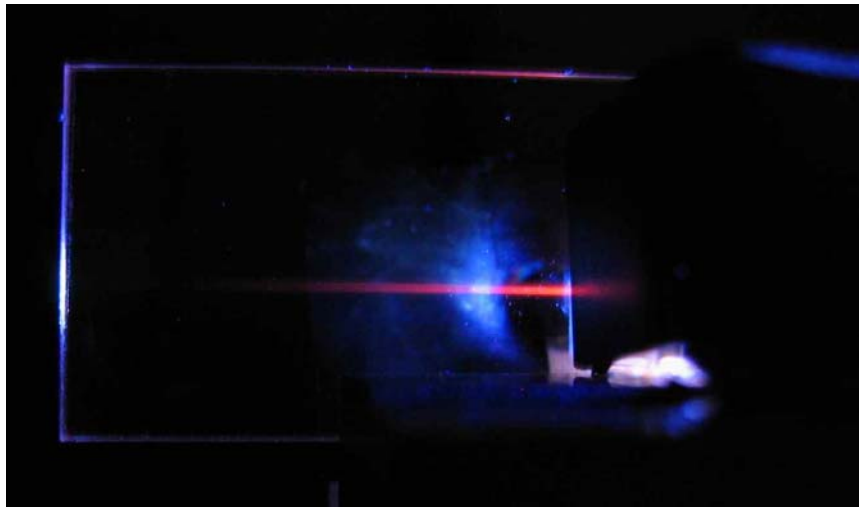
For annealing temperature higher than 900 °C the emission **features typical of Eu³⁺ ion in a crystalline like environment** are predominant, *indicating that most part of Eu³⁺ ions are embedded in SnO₂ nanocrystals*



Enhanced fluorescence from Eu^{3+} in low-loss silica glass-ceramic waveguides with high SnO_2 content

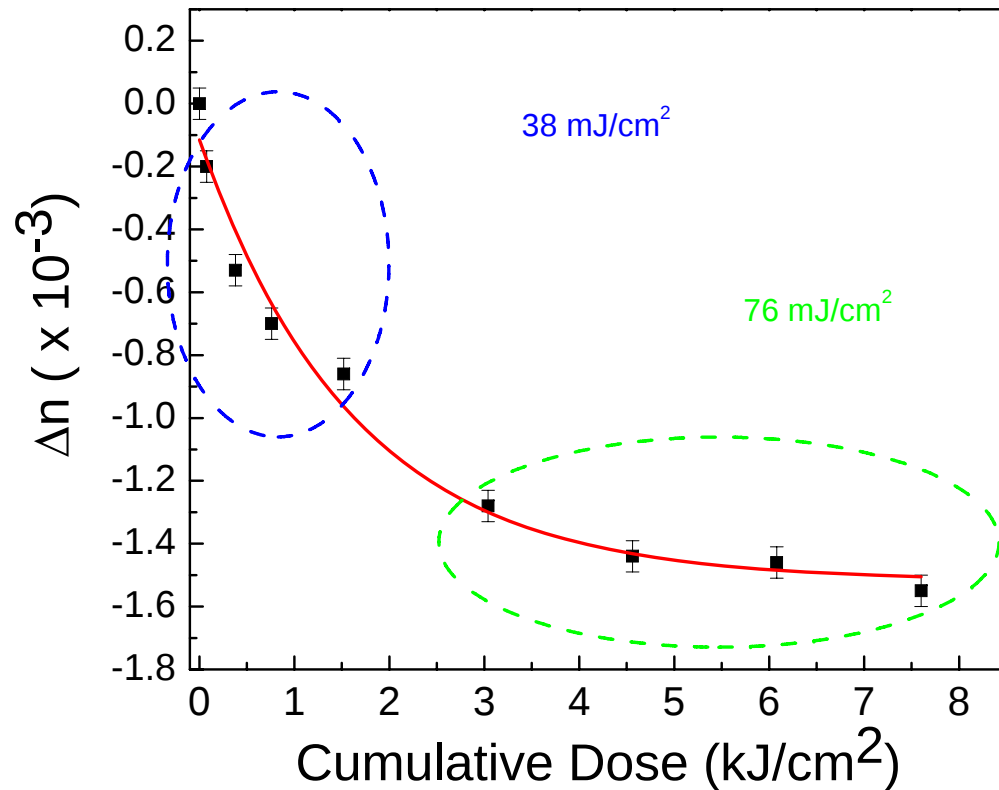
$$\lambda_{\text{ex}} = 351 \text{ nm}$$

S.N.B. Bhaktha, F. Beclin, M. Bouazaoui, B. Capoen, A. Chiasera, M. Ferrari, C. Kinowski, G.C. Righini, O. Robbe, S. Turrell, "Applied Physics Letters 93 (2008) pp. 211904-1/3.



Crack-free and low loss glass ceramic waveguide 75SiO_2 - 25SnO_2 : Eu^{3+} fabricated by sol gel, dip-coating method. Losses remain below 0.8 dB/cm.

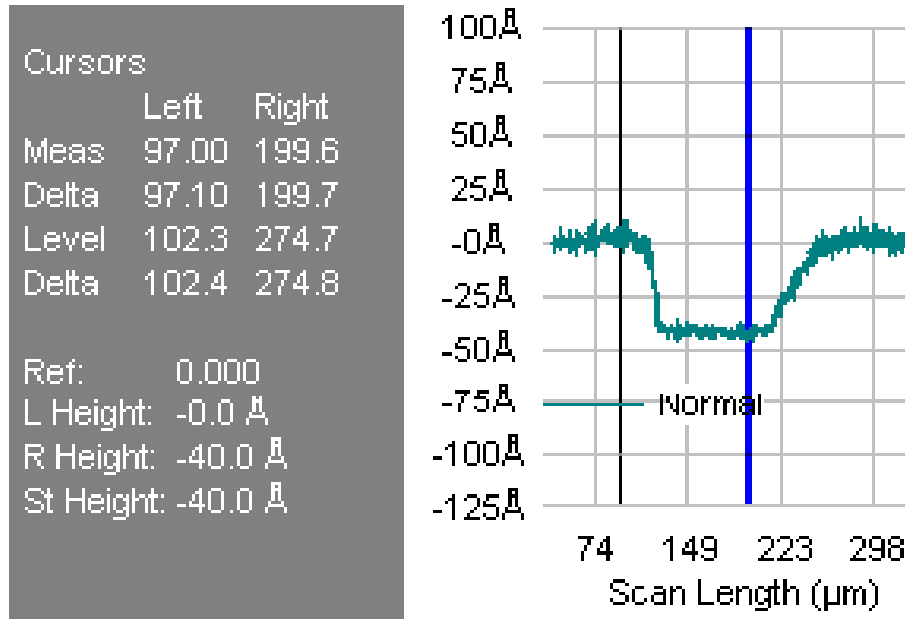
High photosensitivity in low-loss sol-gel $\text{SiO}_2 - \text{SnO}_2$ waveguides



Effective index change at 1550 nm for the single mode waveguide after the UV irradiation at 248 nm as a function of cumulative doses used. The solid line is an help for the eyes.

S. Berneschi, S.N.B. Bhaktha, A. Chiappini, A. Chiasera, M. Ferrari, C. Kinowski, S. Turrell, C. Trono, M. Brenci, I. Cacciari, G. Nunzi Conti, S. Pelli, G. C. Righini "Highly photorefractive Eu^{3+} activated sol-gel $\text{SiO}_2 - \text{SnO}_2$ thin film waveguides"
Proceedings of SPIE Vol. 7604 (2010) pp. 76040Z-1/6

High photosensitivity in low-loss sol-gel $\text{SiO}_2 - \text{SnO}_2$ waveguides



A swelling of about 4 nm
was observed in UV
exposed regions

B.N. Shivakiran Bhaktha, Simone Berneschi, Gualtiero Nunzi Conti, Giancarlo C. Righini, Andrea Chiappini, Alessandro Chiasera, Maurizio Ferrari, Sylvia Turrell
“Spatially localized UV-induced crystallization of SnO_2 in photorefractive SiO_2 - SnO_2 thin film”.
Proceedings of SPIE Vol. 7719 (2010) pp. 77191B-1/5.

S. Berneschi, S.N.B. Bhaktha, A. Chiappini, A. Chiasera, M. Ferrari, C. Kinowski, S. Turrell, C. Trono, M. Brenci, I. Cacciari, G. Nunzi Conti, S. Pelli, G. C. Righini “Highly photorefractive Eu^{3+} activated sol-gel $\text{SiO}_2 - \text{SnO}_2$ thin film waveguides”
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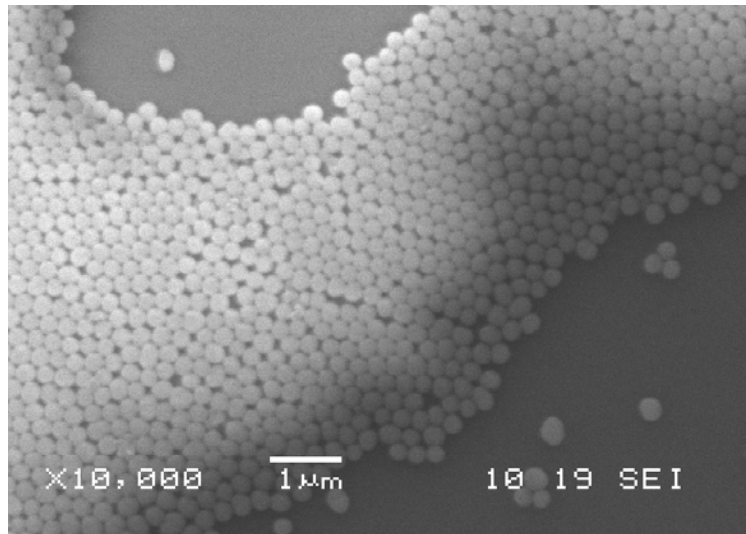
PHOTONIC CRYSTALS

OPALS: A SUITABLE PHOTONIC STRUCTURE FOR LIGHT CONFINEMENT

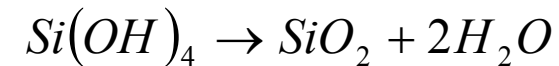
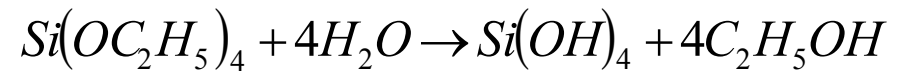
Molar Concentrations

- o $[TEOS] = 0.22\text{ M}$
- o $[NH_3] = 1.0\text{ M}$
- o $[H_2O] = 15.0\text{ M}$

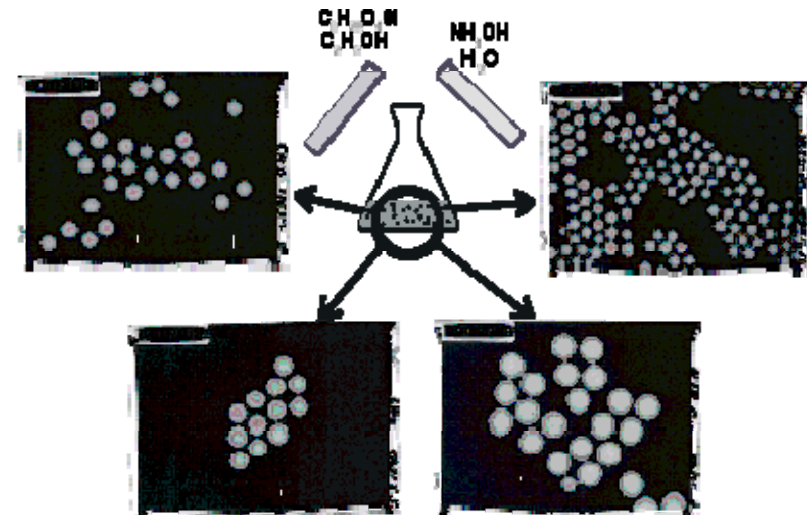
the dimension of : ~255nm



Stober Methods



Silica Spheres



high uniformity of *silica spheres*
(less than 5% of dispersion in diameter)

The reaction takes place at room temperature and

we can control the particle size through the concentration of a single reactive.

3D Photonic Crystals bottom-up approach

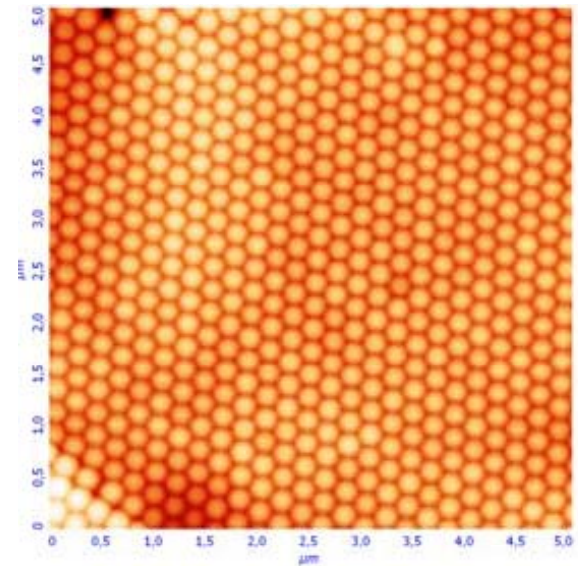
*Self-Assembling
colloidal nano-microspheres*

Low cost

associated with their manufacture

“Easy”

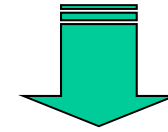
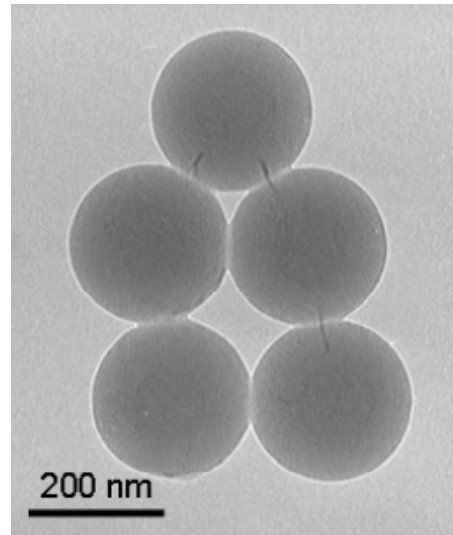
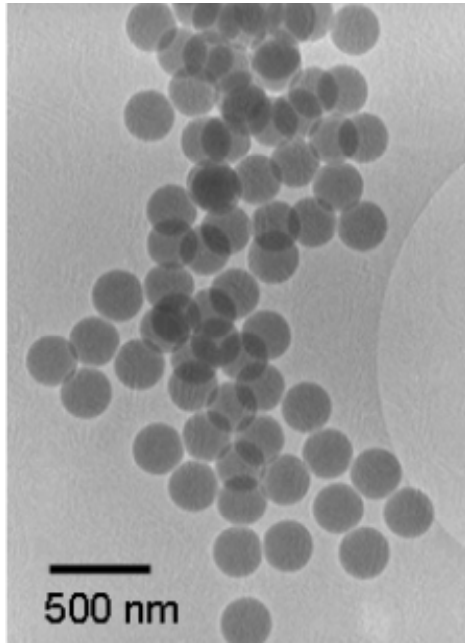
relative easiness of preparation



AFM image of the top surface of the opal formed by spin-coating technique of 236 nm diameter polystyrene spheres.

A. Chiappini, C. Armellini, A. Chiasera, M. Ferrari, L. Fortes, M. Clara Gonçalves, R. Guider, Y. Jestin, R. Retoux, G. Nunzi Conti, S. Pelli, Rui M. Almeida, and G.C. Righini. “An alternative method to obtain direct opal photonic crystals structures” *Journal of Non-Crystalline Solids* 355 (2009) pp. 1167-1170

Polystyrene monosized spheres



Single-stage polymerization process based on formation and growth of polymeric nuclei dispersed in an emulsion constituted by water, styrene, potassium persulfate (KPS) and sodium docecyl sulfate (SdS).

HRTEM characterization of polystyrene spheres synthesized by microemulsion, where an average dimension of 236 nm with a polydispersivity of about 3% is determined

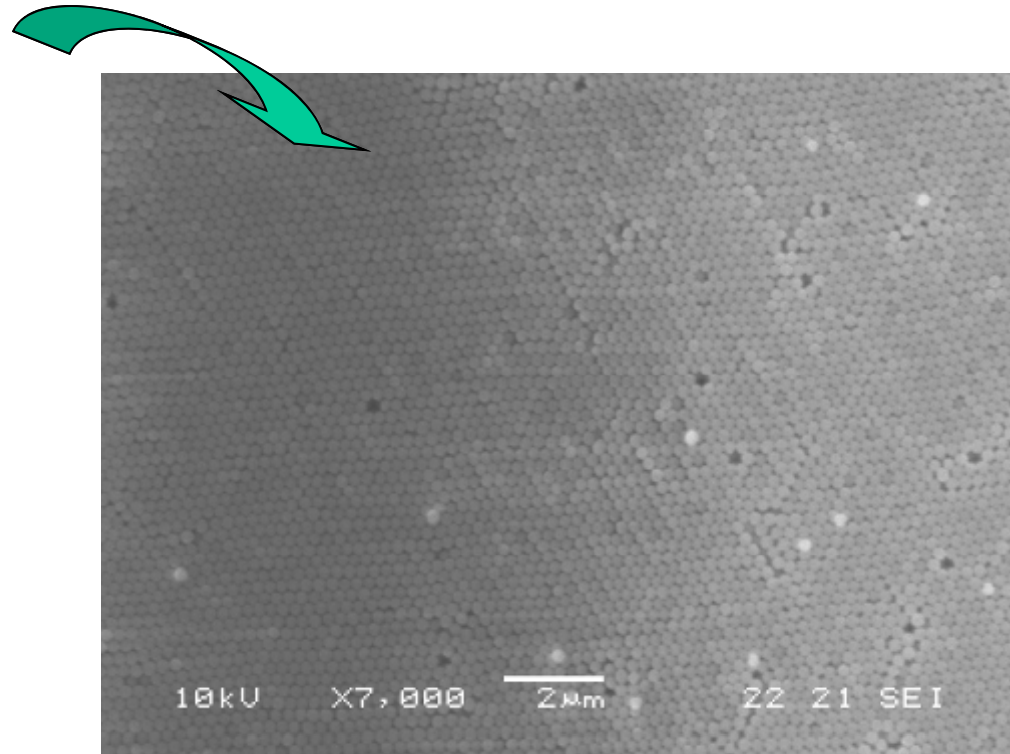
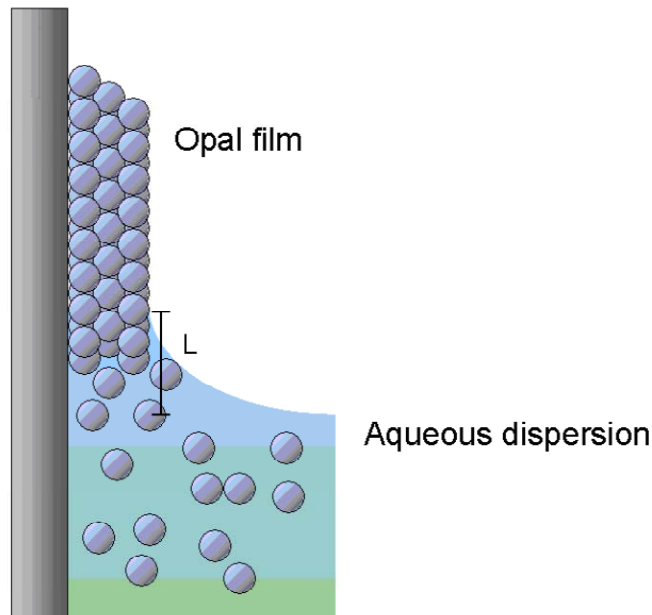
Range 150 ÷ ~ 1000 nm

Control the size of the particles through the concentration of sodium docecyl sulfate.

