

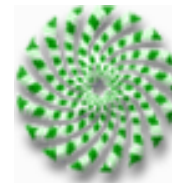
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# NOVEL FIBER OPTIC SENSING ARCHITECTURES BASED ON SENSITIVE NANOFILMS



**upna**  
Universidad  
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Public University of Navarre  
Pamplona; SPAIN  
[www.unavarra.es](http://www.unavarra.es)



*Nanosonic, Inc.*

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Ignacio R. Matias, Francisco J. Arregui and Richard O. Claus

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# Summary

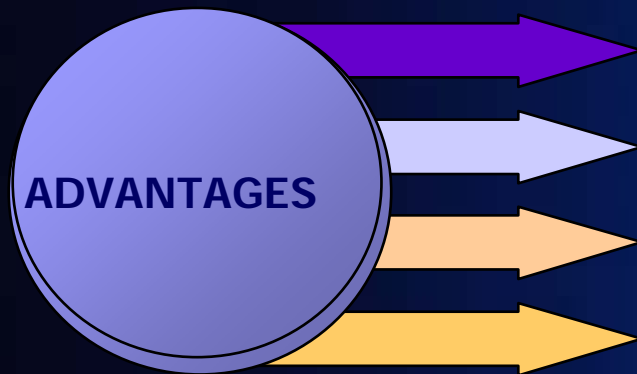
- ❑ Introduction to the fiber optic sensor market
- ❑ Nanotechnology and fiber optic sensors?
- ❑ The Electrostatic Self-Assembled Monolayer Method
- ❑ Possible sensing architectures based on nano-films
  - ❑ Tapered ends
  - ❑ Tapered optical fibers
  - ❑ Hollow core fibers
  - ❑ Long period gratings
  - ❑ Optical fiber gratings
  - ❑ NanoFabry-Perot Cavities
- ❑ Conclusions

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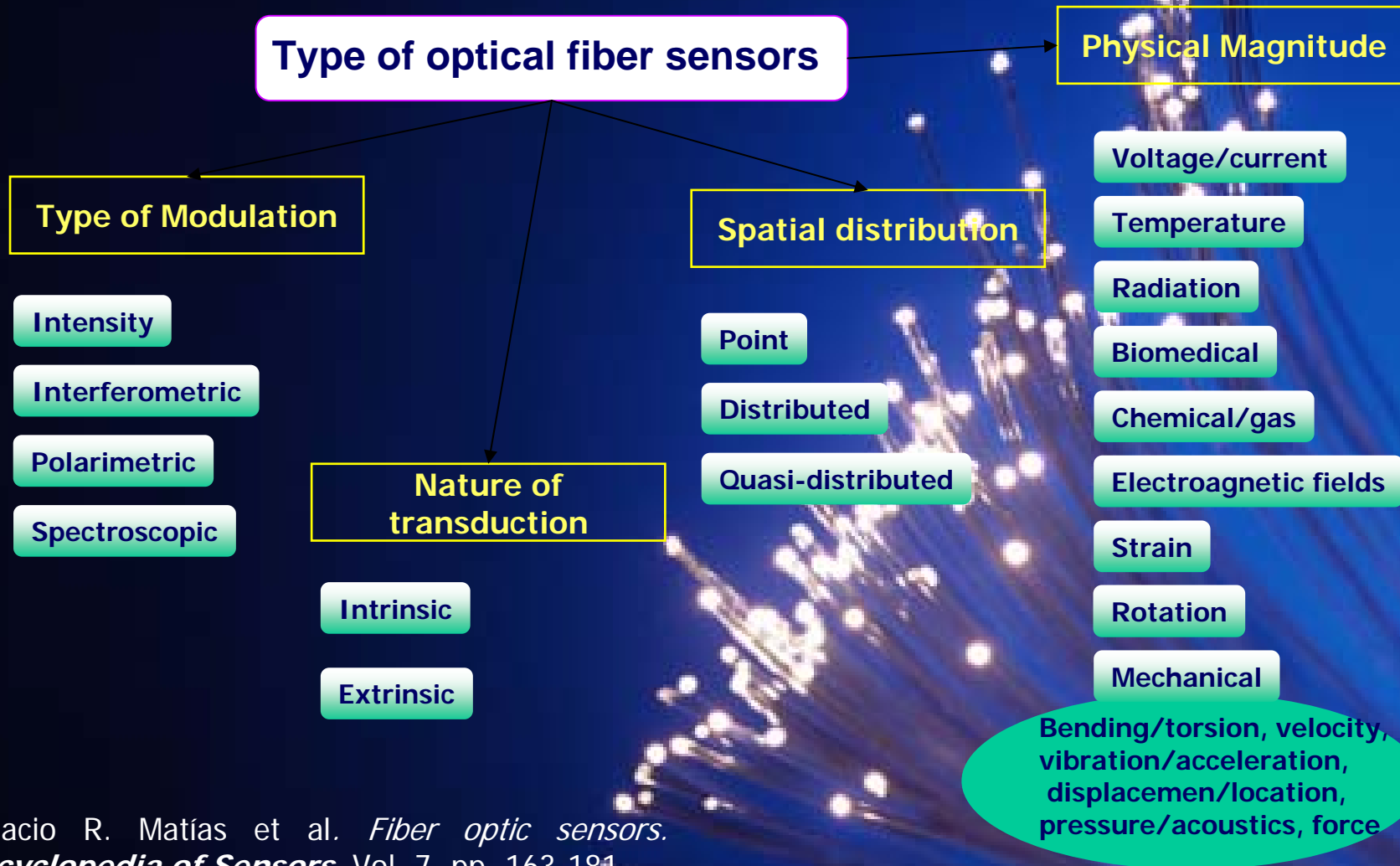
## Technology Overview. Advantages



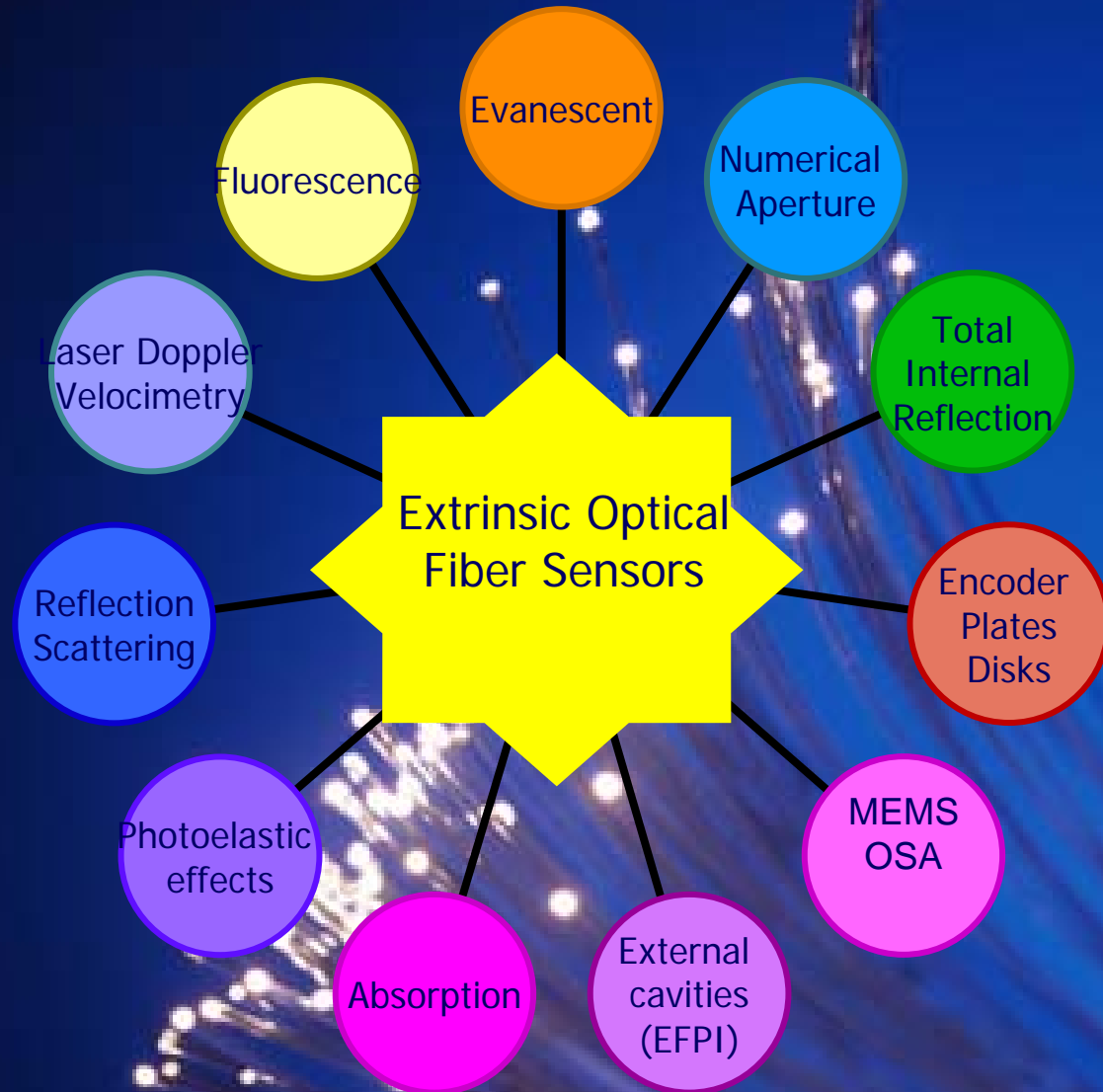
- Small and Lightweight
- Possibility of being embedded in composite materials
- Passive nature
- Large dynamic ranges
- Single- & Multi-Point Sensing Configurations
- Large wideband
- Low attenuation
- Multiplexing techniques
- EMI immunity



## Technology Overview. Classification



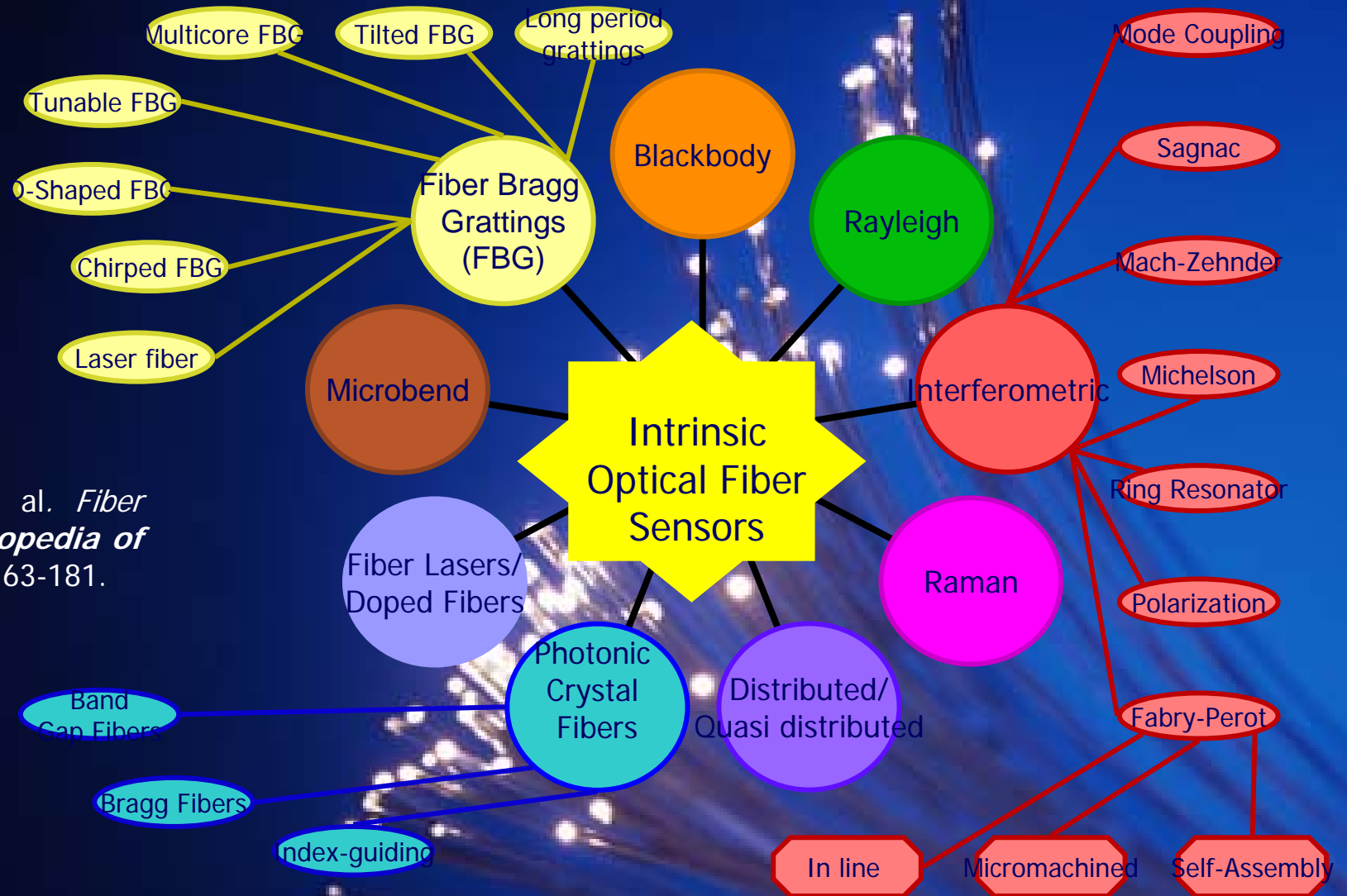
## Technology Overview. Extrinsic sensors



Ignacio R. Matías et al. *Fiber optic sensors. Encyclopedia of Sensors*. Vol. 7, pp. 163-181.

# Novel fiber optic sensing architectures based on sensitive nanofilms

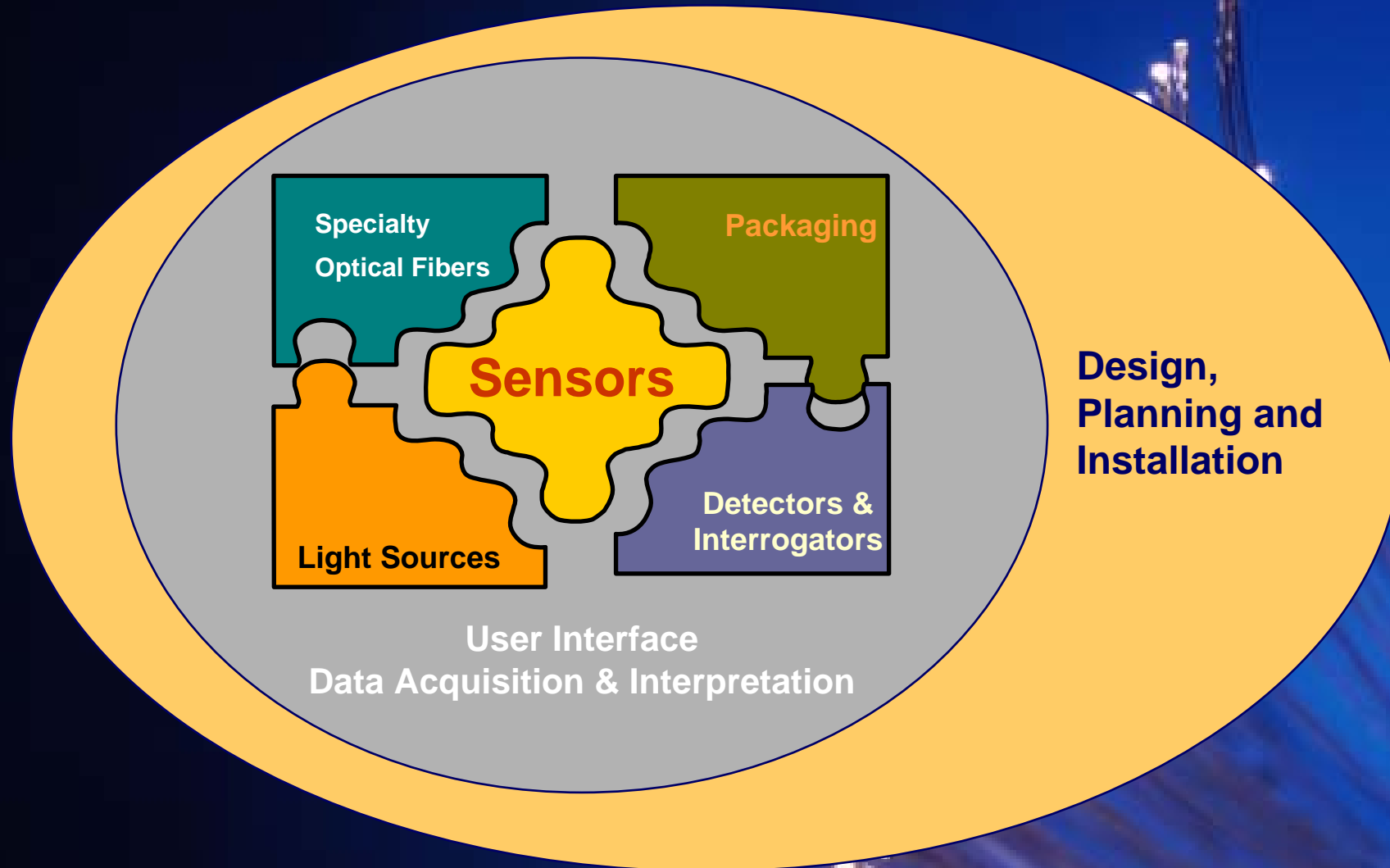
## Technology Overview. Intrinsic sensors



Ignacio R. Matias et al. *Fiber optic sensors. Encyclopedia of Sensors*. Vol. 7, pp. 163-181.

