Optics Communications 315 (2014) 74–78

Contents lists available at ScienceDirect

Optics Communications

journal homepage: www.elsevier.com/locate/optcom

# Fiber optic gas sensors with vanadium oxide and tungsten oxide nanoparticle coated claddings

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ARTICLE INFO

*Article history:*
Received 12 September 2013
Received in revised form
22 October 2013
Accepted 23 October 2013
Available online 13 November 2013

*Keywords:*
Cladding modification
Toxic gases
Fiber optic sensor
Nanocrystalline $WO_3$ and $V_2O_5$
Gas sensors

ABSTRACT

Fiber optic gas sensors with nanoparticles of $V_2O_5$ and $WO_3$ as the cladding of a PMMA fiber have been proposed in this work. The spectral response of these sensors for detection of ammonia, methanol and ethanol under various concentrations has been studied at room temperature. The time response characteristics of the sensors are also presented.

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## 1. Introduction

Fiber optic gas sensors using metal oxides are increasingly explored as they offer several advantages such as low cost, small size and high sensitivity. The basic operation of a fiber optic gas sensor is that when it is exposed to a chemical or physical stimulus, the intensity of light signal traveling through an optical fiber changes. The plastic optical fiber with cladding modification is very attractive for gas sensing because of its large dynamic range and high sensitivity. Optical modes propagating through the fiber interact with core and cladding interface and therefore are more sensitive to the changes in the cladding material for gas sensing [1]. Vanadium oxide ($V_2O_5$) is widely used as catalyst and cathode materials for lithium-ion batteries. Vanadium oxide material has been explored for ammonia, ethanol and humidity sensors using sputtered $V_2O_5$ films, and nanobelts coated-nanotubes [2–5]. The response of $V_2O_5$ and $WO_3$ to NO gas has been studied in thin films [6]. It was found that $V_2O_5$ could detect NO in the range of 50–500 ppm. Carbon nanotubes (CNTs) have been coated on vanadium oxide for improving the gas sensitivity as CNTs exhibit large suface area and chemical stability [7,8]. Tungsten oxide is considered as one of the most interesting materials in the field of gas sensors with respect to conventional metal oxides as it can show very good results in the detection of NO, $NO_2$, $O_3$ and $H_2S$ [9–11]. Tungsten oxide has been also used for the detection of ammonia vapors. Tungsten oxide films exhibited selective response to ammonia in the environments of methane, ethanol and toluene at 300 °C. The response was found to be stable over a long period [12].

A highly sensitive $NH_3$ sensor using Pt catalyzed silica coating over $WO_3$ thick films has been reported [13]. $WO_3$ nano-materials as modified cladding are expected to exhibit better sensitivity and hence they are explored for gas sensing applications. $WO_3$ is a very interesting gas sensor in optical field [14–16] for ammonia and ethanol gases [17–20].

However, all these works use $V_2O_5$ and $WO_3$ either in the form of thick or thin film and some of them are useful only at high temperatures. In this paper, we have used $V_2O_5$ and $WO_3$ nanoparticles as the cladding elements on a fiber, and have characterized their gas sensing properties for detection of ammonia, methanol and ethanol at room temperature.

## 2. Experimental procedure

### 2.1. Sensing mechanism

A schematic diagram of an optical fiber gas sensor is shown in Fig. 1. The sensor consists of multimode step index plastic (PMMA) optical fiber having a length of 42 cm and a diameter of 750 μm. The refractive indices of the core and the cladding are 1.492 and 1.402 respectively. The gas sensing region was obtained by completely removing the clad part of about 3 cm length at the center of the fiber,

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0030-4018/$ - see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.optcom.2013.10.072

to serve as the sensing region. $V_2O_5$ or $WO_3$ nanoparticles were mixed with isopropyl alcohol to form a paste and coated on the sensing region by the dip coating method to a thickness of 30 μm. The coated fiber was dried at room temperature and the sensing part of the fiber was inserted into a gas chamber. White light (Model SL1, Stellar Net Inc., USA, wavelength range 100–2000 nm) was coupled into the fiber source and the output of the fiber was fed into a miniature fiber optic spectrometer (EPP-2000, Stellar Net, USA, spectral range 100–1100 nm) [1]. Ammonia, methanol and ethanol prepared in different ppm levels (0–500 ppm) were passed into the chamber and the intensity variations were recorded using the spectrometer interfaced with a personal computer. The sensor measurements were carried out at room temperature.

