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GLASS-BASED PHOTONIC STRUCTURES: ADVANCES AND PERSPECTIVES

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OUTLINE

- **WELCOME TO THE GLASS AGE**
- **CERAMICS AND ENERGY TRANSFER**
- **WHISPERING GALLERY MODES**
- **♦**1D MICROCAVITIES
- **OPALS**
- **CONCLUSIONS AND PERSPECTIVES**

THE BRIGHT ADVANCEMENT OF GLASS PHOTONICS WELCOME TO THE GLASS AGE

DAVID L. MORSE AND JEFFREY W. EVENSON - CORNING

INTERNATIONAL JOURNAL OF APPLIED GLASS SCIENCE, 7 [4] 409-412 (2016) DOI:10.1111/IJAG.12242

- ➢ Glass is one of the world's most transformative materials.
- Featuring tremendous versatility and distinctive technical capabilities, glass has been responsible for numerous cultural and scientific advancements from windows to optical fiber.
- Today, the pace of glass innovation is accelerating, thanks to scientists' deep understanding of glass physics and chemistry, combined with modern analytic and control technologies.
- > We believe that the world has entered the Glass Age.
- We have an unprecedented opportunity to harness the unique capabilities of glass to solve some of our world's most urgent challenges, such as more effective healthcare, cleaner energy and water, and more efficient communication.
- Realizing the potential of the Glass Age will require collaboration, resources, and support, but it is an opportunity we cannot afford to waste.

GLASS – BASED NANOTECHNOLOGIES

« ...smaller objects in nature are not just scaled replicas of similar big objects and in fact they have improved properties...»

Galileo «Dialogue Concerning Two New Sciences» (1638)



- Light-matter interactions are stronger at small objects (micro- and nanoscale) PHOTONIC GLASS-CERAMICS
- High Q structures, with strong spatial localization of the field, well respond to this principle and receive a great attention in many fundamental processes in photonics. Great application potential in many areas including cavity QED, atom trapping, laser stabilization, microlasers, nonlinear optics, nonlinear-optical thin-film diagnostics, and evanescent-wave sensing. **CONFINED STRUCTURES**

GLASS – BASED NANOTECHNOLOGIES



YOU CAN SAVE ENERGY REDUCING ENERGY CONSUMING

LOW THRESHOLD LASER ACTION



GLASS CERAMICS AND ENERGY TRANSFER

Lidia Zur

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DOWN CONVERSION WITH TB³⁺/YB³⁺



One green photon @ 488nm Two red photon @ 980 nm

DOWN-CONVERTERS SILICA-HAFNIA GLASS CERAMICS

Low phonon energy (~ 700 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

GLASS CERAMICS AS A CLASS OF NANOCOMPOSITE PHOTONIC MATERIALS

 \checkmark Nanoscale structural and optical fluctuation





Validation of the process: hafnia nanocrystals activated by rare earth ions embedded in waveguides – Energy transfer efficiency enhanced by HfO₂ NCs

Tb^{3+}/Yb^{3+} down conversion efficiency

Composition (Yb concentration in mol%)		2%	3%
Estimated transfer efficiency (in glass ceramic)		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%

Pr³⁺/ Yb³⁺ down conversion efficiency

70ZrF ₄ -23.5LaF ₃ -0.5AlF ₃ -6GaF ₃	0.5% $Pr^{3+}/10 Yb^{3+}$	191%
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B. Dieudonné, B. Boulard, G. Alombert-Goget, A. Chiasera, Y. Gao, S. Kodjikian , M. Ferrari "Up- and Down-conversion in Yb³⁺-Pr³⁺ co-doped fluoride glasses and glass ceramics"
Journal of Non-Crystalline Solids 377 (2013) pp. 105-109. doi:10.1016/j.jnoncrysol.2012.12.025

GLASS - BASED INTEGRATED OPTICS TECHNOLOGIES



G.C. Righini, A. Chiappini "Glass optical waveguides: a review of fabrication techniques" Optical Engineering (2014)



SnO₂ nanocrystals

Innovative solution exploiting SnO₂ nanocrystals



SNO_2 NCs as sensitizers for Rare Earth ions







- Energy transfer from SnO₂ into Rare-Earth ions increases the luminescence of Rare-Earth ions
- 2) SnO₂ has a low phonon energy (630cm⁻¹) that **reduces losses** from non-radiative relaxation
- 3) SnO_2 has **transparency** in range from visible to NIR
- 4) SiO_2 -SnO₂ exhibits photorefractivity

 $(\Delta n \sim -10^4 \text{ allowing writing of integrated circuits})$ by UV and visible light