# Novel fiber optic sensing architectures based on sensitive nanofilms

## Fiber Optic Sensing System Key Building Blocks



*Courtesy of Alexis Méndez. MCH Engineering, LLC*



# Novel fiber optic sensing architectures based on sensitive nanofilms

## Fiber Optic Market Status

Fragmented  
Niche markets  
Foothold in niche applications  
Slow adopting industries  
Positive investment environment  
Major franchises emerging  
Positive and continued steady growth  
Important growth in chemical/ bio-detection

- Unfamiliarity with the technology
- Conservative/no-risk attitude of some industries
- Need for a proven field record
- Compatibility with existing equipment
- Cost
- Availability of trained personnel
- Turn-key type systems (total sensing solution)
- Lack of standards
- Quality, performance, packaging & reliability deficiencies across vendors
- Major sensing initiatives likely dominated by wireless

## Market Drawbacks

# Novel fiber optic sensing architectures based on sensitive nanofilms

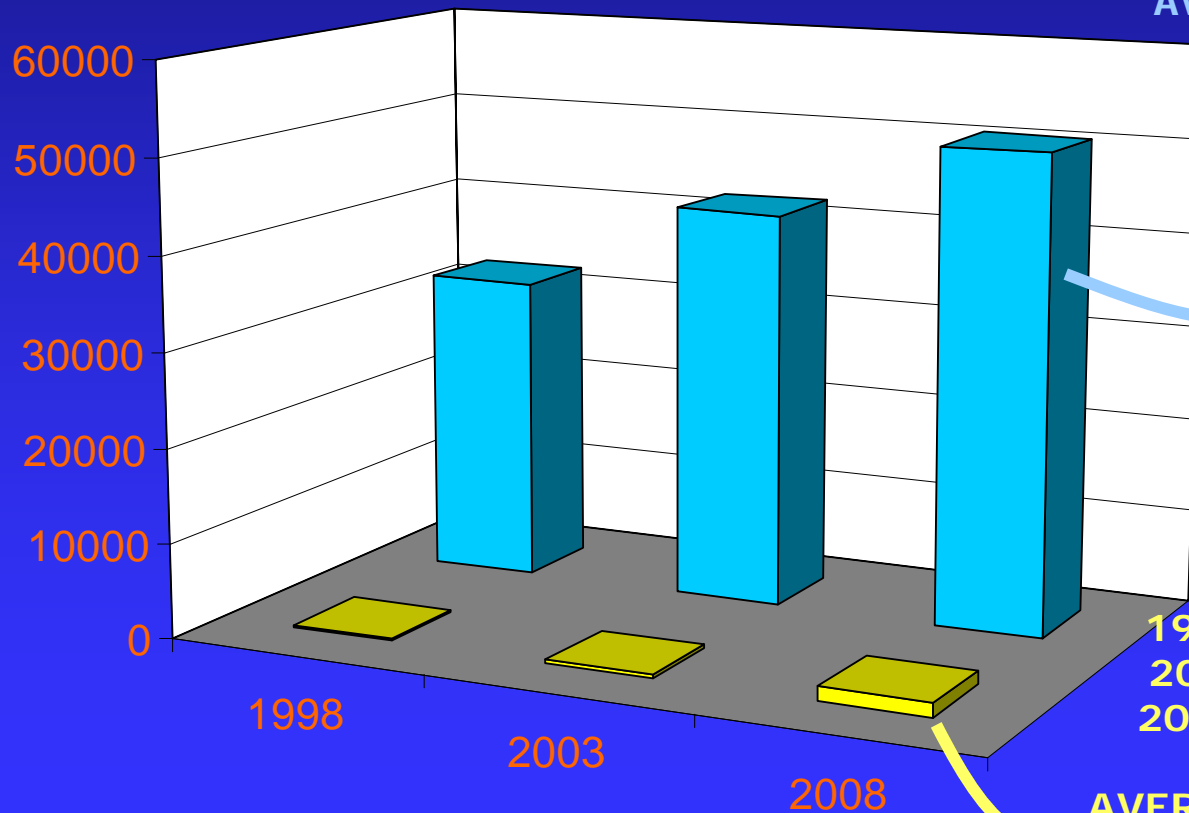
## Sensors Market Size

### Development of the World Market Share of Fiber Optical Sensors until 2008

## SENSORS WORLD MARKET

1998 – U\$32,534.0M  
2003 – U\$42,158.4M  
2008 – U\$50,594.3M

US \$ Million



AVERAGE OF ANNUAL GROWTH RATE – 4,5%

## FOS WORLD MARKET

1998 – U\$ 175 M – MKT SHARE (0,54%)  
2003 – U\$ 283 M - MKT SHARE (0,67%)  
2008 – U\$ 1450 M –MKT SHARE (2,87%)

AVERAGE OF ANNUAL GROWTH RATE – 23,5%

# Novel fiber optic sensing architectures based on sensitive nanofilms

**Civil** (bridges, roads, dams, tunnels)

**Oil & Gas** (Reservoir monitoring, downhole P/T sensing, seismic arrays)



**Transportation**  
(Rail monitoring, Weight in motion, Carriage safety)

## Applications

**Energy Industry** (Power plants, Boilers & Steam turbines, Power cables, Turbines, Refineries)



**Border security and power line monitoring**

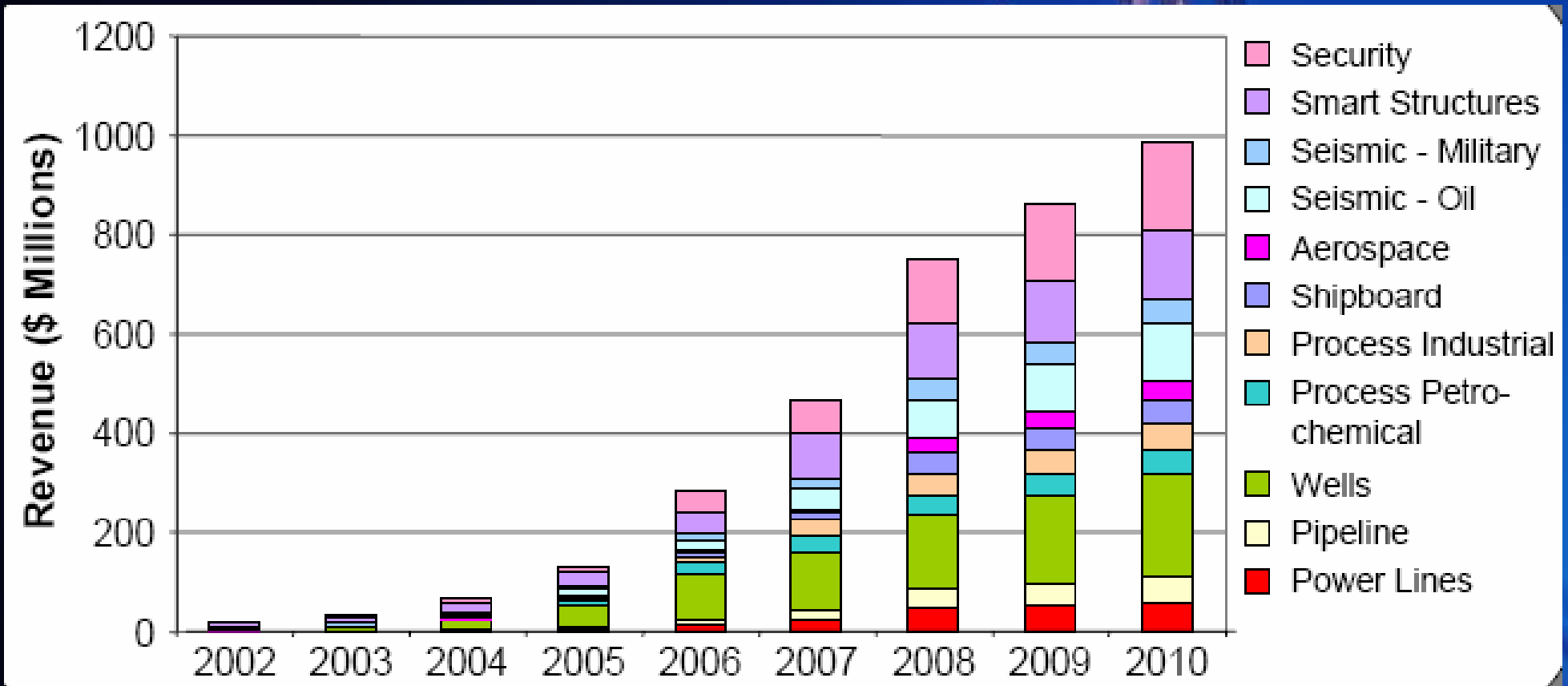


**Aerospace** (Jet engines, Rocket & propulsion systems, Fuselages)

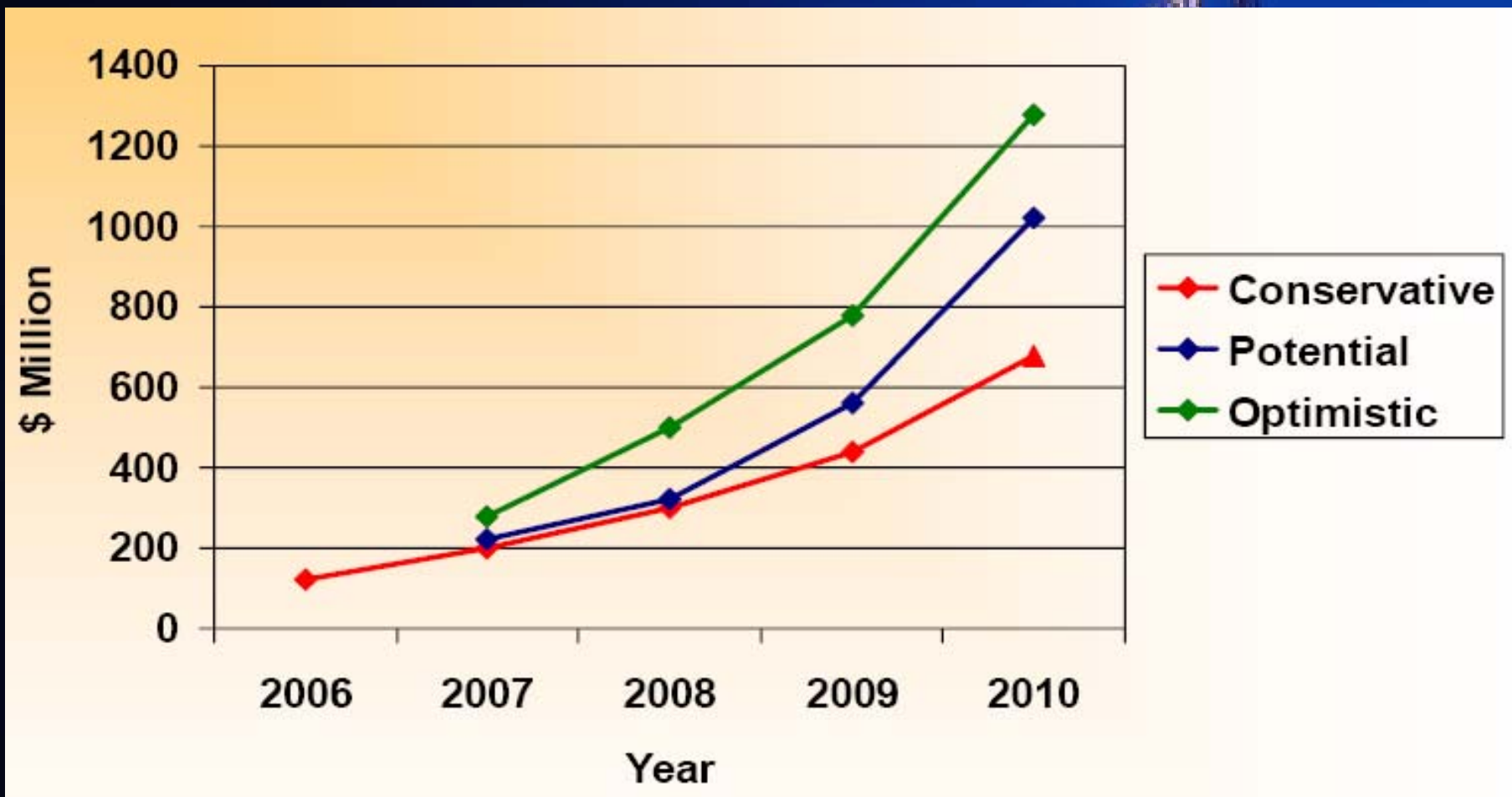


*Courtesy of David Huff and Alexis Mendez*


## Optical Fiber Sensor Market Revenues Breakdown



## Optical Fiber Sensor Market Forecast



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- ❑ Conclusions

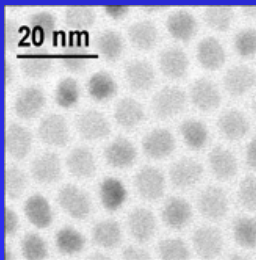


# Novel fiber optic sensing architectures based on sensitive nanofilms

## Nanotechnology and fiber optic sensors?



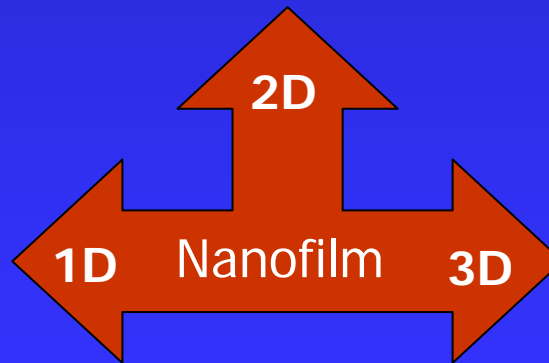
Nanostructure with a size between molecular and microscopic layers of subwavelength thickness (*bottom-up*)



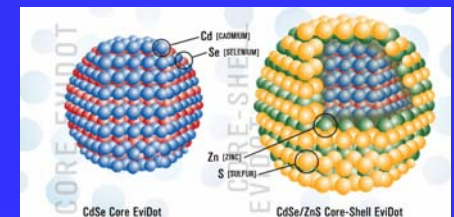
Nanotubes



Nanotextured surfaces



Nanoparticles

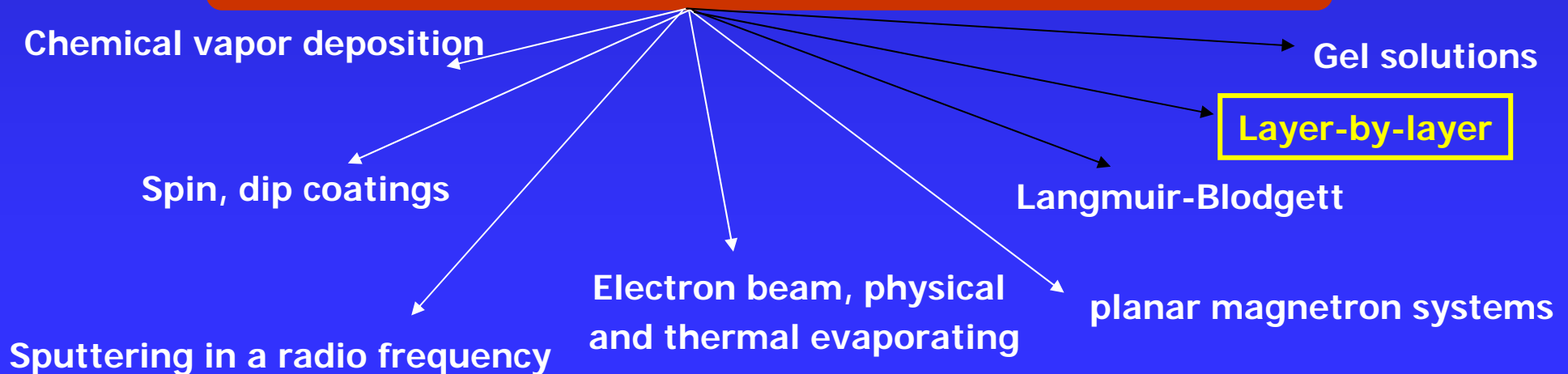


# Novel fiber optic sensing architectures based on sensitive nanofilms

## Nanotechnology and fiber optic sensors?



### Deposition techniques for sensing coatings in OFS





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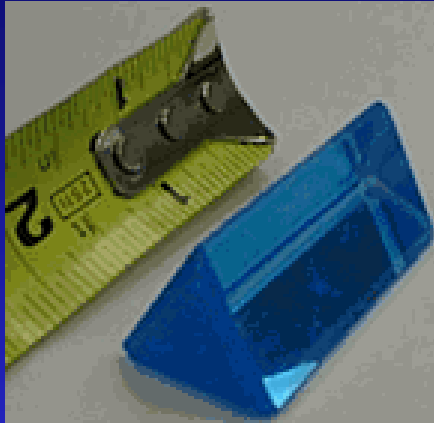
# Novel fiber optic sensing architectures based on sensitive nanofilms

## Introduction

- 1960s: Layer-by-layer adsorption of oppositely charged colloidal particles was first proposed by R. K. Iler R.K. Iler, J. Colloid Interface Sci., 21, 569-594 (1966) "Multilayers of colloidal particles"
  - 1990s: reappearance of works on this topic with Decher and co-workers as the pioneers G. Decher, J.-D. Hong, Makromol. Chem., Macromol. Symp., 46, 321-327 (1991).
  - Today: Layer-by-Layer Electrostatic Self-Assembly (ESA) is one of the most promising techniques for the deposition of nanostructured tailored materials on complex surfaces
- 
- Possible ESA substrates: metals, plastics, ceramics, oxides, semiconductors with different sizes and shapes such as prisms, concave or convex surfaces.
  - Possible ESA coating materials: metals, semiconductors, polymers, dyes, indicators, quantum dots, enzymes and many others (Au, Pt, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, poly(sodium-4-styrenesulfonate) (PSS), poly(diallyldimethyl ammonium chloride) (PDDA), poly acrylic acid (PAA), poly(allylamine hydrochloride) (PAH), poly R-478, poly S-119, Neutral Red, Fluorescein, HPTS, PPV, Prussian Blue, Glucose Oxidase, Silica, Quantum Dots...)

# Novel fiber optic sensing architectures based on sensitive nanofilms

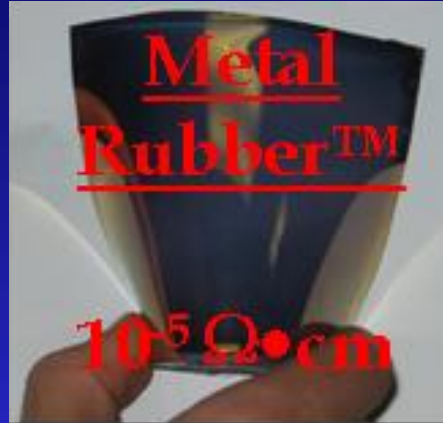
## The ESA Method: diverse applications



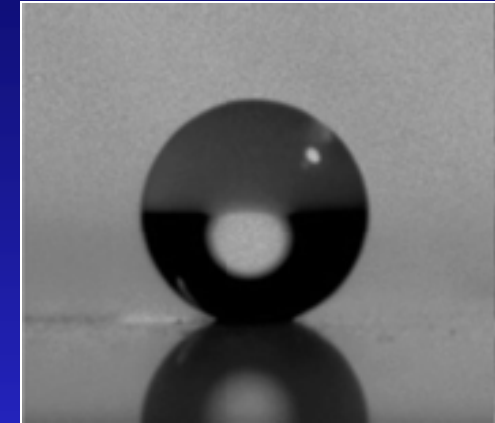
**PRISMS**



**LENS**



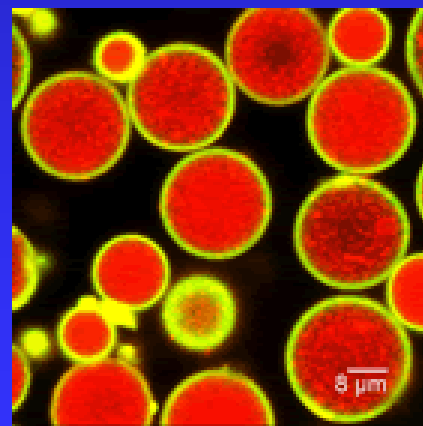
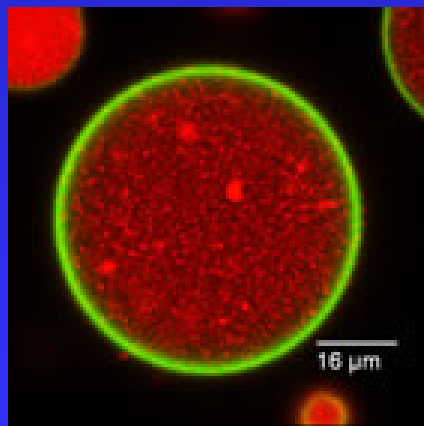
**FLEXIBLE SUBSTRATES**



**SUPERHYDROPHOBIC SURFACES**

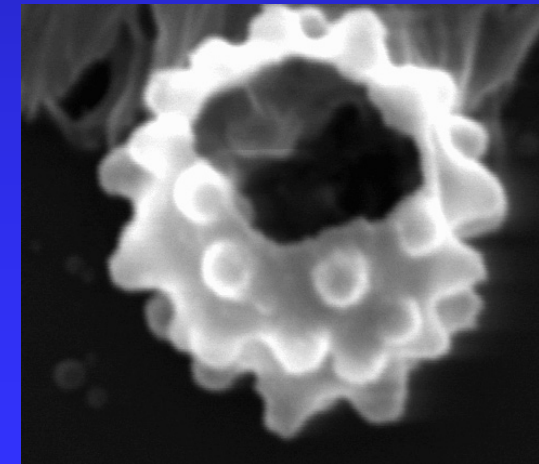
M.I.T., M. F. Rubner et al.

NANOSONIC, INC., R. O. Claus et al.



**MICROSPHERES**


TEXAS A&M, M. McShane et al.



**COATINGS ON BIOLOGICAL CELLS**

University of Melbourne, F. Caruso et al.