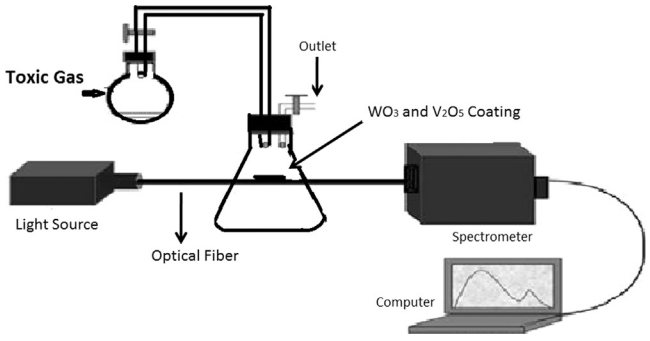


**Fig. 1.** Schematic diagram of a fiber optic gas sensor.

## 3. Results and discussion

### 3.1. XRD and SEM analyses

Vanadium oxide and tungsten oxide nanoparticles were bought commercially. The phase of the samples was analyzed using XRD. Powder X-ray diffraction studies on the nanoparticles were performed with a Rigaku diffractrometer (Model: Ultima III, Japan) using Cu Kα (1.54 Å) radiation. A beam voltage of 40 kV and a beam current 30 mA were used. The data were collected in the $2\theta$ range 10–80° with a continuous scan speed of 0.2°/min.

The XRD analysis showed that vanadium oxide had an orthorhombic phase [Fig. 2(a)]. The estimated crystallite size was about 17 nm. Fig. 2(b) shows the XRD pattern of the $WO_3$ nanoparticles. The data showed that they were in a tetragonal phase and there was no impurity. The crystallite size of tungsten oxide was estimated using Scherrer's equation and found to be about 11 nm. The morphology of the coated cladding was studied using a scanning electron microscope (SEM; Model no. S-3000H, Hitachi, Japan). Fig. 3(a) shows the SEM image of the vanadium oxide coating on the fiber. The oxide exhibits plate like structures comprising nanorods. Fig. 3(b) shows a SEM image of $WO_3$. The particles are found to be agglomerated. The $WO_3$ sample shows an irregular, random shape with non-uniform particle distribution.

### 3.2. Gas sensing characteristics

Fig. 4(a)–(c) shows the output spectral characteristics of a $V_2O_5$ coated sensor exposed to various concentrations of ammonia, methanol and ethanol. The spectra exhibit three peaks around

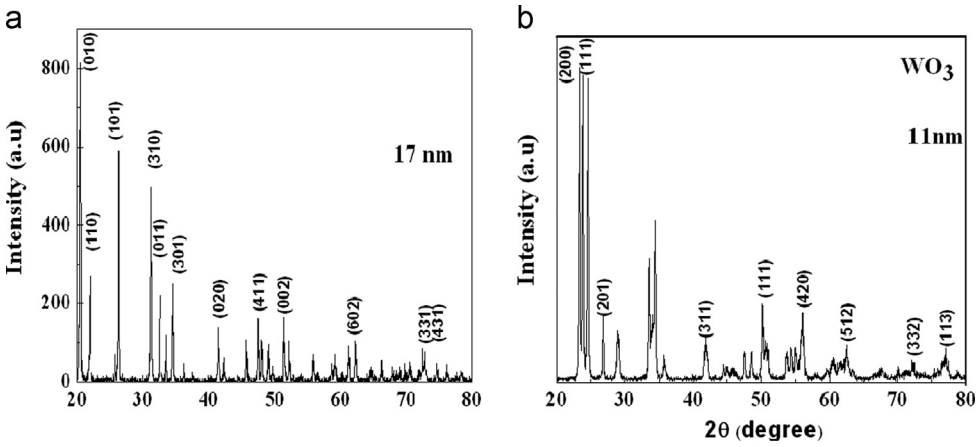


**Fig. 2.** XRD pattern of $V_2O_5$ and $WO_3$.

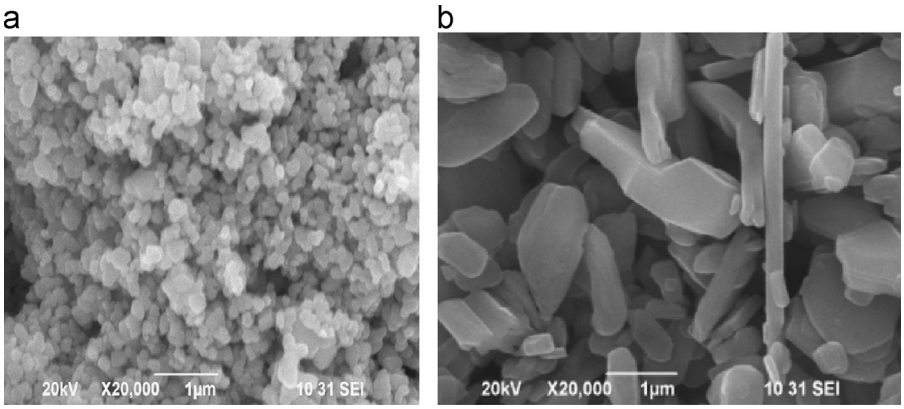


**Fig. 3.** SEM micrograph of $V_2O_5$ and $WO_3$.

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