Sol-gel Derived Nanomaterials for Designing Fiber Optic Gas Sensors

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Outline

• Optical fiber, light guiding and optical fiber chemical sensors
• Sol-gel process for making nanomaterials for hydrogen gas sensing
• Sensor structure
• Examples of optical fiber gas sensors
• Conclusions and further work
• Acknowledgement
A light beam is guided inside an optical fiber through a series of total internal reflections, which requires:

$$\theta < \theta_c = \sin^{-1}(n_1^2 - n_2^2)^{1/2}$$
Light power distribution inside an optical fiber

Light power distribution in an optical fiber

$\frac{d}{dp} = \frac{l}{2n_1[sin^2\theta - (n_2/n_1)^2]^{1/2}}$

>90% of light power is distributed inside the optical fiber core for multimode optical fiber

c.a. 50% of power can be distributed in cladding in single mode optical fibers

Evanescent wave

An evanescent wave is a near-field standing wave formed at each point of total internal reflections

$I = I_0 \exp(-z/d_p)$

$A. \ Katzir, \ et \ al., \ Appl. \ Opt., \ 35, \ 2274 \ (1996)$
OFCS classification

**Evanescent wave (EW) sensor**
- Interaction inside the cladding
- Can use low cost fibers
- Fast response
- Limited sensitivity

**Active core (AC) sensor**
- Interaction inside the fiber core
- Need specially tailored fibers
- High sensitivity

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A selective high temperature H₂ sensor

I: Structure

II: Principle

- A reducing gas (H₂, CO, organic compound) reacts with SnO₂ nanoparticles changes oxygen ion concentration on particle surface
- SnO₂ membrane EW optical spectrometry \( \rightarrow \) selective sensing

Sensor probe preparation

Sn[OC$_3$H$_7$]$_4$ in isopropanol/toluene

(Sn[OC$_3$H$_7$]$_4$ is hydrolyzed in air

Heat treatment (600 °C for 1 hour)
SEM images of SnO2 coating on fiber core surface
Test the sensor for detecting hydrogen in gas sample
Test result

-0.3 0 0.3 0.6 0.9 1.2

Wavelength (nm)

Abs.

10% H₂, 600 °C, 25 min
10% CO, 600 °C
5% CH₄, 600 °C

Absorbance

Wavelength (nm)

300 °C
400 °C
500 °C
600 °C
700 °C
800 °C
Test result

\[
y = 0.08249 \ln(x + 0.17934) + 0.14506
\]

\[
R^2 = 0.99672
\]
Improve selectivity by incorporating reactions to cladding (1 wt% Pt doped SnO₂)
A palladium doped sol-gel silica optical fiber for detecting hydrogen at ambient temperature

1: The process for making palladium nanoparticles doped sol-gel silica optical fiber
A SEM image of a sol-gel derived porous silica optical fiber
III  A SEM image of palladium particles in sol-gel derived porous silica
Test the sensor for detecting hydrogen in gas sample

- pd doped nanocomposite fiber
- epoxy glue
- steel piece for fixing fiber
- silicate fiber
- gas out
- NIR 256-2.1 OceanOptics, Inc.
- computer
- Mikropack Halogen Light Source HL-200
- nitrogen
- 1% hydrogen in nitrogen
V: Test result
V: Test result

![Graph showing test results with time in minutes on the x-axis and intensity in counts/second on the y-axis.]

1% H₂ in
N₂ in

1% H₂ in
N₂ in

1% H₂ in
N₂ in

1% H₂ in
N₂ in

(B)

(A)
Conclusions and further work

• Nanocomposite materials have been synthesized with sol-gel or sol-gel/micelle techniques
• The nanocomposite materials can be coated on surface of optical fiber core or be made into the form of optical fibers for designing gas sensors
• Trace hydrogen in gas samples of different temperature has been detected with developed sensor techniques
• Chemical reactions can be integrated into sensing element to achieve selective sensing
• Further work will be focused on integrating optical spectroscopy and electrochemical methods for developing selective and multi-components sensing techniques.
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