Self Assembly approach

Self Assembly techniques

Vertical Deposition

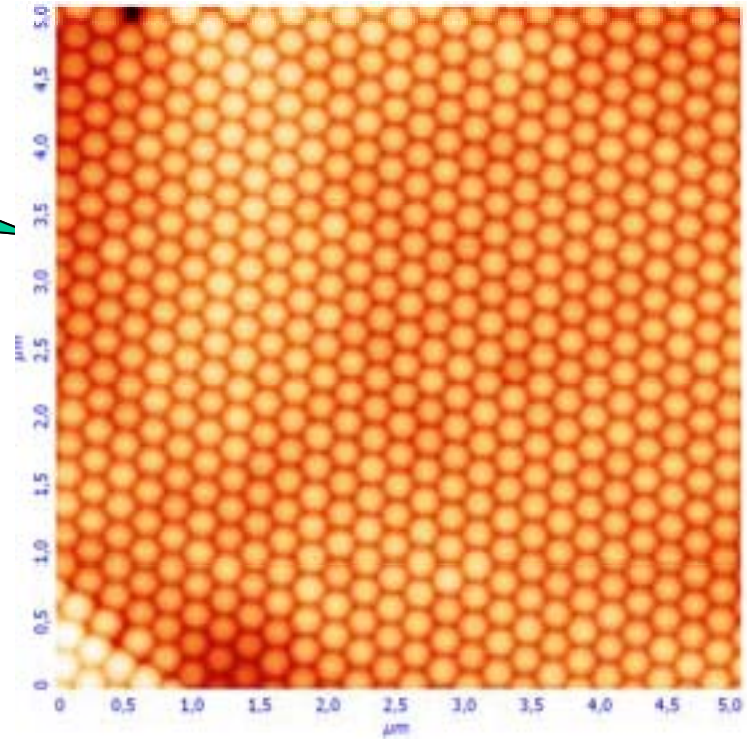


A. Chiappini, C. Armellini, A. Chiasera, M. Ferrari, Y. Jestin, M. Mattarelli, M. Montagna, E. Moser, G. Nunzi Conti, S. Pelli, G.C. Righini, M. Clara Gonçalves, Rui M. Almeida, "Design of photonic structures by sol-gel-derived silica nanospheres" *Journal of Non-Crystalline Solids* 353 (2007) pp. 674–678.

“Bottom up approach”

Self Assembly techniques

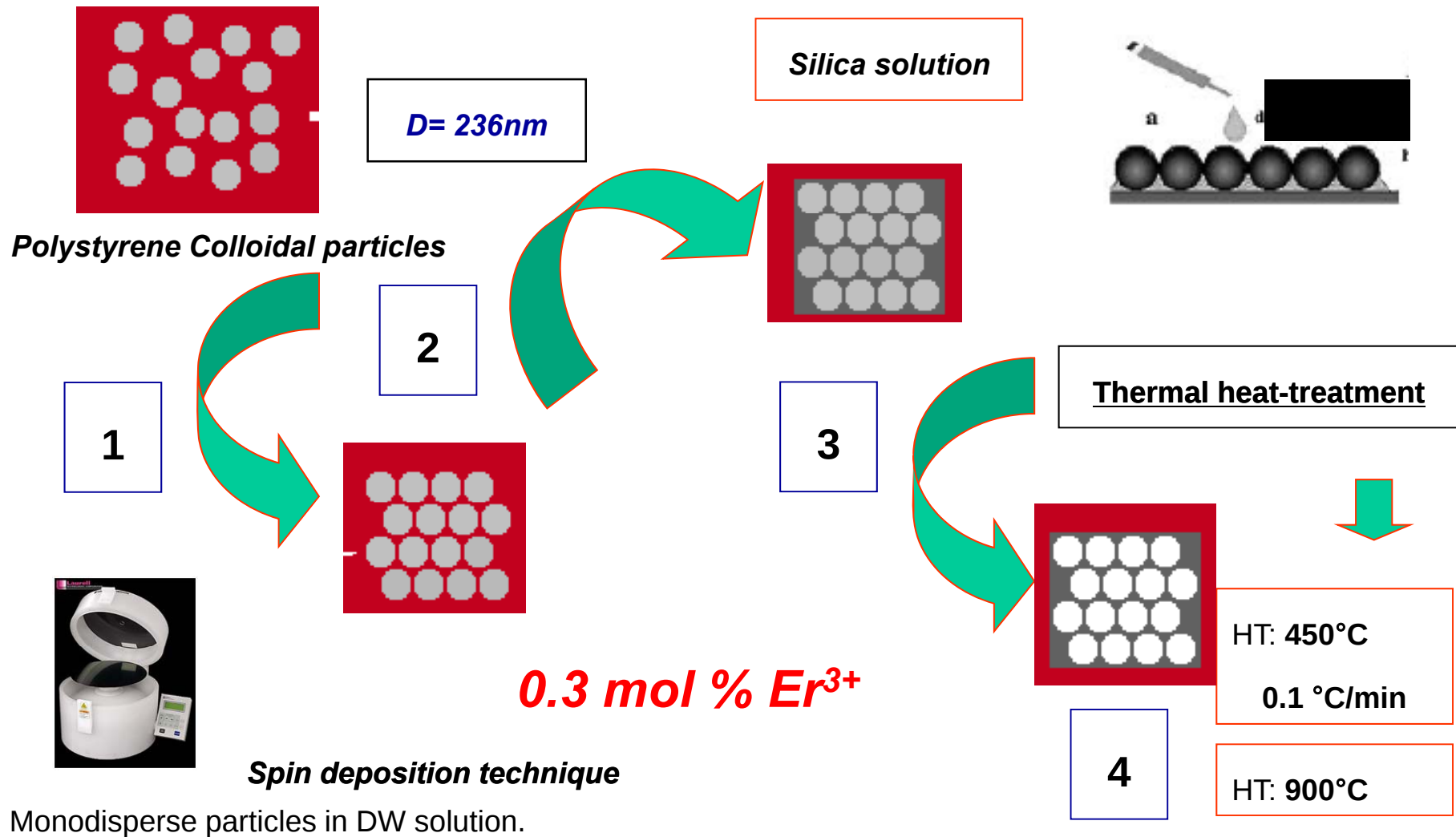
Spin-coating deposition



C. Armellini, A. Chiappini, A. Chiasera, M. Ferrari, Y. Jestin, E. Moser, G. Nunzi Conti, S. Pelli, A. Quandt, G.C. Righini, C. Tosello
“Er³⁺ - activated nanocomposite photonic glasses and confined structures”
Optical Materials 31 (2009) pp. 1071-1074.

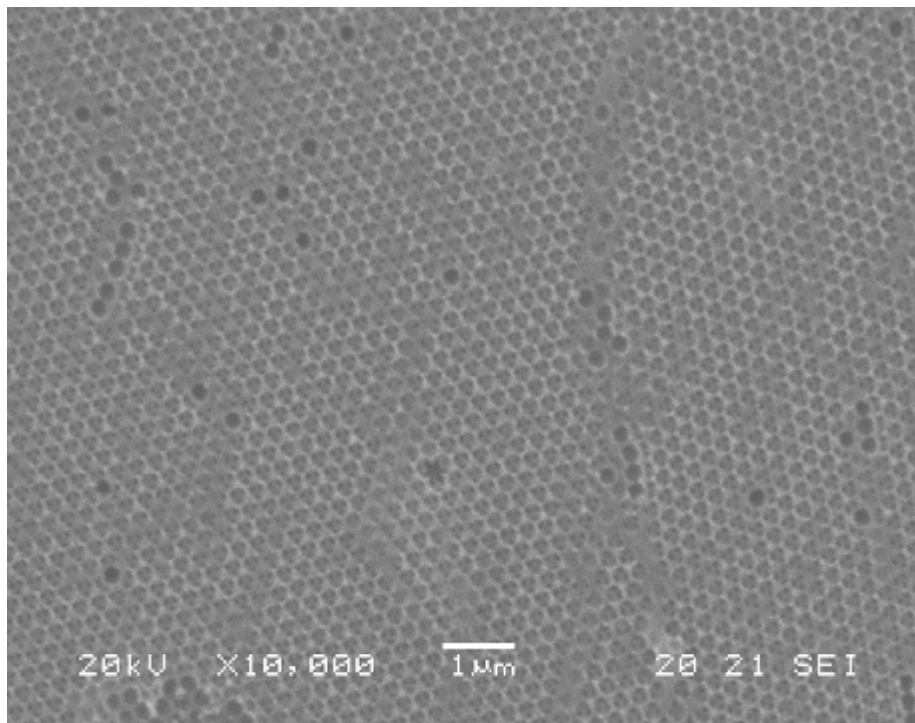
Silica inverse opals

Latex opals as templates

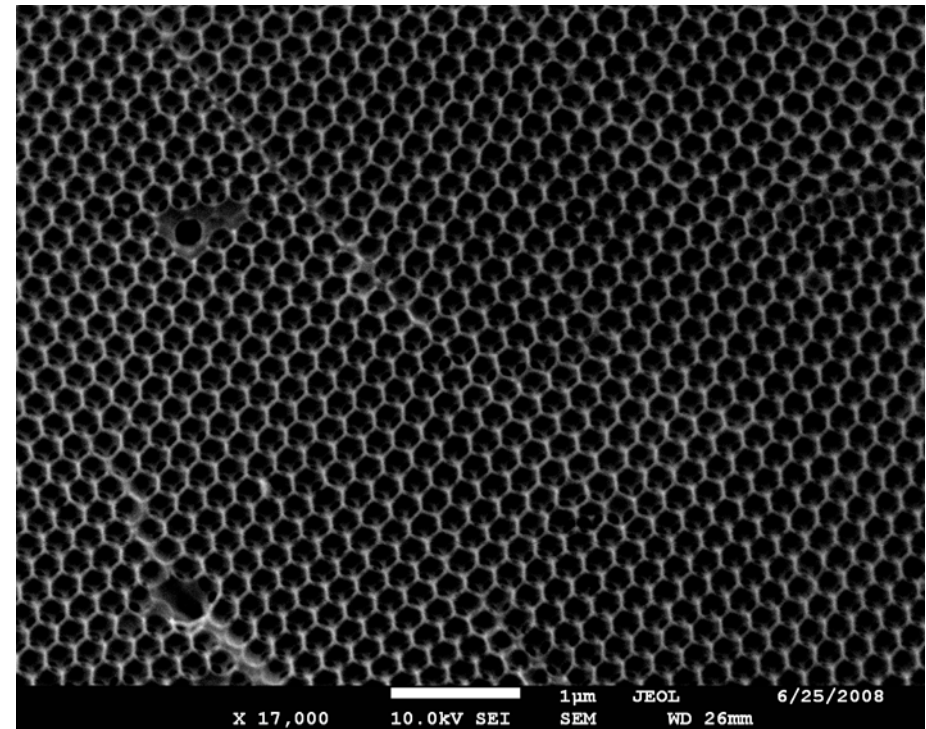


Inverse structure

It's a negative replica of direct opal
(template)



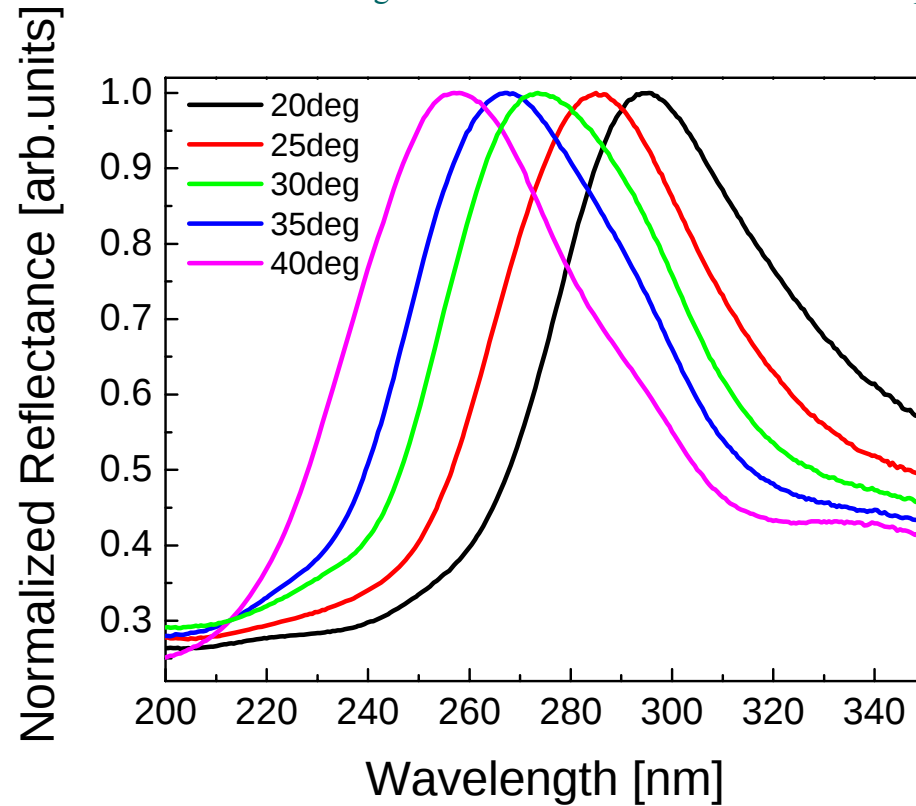
The average dimension of the air-hollows is ~210 nm



Hollow regions of air spheres
well ordered in a triangular lattice
corresponding to the **(111) planes**
of a fcc crystalline structure.

