NOVEL FIBER OPTIC SENSING ARCHITECTURES BASED ON SENSITIVE NANOFILMS

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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 range
- Single- & Multi-Point Sensing Configurations
- Large wideband
- Low attenuation
- Multiplexing techniques
- EMI immunity
Technology Overview. Classification

Type of optical fiber sensors

Type of Modulation
- Intensity
- Interferometric
- Polarimetric
- Spectroscopic

Nature of transduction
- Intrinsic
- Extrinsic

Spatial distribution
- Point
- Distributed
- Quasi-distributed

Physical Magnitude
- Voltage/current
- Temperature
- Radiation
- Biomedical
- Chemical/gas
- Electro magnetic fields
- Strain
- Rotation
- Mechanical
- Bending/torsion, velocity, vibration/acceleration, displacement/location, pressure/acoustics, force

Technology Overview. Extrinsic sensors

Extrinsic Optical Fiber Sensors

- Fluorescence
- Laser Doppler Velocimetry
- Reflection Scattering
- Photoelastic effects
- Absorption
- External cavities (EFPI)
- Total Internal Reflection
- Encoder Plates Disks
- MEMS OSA
- Numerical Aperture
- Evanescent

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Technology Overview. Intrinsic sensors

Fiber Bragg Gratings (FBG)
- Multicore FBG
- Tunable FBG
- D-Shaped FBG
- Chirped FBG

Long period gratings
- Laser fiber

Fiber Lasers/Doped Fibers

Rayleigh

Blackbody

Microbend

Interferometric

Mode Coupling
- Sagnac
- Mach-Zehnder
- Michelson
- Ring Resonator
- Fabry-Perot

Raman

Photonic Crystal Fibers

Distributed/Quasi distributed
- In line
- Micromachined
- Self-Assembly

Intrinsic Optical Fiber Sensors

Band Gap Fibers
- Bragg Fibers
- Index-guiding

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Fiber Optic Sensing System Key Building Blocks

- Specialty Optical Fibers
- Packaging
- Light Sources
- Detectors & Interrogators
- User Interface
- Data Acquisition & Interpretation

Design, Planning and Installation

Courtesy of Alexis Méndez, MCH Engineering, LLC
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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

Market Drawbacks

- 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

 Courtesy of David Huff (Oida) Source: Quorex
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Sensors Market Size

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

US $ Million

<table>
<thead>
<tr>
<th>Year</th>
<th>Market Size (US $ Million)</th>
<th>Market Share</th>
</tr>
</thead>
<tbody>
<tr>
<td>1998</td>
<td>175</td>
<td>0.54%</td>
</tr>
<tr>
<td>2003</td>
<td>283</td>
<td>0.67%</td>
</tr>
<tr>
<td>2008</td>
<td>1450</td>
<td>2.87%</td>
</tr>
</tbody>
</table>

Average of annual growth rate: 23.5%

Source: INTECHNO CONSULTING
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Applications

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

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

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

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

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

**Border security and power line monitoring**

Courtesy of David Huff and Alexis Mendez
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Optical Fiber Sensor Market Revenues Breakdown

![Graph showing Optical Fiber Sensor Market Revenues Breakdown from 2002 to 2010.]

Courtesy of David Huff (Oida). Source: Light Wave Venture
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Optical Fiber Sensor Market Forecast

![Graph showing Optical Fiber Sensor Market Forecast from 2006 to 2010. The graph includes three trends: Conservative, Potential, and Optimistic, with the Optimistic trend showing the highest growth.]( Courtesy of David Huff (Oida). Source: Light Wave Venture)
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- The Electrostatic Self-Assembled Monolayer Method
- Possible sensing architectures based on nano-films
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  - Tapered optical fibers
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  - Long period gratings
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Nanotechnology and fiber optic sensors?

Nanostructure with a size between molecular and microscopic layers of subwavelength thickness (*bottom-up*).
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Nanotechnology and fiber optic sensors?

Deposition techniques for sensing coatings in OFS

- Chemical vapor deposition
- Spin, dip coatings
- Sputtering in a radio frequency
- Gel solutions
- Layer-by-layer
- Langmuir-Blodgett
- Electron beam, physical and thermal evaporating
- planar magnetron systems
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Introduction


- **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, oxydes, 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, Al₂O₃, Fe₃O₄, SiO₂, TiO₂, ZrO₂, 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...
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The ESA Method: diverse applications

- **PRISMS**
  - NANOSONIC, INC. R. O. Claus et al.
- **LENS**
- **FLEXIBLE SUBSTRATES**
  - SUPERHYROPHOBIC SURFACES
    - M.I.T., M. F. Rubner et al.
- **MICROSHERES**
  - TEXAS A&M, M. McShane et al.
- **COATINGS ON BIOLOGICAL CELLS**
  - University of Melbourne, F. Caruso et al.
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Nanostructured coatings onto tapered ends of optical fibers