# Summary

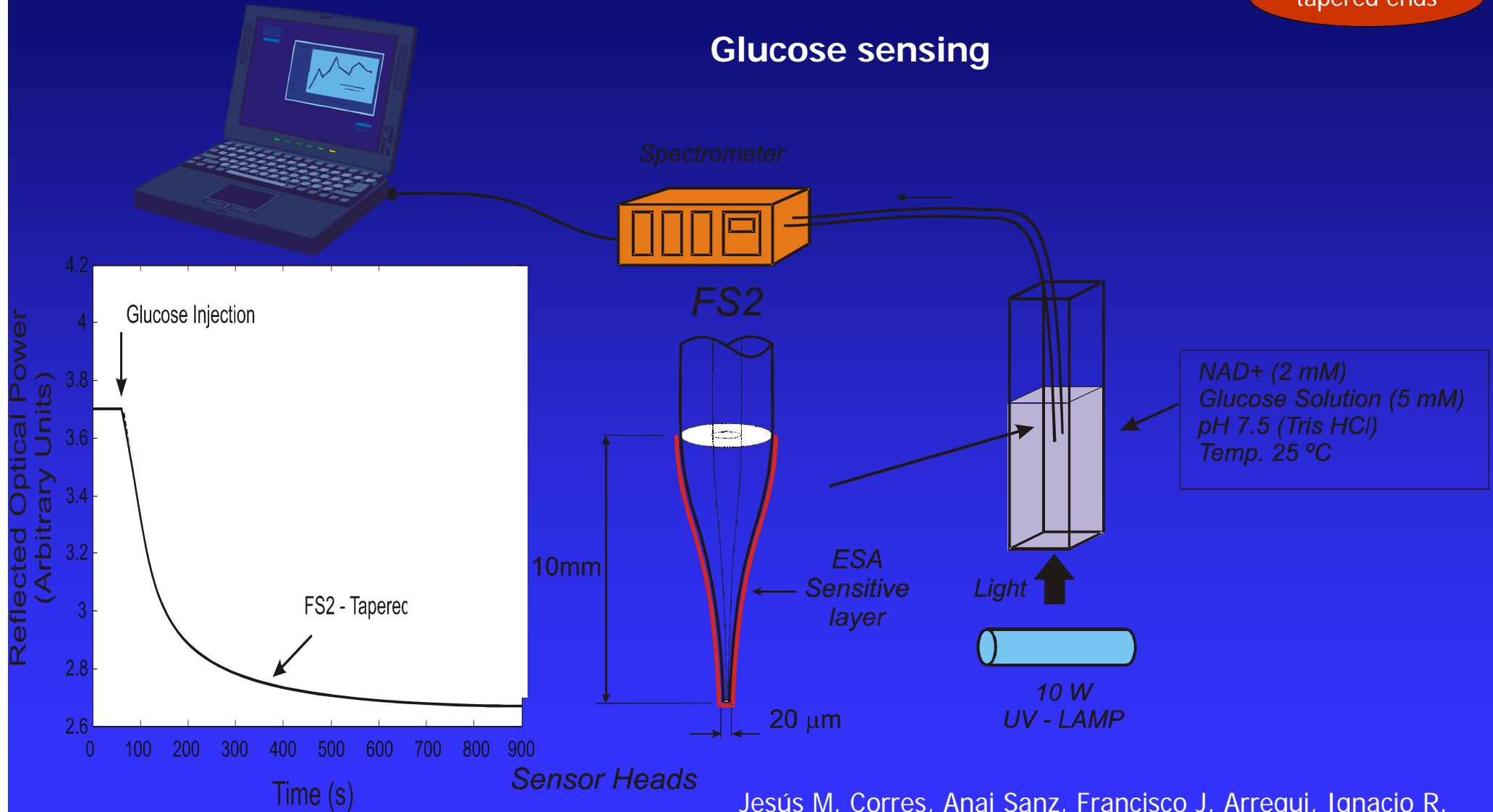
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# Novel fiber optic sensing architectures based on sensitive nanofilms

## Nanostructured coatings onto tapered ends of optical fibers


tapered ends

### Glucose sensing



Jesús M. Corres, Anai Sanz, Francisco J. Arregui, Ignacio R. Matías and Joaquín Roca. "Fiber optic glucose sensor based on bionanofilms". *Sensors and Actuators B*. Not published yet

# Summary

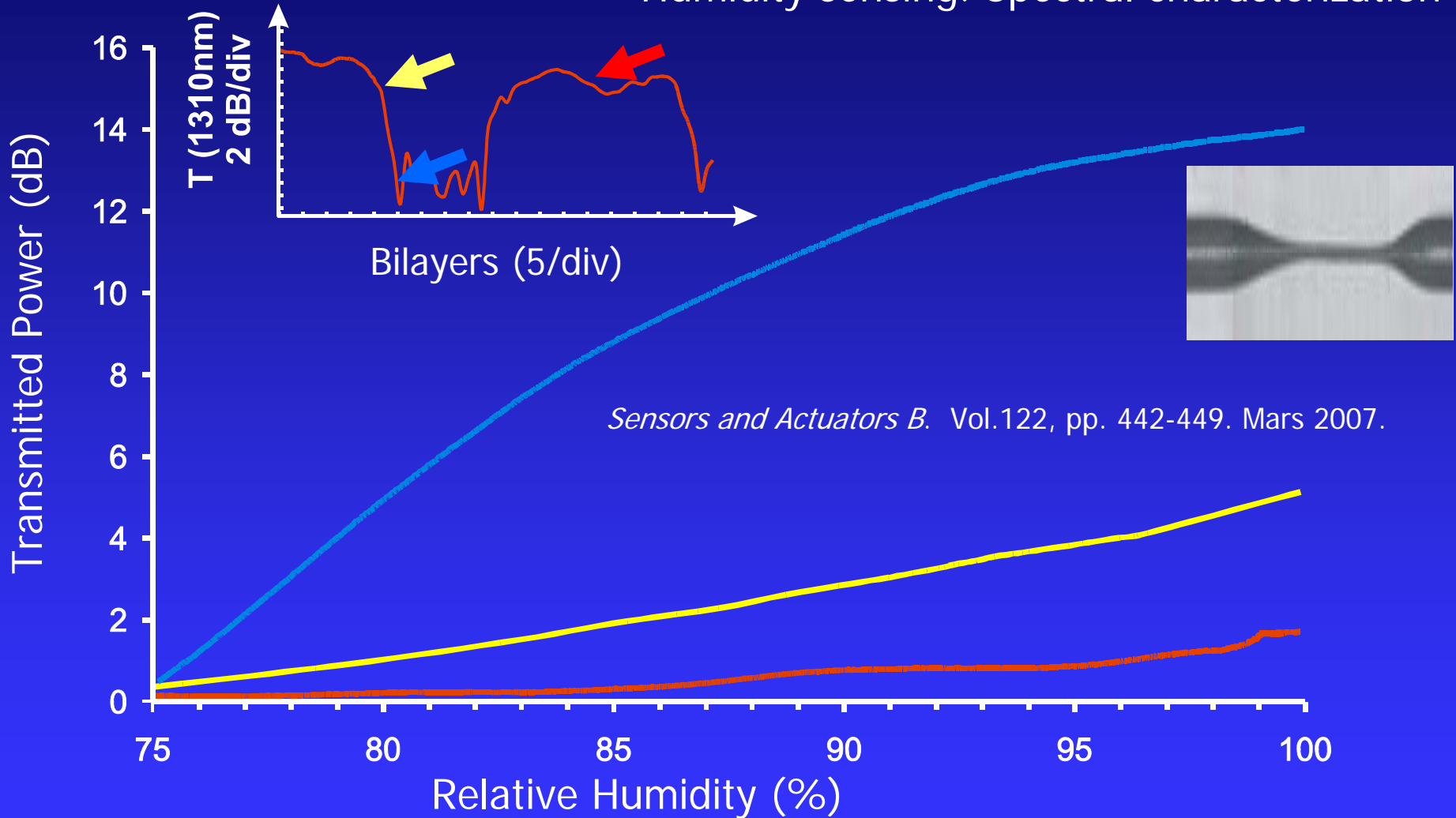
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# Novel fiber optic sensing architectures based on sensitive nanofilms

## Sensors based on Tapered Optical Fibers

TOF

Humidity sensing. Spectral characterization

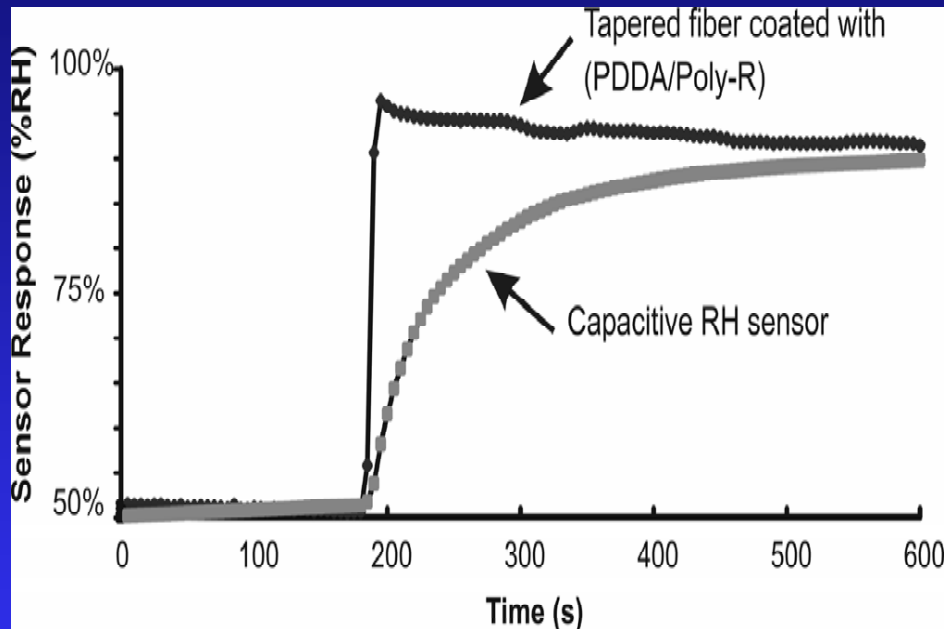


Experimental response of a 20m waist diameter TOF-based humidity sensors to RH corresponding to three working points of coating thicknesses: 23, 26 and 62 bilayers

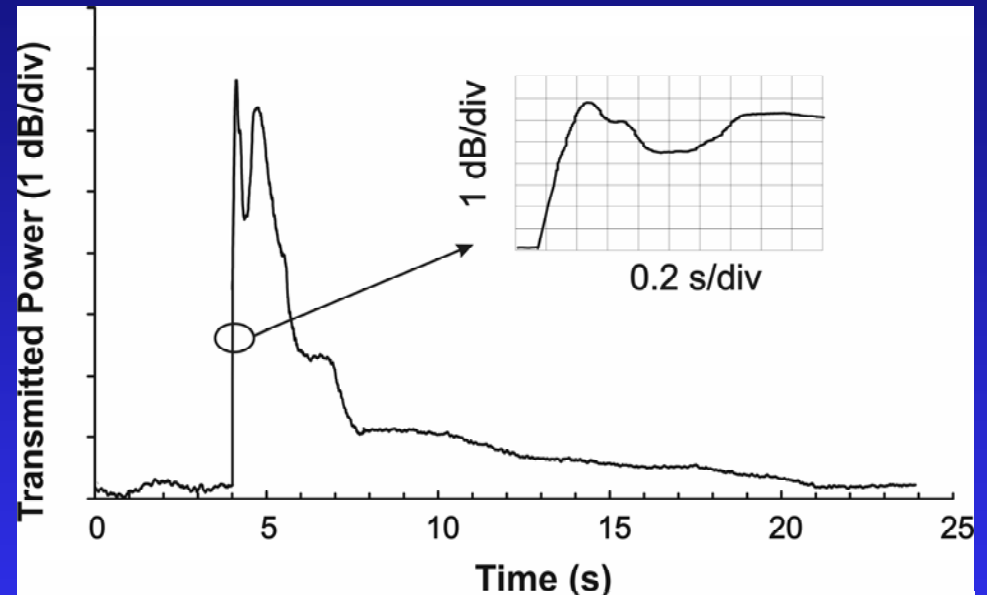
# Novel fiber optic sensing architectures based on sensitive nanofilms

## Sensors based on Tapered Optical Fibers

### Humidity sensing. Response time



Humidity response time compared to a commercial one  
(Blue box humidity sensor T12000/6, from Philip Harris)



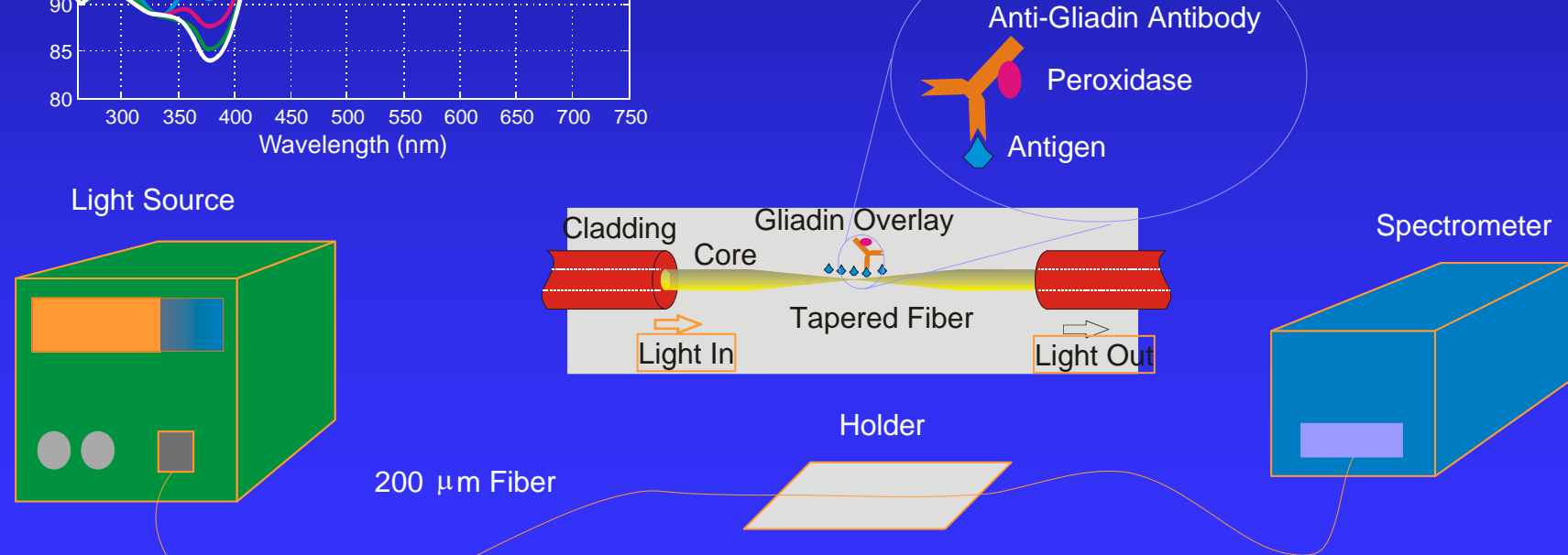
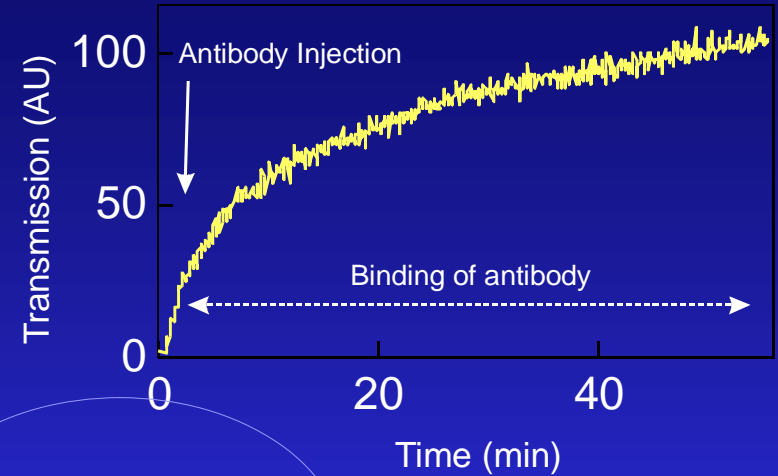
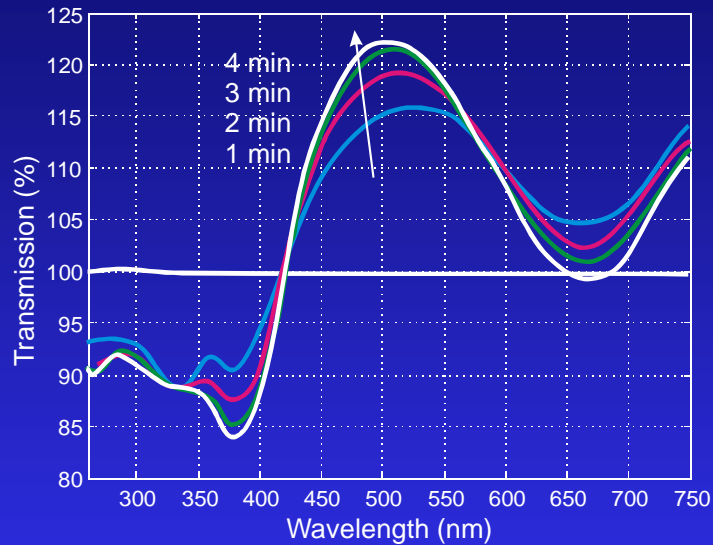
Experimental response to the human breath



# Novel fiber optic sensing architectures based on sensitive nanofilms


## Sensors based on Tapered Optical Fibers

### Gliadin sensing (gluten detection)



Tapered Optical Fiber Biosensor for the Detection of Anti-Gliadin Antibodies". IEEE Sensors Conference 2007.

# Summary

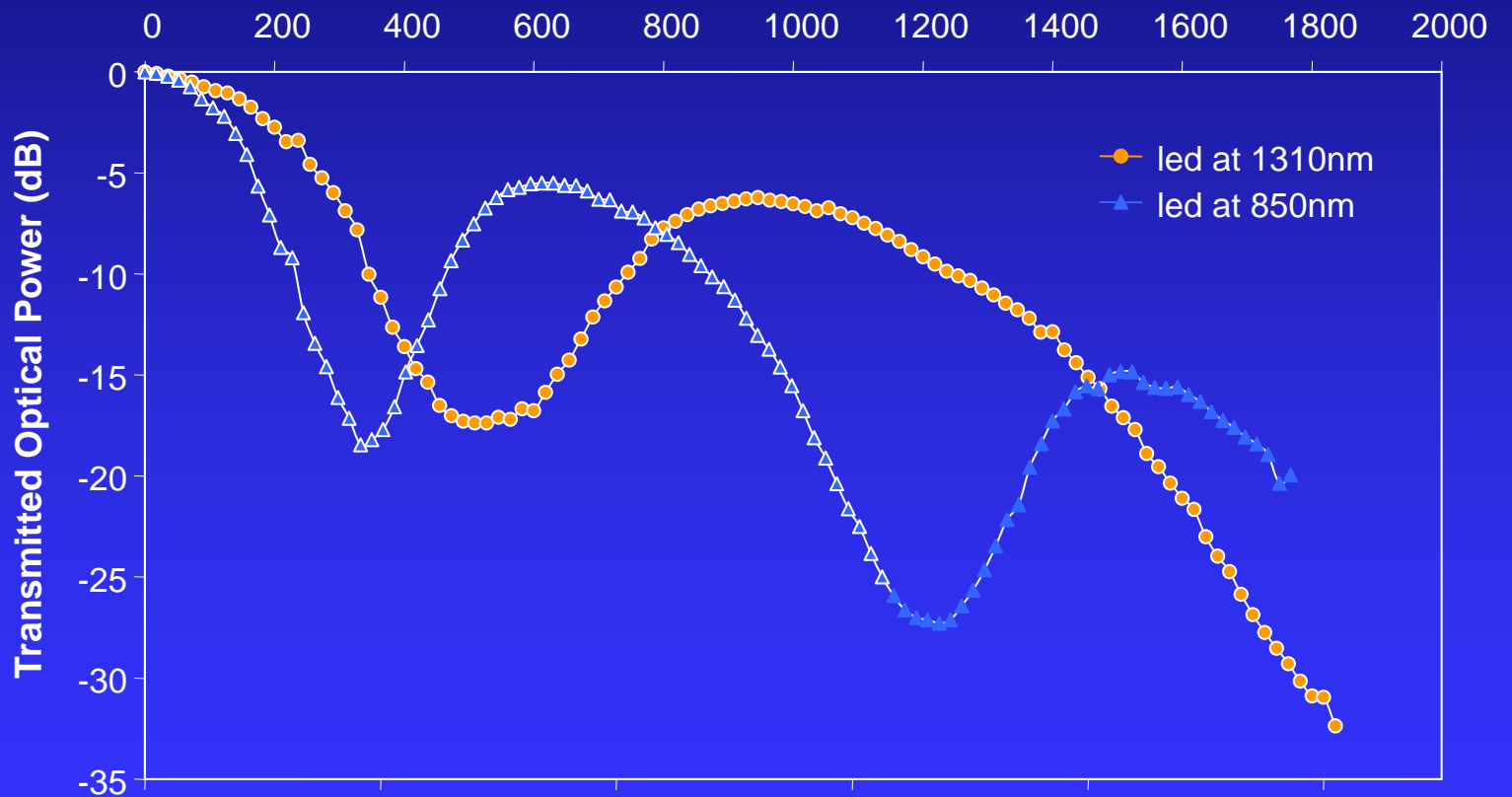
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## Sensors based on Hollow core fibers