Lidia Zur, Thi Ngoc Lam Tran, Marcello Meneghetti, Thi Thanh Van Tran, Anna Lukowiak, Alessandro Chiasera, Daniele Zonta, Maurizio Ferrari, Giancarlo C. Righini "Tin-dioxide nanocrystals as Er³⁺ luminescence sensitizers: formation of glass-ceramics thin films and their characterization" Optical Materials 63 (2017) pp.95-100 Special Issue to Celebrate G. Boulon doi: 10.1016/j.optmat.2016.08.041

TEM

Homogeneous distribution



About 4 nm NCs



Tran T.T. Van, S. Turrell, B. Capoen, Lam Q. Vinh, O. Cristini-Robbe, M. Bouazaoui, F. d'Acapito, M. Ferrari, D. Ristic, A. Lukowiak, R. Almeida, L. Santos, and C. Kinowski "Erbium-doped tin-silicate sol-gel-derived glass-ceramic thin films: Effect of environment segregation on the Er³⁺ emission" Science of Advanced Materials 7 (2015) pp. 301-308 doi: 10.1166/sam.2015.2022

Excitation spectra

Room temperature excitation spectra of ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$ emission at 613 nm



The broad and strong band observed at 310 nm (4.0 eV) corresponds to the SnO_2 band-gap energy.

PL spectra λ_{ex} =351nm



For annealing temperature higher than 900 °C the emission features typical of Eu^{3+} ion in a crystalline like environment are predominant, *indicating that most part of Eu^{3+} ions are embedded in SnO₂ nanocrystals*





Enhanced fluorescence from Eu^{3+} in lowloss silica glass-ceramic waveguides with high SnO_2 content $\lambda_{ex} = 351 \text{ nm}$

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 $75SiO_2$ - $25SnO_2$: Eu³⁺ fabricated by sol gel, dip-coating method. Losses remain below 0.8 dB/cm.

High photosensitivity in low-loss sol-gel SiO₂ – SnO₂ 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.

Anna Lukowiak, Lidia Zur, Thi Ngoc Lam Tran, Marcello Meneghetti, Simone Berneschi, Gualtiero Nunzi Conti, Stefano Pelli, Cosimo Trono, B.N. Shivakiran Bhaktha, Daniele Zonta, Stefano Taccheo, Giancarlo C. Righini, Maurizio Ferrari "Sol–Gel-Derived Glass-Ceramic Photorefractive Films for Photonic Structures" Crystals 7 (2017) pp. 61_1/7 doi:10.3390/cryst7020061

High photosensitivity in low-loss sol-gel SiO₂ – SnO₂ waveguides



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

Lidia Zur, Thi Ngoc Lam Tran, Marcello Meneghetti, Maurizio Ferrari "Sol-gel derived SnO2-based photonic systems"

in Handbook of Sol-Gel Science and Technology (2017) pp. 1-19, Springer International Publishing AG Eds. Lisa Klein, Mario Aparicio, Andrei Jitianu Print ISBN: 978-3-319-19454-7 Online ISBN: 978-3-319-19454-7 doi: 10.1007/978-3-319-19454-7_116-1

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 $SiO_2 - SnO_2$ thin film waveguides" Proceedings of SPIE Vol. 7604 (2010) pp. 76040Z-1/6

DESIGN FOR PLANAR PHOTONIC STRUCTURES





- i. active Fabry-Perot cavity with passive Distributed Bragg Reflectors (DBR- FP);
 ii. active Distributed-Feedback structure (DFB);
- iii.DFB with Quarter-Wave phase Shift (QWS-DFB);
- iv.DFB with Multiple Phase Shifts distributed along the cavity (MPS-DFB)

SnO₂-SiO₂:Er³⁺ PLANAR WAVEGUIDES

70%SiO₂-30%SnO₂-0.5%Er³⁺, HT@ 1000°C for 1h



TE



INCREASED REFRACTIVE INDEX



70%SiO₂-30%SnO₂-0.5%Er³⁺, HT@ 1000°C for 1h



SnO₂-SiO₂:Er³⁺ PLANAR WAVEGUIDES







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WHISPERING GALLERY MODES

Davor Ristic

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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. Cibiel, 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

LASER WGMS COATED SPHERICAL RESONATORS

> The effect of the coating on the whispering gallery modes was studied > The coating used was 70 % SiO₂ – 30% HfO₂ doped with 0.3 mol % Er³⁺ > The spheres (D=140±10 μ m) were characterized using a tapered fiber



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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

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

 λ = 1550 nm (wavelength of the signal)

FSR is 3.7 nm corresponding to a microsphere of about 130 μ m Q-factor is greater than the resolution of our detector (>3x10⁴)