Optical Properties

Variable incident angle reflectance measurements of inverse opals



Bragg's Law

$$\lambda = 2 \times 0.816 D (n_{eff}^2 - \sin^2 \theta)^{1/2}$$

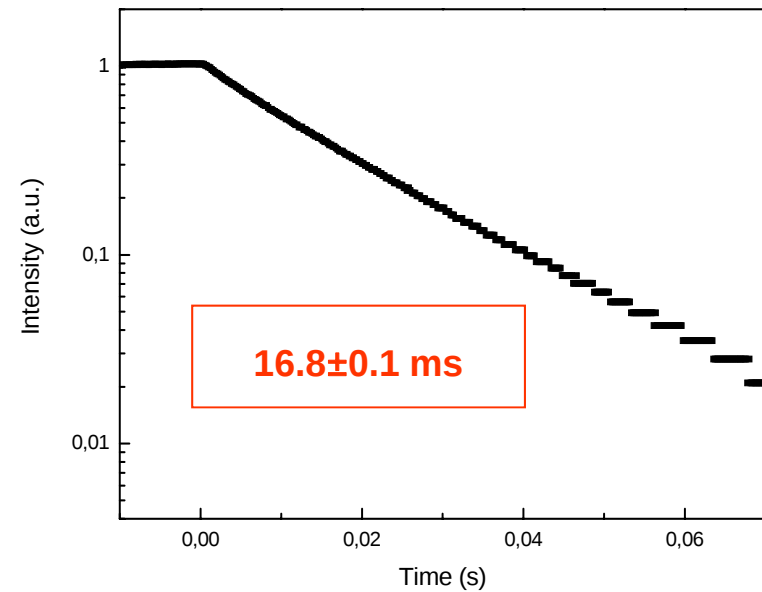
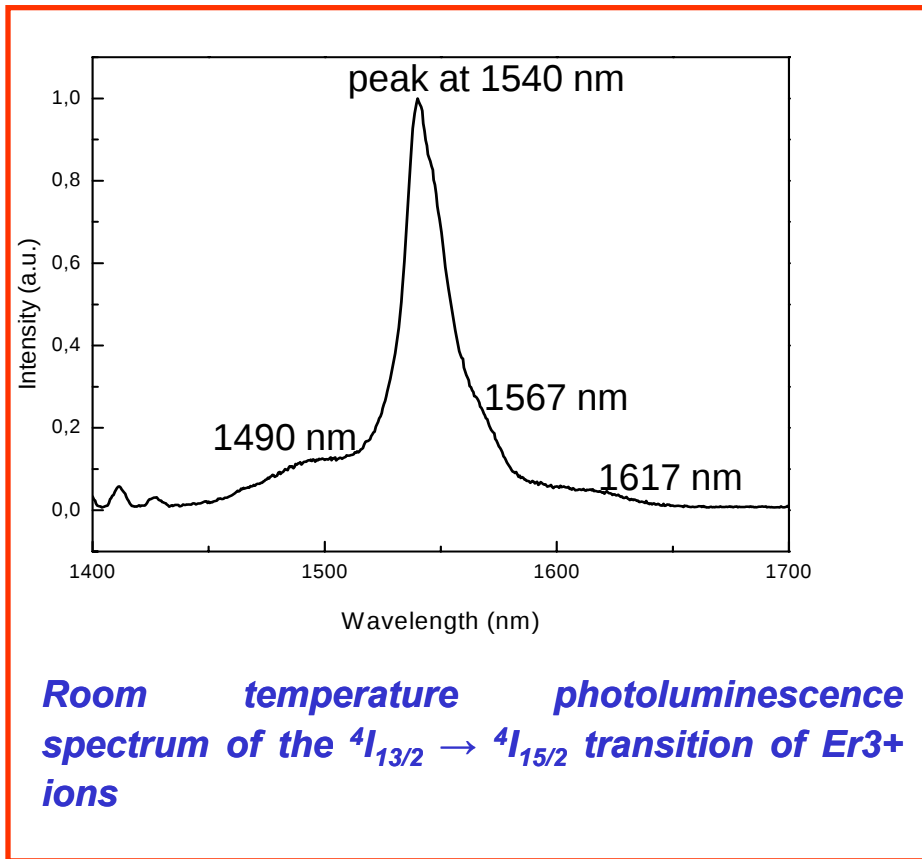
$$n_{eff}^2 = n_{silica}^2 \cdot f + n_{air}^2 \cdot (1 - f)$$



Filling Factor 23%

Max 26%

Luminescence at 1.5 μ m



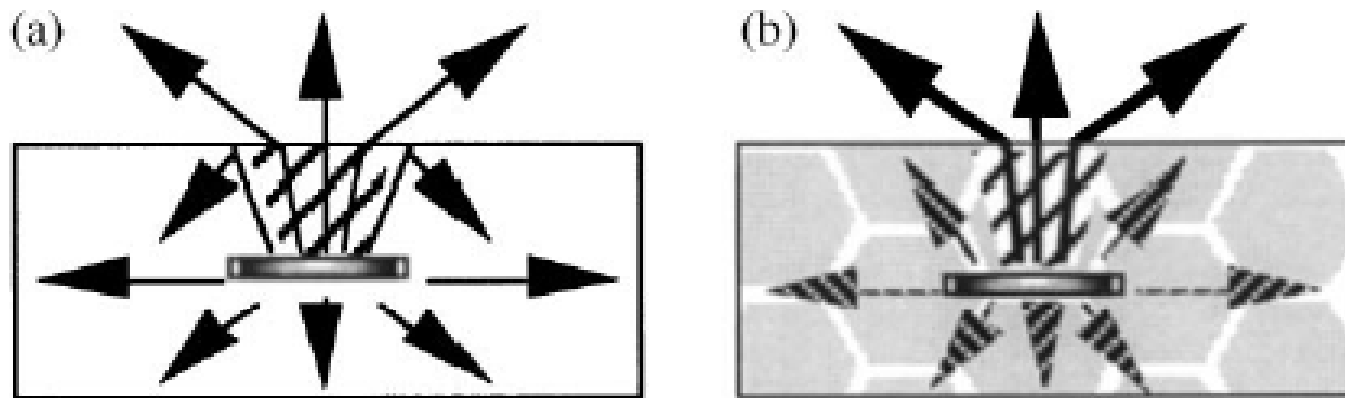
$$\eta = 90\%$$

C. Armellini, A. Chiappini, A. Chiasera, M. Ferrari, Y. Jestin, E. Moser, R. Retoux, G. Speranza, L. Minati, G. Nunzi Conti, S. Berneschi, I. Cacciari, S. Pelli, G.C. Righini.
“Fabrication and Characterization of Silica Opals”
Advances in Science and Technology Vol. 55 (2008) pp 118-126.

Photonic Crystals - fundamentals

Why are Photonic Materials special?

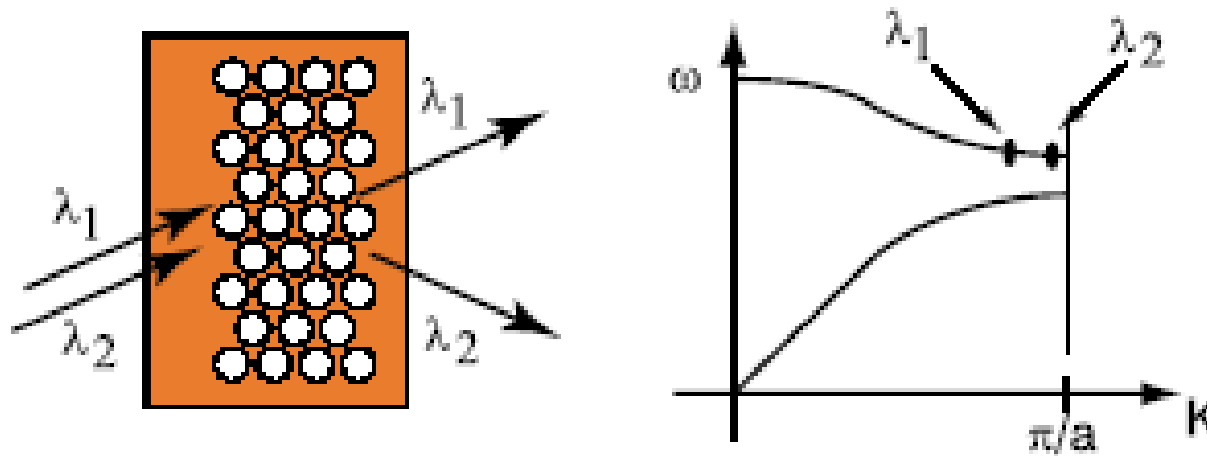
Improvement of LED efficiency.



(b) *microstructured emitter: light that would not radiate into the emission cone is suppressed by the photonic crystal, i.e. there are no modes for the emission to radiate into, so **all spontaneous emission is channeled into the “useful” modes of the device.** In principle, the external efficiency of such a device can reach the internal efficiency of the material of $> 90\%$.*

Photonic Crystals - application

Superprism effect

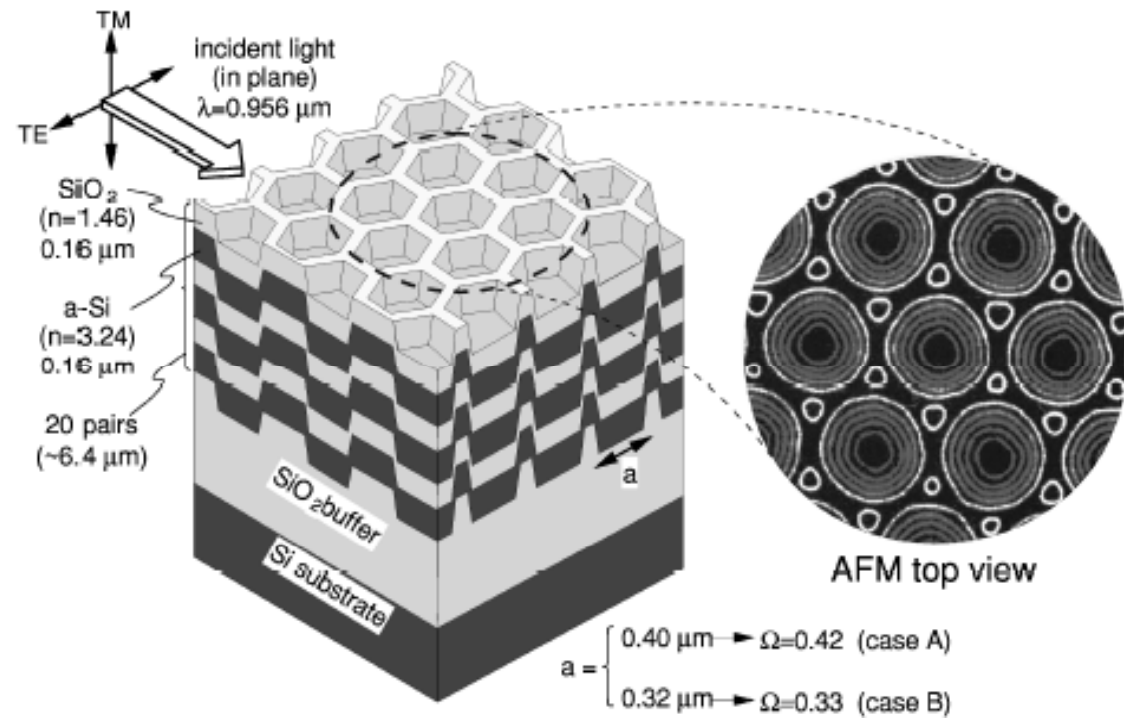


The figure shows how the dispersion properties of a photonic crystal can be used. Due to the high dispersion of wavelengths near the band edge, it is possible to separate the spectral contents of an incoming multi-wavelength signal. This particular effect is known as "superprism"

Photonic Crystals - application

Superprism effect

The self-organized 3D photonic crystal, with the stacking structure analogous to that of simple hexagonal graphite, employed to **demonstrate superprism effect** in the optical frequency region.

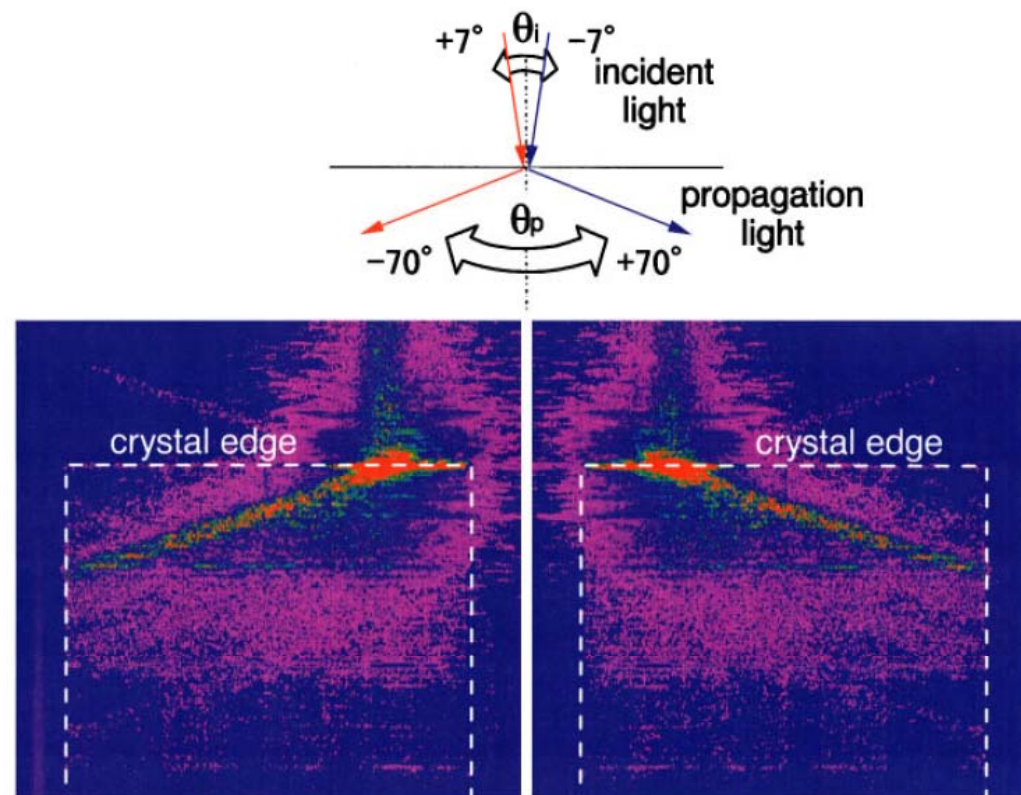


✓ H. Kosaka, T. Kawashima, A. Tomita, M. Notomi, T. Tamamura, T. Sato, and S. Kawakami “Superprism phenomena in photonic crystals”, Phys. Rev. B 58 (1998) pp. R10 096-R10 099.

Photonic Crystals - application

Superprism effect

Photographs showing light-beam swing inside the photonic crystal. The tilting angle of the incident light was slightly altered from $+7^\circ$ (left) to -7° (right). Both paths show negative bending. The incident light has a wavelength 956 nm ($\lambda/a=0.33$) with TM polarization. The crystal size is 0.5 mm x 0.5 mm.

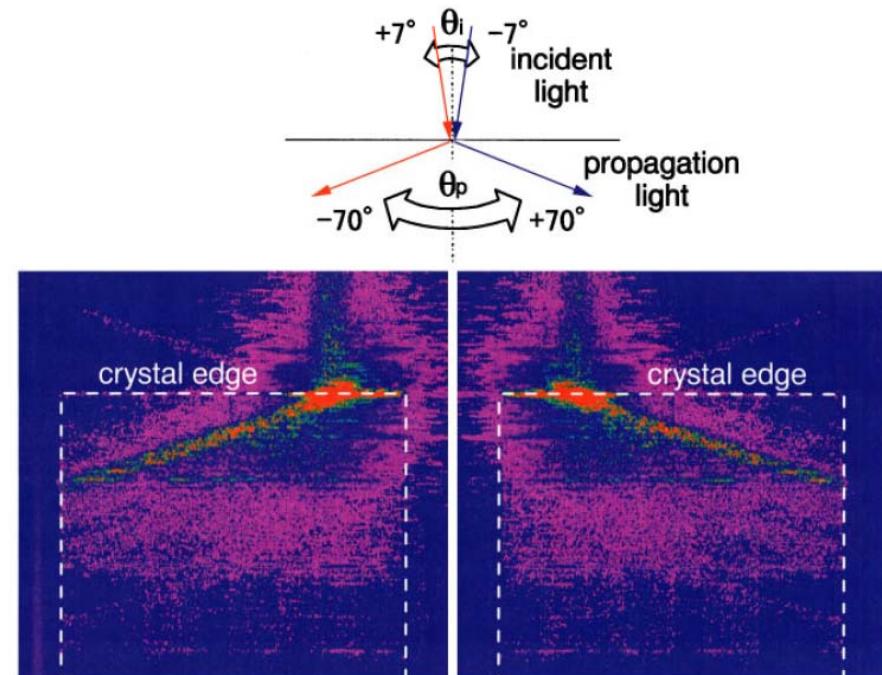


✓ H. Kosaka, T. Kawashima, A. Tomita, M. Notomi, T. Tamamura, T. Sato, and S. Kawakami "Superprism phenomena in photonic crystals", Phys. Rev. B 58 (1998) pp. R10 096-R10 099.

Photonic Crystals - application

Superprism effect

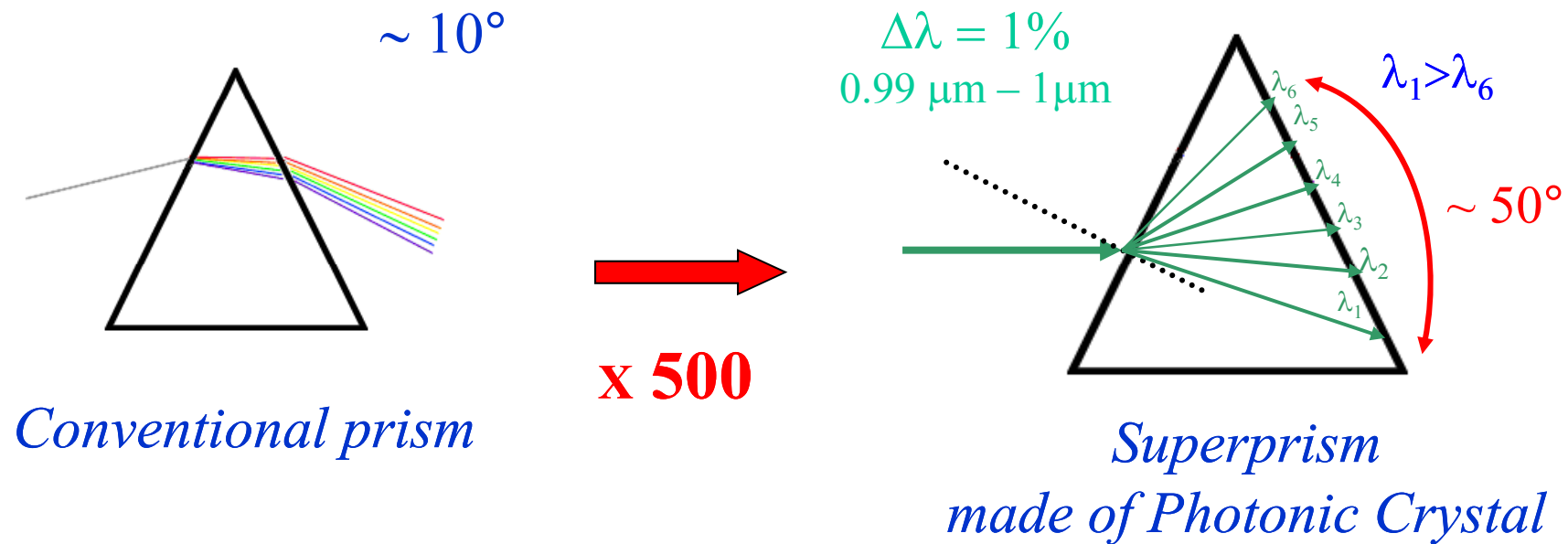
If Snell's law is applied without regard to the photonic band anisotropy, this phenomenon implies a *negative refractive index*.



✓ H. Kosaka, T. Kawashima, A. Tomita, M. Notomi, T. Tamamura, T. Sato, and S. Kawakami "Superprism phenomena in photonic crystals", Phys. Rev. B 58 (1998) pp. R10 096-R10 099.