Humidity sensing based on evanescent wave

### Spectral characterization

Thickness of nanofilm (nm)



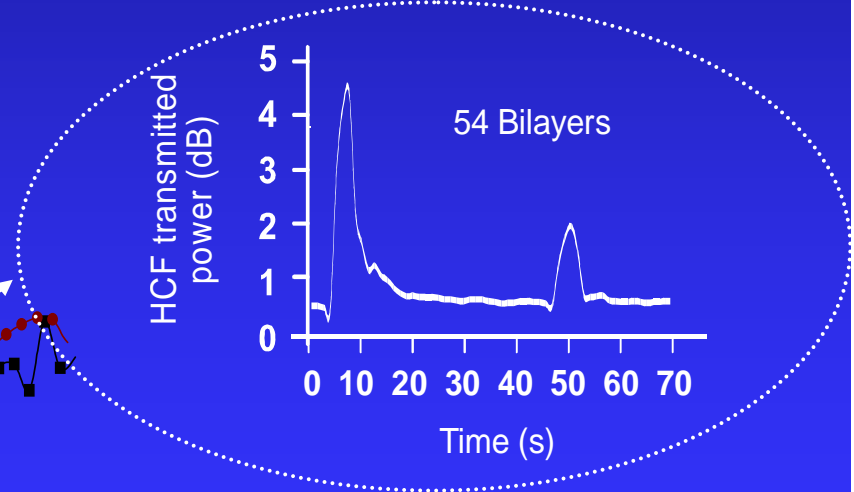
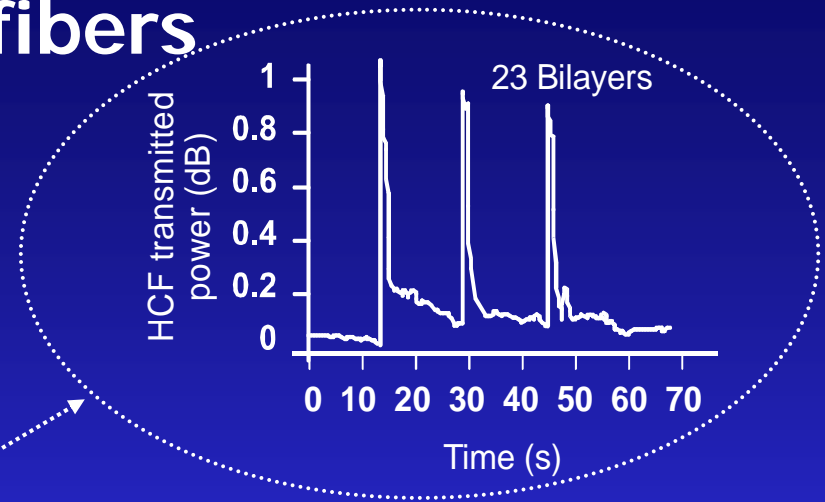
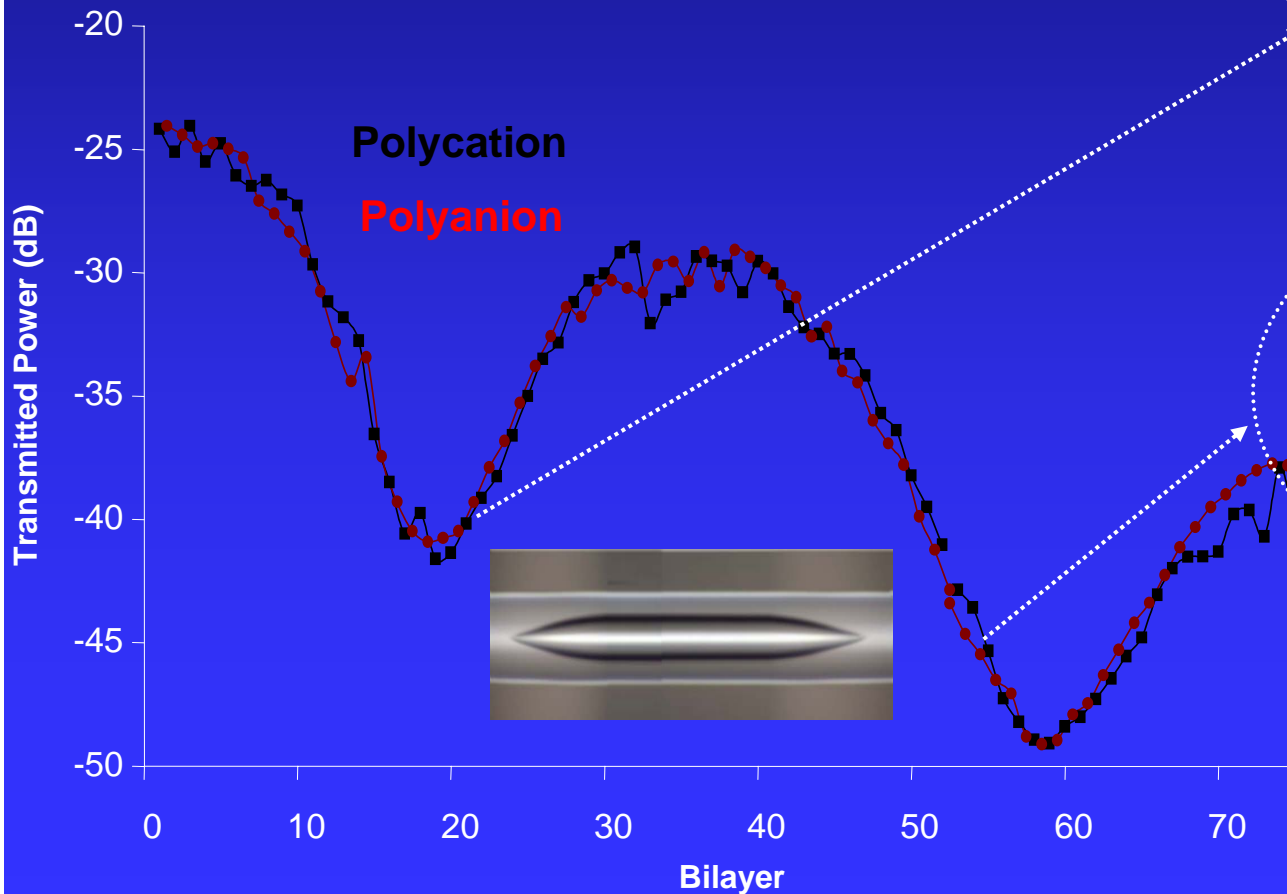
"Nanofilms on hollow core fiber-based structures: an optical study".  
*IEEE J. of Light. and Tech.* Vol. 24 (5); pp. 2100-2107. May 2006

Number of bilayers


## Sensors based on Hollow core fibers

Humidity sensing based on evanescent wave

breathing monitoring



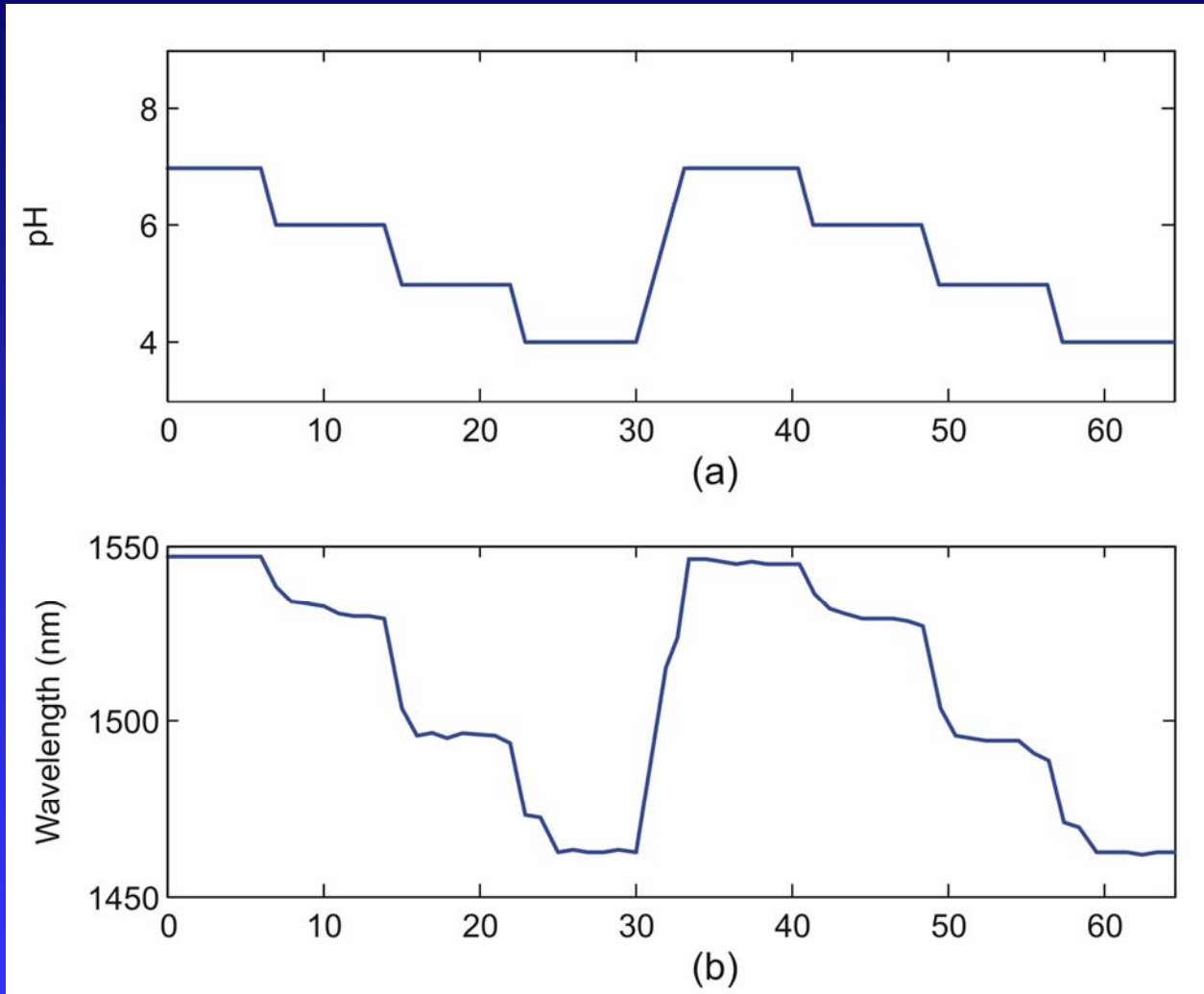
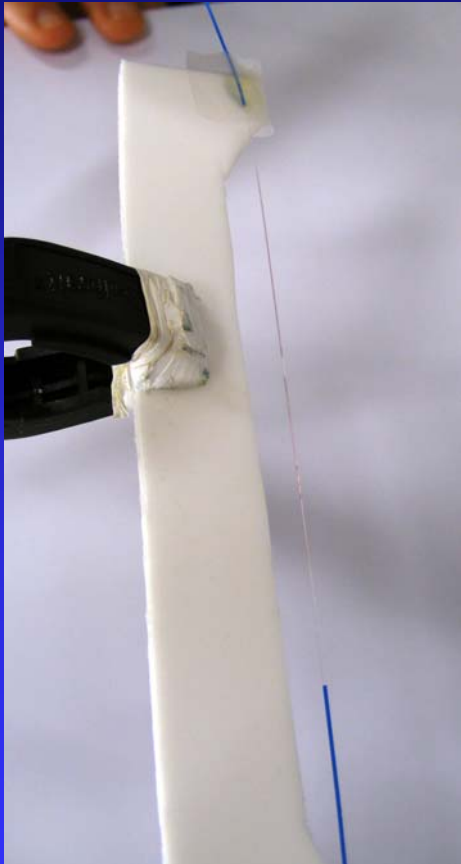
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# Novel fiber optic sensing architectures based on sensitive nanofilms


## Nanostructured coatings on Long Period Gratings: a pH sensor

LPG overlays



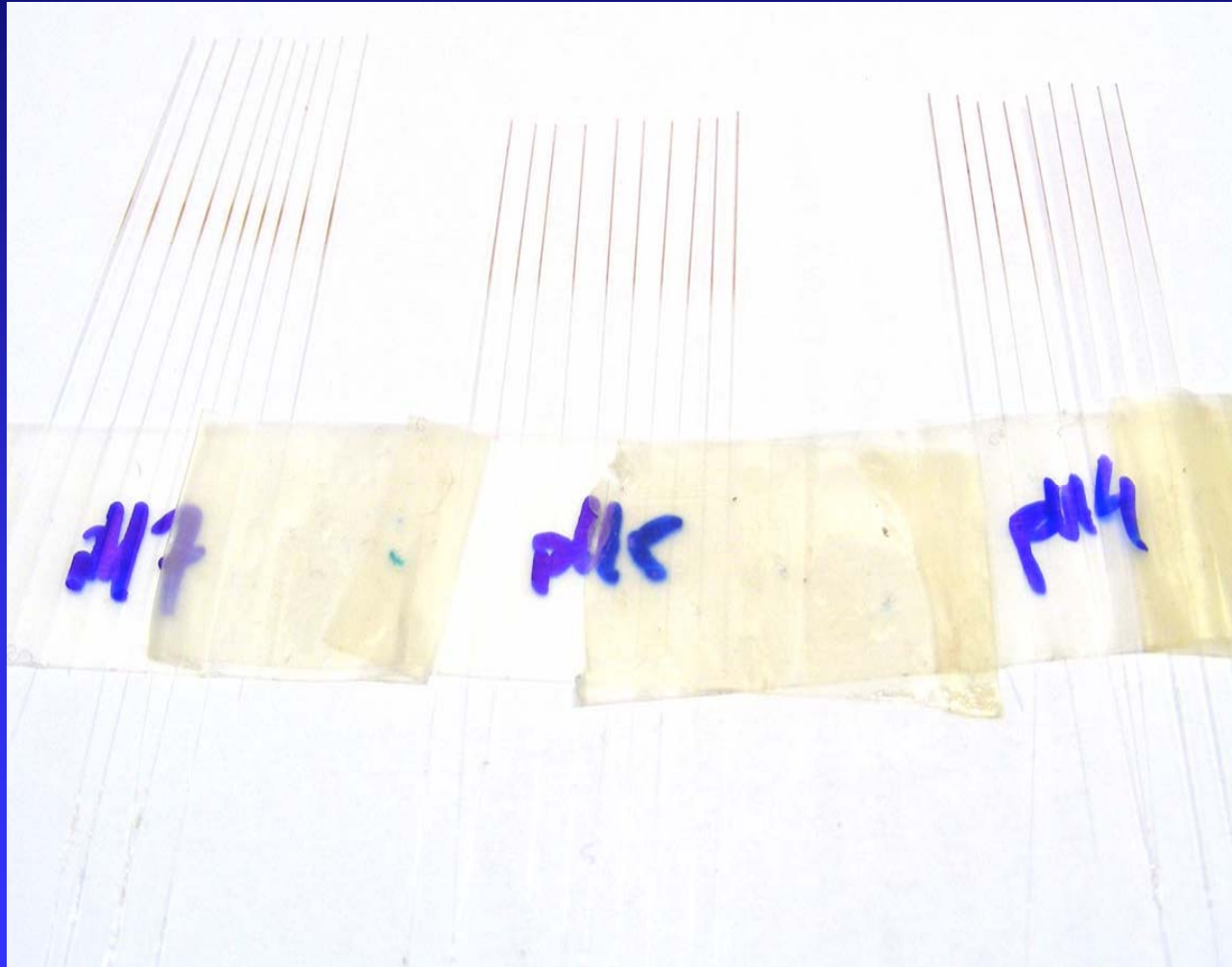
Optical response of one of the attenuation bands of a LPG coated with [PAH/PAA] coatings when is submitted to pH changes

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## Optical fiber microgratings

gratings



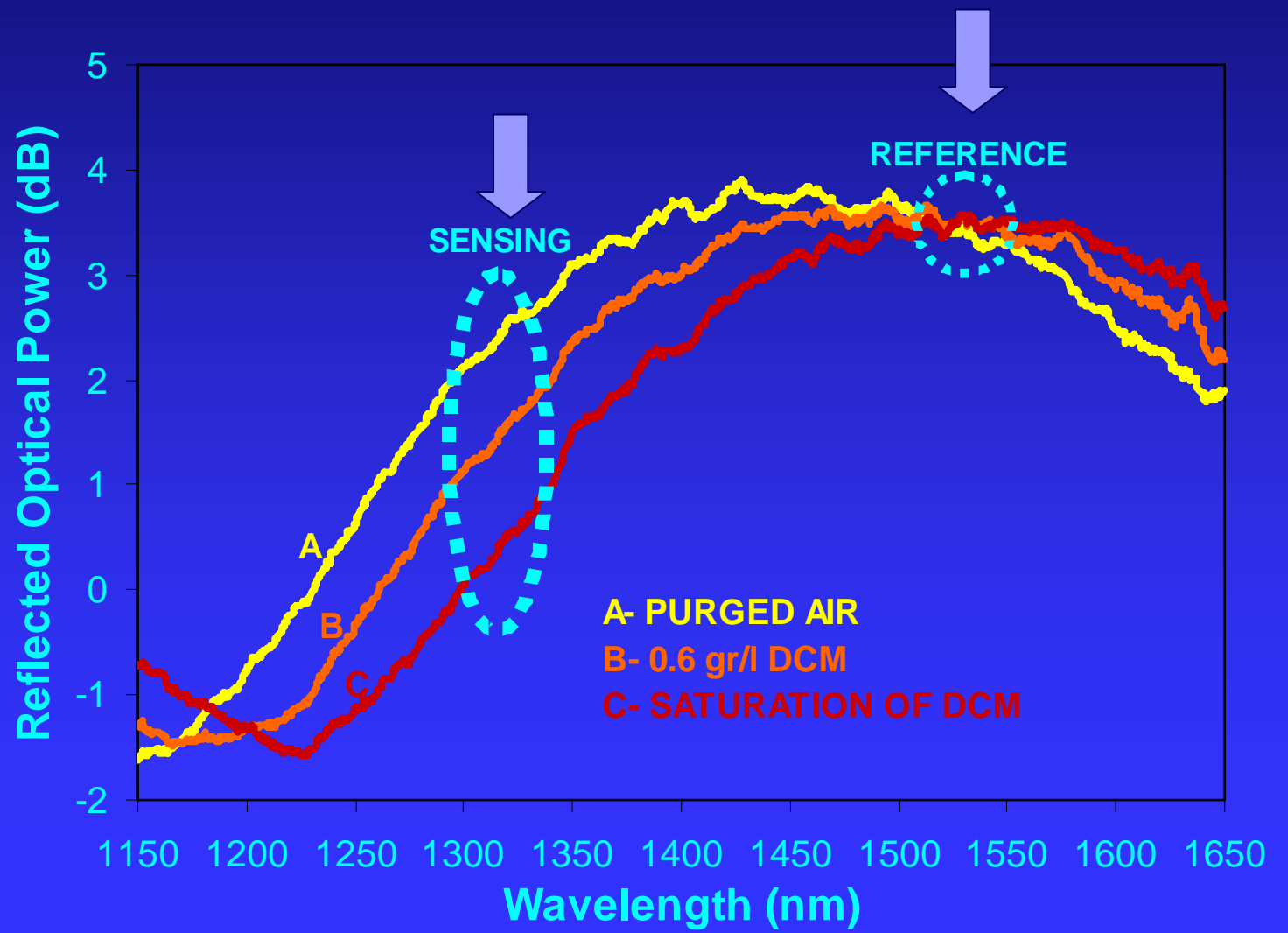
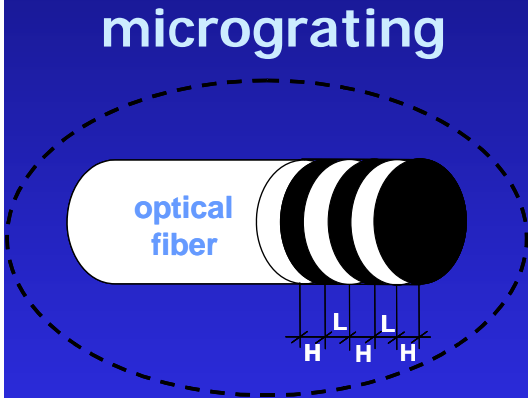
Fabrication of Microgratings on the Ends of Standard Optical Fibers by the Electrostatic Self-Assembly Monolayer Process. *Optics Letters*, vol. 26 (3); pp. 131-133, 2001.