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N=1.6

Biosensor for protein detection





Figure 2: Binding of e.g. Proteins or DNA (represented as squares) on the microsphere surface increases the initial microsphere radius R and leads to a red shift of a given optical resonance wavelength λ

http://www.photonicatomsensors.com

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1D MICROCAVITIES





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GLASS-DERIVED 1-D PHOTONIC CRYSTALS

One-dimensional photonic crystals

outstanding tool for *new photonics*, being the *simplest system* to exhibit a so-called *photonic bandgap* and therefore one of the easiest to handle in order to obtain *tailored optical devices*



LUMINESCENCE ENHANCEMENT PHOTON MANAGEMENT

- One of the interesting features of the 1-D microcavities is the possibility to enhance the luminescence, resonant with the cavity, when the defect layer is activated by a luminescent species.

- When the cavity dimensions approach the wavelength of the emission the density of electromagnetic states inside the cavity are strongly perturbed and can lead to significant enhancement of the luminescence quantum yield.

- This enhancement is achieved by increasing the number of the localized modes coupled with the emitter

1-D PHOTONIC CRYSTALS FOR

LOW THRESHOLD LASER ACTION

When the spontaneous emission of the emitter, embedded in the

defect layer of a 1-D photonic crystal, is strongly enhanced the

possibility of low threshold lasing could take place.



Hybrid 1-D microcavity: fabrication



2) Active layer deposition (sol-gel)

Hybrid 1-D microcavity: fabrication



PMMA polymer matrix containing CdSe-CdS-ZnS
Hybrid 1-D microcavity: Laser action



Hybrid 1-D microcavity: Laser action



Coherent emission from fully Er³⁺ doped monolithic 1-D dielectric microcavity fabricated by rf-sputtering

Morphology



SEM micrograph of the Er^{3+} doped 1D dielectric microcavity cross section. The bright and dark region corresponds to TiO_2 and SiO_2 layers, respectively. The substrate is located on the bottom of the images and air on the top.

Transmission



Transmission spectrum of the cavity with two Bragg mirrors, each one consisting of ten pairs of SiO_2/TiO_2 layers in the region between 450 nm and 2500 nm. The first order stop band ranges from 1300 nm to 1850 nm. The first order cavity resonance corresponds to the sharp maximum centered at 1559.2 nm.

${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ photoluminescence spectrum



 ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ photoluminescence spectrum of the cavity activated by Er³⁺ ions in 1D dielectric microcavity. The emission is recorded at 0 degree from the normal on the samples upon excitation at 514.5 nm at the input power of 185 mW (red line) and 24 mW (blue line). 30 degree of excitation angle for both the measurements.

Peak intensity and FWHM vs Pump Power Exciting at 514.5 nm / 30°



 ${}^{4}I_{13/2} \rightarrow {}^{4}I_{15/2}$ photoluminescence peak intensity and FWHM (blue line) at 1560 nm as a function of 514.5 nm pump power with 0 degree of detection angle and 30 degree of excitation angle. Red and green line are the results of linear fit while the blue line is a guide for the eyes.

Structure of the microcavity

NEW approach



Active Bragg Mirror: 10 alternated quarter wave layers TiO₂ (172 nm) and SiO₂ (262 nm) activated with 0.2 mol % of Er³⁺. Active layer: half wave (525 nm) SiO₂ activated with 0.2 mol % of Er³⁺. The dark regions corresponds to SiO₂ and the white regions corresponds to TiO₂

Transmission



Emission Features – Bragg reflectors with 10 layers



Emission Features – Bragg reflectors with 10 layers



Emission Features – Bragg reflectors with 14 layers



Disordered 1-D photonic structures



SEM micrograph of the microcavity composed by 14 couple of $\text{TiO}_2/\text{SiO}_2$ layers. To realize the disordered photonic structure we have alternated layers of SiO_2 and TiO_2 , with a thickness of (80 + n) nm, where *n* is a random integer $0 \le n \le 40$. In this way we obtain a random sequence of thicknesses, between 80 and 120 nm.

BROAD BAND MIRRORS BASED ON DISORDERED 1-D PHOTONIC STRUCTURES



Reflection of the CIE 1931 diagram by a 1-D photonic crystal (a) and a disordered 1-D photonic structure (b)

average transmittance value of 0.7 was obtained for the 300 - 1200 nm transmission spectrum

This appealing behavior is due to interference between waves traveling in regions with different optical paths, determined by the disordered distribution of stacked layer thicknesses.

Optical cavity with graphene



Optical cavity with graphene: It is possible to fabricate a monolithic systems?