Photonic Crystals - application

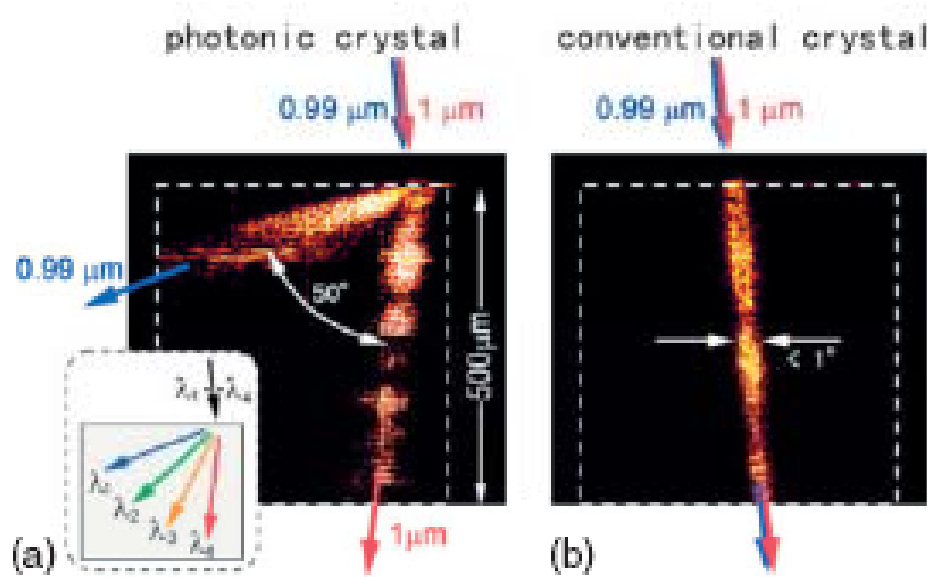
Superprism effect



✓ H. Kosaka, T. Kawashima, A. Tomita, M. Notomi, T. Tamamura, T. Sato, and S. Kawakami “Superprism phenomena Superprism Phenomena in Photonic Crystals: Toward Microscale Lightwave Circuits”, Journal of Lightwave Technology, 17 (1999) pp. 2032-2038.

Photonic Crystals - application

Superprism effect



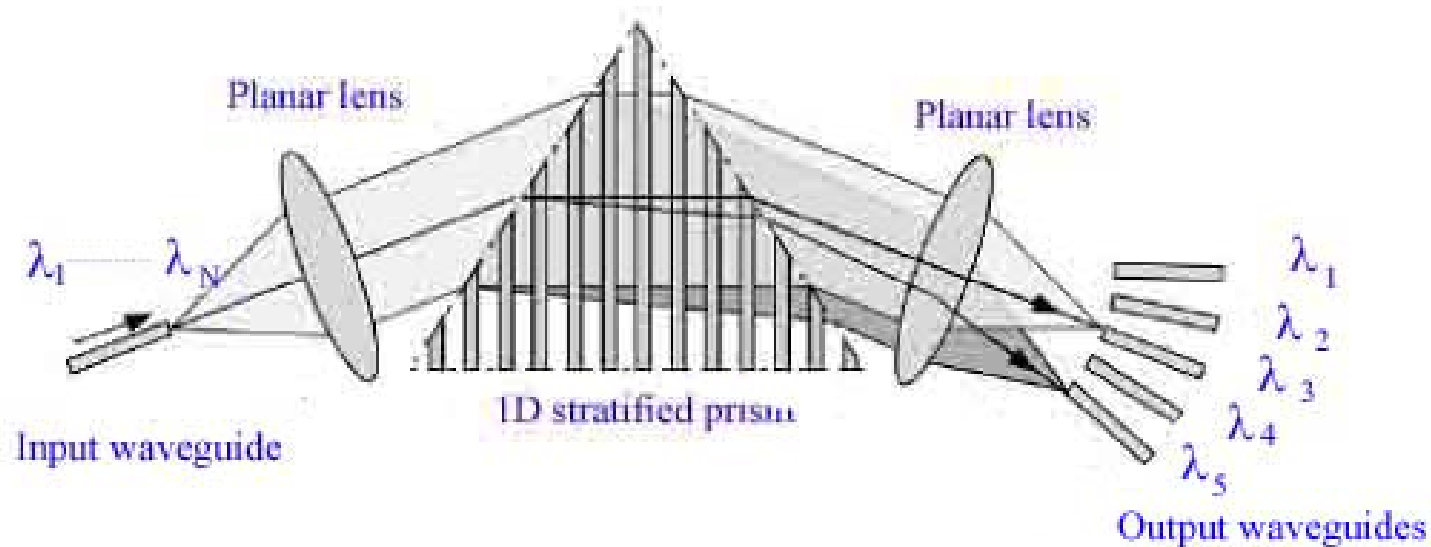
✓ H. Kosaka, T. Kawashima, A. Tomita, M. Notomi, T. Tamamura, T. Sato, and S. Kawakami "Superprism phenomena Superprism Phenomena in Photonic Crystals: Toward Microscale Lightwave Circuits", Journal of Lightwave Technology, 17 (1999) pp. 2032-2038.

(a) light paths inside a PC with incident light of $0.99\text{-}\mu\text{m}$ and $1.0\text{-}\mu\text{m}$ wavelengths. A large swing reaching 50° was achieved with a slight wavelength change of 1%. The incident light was polarized in the TM mode and tilted at 15° from normal to the crystal edge and

(b) light propagation in a conventional Si crystal under the same condition as (a). The two beams with different wavelengths traced almost the same paths.

Photonic Crystals - application

Superprism effect: 1D superprism (plan view)



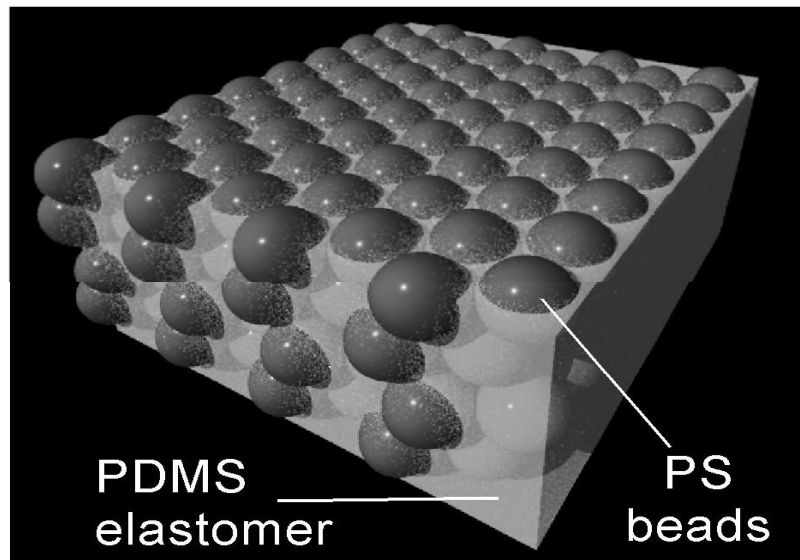
✓ A. Bakhtazad, A.G. Kirk, “**Superprism effect with planar 1-D photonic crystal**”, Proceedings of the SPIE, Volume 5360, pp. 364-372 (2004)

Strain Sensor

Material: *Changing the structural color*

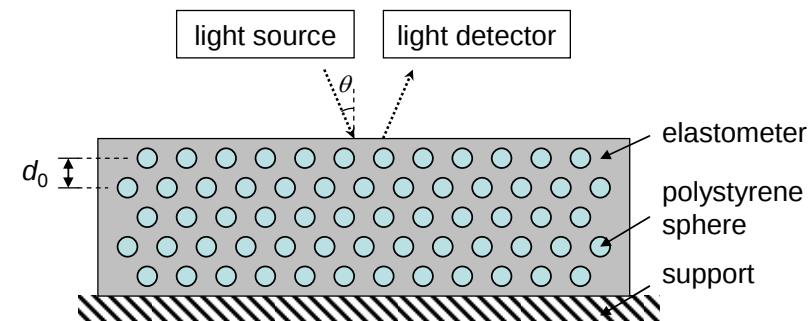
external stimulus

naked eyes

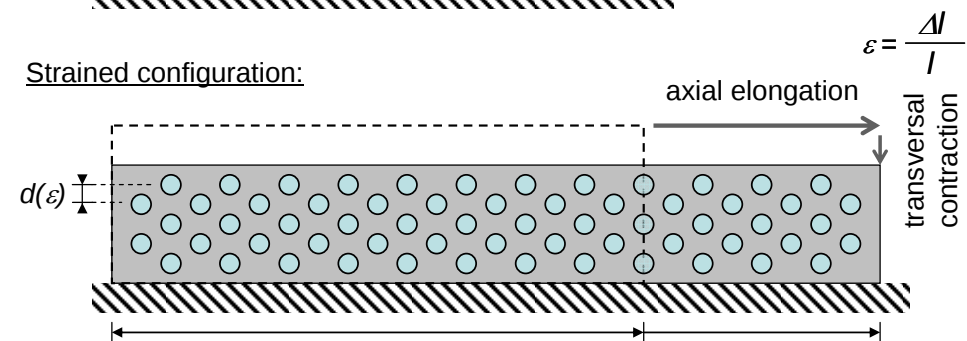


How it works?

Initial configuration:



Strained configuration:

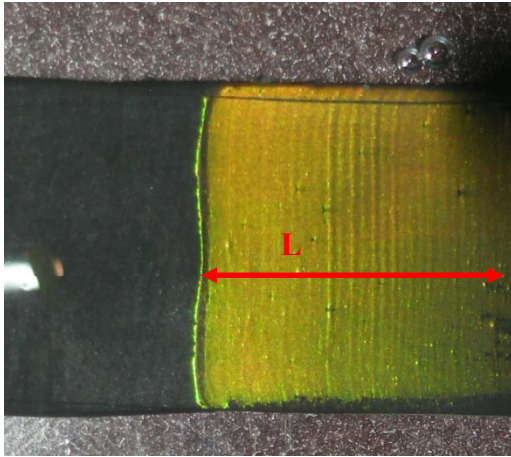


$$\lambda = 2 \cdot d_{111} \cdot n_{eff}$$

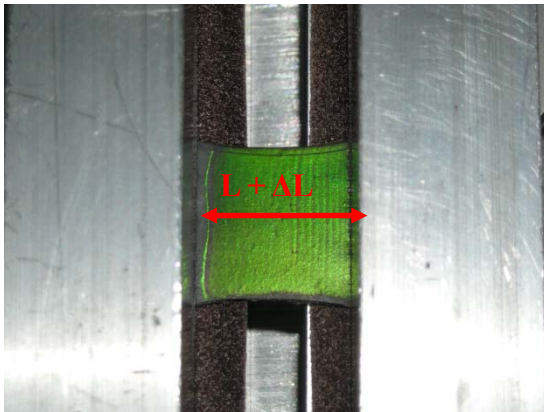
Blue shift of the wavelenght of the diffraction peak as a function of the *applied strain*

Experimental validation

Initial

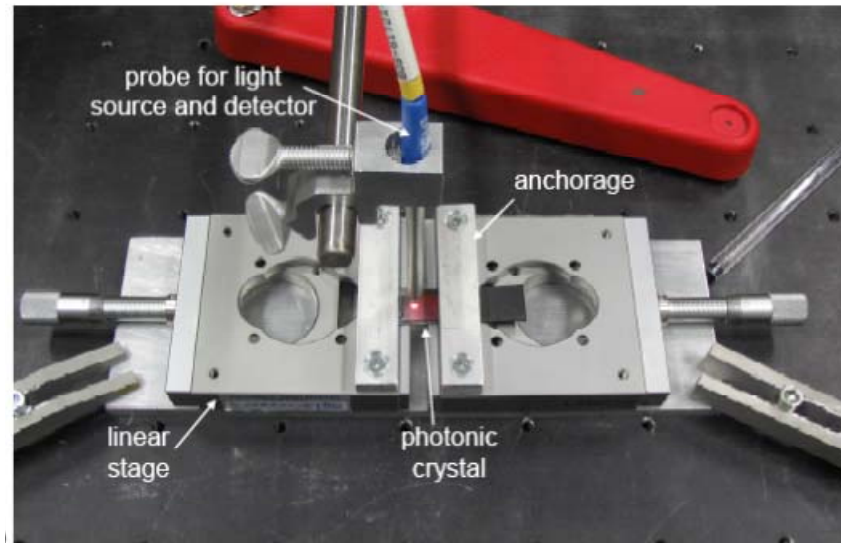
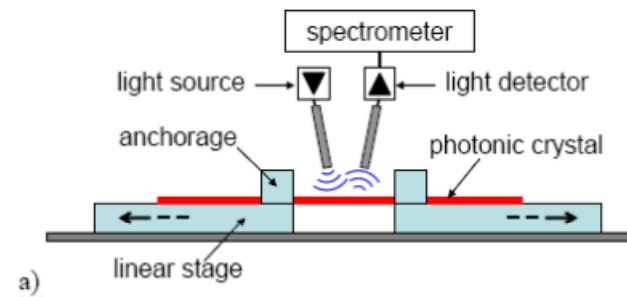


Applied strain



Strain Sensor

Experimental set-up

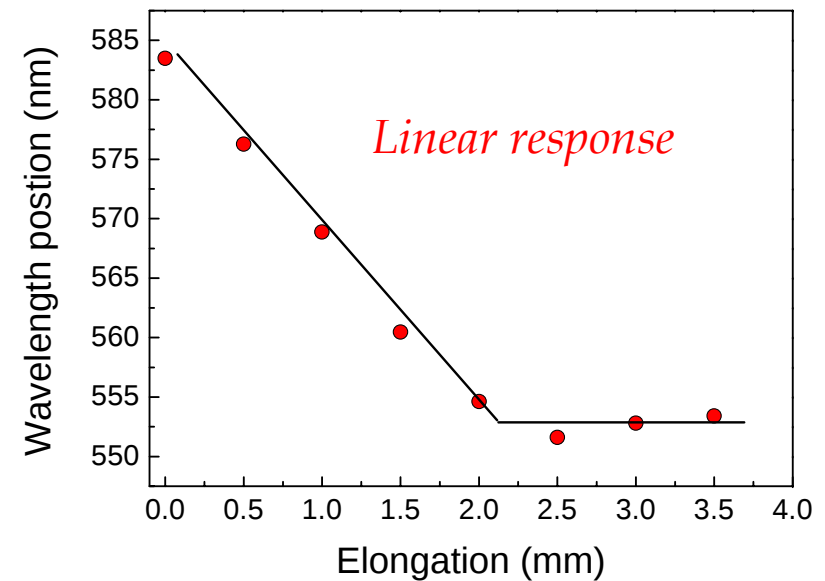
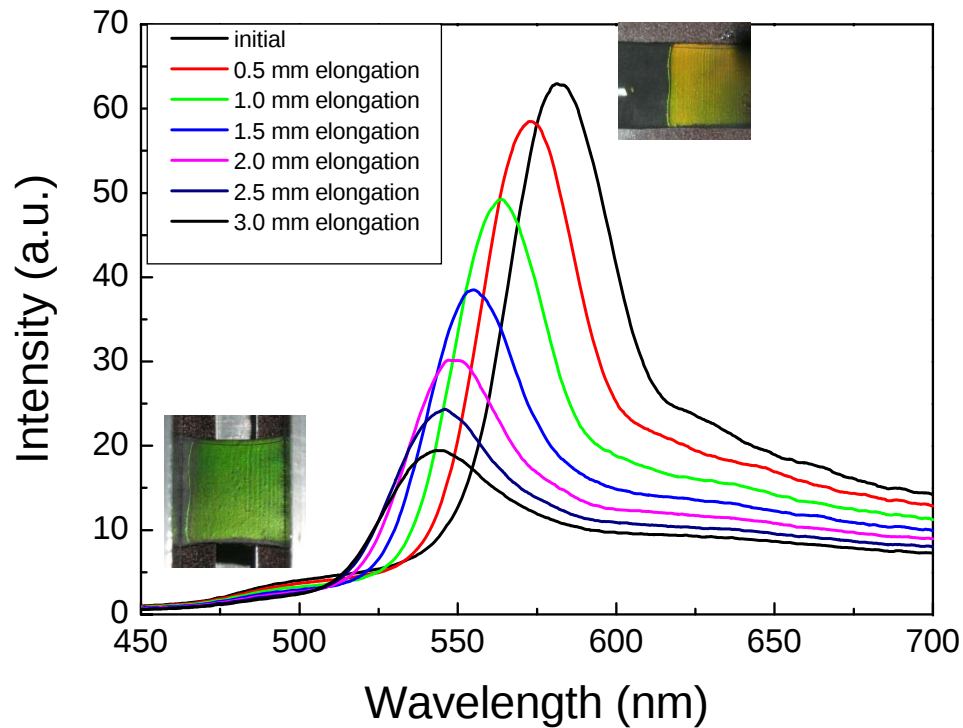


[D. Zonta, A. Chiappini, A. Chiasera, M. Ferrari, M. Pozzi, L. Battisti, M. Benedetti, Proc. of SPIE 7292 \(2009\) pp. 729215-1 - 729215-10.](#)

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Strain Sensor

Response Characterisation

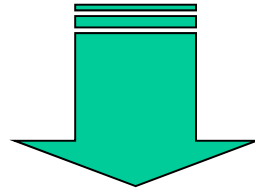


Peak position *blue shifts* 583 to 550 nm

[D. Zonta, A. Chiappini, A. Chiasera, M. Ferrari, M. Pozzi, L. Battisti, M. Benedetti, Proc. of SPIE 7292 \(2009\) pp. 729215-1 - 729215-10.](#)

Possible applications

– *SERS substrate*



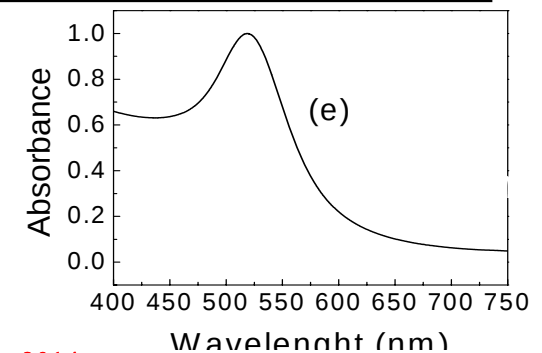
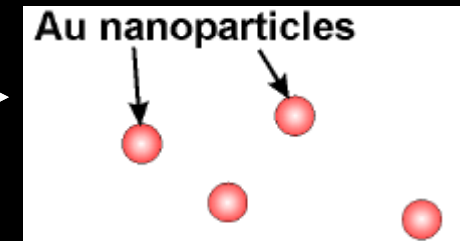
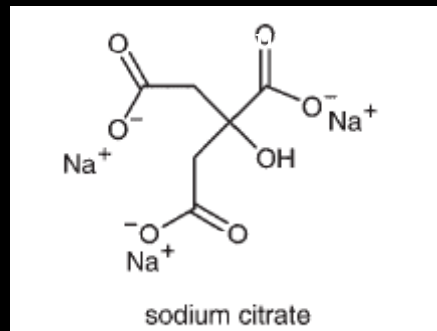
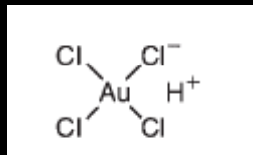
Metallo – dielectric structures

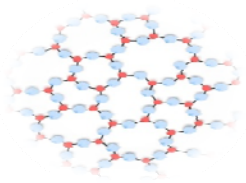
Gold nanoparticles

How to prepare?

