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Gas sensing based on detection of light radiation from a region of modified cladding (nanocrystalline ZnO) of an optical fiber

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ABSTRACT

A new type of fiber optic gas sensor is proposed by detecting a light radiated from a region of cladding modified with metal oxide (nanocrystalline ZnO). The intensity of radiated light is found to vary with different gasses and concentrations. Sensing characteristics are studied for ammonia, methanol, ethanol and acetone gasses. Gas sensitivity of the proposed sensor is compared with clad-modified fiber optic gas sensor. The new sensor exhibits enhanced sensitivity. Time response characteristics of the sensor are reported.

1. Introduction

Metal oxides (ZnO and SnO_2) based gas sensors are widely used for sensing gasses such as NH_3, CO, NO_2, ethanol and methanol [1–8], in which, electrical resistivity changes when it is exposed to the gas. However, gas sensitivity, which is defined as a change in the output of the sensor to the change in the gas concentration, is found to be poor at room temperature and the operating temperature is greater than 250 °C [3,6]. Many studies have been carried out to enhance the gas sensitivity by multilayer deposition [9] adding volatile material as a dopant in sensing materials [10,11] or by a design of a new type of sensor.

Optical fiber gas sensors using metal oxides as sensing medium have been attempted for room temperature operation [11,12]. A side polishing optical fiber [13], coating optical fibers with nanocrystalline structure material [12], clad-modified Fiber optic gas sensors [11–14] have been proposed. Fiber optic sensors have many advantages such as immune to electromagnetic interference, good corrosive resistance and can be used in hostile environments. They have additional features such as low cost and smaller in size [15–17].

In clad-modified fiber optic sensors, light is passed through one end of the clad-modified optical fiber and the transmitted light is received at the other end as output (transmitting mode) [11,12]. Part of the light propagating through the fiber is found to be leaked through the modified clad region. The changes in the output light intensity are related to interactions among modified clad surface, a presence of gas at the surface and leaked light [11].

In the transmitting mode, only a small amount of light undergoes a leakage and most of the light is transmitted through the fiber. It is found that the leakage leads to light radiation at the modified clad surface [11]. Since, the light emanating from the clad surface may undergo variations in the presence of gasses, the detection of light at the surface may lead to a gas sensor. In this study, it is explored for detecting a light radiation from the clad-modified region (Radiation mode) for different gasses. The gas sensitivity obtained is compared with that of the transmitting mode.

In recent years, nanocrystalline ZnO is widely studied in gas sensing applications due to significant physical properties [18,19]. ZnO exhibits high chemical and thermal stabilities. The optical properties of ZnO have not been much explored for optical sensing applications [20,21] and it has about 80% transparency in visible and UV regions [18,21] with a refractive index of 1.901. In this study, therefore, ZnO is used as gas sensing medium.

1.1. Gas sensing principle (Radiation mode)

Fig. 1 shows working principle of radiation mode sensing. The sensor consists of two optical fibers, in which one is used to transmit a light (transmitting fiber) and other is used to detect light radiation (detecting fiber). In the transmitting fiber, a part of the clad is removed and replaced with a metal oxide (ZnO) to sense a gas.

Light is launched through the fiber at all angles and propagates through a transmitting fiber at different angles (Fig. 2). The light which incidents at the interface A, is found to undergo partially reflection and refraction (leakage of light into the clad modified region through interface A) as a refractive index of the core (glass −1.4589) of the optical fiber is lesser than the clad-modified region (ZnO −1.901).

Since, ZnO has very high transmission co-efficient (about 80%), the

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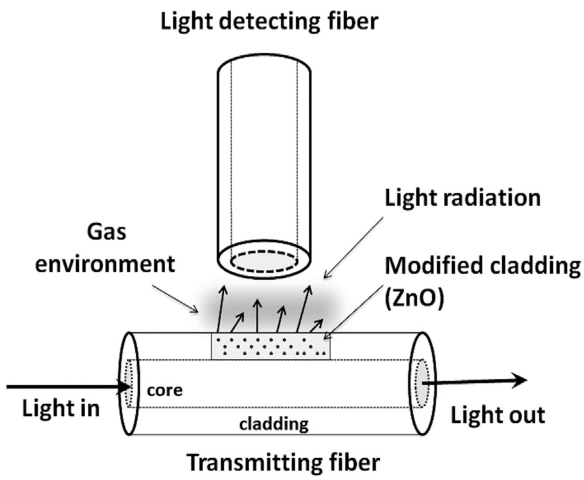

Fig. 1. Principle of radiation mode sensing.