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Monolithic 1D Photonic crystal with graphene layer in the defect



Monolithic 1D Photonic crystal with graphene layer in the defect



Hybrid 1-D microcavity: acoustic sensor



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OPALS AS STRAIN SENSORS

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OPALS



$$\lambda = 2 \cdot \sqrt{\left(n_{eff}\right)^2 - (\sin\theta)^2} \cdot d$$
$$n_{eff}^2 = n_{spheres}^2 \cdot f + n_{medium}^2 (1 - f)$$
$$f = 74\%$$
$$d = \sqrt{\frac{2}{3}} D$$

If $\theta = 0$, then $\lambda = 2 \cdot n_{eff} \cdot d$

fcc structure provides stable system from thermodynamical point of view

 θ – incident angle, d – interplanar distance, l_0 – initial length, D – spheres diameter

FABRICATION: 3D PC

1. Producing polystyrene spheres (PSs):

- mixture of water, surfactant (SDS), styrene, polymerization initiator ($K_2S_2O_6$)
- desired dimension $(D_{PS} = 230nm)$ with low polydispersivity

2. Arranging the PS spheres into ordered structure

• <u>vertical</u> deposition of PSs onto Viton substrate

(50mm×15mm×1mm)

• constant temperature T = 50°

3. Infiltration with PDMS

- base : curing agent (3:1)
- 4h for curing at $T = 65^{\circ}$.





SENSITIVITY TO STRAIN



$$\lambda = 2 \cdot n_{eff} \cdot d$$

$$\varepsilon_{z} = -\upsilon \varepsilon_{x}$$
$$\Delta \lambda = 2 \cdot n \cdot \varepsilon_{z} \cdot d_{0}$$
$$\Delta \lambda = \underbrace{-2 \cdot n \cdot d_{0}}_{\lambda_{0}} \upsilon \varepsilon_{x}$$

 ΔI – elongation Δd - change of interplanar distance

 $\begin{bmatrix} \lambda \\ d \end{bmatrix}$ values after deformation

υ - Poisson coefficient; $ε_x$, $ε_z$ - strain value along *X*,*Z* axis

TESTING 3D STRUCTURE



Unloaded Sample ($\lambda = 583$ nm)

Elongated Sample ($\lambda = 550$ nm)































[mɛ]



Experimental performances

 sensitivity to strain 	-288 pm/με
 inverse sensitivity 	3.47 με/pm
 resolution of the reflected peak 	~100 pm
 instrumental resolution 	~350 µε
 max measurable elongation 	>150 µε [= 15%]
 to appreciate a change in colour: 	Δλ > 10 nm >35 με [= 3.5%]

PCs vs. FBGs

	Photonic crystals		FBGs
•	resolution of the reflected peak in the order of ~100 pm	•	width of the reflected peak in the order of ~20pm
•	reflected band depends on the transversal strain	•	the grating is deformed directly by the strain of the support
	$\Delta \lambda \approx 2n_{eff} d_0 v \varepsilon_x$		$\Delta \lambda \approx 2n_{eff} d_0 \varepsilon_x$
•	work in the visible ($\lambda_0 \sim 400-600$ nm)	•	work in the infrared ($\lambda_0 \sim 1550$ nm)

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OPALS FOR PHOTONS MANAGEMENT

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Silica inverse opals Latex opals as templates



Luminescence at 1.5µm


Assessment of the chromatic behavior of colloidal sensors



We assume an **isotropic displacement** of the spheres assembled in the periodic lattice fcc

Sketch of the isotropic displacement of the spheres assembled in the periodic lattice fcc of the colloidal crystal after solvent initial application. Clock wise: state (green colour), intermediate state (orange colour), and final state (red colour) with their respective reflectance peak shift.

The process is fully reversible

The assumption and the analytical model have been verified by the response of specific polar solvents



• Optical shift of the reflected peak

Validation

$$S = \frac{(d_{111})_s}{(d_{111})_0}$$

Analyte	Swelling Tabulated*	Swelling Calculated
Methanol	1.02	1.03
Tert-butyl	1.21	1.19

J. Lee et al. Analytical Chemistry 75 (2003) 6544-6554.



Agreement between the values

chromatic changes are caused by the displacement of the spheres which at first approximation may be considered a homothetic expansion

Isomers and alcohol concentration





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Conclusions and Perspectives

Cutting-edge glass based research is present in a huge amount of

areas crucial for the improvement of the quality of life:

- ► ICT
- ≻ Lighting
- ≻ Laser
- ➢ Sensing
- ➢ Energy
- Environment
- > Biological sciences
- Medical sciences
- ➢ Quantum optics

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