Turkevich Method (1951)

Reduction of Gold ion by citrate ions





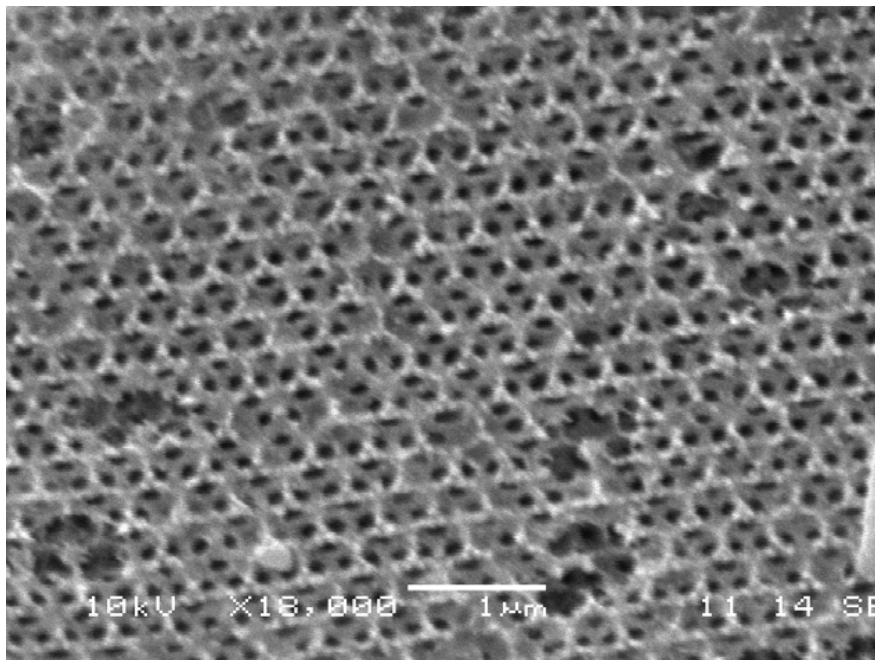
Structural Properties

Inverse ordered structure is still present after immersion process

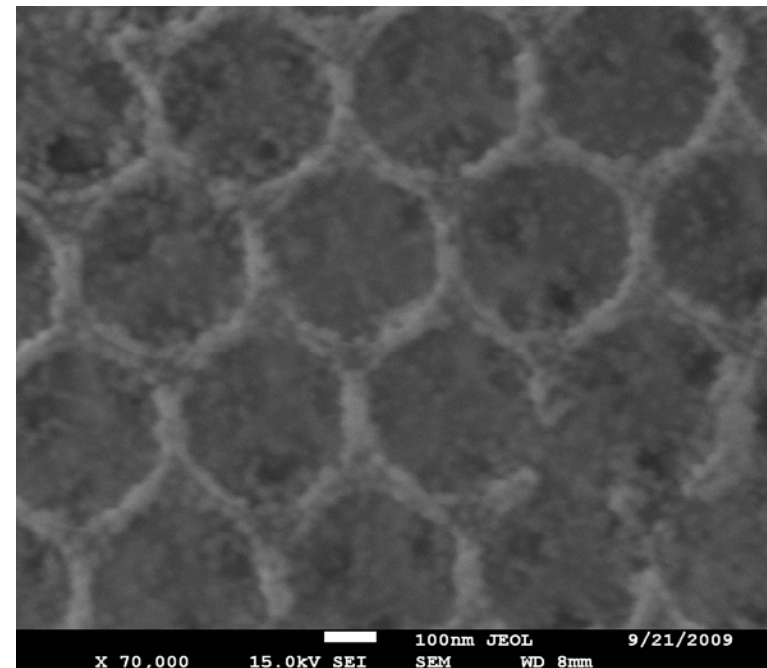


hollow regions of the air spheres

inner dark holes

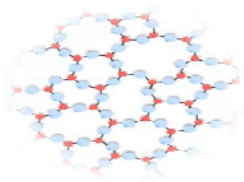


After 2 h immersion time



After 10 h immersion time

Au Nps are attached/immobilized on the network of the ISO system

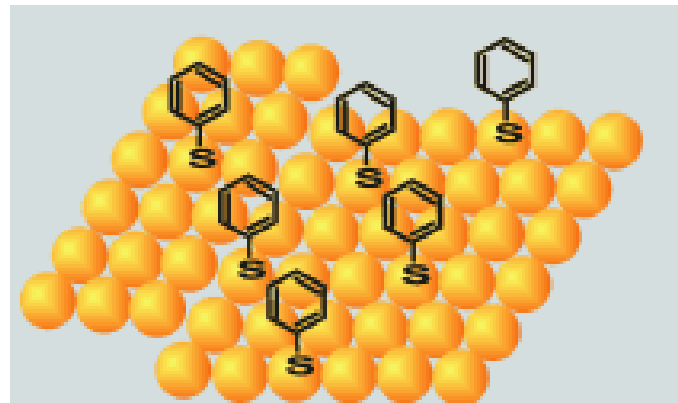
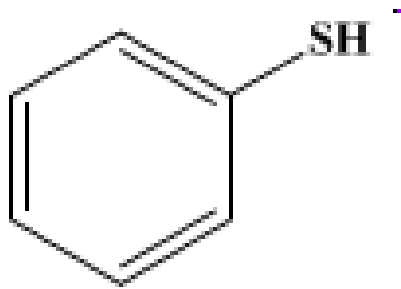


Raman Signal Enhancement

SERS substrate

benzenethiol (BT) as a probe molecule

$1 \times 10^{-3} M$



using a 10 s accumulation time, incidence power 100 μW .

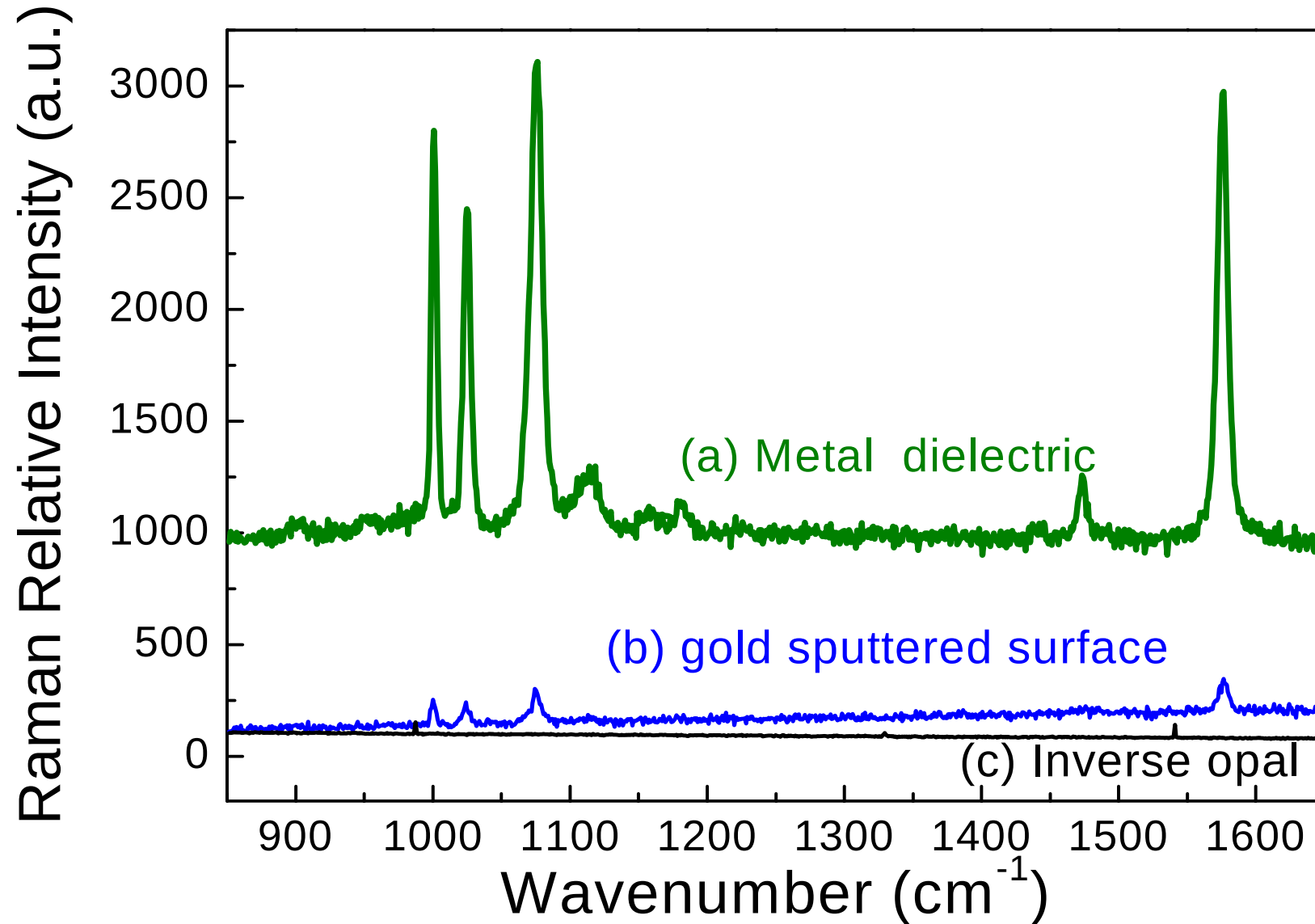
to evaluate the efficiency of the MDCS

we have compared the Raman signal obtained for these structures with those collected :

b) on a gold film (GF) deposited by sputtering

c) on ISO spotted with a 5 μl drop of pure BT and left to dry out (ISO_BT).

Raman Signal Enhancement





Scienze fisiche e tecnologie della materia

CSMFO Lab. <http://www.tn.ifn.cnr.it/>



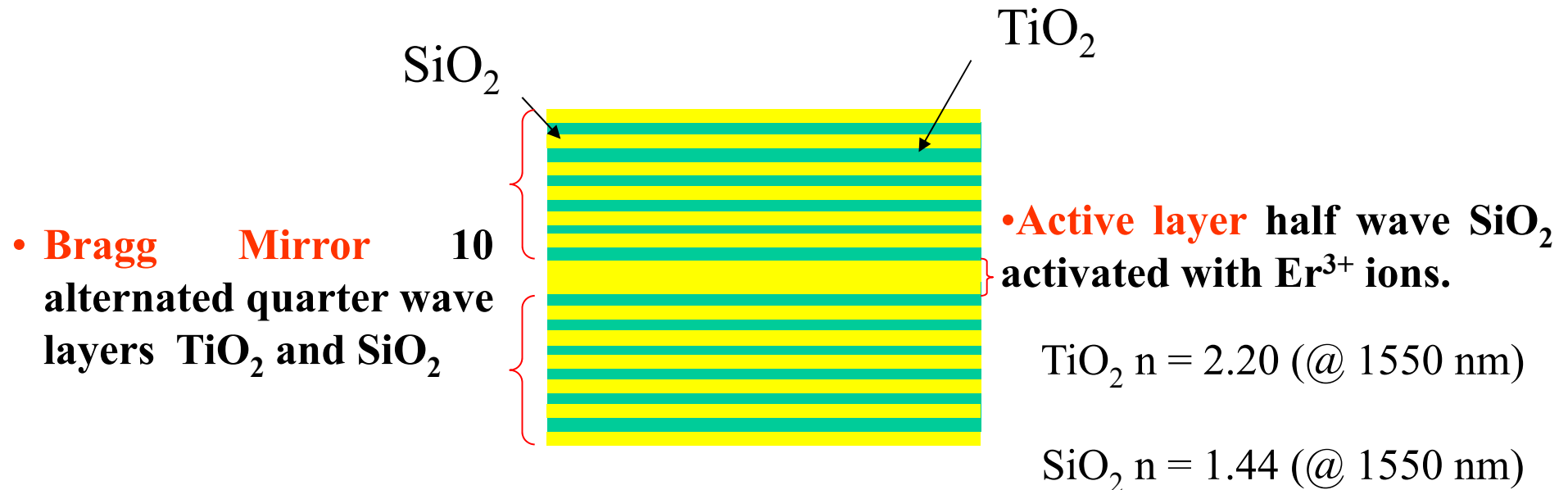
Consiglio Nazionale delle Ricerche
Istituto di Fotonica e Nanotecnologie - Unità di Trento



PHOTONIC CRYSTALS

HIGH QUALITY FACTOR 1-D Er^{3+} - ACTIVATED DIELECTRIC MICROCAVITY FABRICATED BY RF-SPUTTERING

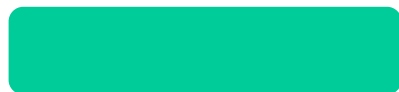
Fabrication Protocol – Target composition



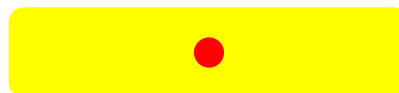
SiO_2 Target



TiO_2 Target



$\text{SiO}_2 + \text{Er}$ Target

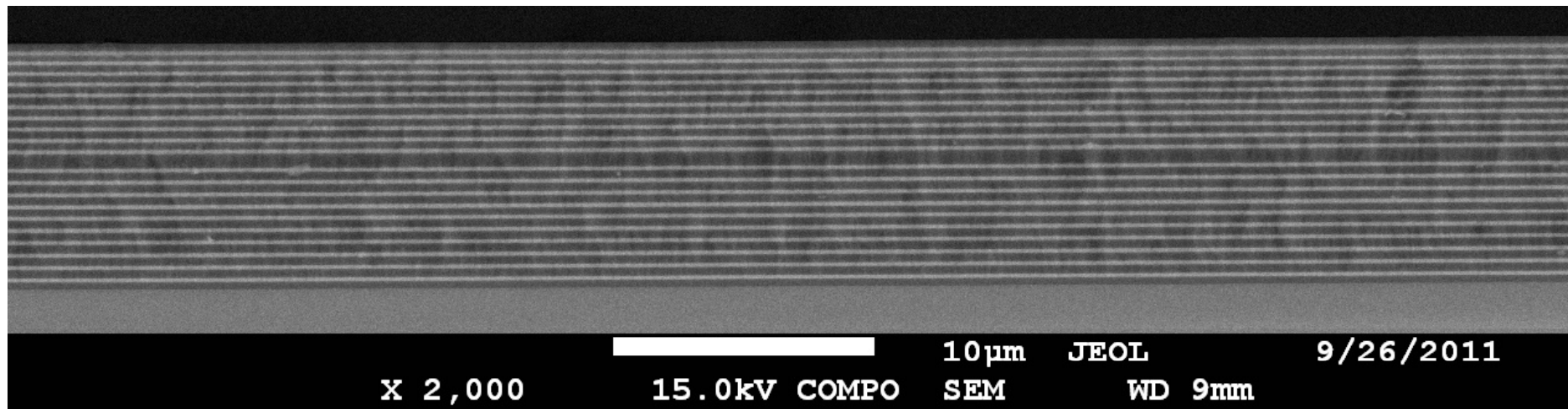


Substrate: v- SiO_2 , dim. 7 x 3cm

Deposition is performed by **sputtering** alternatively the targets of silica and titania. The depositions were tailored to reach the appropriate thicknesses to obtain a **cavity resonance centred at 1.7 μm**

Development and optical and spectroscopic diagnostic of photonic materials and structures

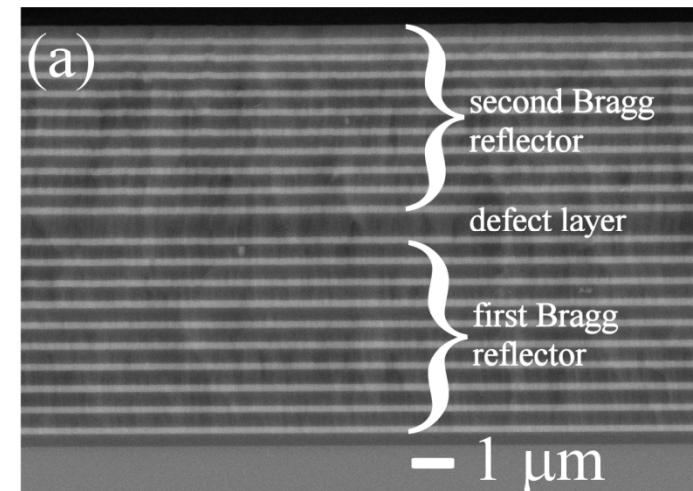
1-D microcavities fabricated by RF-sputtering



Bragg Mirror: 20 alternated quarter wave layers TiO_2 (170 nm) and SiO_2 (320 nm).

Active layer: half wave (640 nm) SiO_2 activated with 0.2 mol % of Er^{3+} .