# Novel fiber optic sensing architectures based on sensitive nanofilms

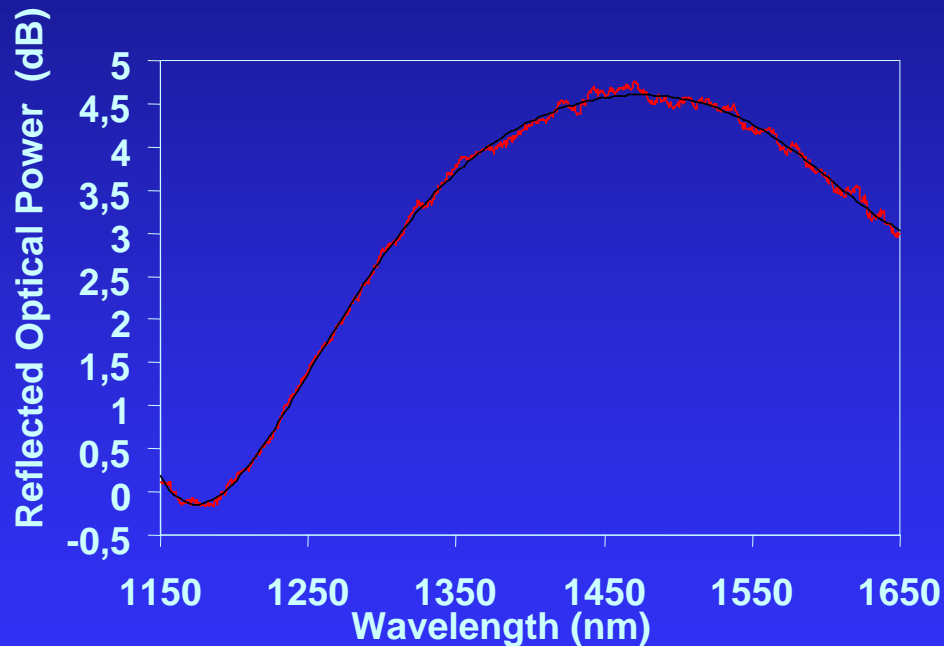
## EXPERIMENTAL RESULTS -VOLATILE ORGANIC COMPOUND SENSOR DICHLOROMETHANE (DCM)

gratings

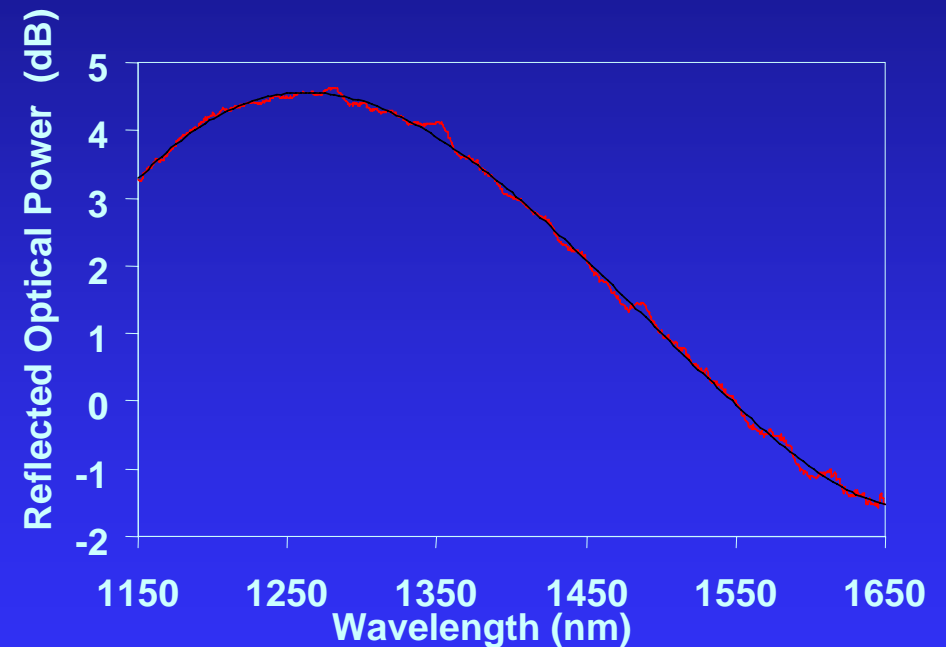


## Different spectral responses

Sensor 1



Sensor 2

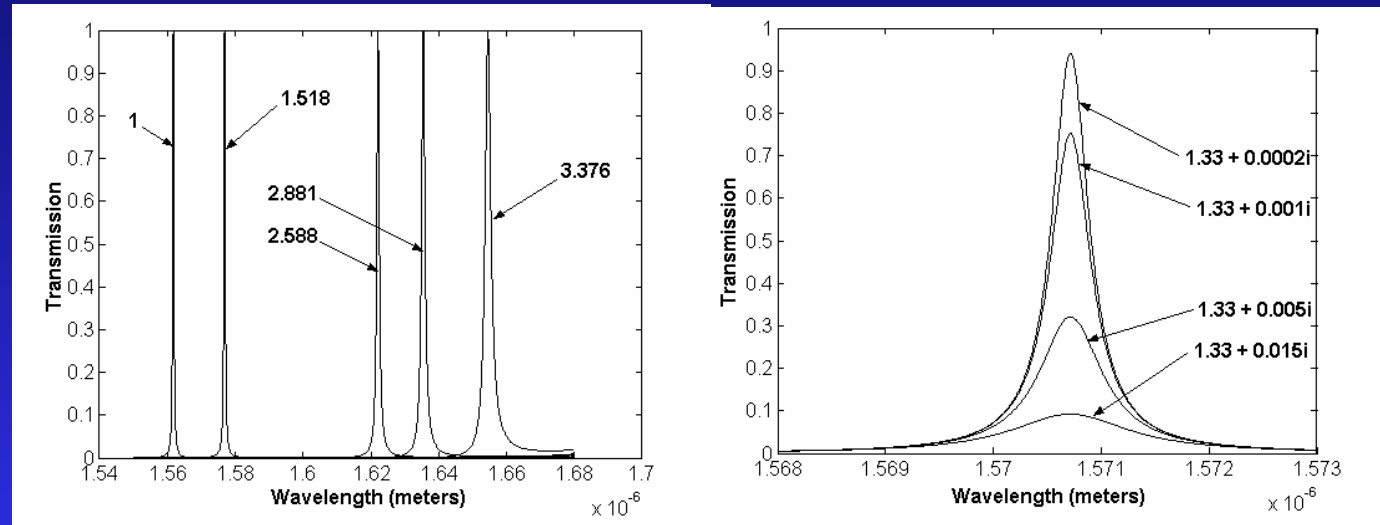


# Novel fiber optic sensing architectures based on sensitive nanofilms

## 1D PBG with defects

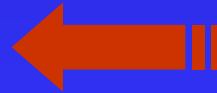
others

Refractometer



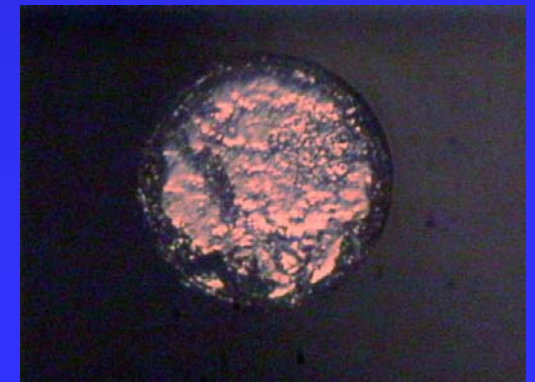
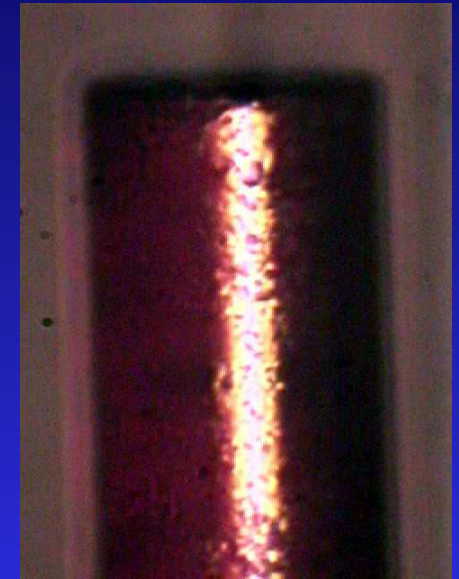
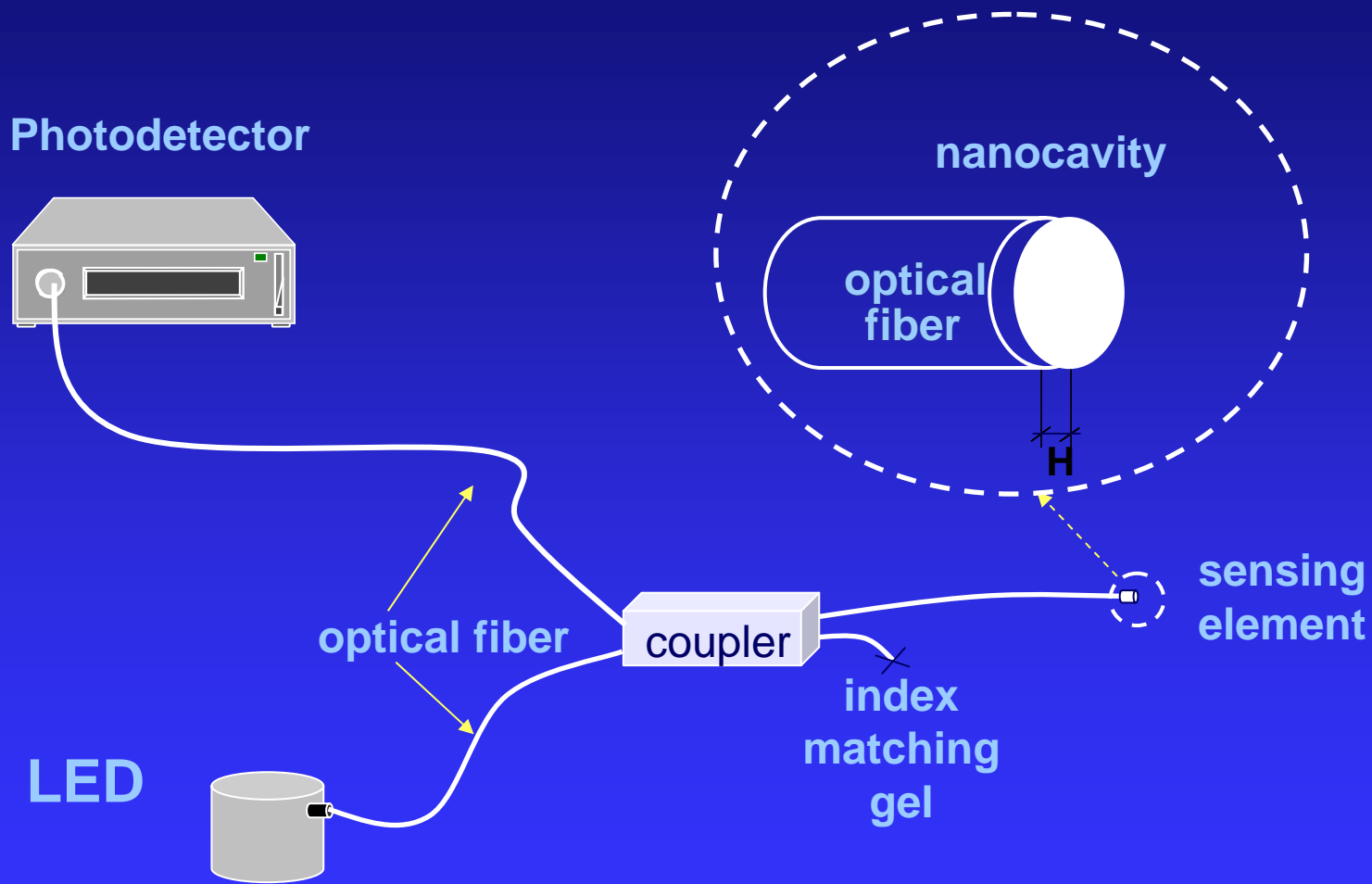
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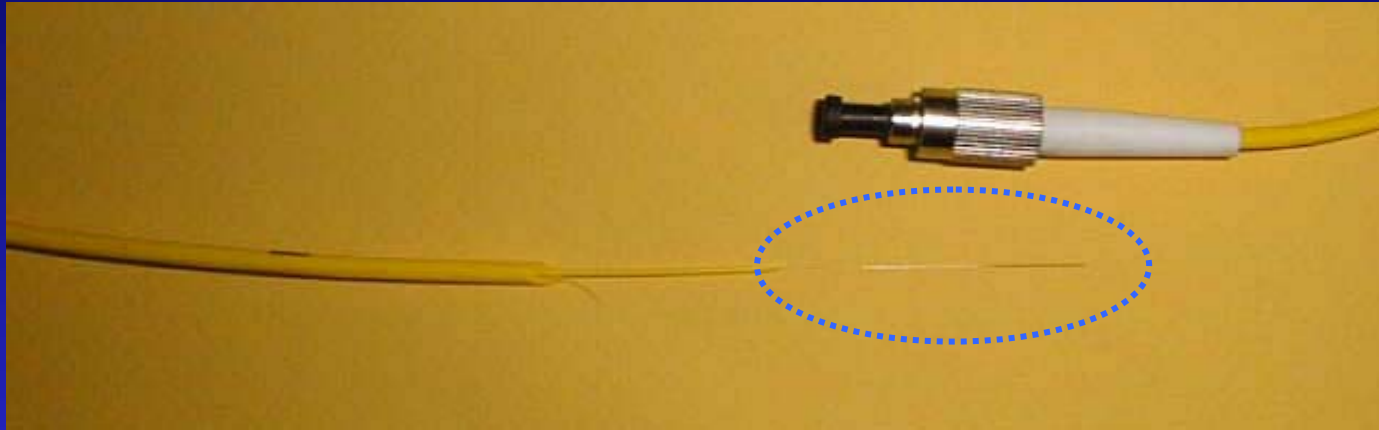
# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Experimental set-up



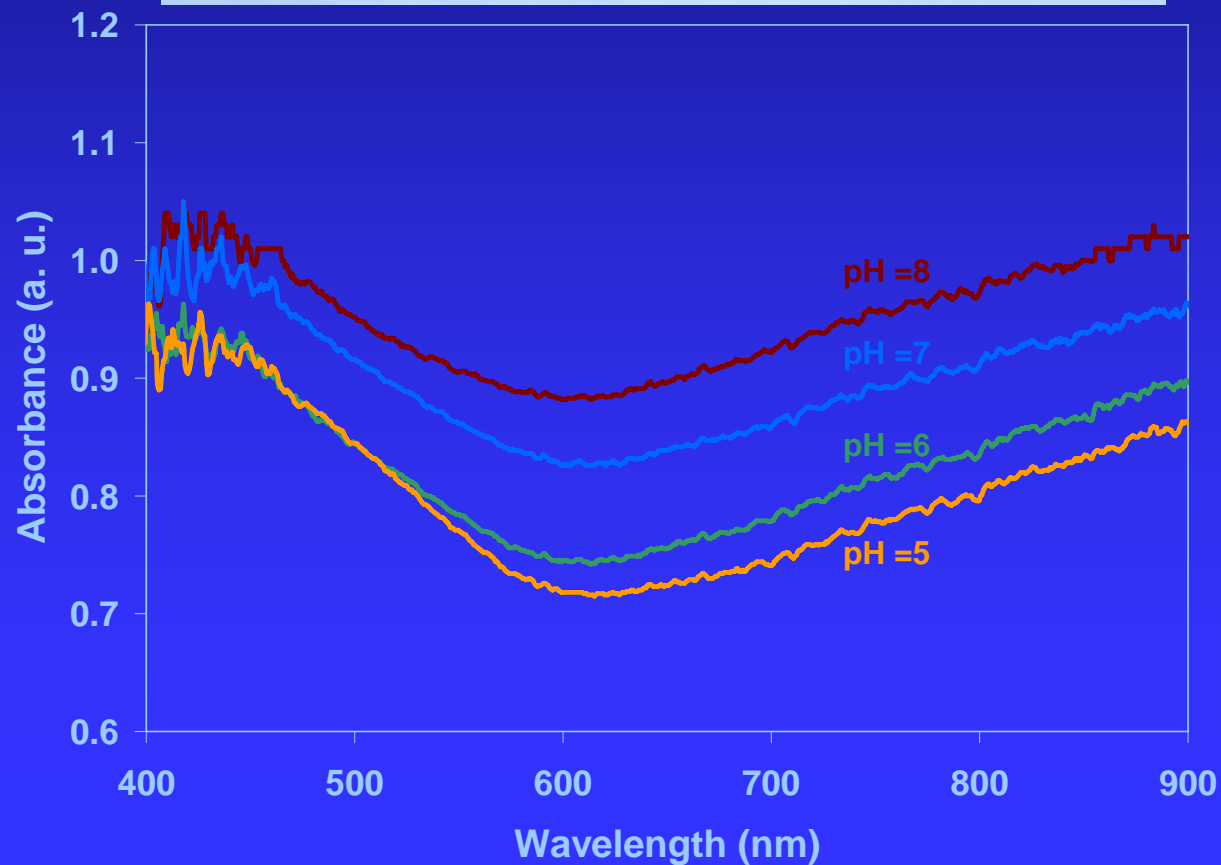
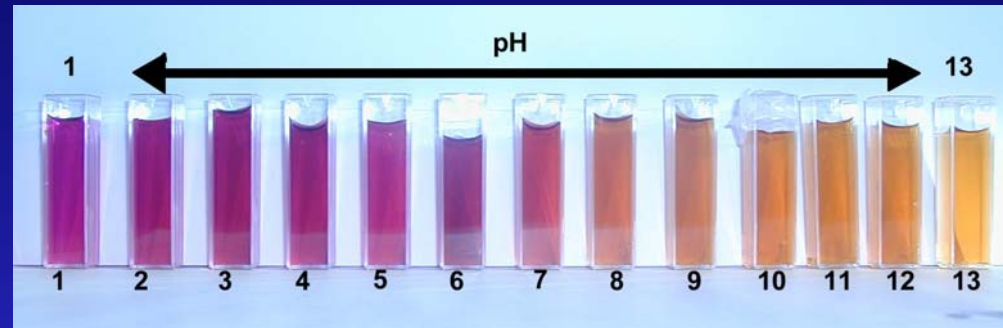
nanoFabry-Perots