Glucose sensing

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Sensors based on Tapered Optical Fibers

Humidity sensing. Spectral characterization

Transmitted Power (dB)

Relative Humidity (%)

Bilayers (5/div)

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

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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

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Sensors based on Tapered Optical Fibers

Gliadin sensing (gluten detection)

Antibody Injection

Binding of antibody

Transducer

Anti-Gliadin Antibody

Peroxidase

Antigen

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Sensors based on Hollow core fibers

Humidity sensing based on evanescent wave

Spectral characterization

Transmitted Optical Power (dB)

Thickness of nanofilm (nm)

Number of bilayers

“Nanofilms on hollow core fiber-based structures: an optical study”. 
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Sensors based on Hollow core fibers

Humidity sensing based on evanescent wave

breathing monitoring

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Nanostructured coatings on Long Period Gratings: a pH sensor

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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EXPERIMENTAL RESULTS - VOLATILE ORGANIC COMPOUND SENSOR
DI CHLOROMETHANE (DCM)

A - PURGED AIR
B - 0.6 gr/l DCM
C - SATURATION OF DCM
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Different spectral responses

Sensor 1

Sensor 2

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1D PBG with defects

Refractometer

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NFP Cavities. Experimental set-up

Photodetector

LED

nanoFabry-Perots

optical fiber

coupler

nanocavity

index matching gel

sensing element
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**NFP Cavities. pH Sensors**

- nanofabry-perots
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**NFP Cavities. pH Sensors**

![Graph showing absorbance over time for pH 7 and pH 5](image)
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NFP Cavities. pH Sensors

Graph showing reflected optical power (dB) over time (min) with peaks at pH = 6, pH = 5, and pH = 4.
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**NFP Cavities. Ammonia sensors**

- Spectral response to:
  - 11 %RH
  - 43 %RH
  - 98 %RH
  - Acetone
  - Ethanol
  - Dichloromethane

- < 4% of cross-sensitivity to other compounds

- 5.6 dB with NH$_3$

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**NFP Cavities. Ammonia sensors**

Dynamic response of a 25 bilayers sensor to Ammonia
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NFP Cavities. Ammonia sensors

Recovery time < 4 seconds

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**NFP Cavities.** Volatile organic compounds sensors

\[ \text{[Vap+PAH^+/PAA^-]} \]

![Graph showing reflected optical power vs. layer number for PAH+Vap and PAA-](image)

Sensors and Actuators B. Vol. 115 (1); pp. 444-449, 2006
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**NFP Cavities.** Volatile organic compounds sensors

Absorbance spectra of the sensor after 40 minutes exposure
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**NFP Cavities.** Volatile organic compounds sensors

Response of the sensor for different methanol concentrations

Reflected Optical power (dB)

<table>
<thead>
<tr>
<th>Time (minutes)</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>31 mmol/l (methanol)</td>
<td>-3</td>
<td>-2.5</td>
<td>-2</td>
<td>-1.5</td>
<td>-1</td>
<td>-0.5</td>
<td>0</td>
</tr>
<tr>
<td>62 mmol/l (methanol)</td>
<td>-3</td>
<td>-2.5</td>
<td>-2</td>
<td>-1.5</td>
<td>-1</td>
<td>-0.5</td>
<td>0</td>
</tr>
<tr>
<td>125 mmol/l (methanol)</td>
<td>-3</td>
<td>-2.5</td>
<td>-2</td>
<td>-1.5</td>
<td>-1</td>
<td>-0.5</td>
<td>0</td>
</tr>
</tbody>
</table>
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NFP Cavities. Volatile organic compounds sensors

Response of the sensor for different ethanol concentrations

- 21.5 mmol/l (ethanol)
- 43 mmol/l (ethanol)
- 86 mmol/l (ethanol)
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**NFP Cavities.** Volatile organic compounds sensors

Comparison between ethanol and methanol

![Graph comparing reflected optical power between ethanol and methanol](image-url)
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
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**NFP Cavities.** Human breathing

Sensor and opto-electronic units

Face mask and sensor
NFP Cavities. Human breathing

IEICE Transactions on Electronics, vol. E83-C (3); pp. 360-365, 2000
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**NFP Cavities.** Interrogator system for NFP reflexive sensors

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**NanoSonic, Inc.**
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NFP Cavities. Humidity sensors using silica nano-spheres

Caracterization (AFM)

50 nm SiO$_2$ nanoparticles
Contact angle: 5°
Surface: Silica
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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.
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