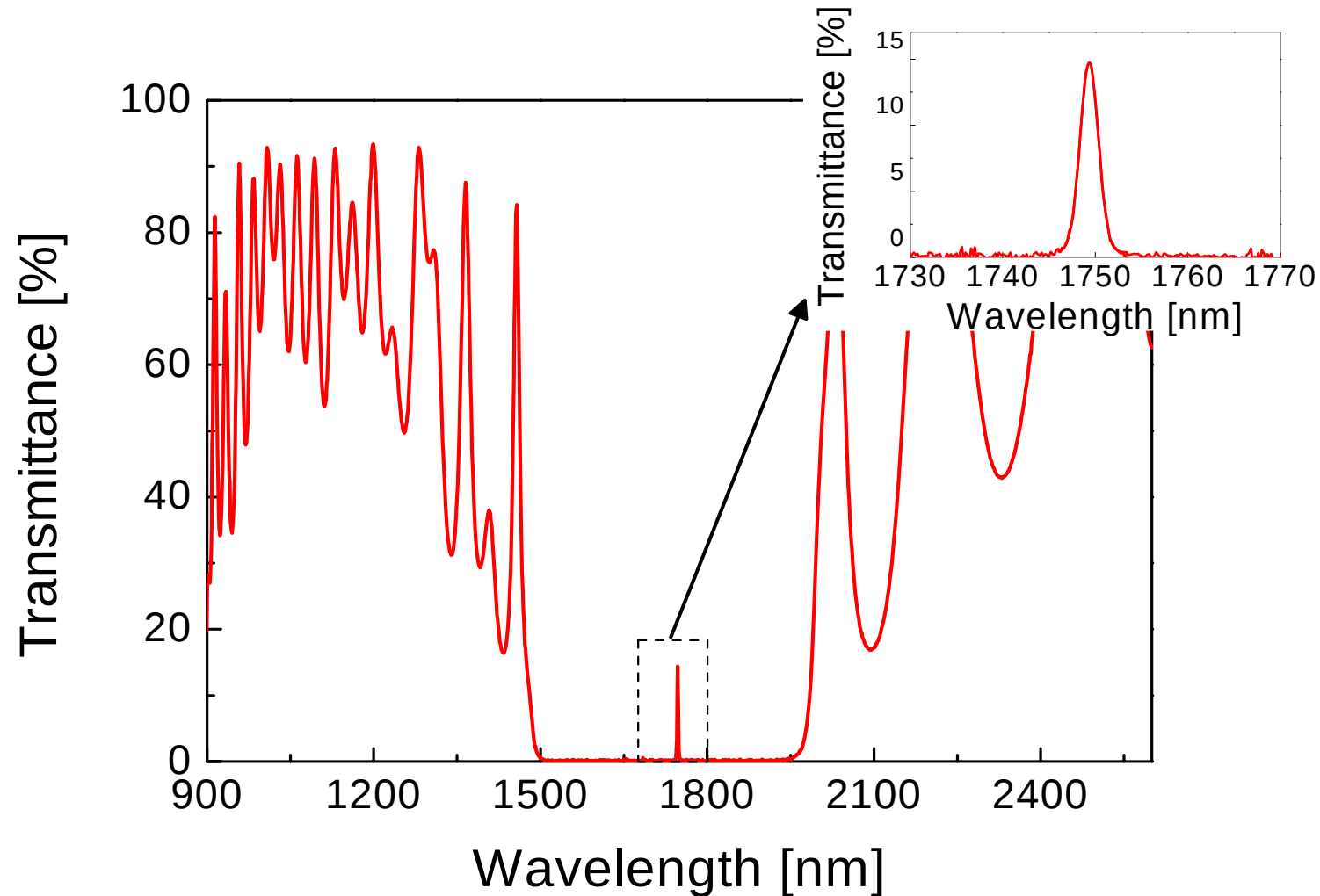
The dark regions corresponds to SiO_2 and the white regions corresponds to TiO_2



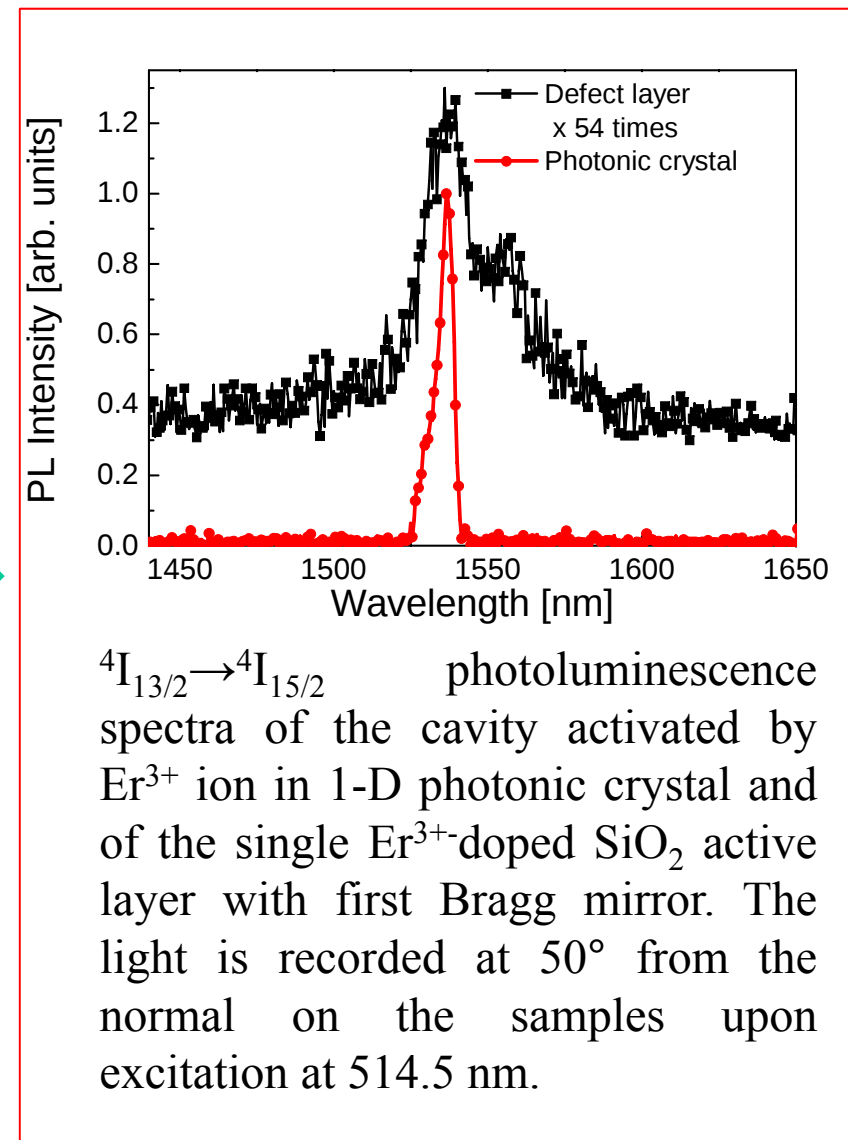
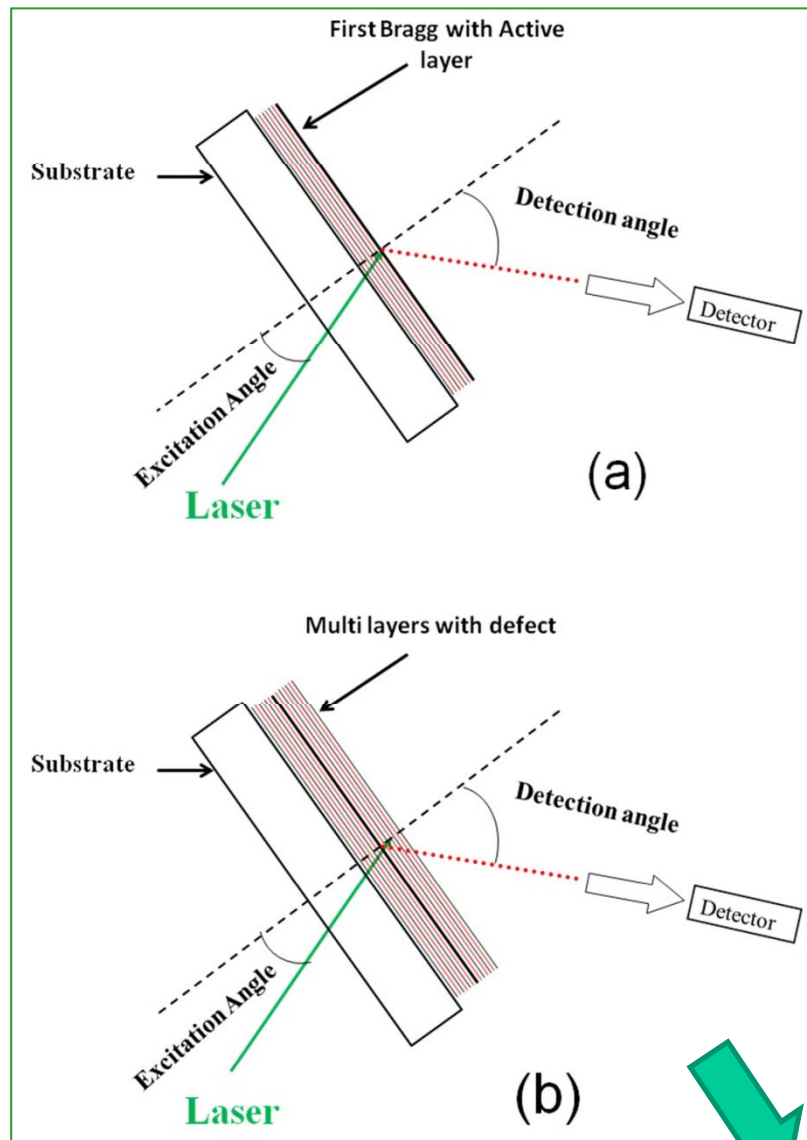
S. Valligatla, A. Chiasera, S. Varas, N. Bazzanella, D.N. Rao, G.C. Righini, M. Ferrari, "High quality factor 1-D Er^{3+} -activated dielectric microcavity fabricated by RF-sputtering", Optics Express, Vol. 20 Issue 19, pp.21214-21222 (2012)

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Transmission measurements



Transmittance spectrum of the cavity with two Bragg reflectors. The stop band range from 1490 to 1980 nm. The cavity resonance corresponds to the sharp maximum centred at 1.749 μm . The FWHM of the resonance is 1.97 nm that correspond to a quality **Q factor of 890**.



Schematics of the excitation and detection geometries employed for a reliable assessment of the influence of the cavity on the 1.5 μm emission band of Er^{3+} ion.

Fig. (a): configuration employed for the Er^{3+} -activated reference sample:

Fig. (b): configuration employed for the Er^{3+} -activated 1-D microcavity.



Scienze fisiche e tecnologie della materia

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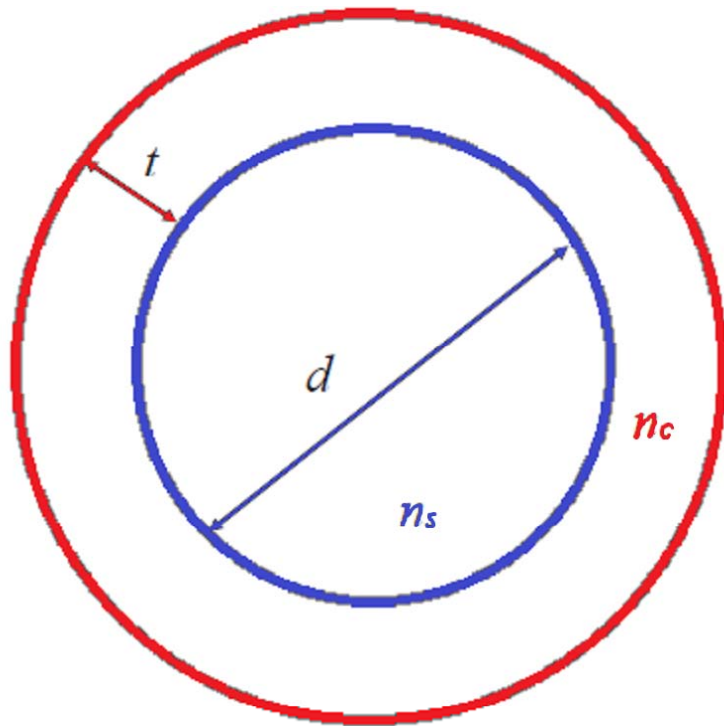
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SPHERICAL MICRORESONATORS

WHISPERING GALLERY MODES LASER

Spherical microresonators WGMs



Microsphere of diameter d and refractive index n_s coated by a film of thickness t and refractive index n_c .

G.C. Righini, Y. Dumeige, P. Féron, M. Ferrari, G. Nunzi Conti, D. Ristic, S. Soria., “Whispering gallery mode microresonators: fundamentals and applications”, *Rivista del Nuovo Cimento* 34 (2011) pp. 435-488.

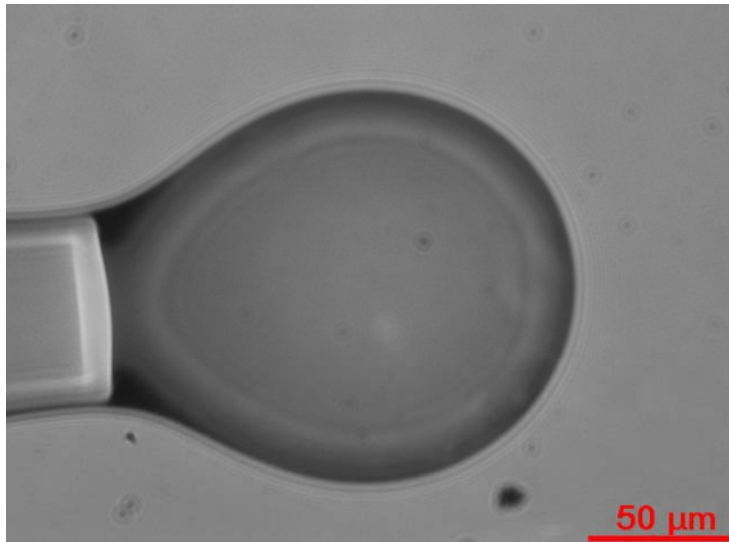
D. Ristić, A. Rasoloniaina, A. Chiappini, P. Féron, S. Pelli, G. Nunzi Conti, M. Ivanda, G.C. Righini, G. Cibieli, and M. Ferrari “About the role of phase matching between a coated microsphere and a tapered fiber: experimental study” *Optics Express*, 21 (2013) pp 20954-20963

Glass coated microspheres

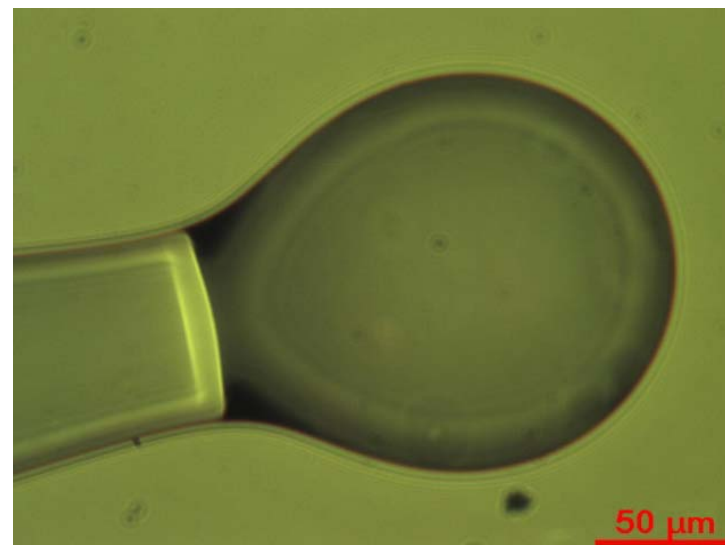
Silica microspheres fabricated by melting a SMF-28 rod. This glass presents a very low concentration of impurities (<0.3 ppm) and OH groups (<0.2 ppm), the attenuation coefficient is very low ($\cong 0.2$ dB/km at 1550 nm) The diameters and ellipticity are also reported

	F1	F2	F3	F4	F5	F6	F7	F8	F9
d/ μm [$\pm 5 \mu\text{m}$]	155	135	140	140	140	145	130	155	145
Ellipticity [$\pm 1\%$]	4%	7%	4%	5%	7%	5%	4%	3%	3%

Microscope images of the F5 microsphere before and after coating



Before coating



After coating

Glass coated microspheres

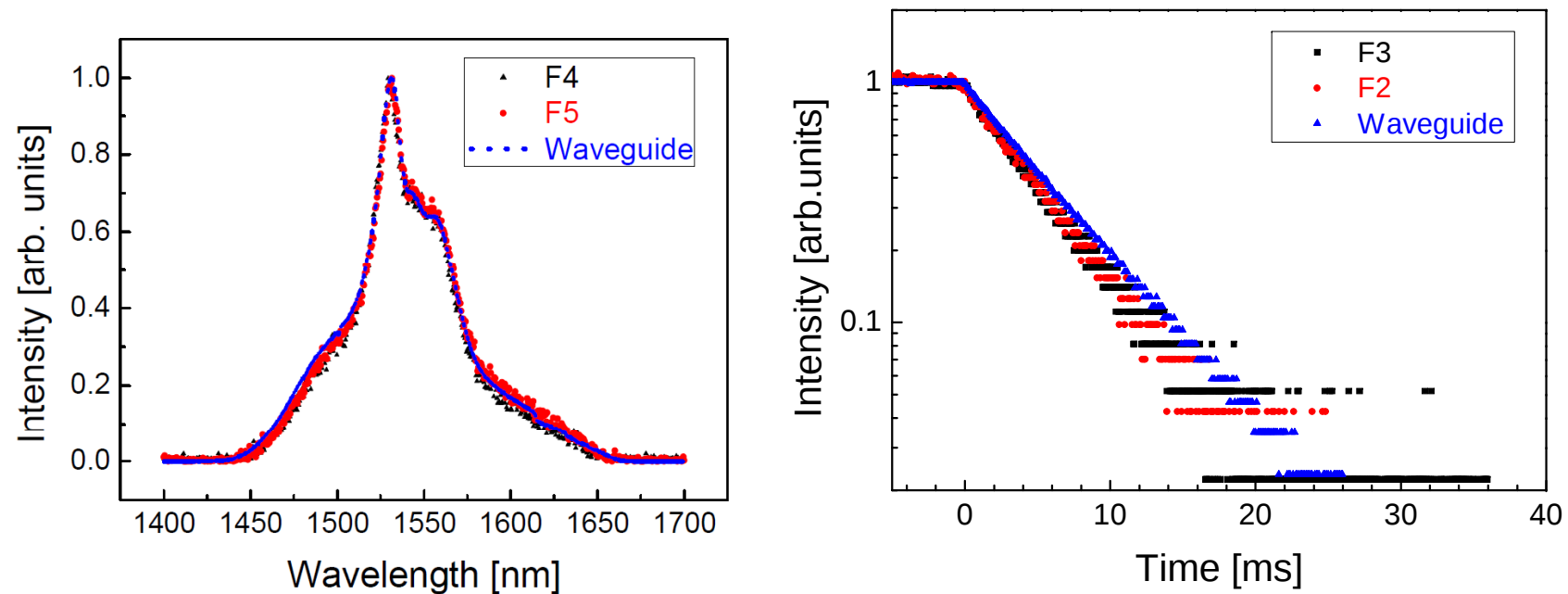
A waveguide was prepared as reference using the same sol and the same dip coating technique as for the microsphere in order to estimate the thickness and the refractive index of the coating

Wavelength [nm]	Mode polarization	number of modes	refractive index	thickness [μm]
632.8	TE	3	1.6165 ± 0.0003	1.25 ± 0.01
543.5	TE	3	1.6215 ± 0.0003	1.25 ± 0.01

Thickness, refractive index, and modal parameters of the planar waveguide used as reference for the sol gel coating of the microsphere. Values are obtained by m-line measurement. The film composition was $70\text{SiO}_2\text{-}30\text{HfO}_2$ activated with 0.3 mol% Er^{3+} .