# Novel fiber optic sensing architectures based on sensitive nanofilms



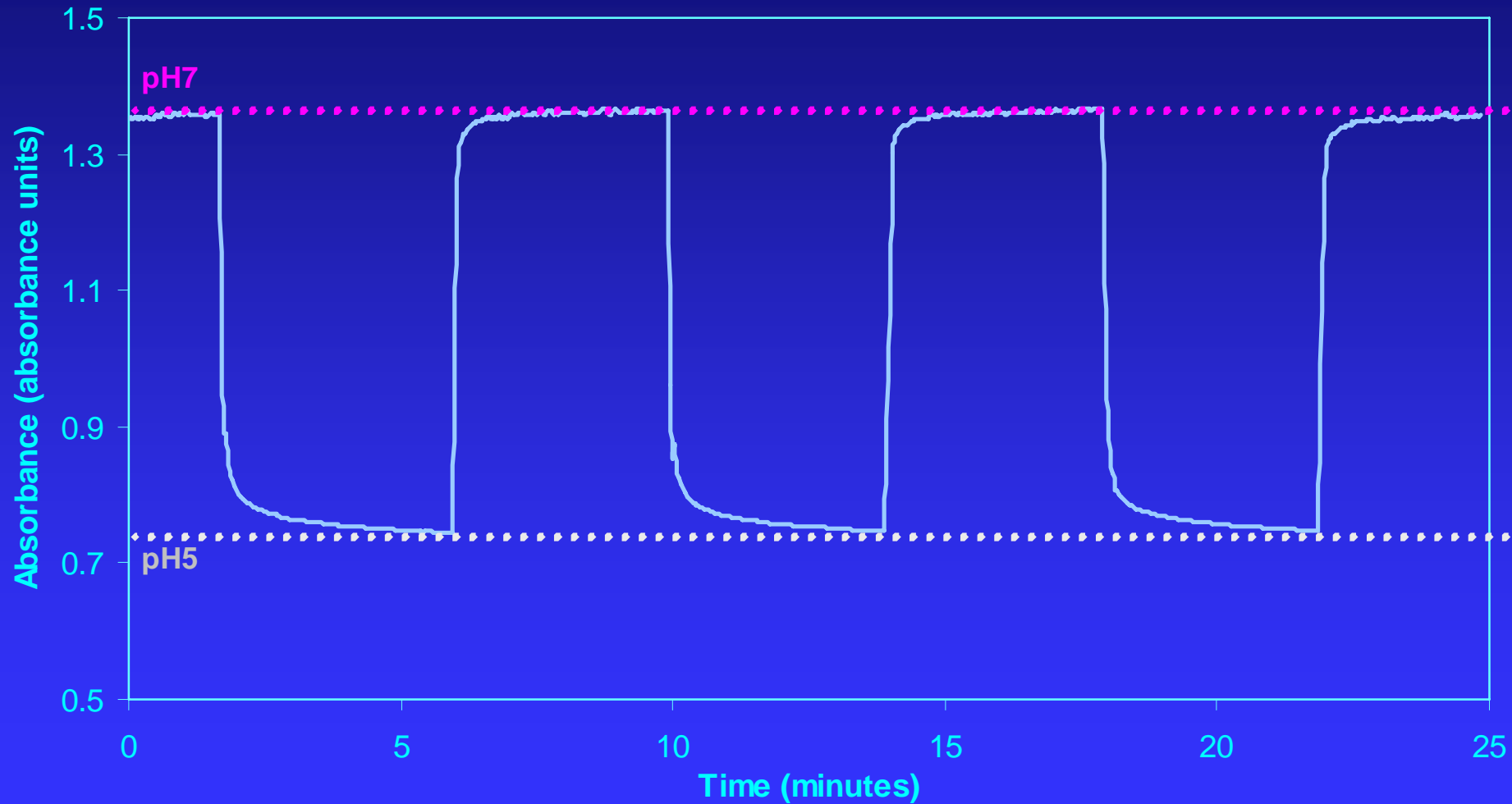
# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. pH Sensors



# Novel fiber optic sensing architectures based on sensitive nanofilms

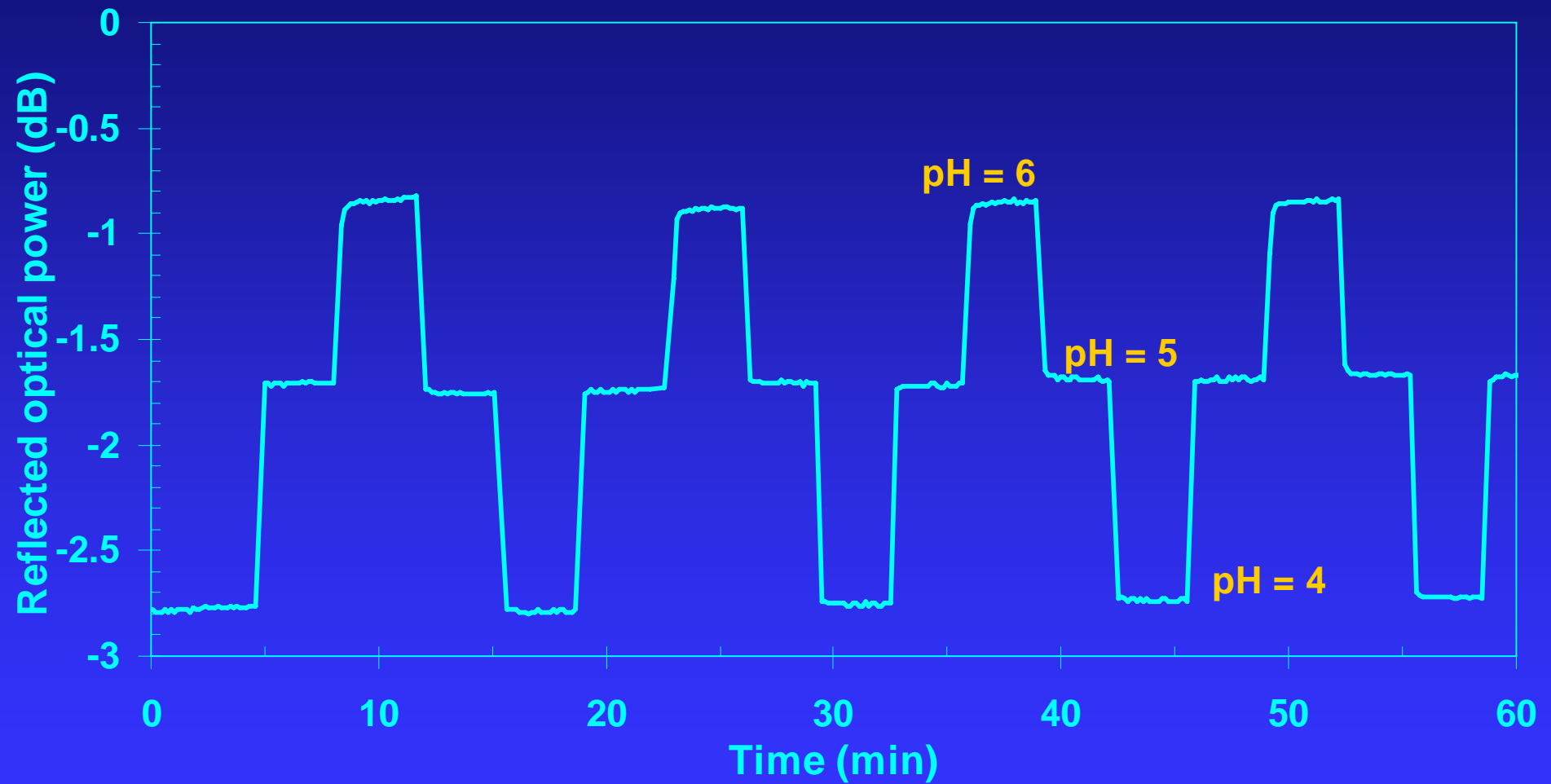
## NFP Cavities. pH Sensors





## NFP Cavities. pH Sensors

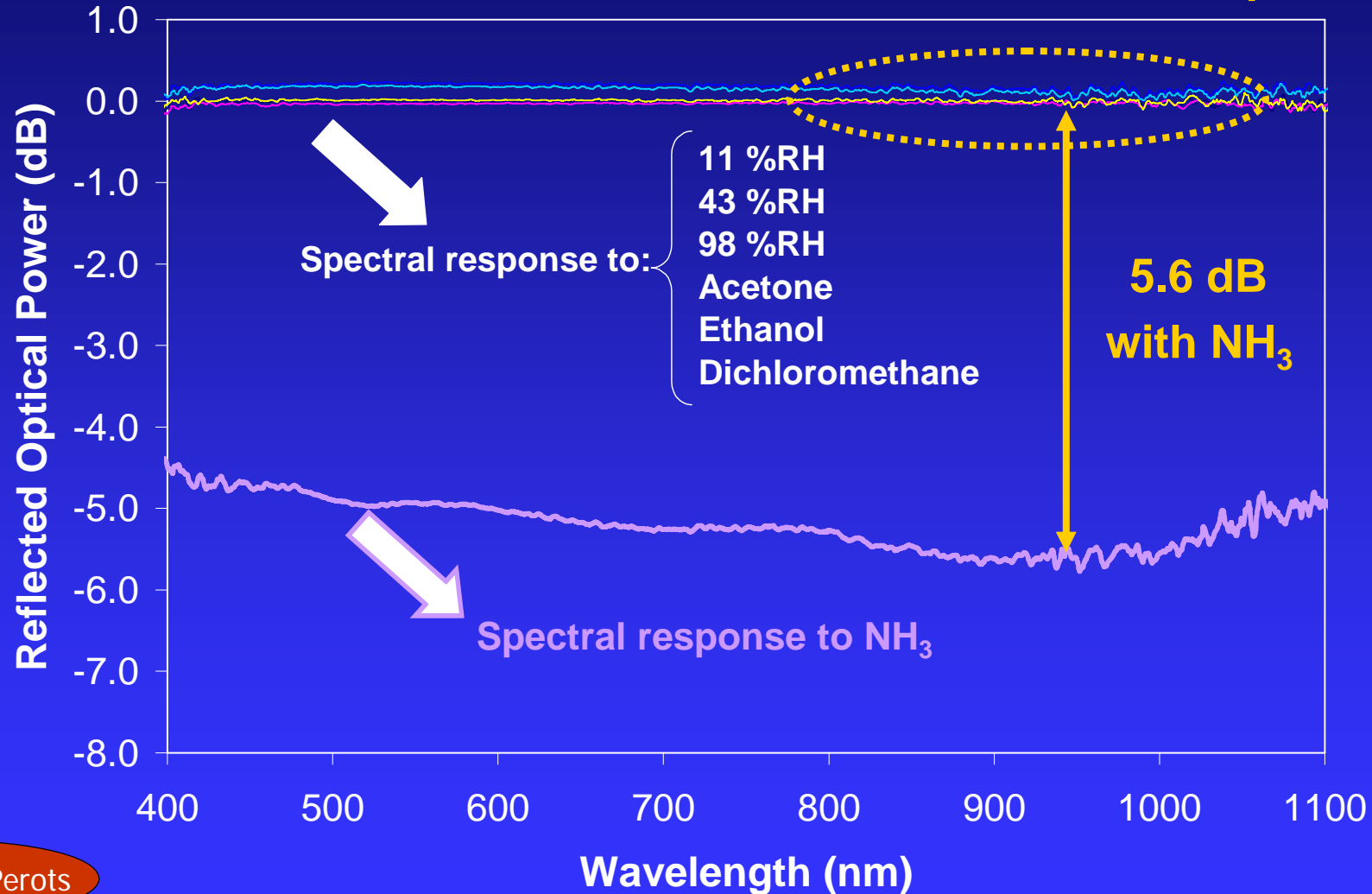
nanoFabry-Perots



# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Ammonia sensors

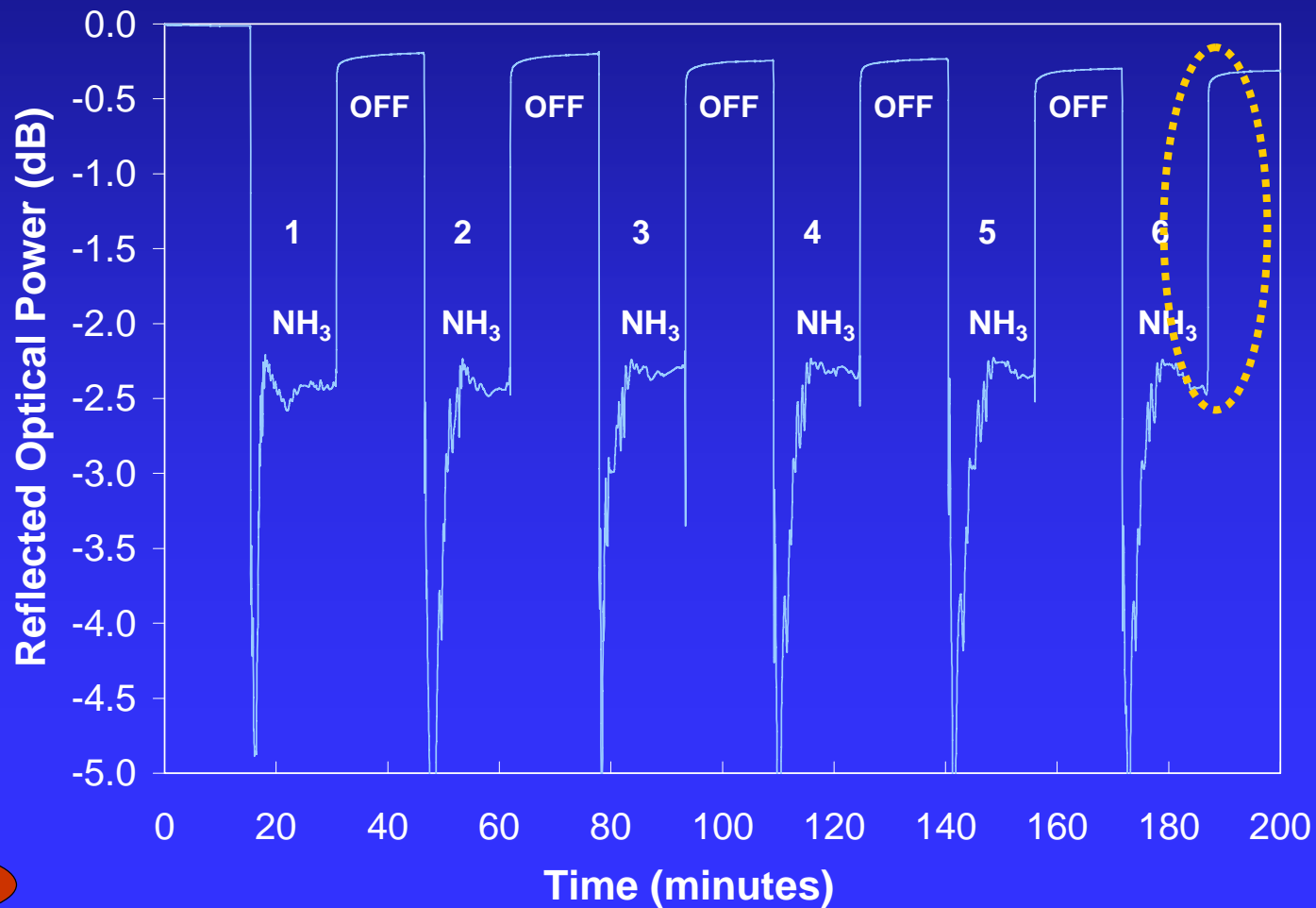
< 4% of cross-sensitivity  
to other compounds



nanoFabry-Perots

## NFP Cavities. Ammonia sensors

Dynamic response of a 25 bilayers sensor to Ammonia

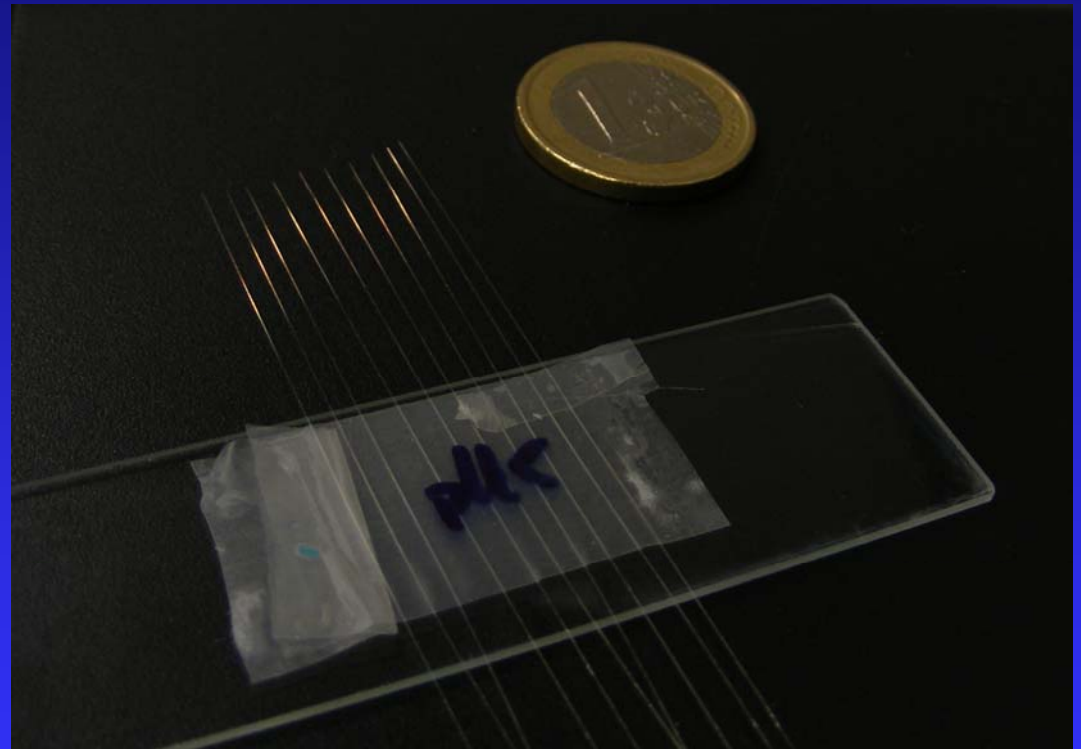
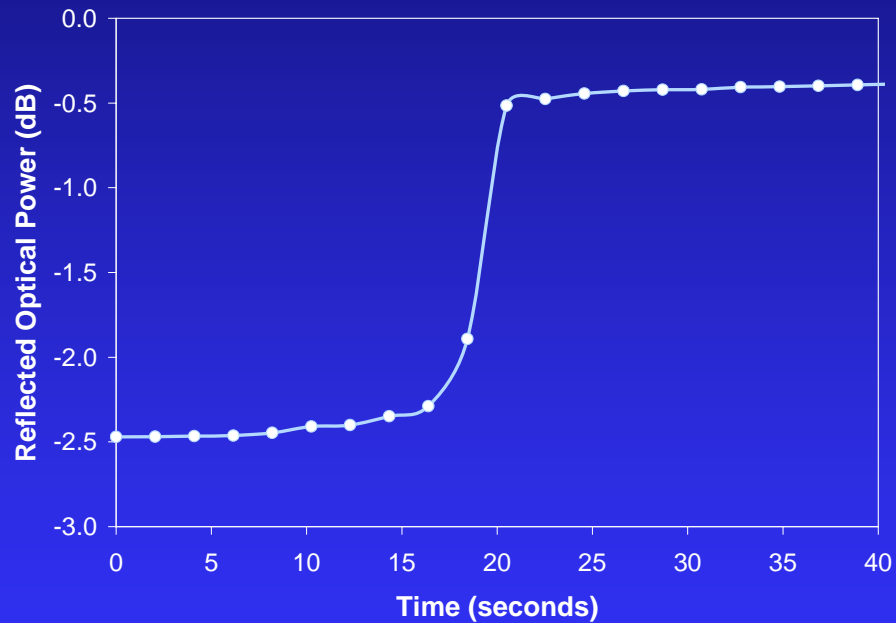


# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Ammonia sensors

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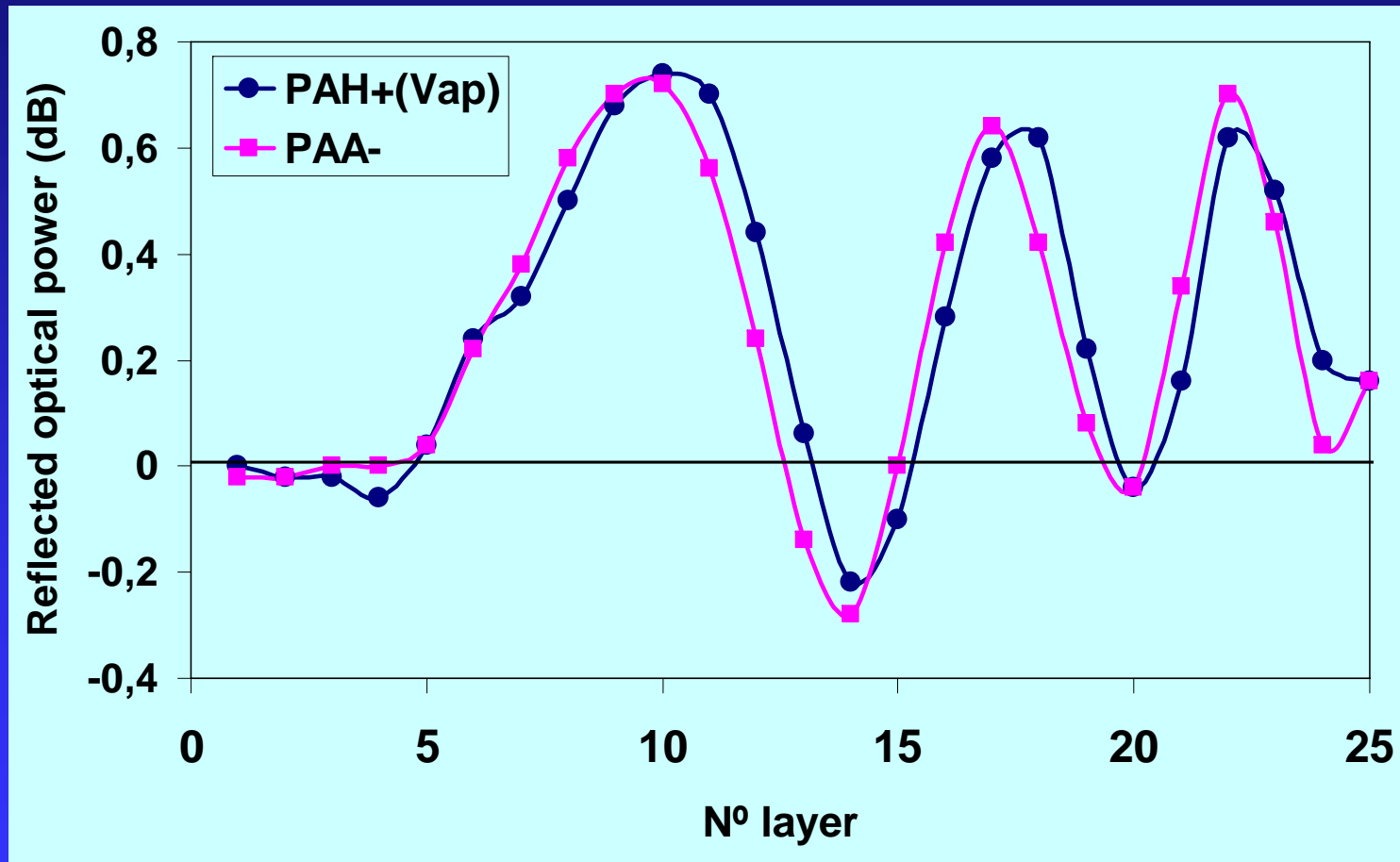
Recovery time < 4 seconds



# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Volatile organic compounds sensors

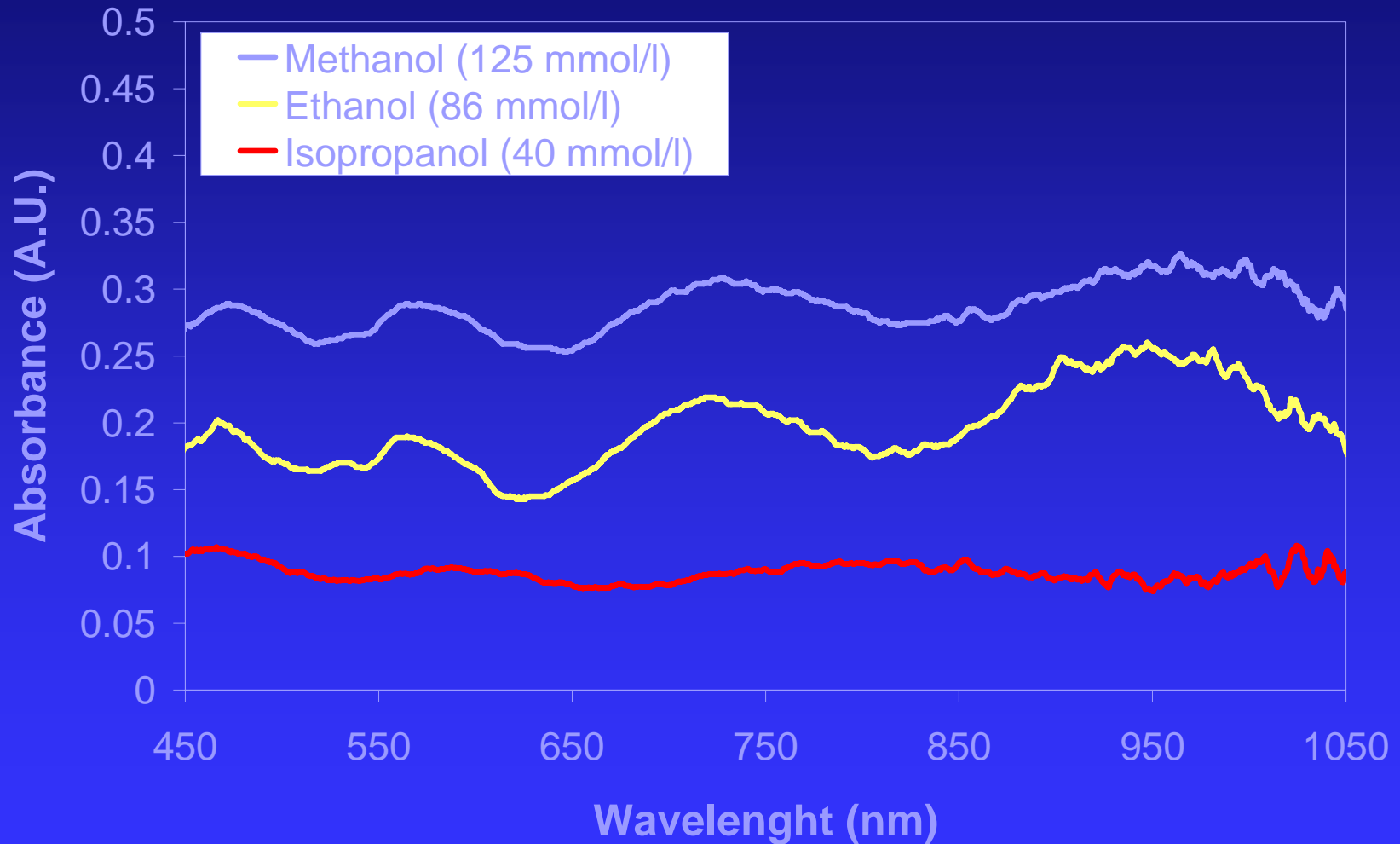
[Vap+PAH+/PAA-]



# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Volatile organic compounds sensors

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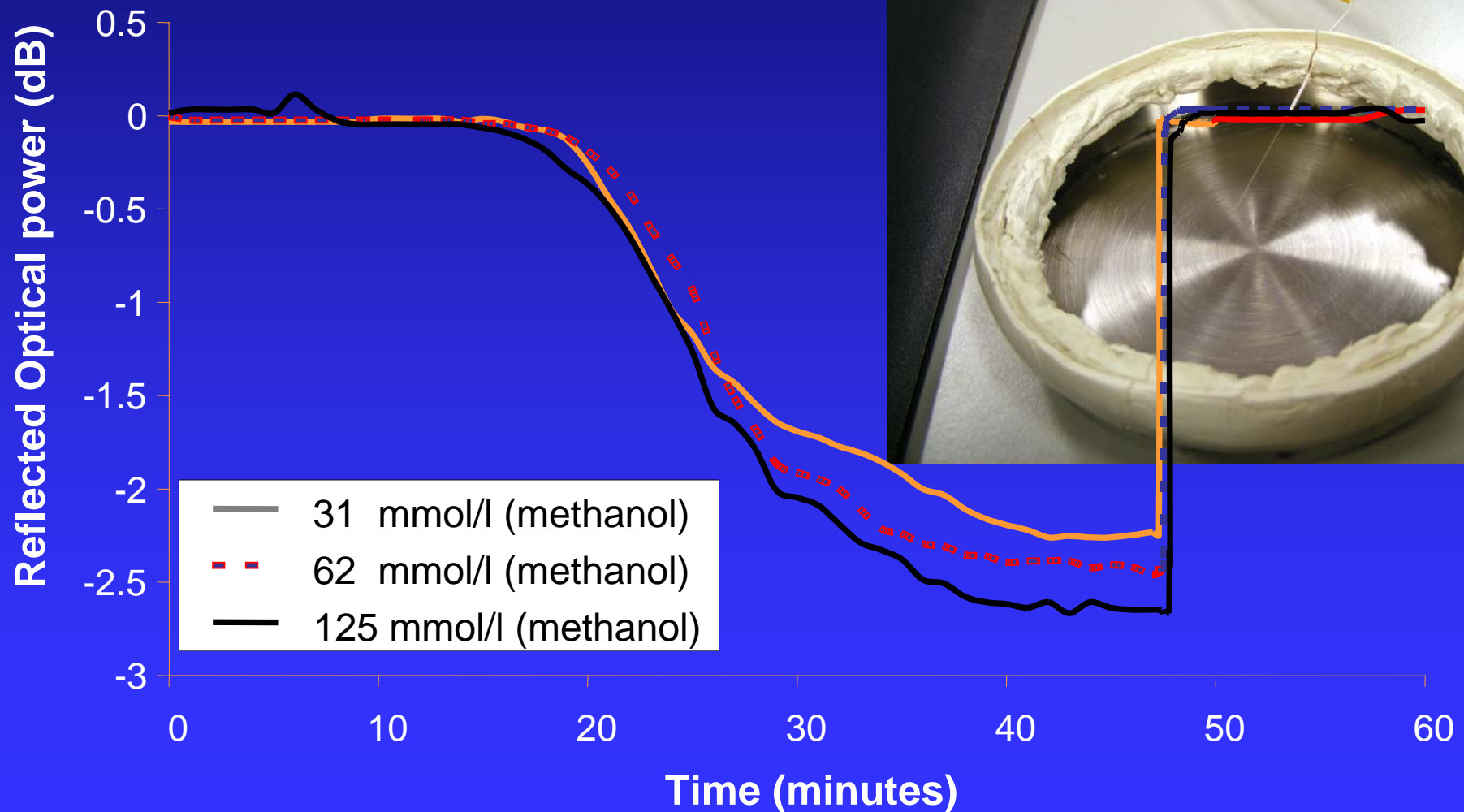
Absorbance spectra of the sensor after 40 minutes exposure

# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Volatile organic compounds sensors

nanoFabry-Perots

Response of the sensor for different methanol concentrations

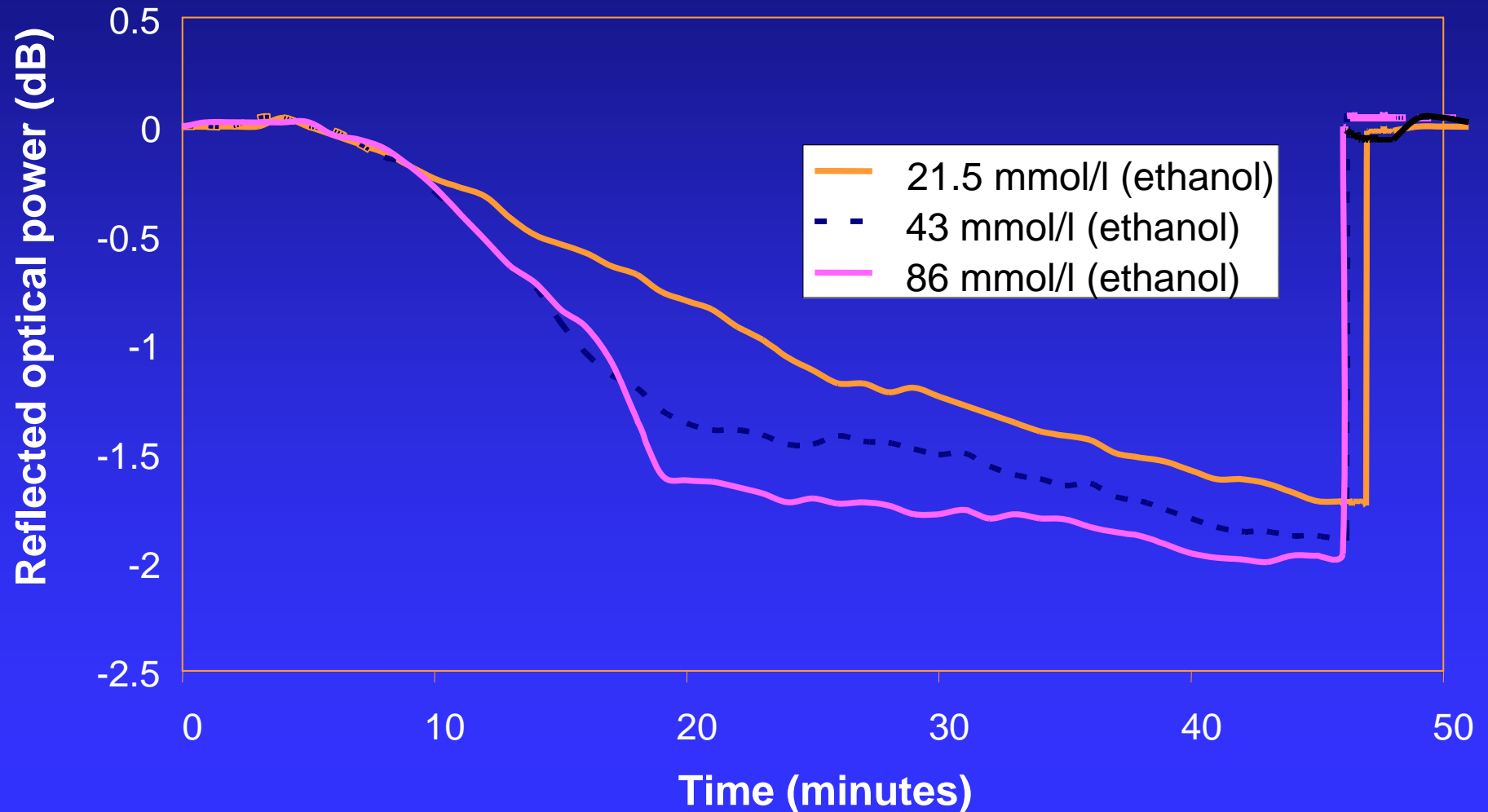


# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Volatile organic compounds sensors

nanoFabry-Perots

Response of the sensor for different ethanol concentrations

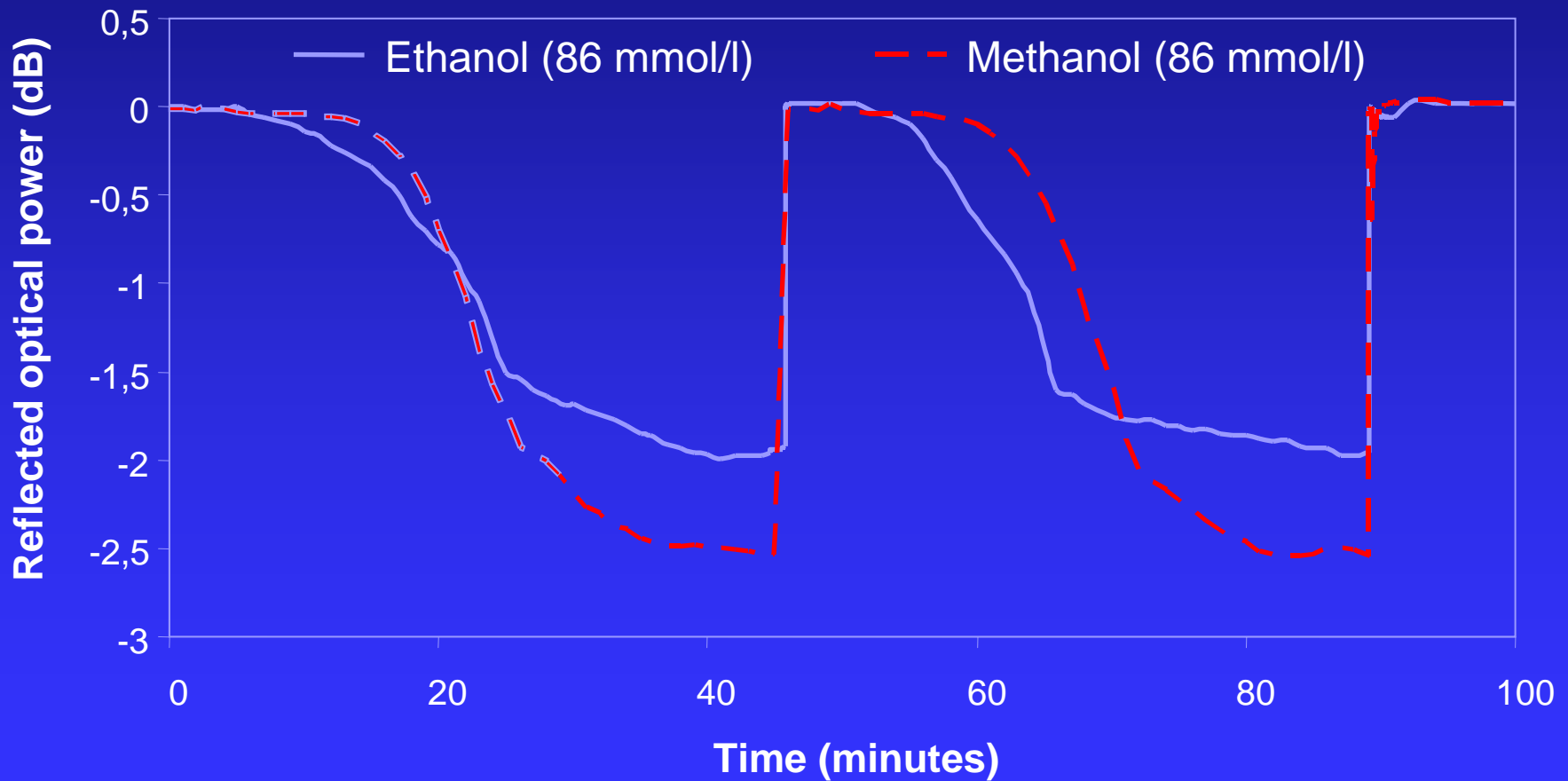




## NFP Cavities. Volatile organic compounds sensors

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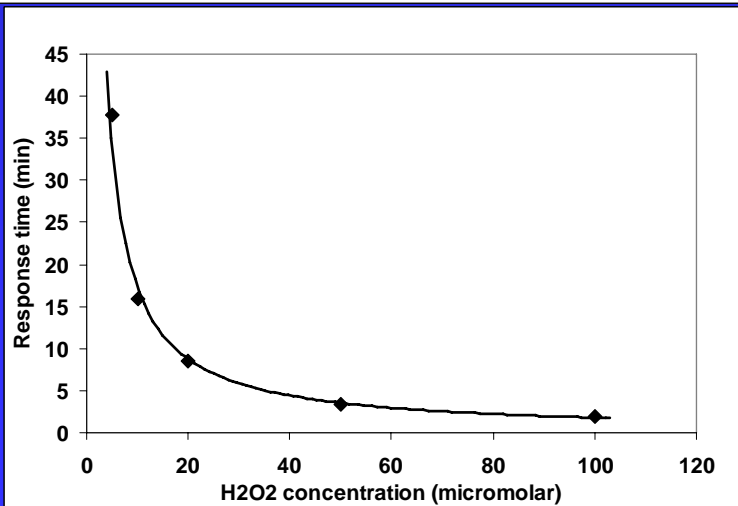
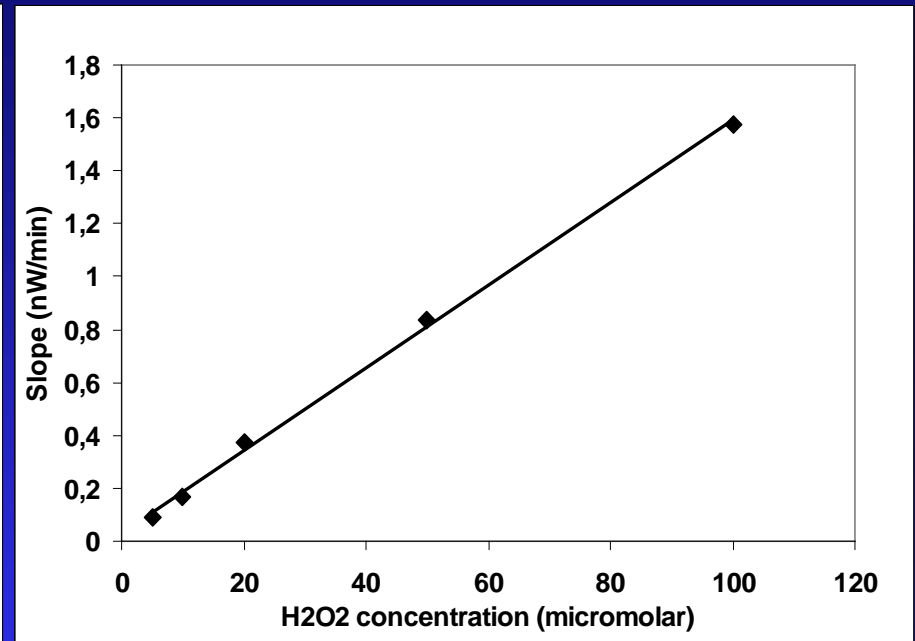
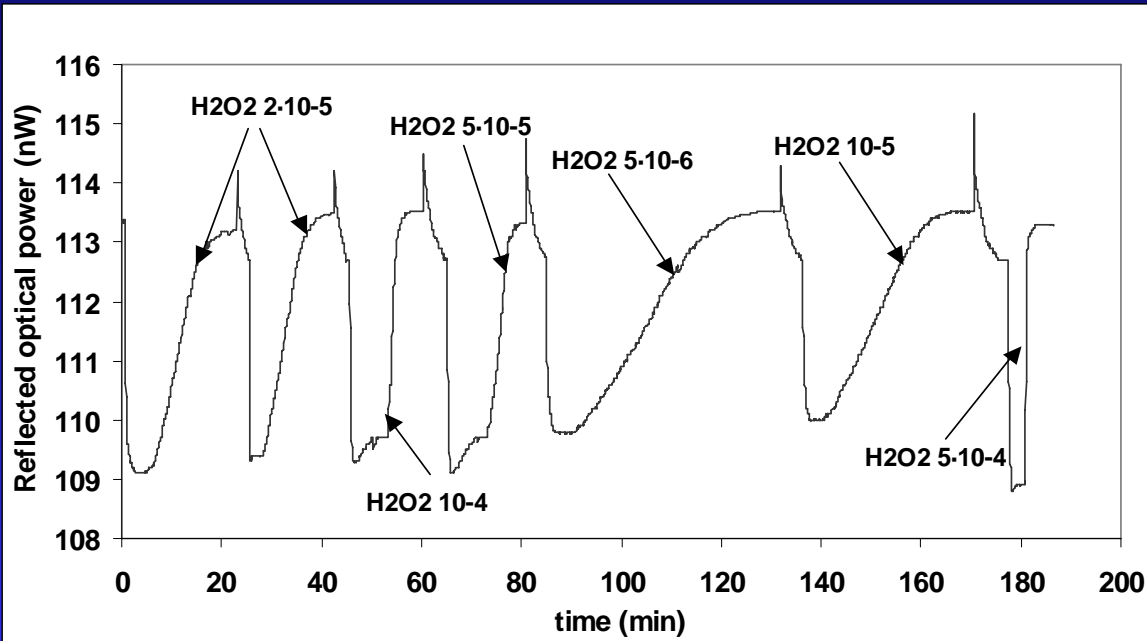
Comparison between ethanol and methanol



# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Hydrogen peroxide sensor

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Response of the sensor for different concentrations of hydrogen peroxide at pH 4:

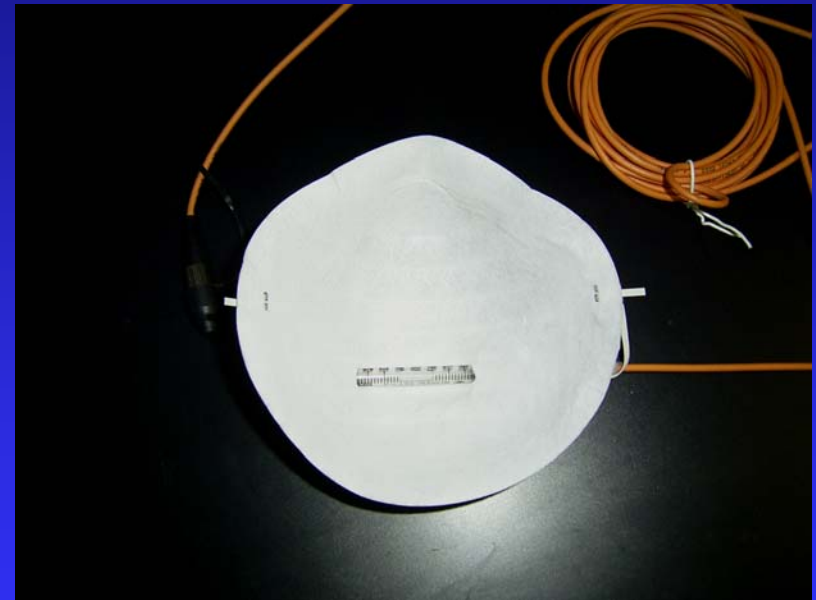
- 1.- Reflected optical power
- 2.- Slope of the change in the reflected power
- 3.- Response time

# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Human breathing



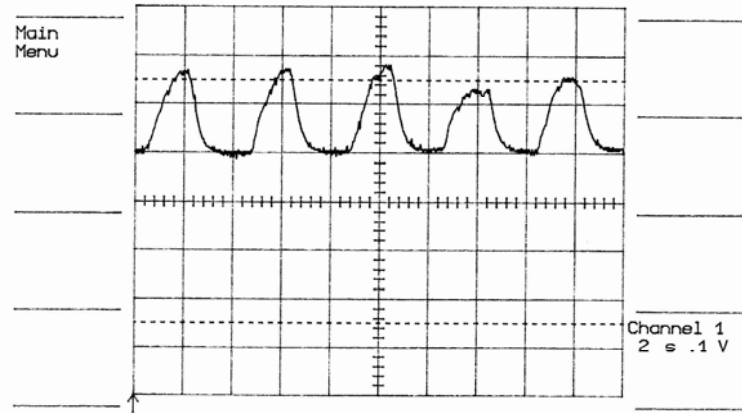
Sensor and opto-electronic units



Face mask and sensor

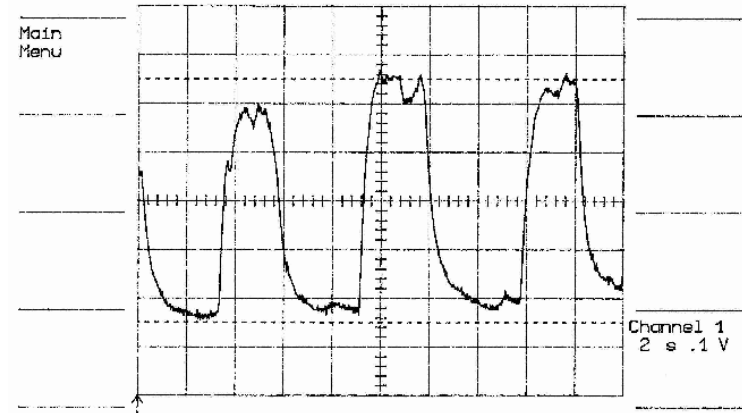
# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Human breathing



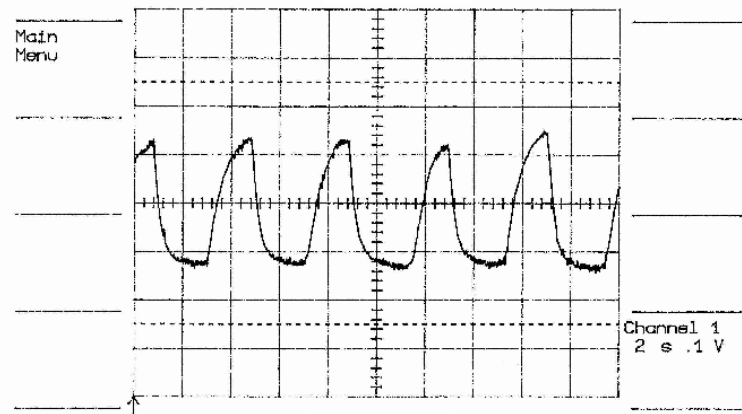
(a)

Ch1 .1 V =  
T/div 2 s Ch2 20mV =  
Trig 5.00 div+CHAN 1~



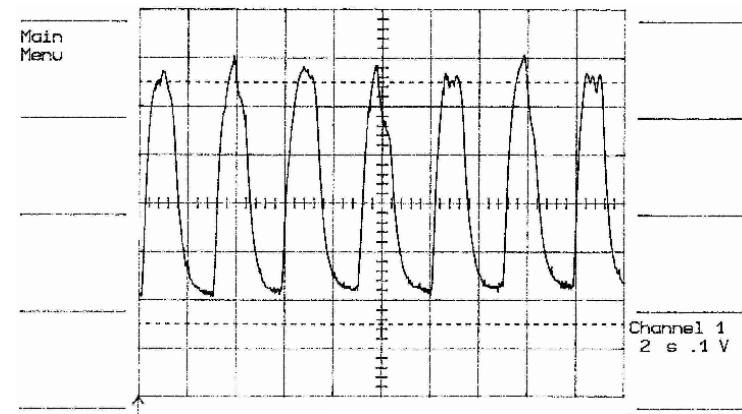
(b)

Ch1 .1 V =  
T/div 2 s Ch2 20mV =  
Trig 5.00 div+CHAN 1~



(c)

Ch1 .1 V =  
T/div 2 s Ch2 20mV =  
Trig 5.00 div+CHAN 1~



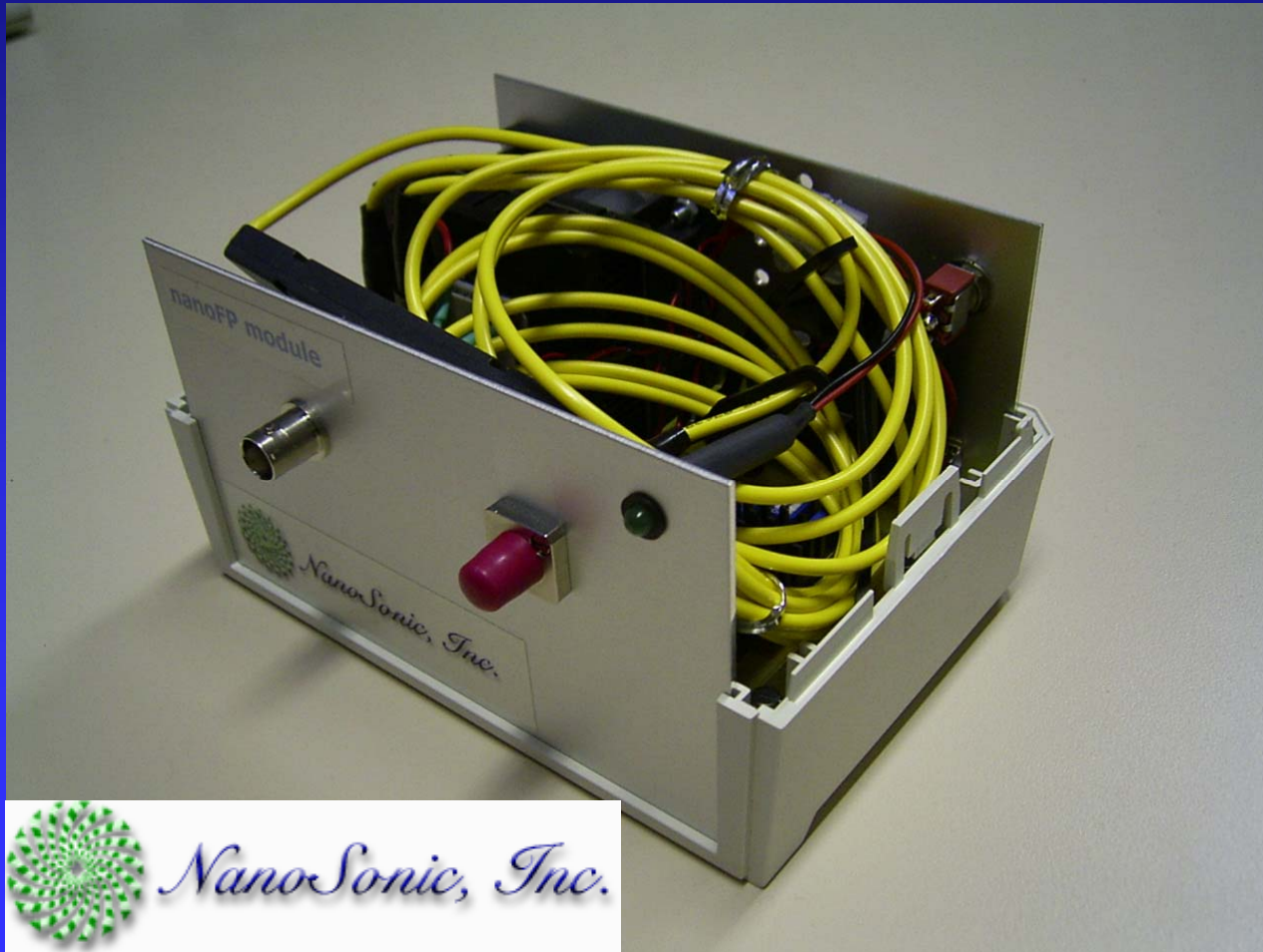
(d)

Ch1 .1 V =  
T/div 2 s Ch2 20mV =  
Trig 5.00 div+CHAN 1~

# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Interrogator system for NFP reflexive sensors

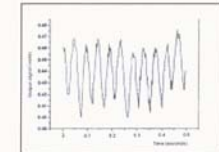
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NanoSonic NanoFP™ optical fiber sensor instrumentation system.



Typical NanoFP™ optical fiber sensor instrumentation system output data for rapid small variations in local relative humidity at optical fiber sensor tip.

- For use in a wide range of environmental, chemical, and industrial applications and installations.
- Exhibits sub-millisecond response to humidity changes at the distal end of the filter probe.
- Ultrasmall size with ultrafast response speed.
- Environmentally rugged sensing element geometry and coating chemistry.

NanoFP™ Specifications	
Range	10-90% RH
Accuracy	± 1%
Response (10-90%)	< 1 millisecond
Repeatability	< 1%
Output Voltage	0 to 4 V for 0 to 100% RH
Analog Output Connector	BNC
Sensor Diameter	2.5 mm
Dimensions	15 cm x 10 cm x 8.8 cm
Weight	14 oz.
Power Supply Input	115 - 220 V, 50-60 Hz
Power Supply Output	5 VDC
Price	\$2995

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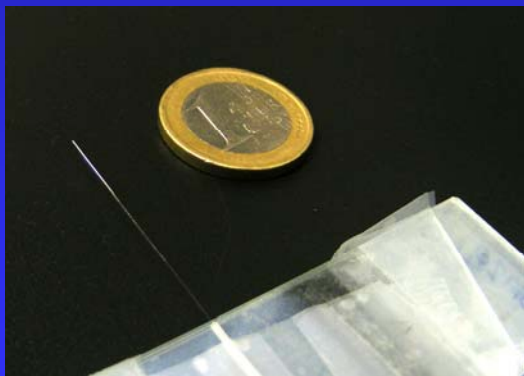
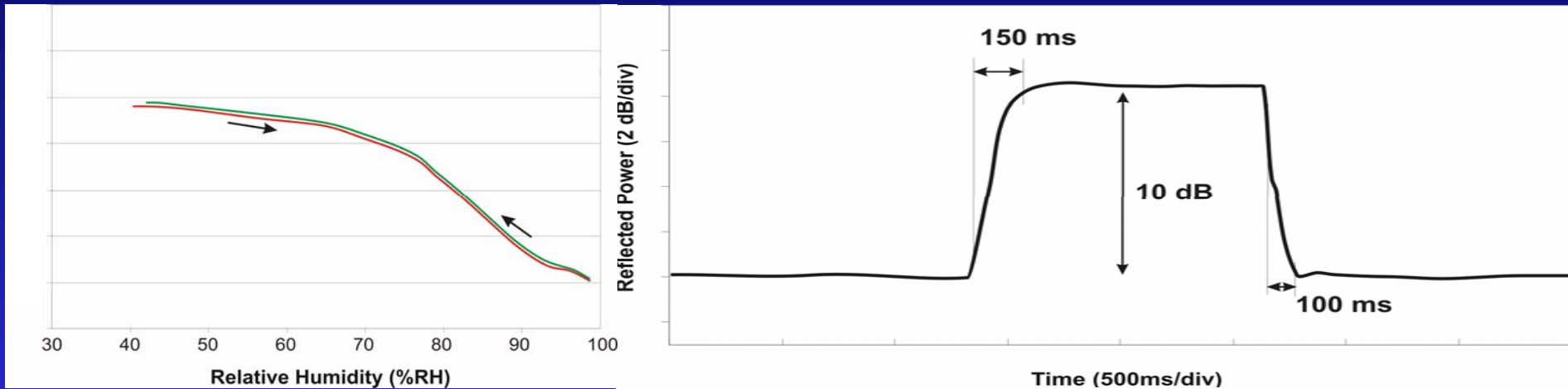
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# Novel fiber optic sensing architectures based on sensitive nanofilms

## NFP Cavities. Humidity sensors using silica nano-spheres

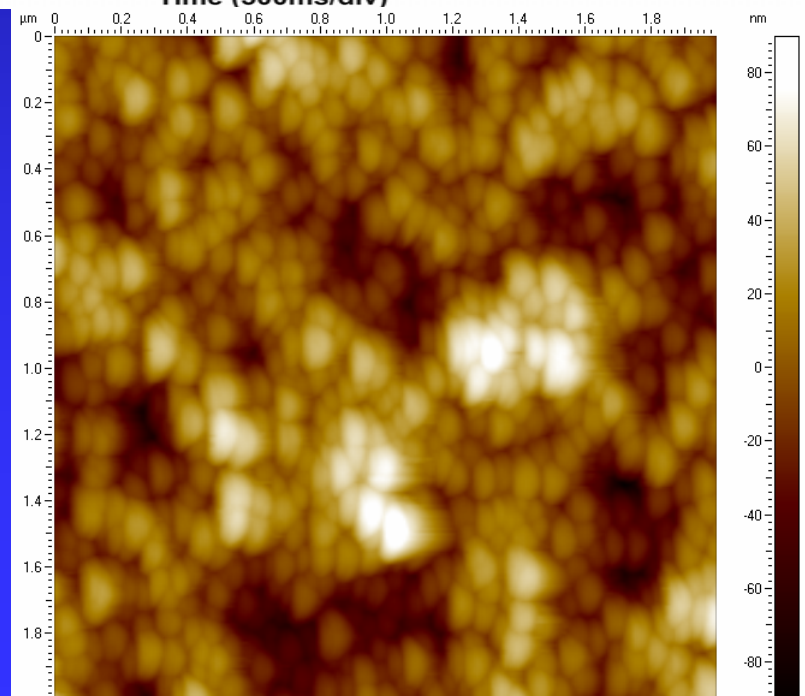


Characterization (AFM)

50 nm SiO<sub>2</sub> nanoparticles


Contact angle: 5°

Surface: Silica



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# Summary

- ❑ Introduction to the fiber optic sensor market
- ❑ Nanotechnology and fiber optic sensors?
- ❑ The Electrostatic Self-Assembled Monolayer Method
- ❑ Possible sensing architectures based on nano-films
  - ❑ Tapered ends
  - ❑ Tapered optical fibers
  - ❑ Hollow core fibers
  - ❑ Long period gratings
  - ❑ Optical fiber gratings
  - ❑ NanoFabry-Perot Cavities
- ❑ Conclusions 

## CONCLUSIONS

- The Layer-by-Layer Electrostatic Self-Assembly Method has been presented as a useful tool for fabricating nano-structured sensing coatings, not only fiber optic sensors.
- These coatings can be deposited on substrates of different shapes: flat, cylindrical or conical
- Different optical fiber sensors have been already experimentally demonstrated (humidity, volatile organic compounds, ammonia, glucose, etc.) and the possible applications of this technique in the sensing field are very promising.
- The sensors have a very fast response time, can operate at room temperature and it is possible to find a suitable architecture depending on the specific application.
- Several different optical fiber structures to fabricate sensors have been proposed: Tapered ends, Tapered optical fibers, Hollow core fibers, Long period gratings, Optical fiber gratings, NanoFabry-Perot Cavities, 1D PBG with defects, etc.). All of them are feasible to be implemented using ESA technique with different sensing properties and final performances.
- It is possible to design specific sensors for specific applications by varying any of the design parameters: materials, thickness, number of bilayers, structures, etc.



# ACKNOWLEDGEMENTS

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All the people at Nanosonic, Inc  
[www.nanosonic.com](http://www.nanosonic.com)



All the people at Virginia Tech

Fiber & Electro-Optics  
RESEARCH CENTER 



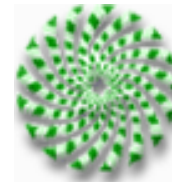
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# NOVEL FIBER OPTIC SENSING ARCHITECTURES BASED ON SENSITIVE NANOFILMS



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