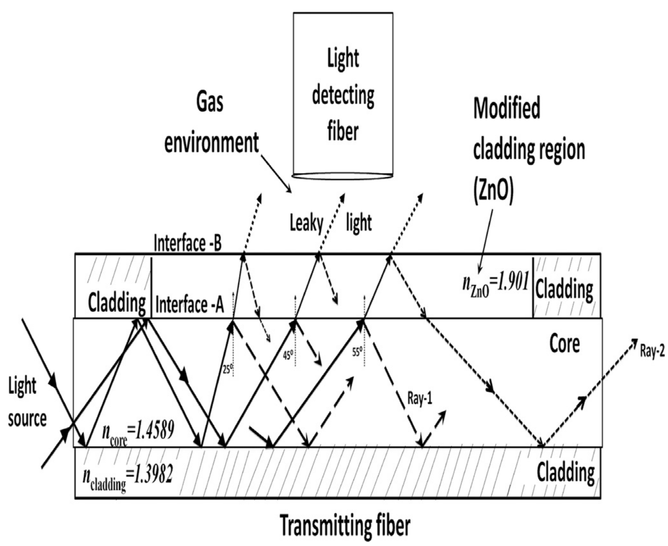

Fig. 2. Light radiation (evanescent field) from clad-modified region.

refracted light undergoes little attenuation through a clad-modified region and reaches out the Interface –B (modified clad surface - air/gas medium) [11]. The refractive index of outer medium (air medium in the absence of gas) is lesser than the modified cladding region. The refracted light arriving at this interface (B) at different angles undergoes reflection, refraction and total internal reflection. The refracted light from interface A, whose incident angles are greater than the critical angle, undergoes total internal reflection where as others reflection and refraction. Hence, a light would be radiated out of the clad-modified surface of the fiber (interface B). The detecting fiber, which is kept nearer to this surface (B), will detect a light radiation. The intensity of the detected light may vary for different gas environments. Simultaneously, the intensity of light received from the transmitting fiber also may change (transmitting mode).

2. Experimental procedure

2.1. Sensing probe preparation

Multimode fibers (Model FT400EMT-Thorlabs) were used as light transmitting fiber and detecting fiber. The core of the fiber is made of pure silica. The fiber core was covered with cladding made of Technology Enhanced Clad Silica (TECS) and tefzel polymer as a buffer cover. The core diameter is 400 µm and cladding diameter is 425 µm. The buffer diameter is 730 µm. The refractive index of core and cladding are 1.4589 and 1.3982, respectively, at the wavelength of 589.3 nm. It has a numerical aperture of 0.39. The lengths of the fibers used were about 25 cm.

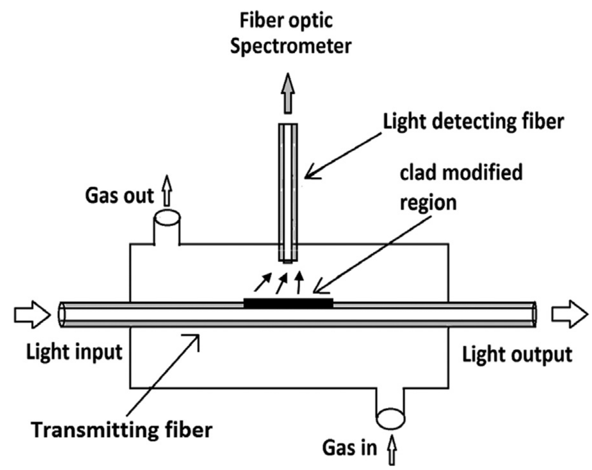

Fig. 3. Schematic diagram of gas sensor set-up.

Gas sensing region was prepared in the transmitting fiber by side polishing up to the core region about 5 mm length. Initially, the tefzel layer was removed by scratching with silicon carbide blade (Model F-CL1, Newport) manually. Then, the TECS cladding was removed by dissolving in acetone solution (Thorlabs - data sheet). The complete removal of cladding was ascertained by measuring the diameter of the fiber, which was found to be around 565 µm.

ZnO powder was mixed with isopropyl alcohol in the ratio of 1:2 and a slurry was formed. Then the clad removed region of the transmitting optical fiber was dipped in the slurry and dried at room temperature for 3 to 4 h. The thickness of the coating was about 250 µm. which was measured using travelling microscope by measuring the diameter before (about 565 µm) and after dip coating process (about 815 µm).

2.2. Gas sensing setup

The schematic diagram of an optical fiber gas sensor is shown in Fig. 3. The clad modified fiber was inserted into a gas chamber. The detector fiber was placed vertically to the transmitting fiber at the clad-modified region with a gap of about 1 mm. The transmitting fiber was connected to a Tungsten - Halogen lamp (SL1, Stellarnet Inc, USA) having a spectral range of 350–2200 nm. The detecting fiber was connected to Fiber optic spectrometer (EPP2000, Stellarnet Inc, USA) having spectral response 200–1200 nm.

Initially different concentrations of ammonia, ethanol, methanol and acetone solutions (100–500 ppm) were prepared by dilution method using distilled water. Then, 25 ml of solution was taken in a round bottom flask and the vapour produced in it, was sent to the gas chamber along with carrier gas (atmospheric air (1 l/min, 101.383 kPa)). The air was pumped into the solution using low powered air pump. This produced a mixture of vapour and air. Spectral measurements were made for different concentrations after three minutes in order to stabilize gas concentration inside the chamber. Experiments were performed at room temperature with a relative humidity of 71%.

3. Results and discussion

3.1. XRD analysis of ZnO nanomaterial

ZnO was purchased from Sigma-Aldrich and used without any further purification. XRD study was carried out using Rigaku X-ray diffractometer (Ultima III - Japan). CuKα target was used to generate