Glass coated microspheres

Room temperature photoluminescence spectra and decay curves of the $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition of Er^{3+} ions for the sol gel coated spherical microresonators and the planar waveguide used as reference. The film composition was $70\text{SiO}_2\text{-}30\text{HfO}_2$ activated with 0.3 mo% Er^{3+} .

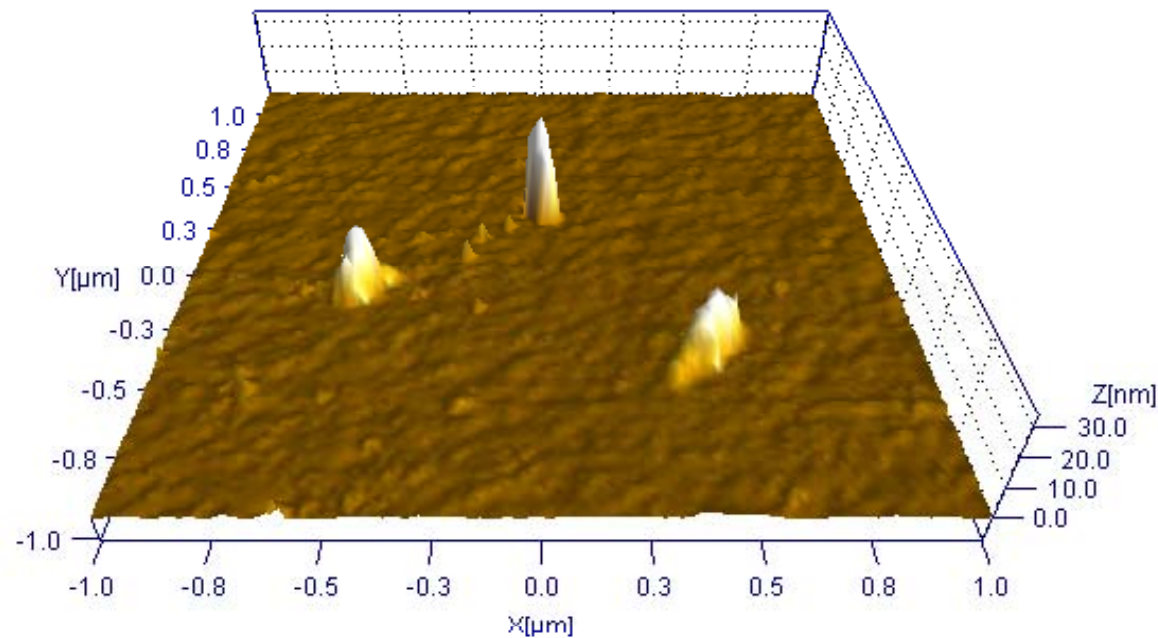


All the photoluminescence spectra showed that the deposited films are amorphous, and that there is no difference between the shape of the spectra of different microspheres and the waveguide, indicating that the coating procedure is reproducible.

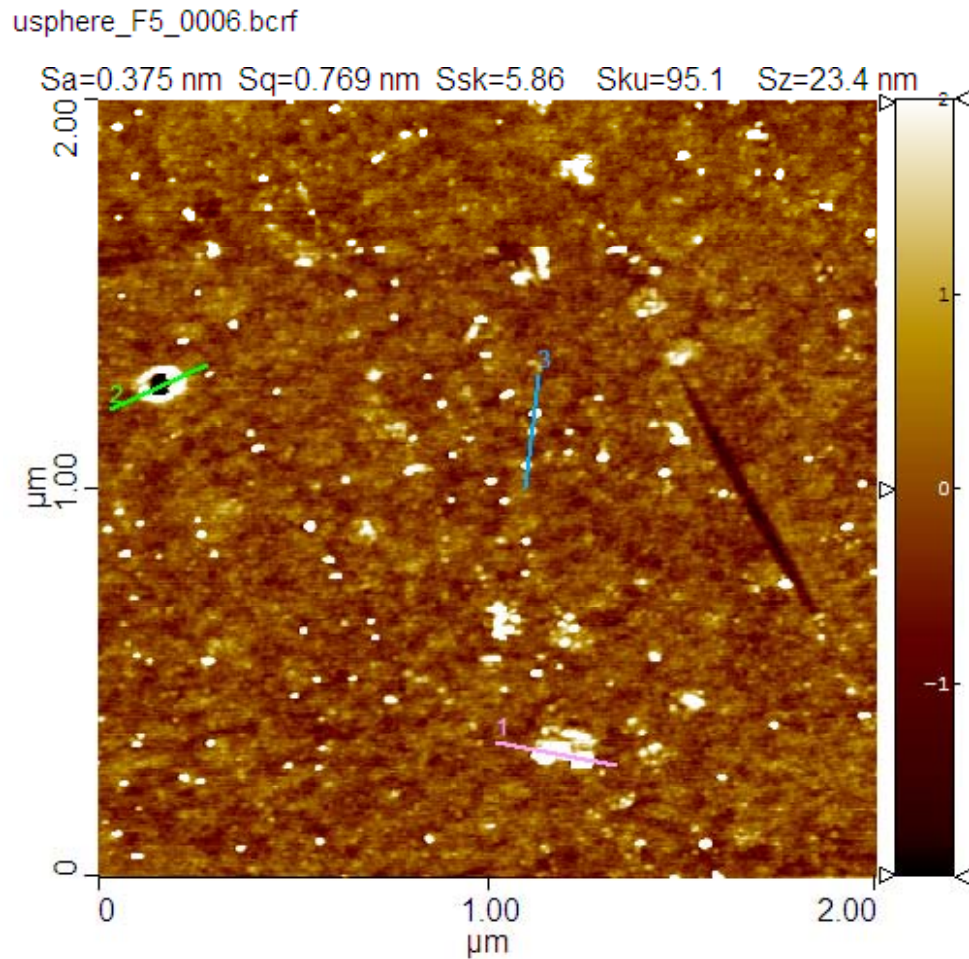
Glass coated microspheres – AFM

Two quantities were measured:

- The surface roughness
- The number of defects/ μm^2

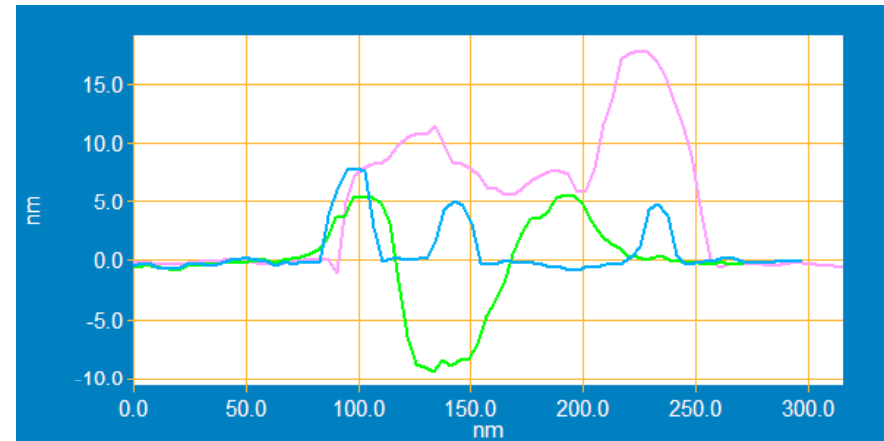


Glass coated microspheres – Defects



Defect types

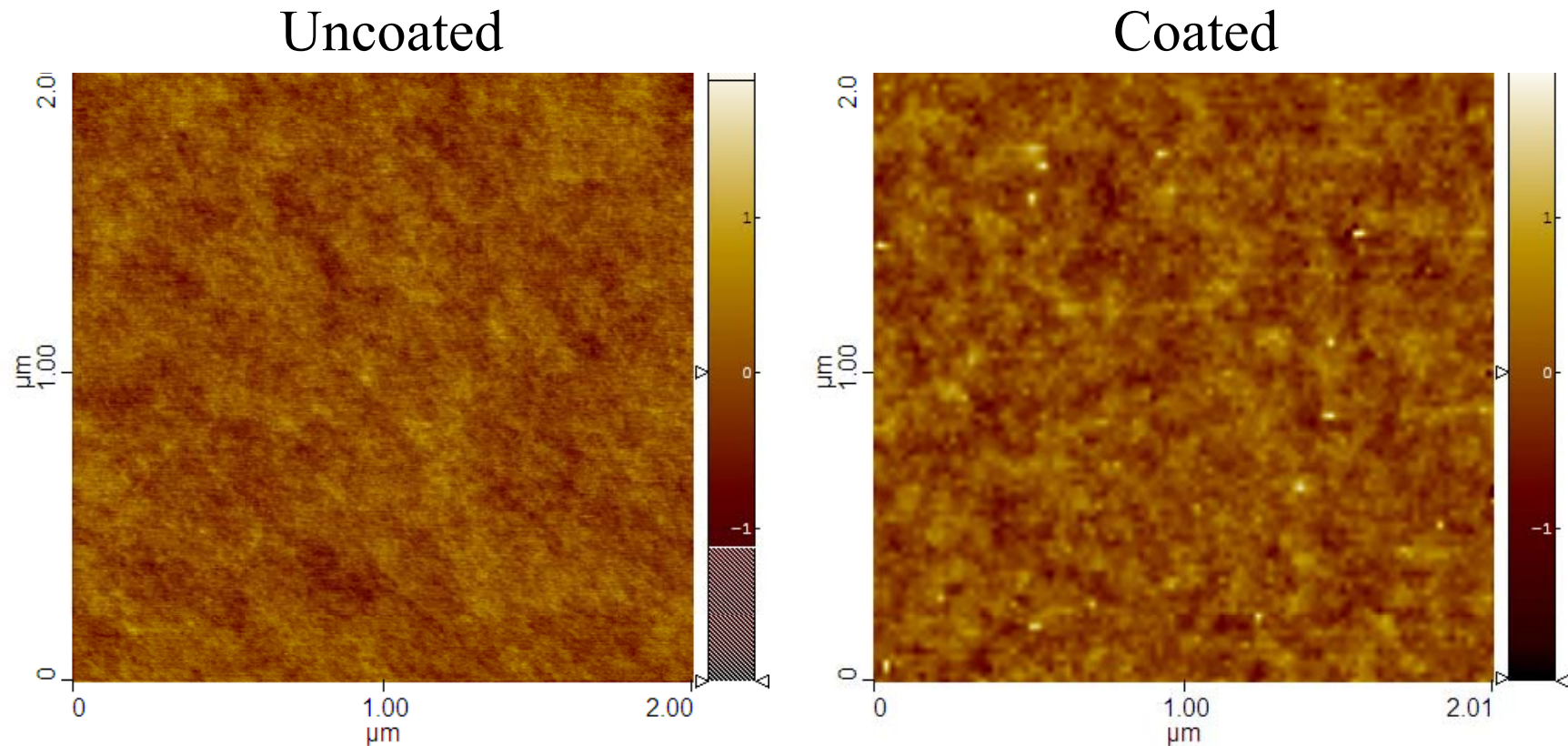
- small defects (around 5 nm in height)
- Big defects (>15 nm in height)
- Holes (10 nm in depth)



A low number of defects is needed to achieve high Q-factors

Glass coated microspheres – Roughness

For the surface roughness measurements the areas with no defects were chosen



Glass coated microspheres – Surface assessment

Roughness calculated on $2 \times 2 \mu\text{m}^2$ areas

Defects counted on a

➤ $20 \times 20 \mu\text{m}^2$ image

➤ $10 \times 7 \mu\text{m}^2$ image

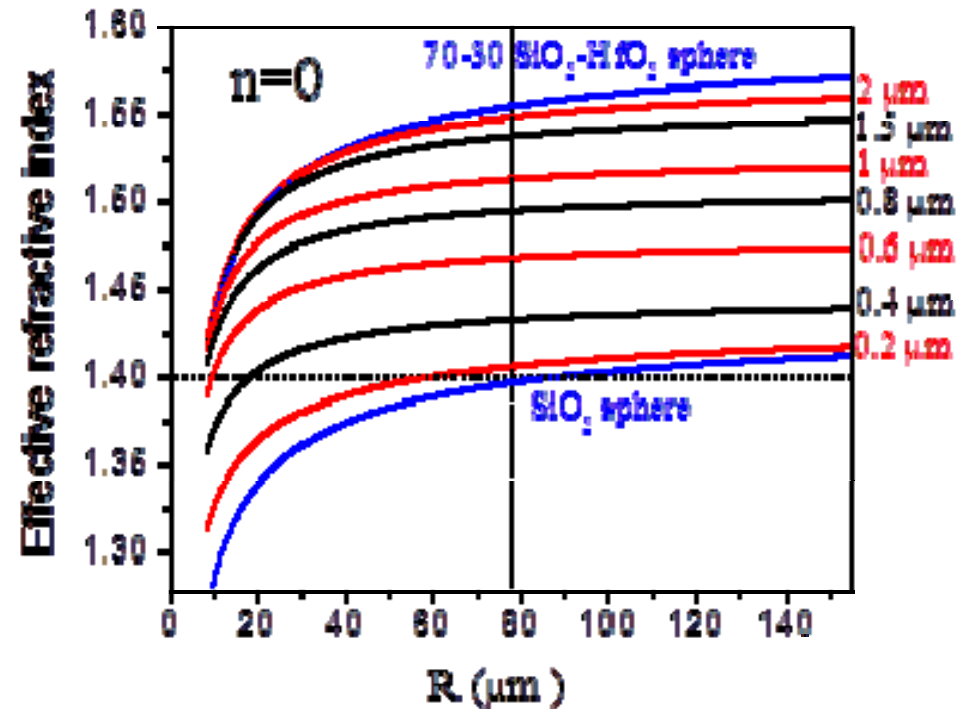
A 4th order bidimensional subtraction plus an histogram line-by-line levelling has been applied before computing the roughness

Defect density definition: (objects with $Z_{\text{max}} > 2 \text{ nm}$) numb./ μm^2

Microsphere F5	Average absolute value surface roughness [nm]	Root mean square surface roughness [nm]	Defect density [numb./ μm^2]
Uncoated	0.191 ± 0.004	0.242 ± 0.006	2.00×10^{-2}
Coated	0.29 ± 0.04	0.48 ± 0.19	6.24

Glass coated microspheres – Near Field

Effect of the coating thickness on the coupling efficiency

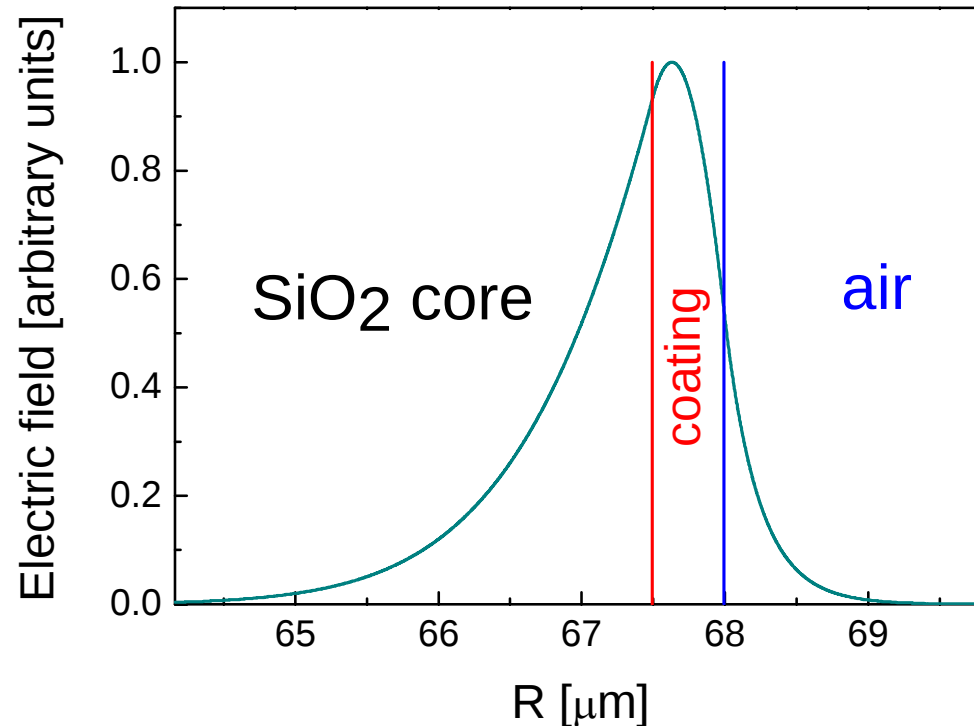


The effective refractive index of the 70 SiO_2 -30 HfO_2 coated microsphere as a function of the radius of the silica sphere for the fundamental equatorial ($n=0$; $l=|m|=0$) TE mode at 1560 nm. The refractive index of the sphere is 1.44 and of the coating 1.6. The coating thicknesses, given in μm , are also reported in the figure. The blue lines correspond to the border cases of no coating (blank sphere) or of very thick coating (bulk sphere with $n=1.6$). The horizontal dashed line corresponds to the effective refractive index of the propagation mode of a silica fiber taper with a waist of 3 μm . The vertical dashed line corresponds to a silica sphere diameter of 155 μm which is the diameter of the sphere coated in our experiment.

Optics Express, 21 (2013) pp 20954-20963 “About the role of phase matching between a coated microsphere and a tapered fiber: experimental study “

Glass coated microspheres – Near Field

Effect of the coating thickness on the coupling efficiency



$R = 68 \mu\text{m}$

$d = 0.5 \mu\text{m}$

$l = 400$

$N = 0$

We need:

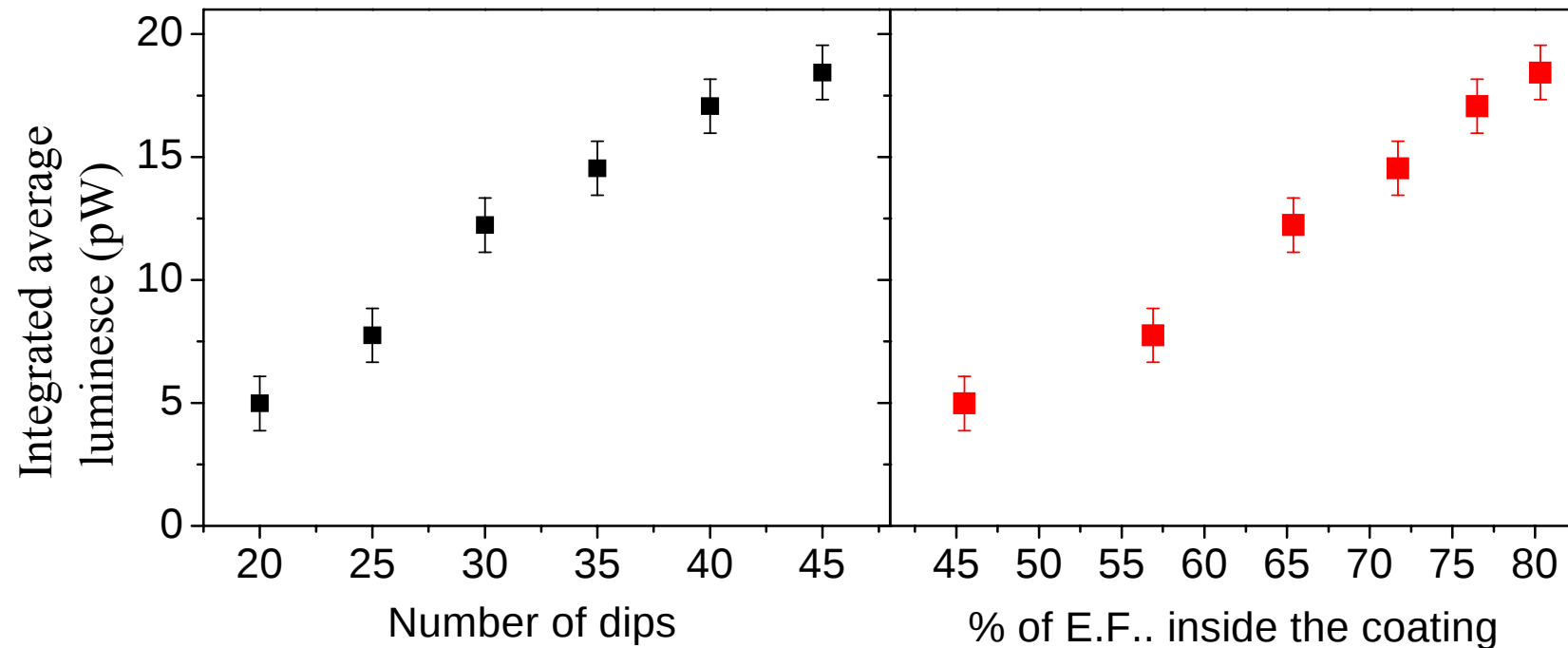
➤ high amount of E. field in the coating

➤ a reasonably long evanescent tail

$d [\mu\text{m}]$	l	$R [\mu\text{m}]$	E (sphere)	E (coating)	E (air)
0.2	388	68.0056	0.88637	0.05826	0.05537
0.3	391	68.0616	0.79013	0.13292	0.07695
0.5	400	67.9925	0.55117	0.3473	0.10153
0.7	409	67.996	0.38182	0.52335	0.09483
1	418	67.9822	0.23697	0.68933	0.07371
1.3	423	67.9418	0.15764	0.78497	0.05738

Glass coated microspheres – Laser WGMs

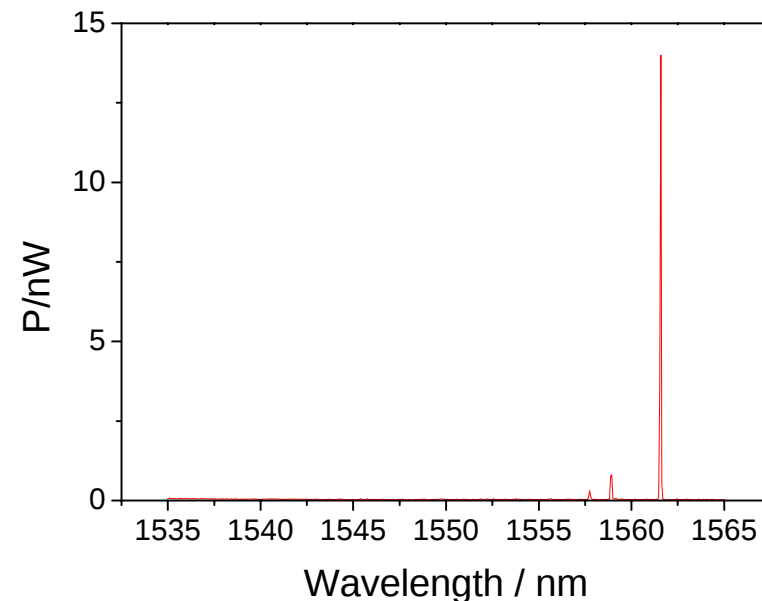
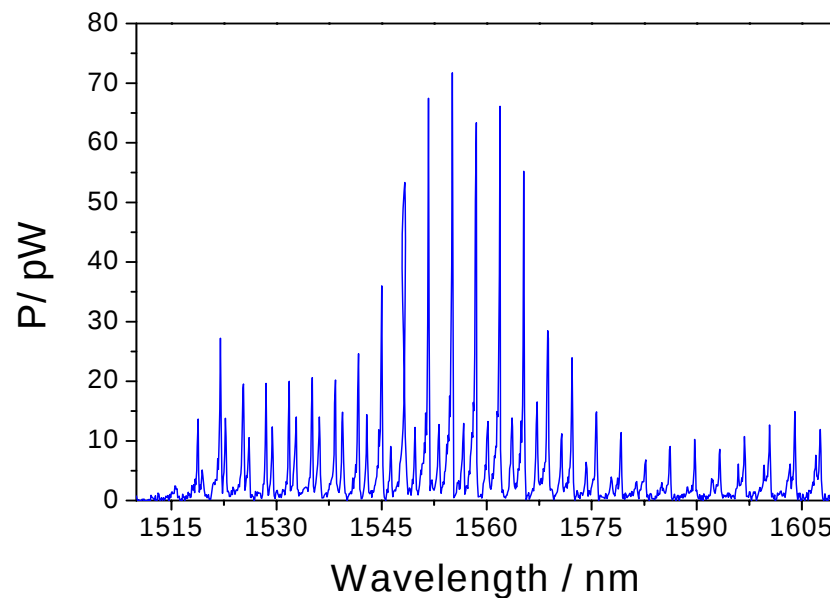
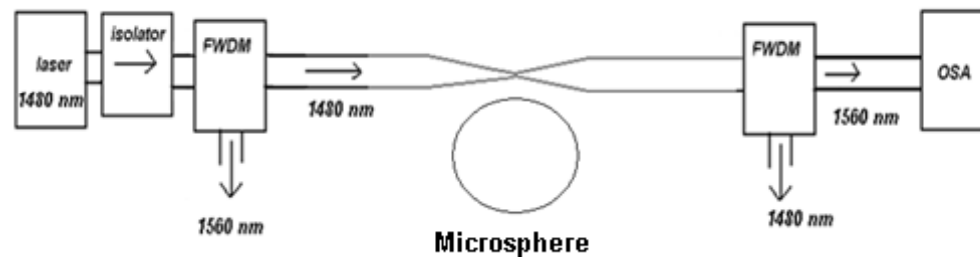
Effect of the coating thickness on the coupling efficiency



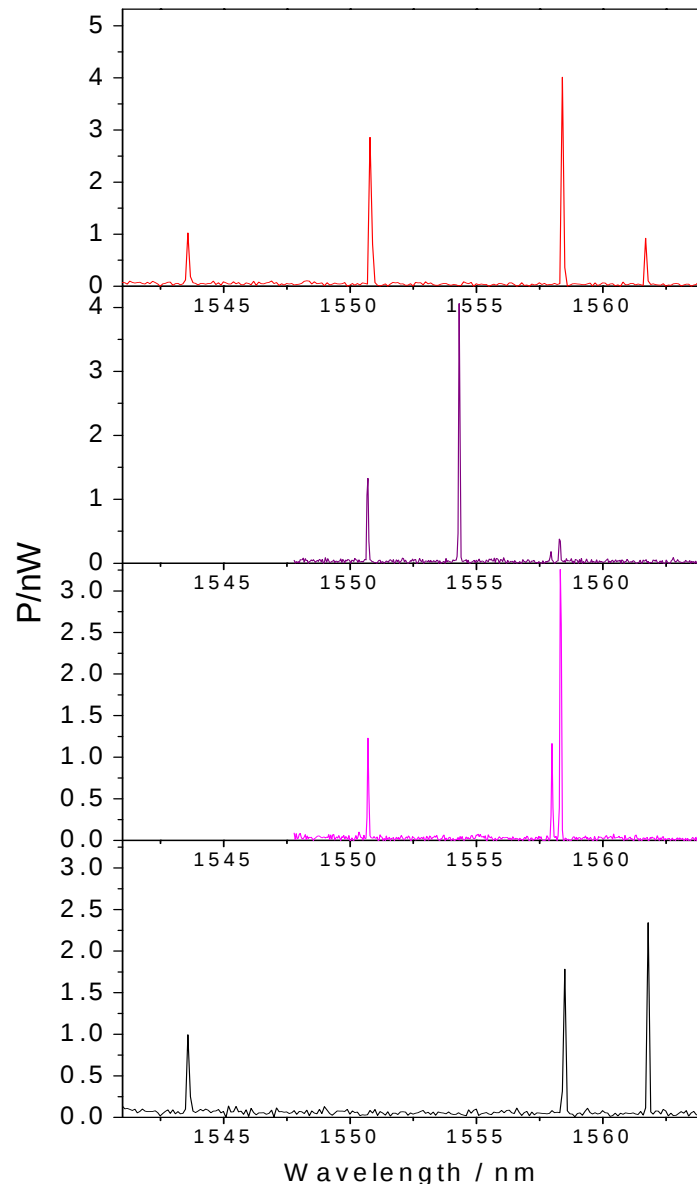
The average integrated luminescence in the 1535-1585 nm range for the 0.3 mol% Er^{3+} 70SiO_2 - 30HfO_2 coated sphere in respect to the number of dips and to the percentage of the fundamental WGM electric field (e. f.) inside the coating as calculated from the number of dips.

Glass coated microspheres – Near Field

- The effect of the coating on the whispering gallery modes was studied
- The coating used was 70 % SiO_2 – 30% HfO_2 doped with 0.3 mol % Er^{3+}
- The spheres ($D=140\pm 10\text{ }\mu\text{m}$) were characterized using a tapered fiber



Glass coated microspheres – Laser WGMs



Excitation at 1.48 μm

Laser power: ≈ 120 mW.

The peak power of the detected modes was always in the range of nanowatts, the highest power detected being 30 nW.

WGM modes are clearly visible

Different modes are excited depending on the position of the taper

$$FSR_{l,l\pm 1} = \frac{\lambda^2}{\pi N d}$$

$N=1.6$

$\lambda = 1550$ nm (wavelength of the signal)

FSR is 3.7 nm corresponding to a microsphere of about 130 μm

Q-factor is greater than the resolution of our detector ($>3 \times 10^4$)

Thermal effects of coatings

Another important application of transparent coatings onto spherical microresonators concerns the possibility of reducing the thermal shift of the WGM resonances due to microsphere heating by the dissipated pump energy.

Actually, the shift is due both to the **temperature-induced change in the refractive index** (the thermo-optic coefficient) and to the **temperature-induced increase of the diameter** of the sphere (coefficient of thermal expansion)

Alessandro Chiasera, Yannik Dumeige, Patrice Féron, Maurizio Ferrari, Yoann Jestin, Gualtiero Nunzi Conti, Stefano Pelli, Silvia Soria, and Giancarlo C. Righini. "Spherical whispering-gallery-mode microresonators" *Laser & Photonics Reviews* 4 (2010) pp. 457-482

Thermal effects of coatings

$$\frac{1}{\lambda} \frac{d\lambda}{dT} = \alpha + \frac{1}{N} \frac{dN}{dT} ,$$

λ is the WGM wavelength

T is the temperature

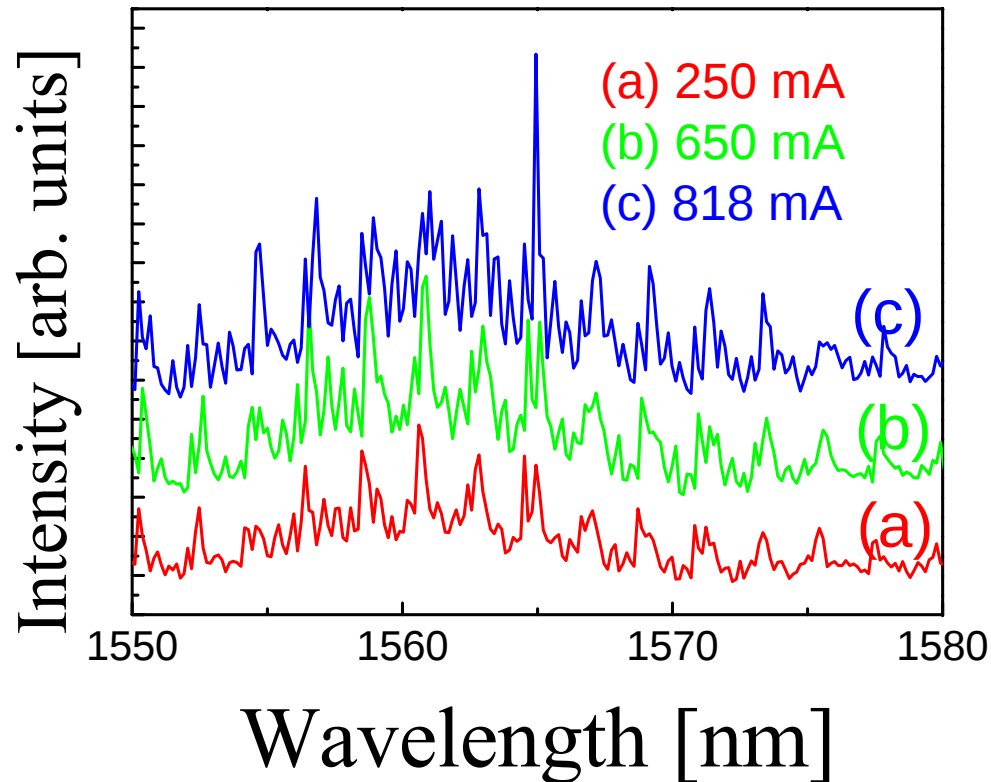
α is the thermal-expansion coefficient

N is the refractive index of the microsphere

One can also take advantage of this characteristic of the spherical WGMRs to accurately measure the thermo-optic coefficient of the material of which the microresonator is made

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Thermal effects of coatings



**No red shift
increasing
pump power**



**due to the
silica-hafnia
coating**

Whispering gallery modes emission spectra of a silica microsphere of 250 μm in diameter and coated by a film of $70\text{SiO}_2\text{-}30\text{HfO}_2$ activated with 1 mol% Er^{3+} . The thickness of the coating is 0.8 μm . Spectra are obtained at different current of the 1480 nm pump laser.

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Conclusions and Perspectives

- Photonic crystals that exhibit specific morphologic, structural, and optical properties allow to develop interesting new physical concepts, as well as novel photonic devices based on the control of the light.
- RF – sputtering and colloidal route are complementary techniques suitable for fabrication of high Q 1-D photonic microcavities and versatile 3-D photonic opals activated by rare earth ions, respectively.
- WGMs laser or frequency combs can be tailored by suitable coating of spherical microresonators

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- SiMeCro research project (2012-2013) Caritro Foundation
- MAE Significant Bilateral Project between Italy and Egypt titled “Smart optical nanostructures for green photonics (2013-2015)”

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