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Detection of Emanated Light at the Modified Clad Region (Sno_2) of the Optical Fiber for Methanol Gas Sensing

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Abstract: ***A leakage detecting sensor (radiation mode (or) double fiber mode) is proposed to monitor the light rays propagates from the surface of the modified clad region (SnO_2) of the optical fiber. The output intensity increases and decreases for the varying gas concentration (0-500 ppm) with the presence of different gases (methanol, ethanol, acetone and ammonia) at room temperature. The received output intensity from the clad modified surface increases for ammonia and methanol, whereas, it decreases for acetone and ethanol gases for the increase in the gas concentration. However, in the transmitting mode (single fiber mode) the output light intensity decreases for all the gases with varying gas concentration. The output gas sensitivity of the proposed sensor (double fiber mode) is compared with the transmitting mode sensor and the sensor shows superior response for methanol over other gases. The dynamic characteristics of the sensor are reported.***

Keywords: Leakage sensing, Optical fiber, Acetone sensor, Refracted light sensing, Clad replaced.

I. INTRODUCTION

SnO_2 is extensively investigated and used to detect a variety of gases for practical application. SnO_2 nanomaterials with different morphologies and spatial assemblies have been fabricated in order to improve the gas sensing performances[1]. Recently, SnO_2-based composites, including SnO_2-inorganic metal oxide, SnO_2-carbon nanomaterials, SnO_2-noble metals, SnO_2-polymer, and SnO_2-other materials have been reported based on electrical conductivity type of gas sensors [2]. These studies shows that gas sensing of this material could be greatly improved, such as high sensitivity, low working temperature, quick response, excellent stability or low detection limit [2].

Tin oxide is one of the transparent and conductive oxide materials with high stability, high resistance to moisture and acids [3,4]. Clad modified Fiber optic gas sensors were reported for ammonia and ethanol. SnO_2 clad modified fiber optics sensor was reported for ammonia detection [5]. An evanescent fiber optic gas sensor with a single-mode silica fiber clad modified with nano sized amorphous tin oxide, was studied to detect ethanol vapor in the range of 1000 to 20,000 ppm at room temperature on the telecommunication wavelength of 1.55 micron. The response time was found to be about 10 s for 1000 ppm [6].Ethanol sensitivities of SnO_2 and SnO_2:CuO nanoparticles were studied using a clad-modified fiber-optic sensor. SnO_2: CuO exhibits higher ethanol sensitivity compared to pure SnO_2. It is found that the addition of CuO into SnO_2 allows the effective formation of more number of oxygen vacancies, hetero junction, and high crystalline nanoparticles, which enhance its sensitivity to about three times than that of pure SnO_2[7].

In this work, gas sensing properties of SnO_2 clad modified fiber optic sensor is reported in radiation mode for ammonia, ethanol, methanol and acetone gases at room temperature. The sensitivity, selectivity and time response of the sensor explained.

II. PROCEDURE FOR PAPER SUBMISSION

A. Probe Preparation

Initially, the clad was removed in the middle of the transmitting fiber which was replaced with the sensing material (SnO_2). The sensing material was mixed with isopropyl alcohol to form the slurry and it was coated and dried at room temperature. While measuring the diameter before (525 µm) and after (755 µm) coating the thickness of the sensing material was found to be about 250 µm. Measurements were carried out at room temperature with a relative humidity of 71%.

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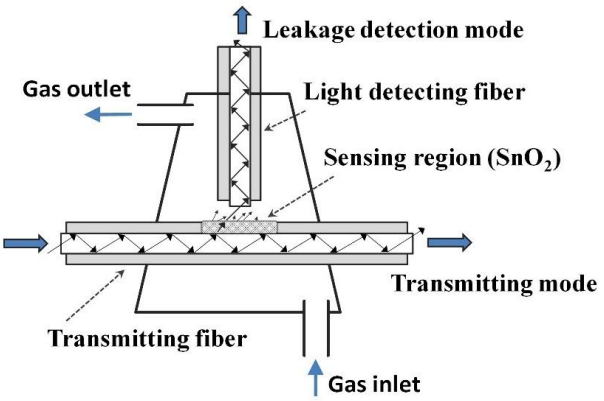


Fig. 1: Schematic diagram of radiation detection mode

B. Gas Sensing Setup

The radiation detecting fiber was fixed upright to the transmitting fiber to detect the radiation (leakage light) from the modified clad surface (sensing medium), with the gap distance of about 1mm (Fig.1). The radiation detection fiber was connected to the optical spectrometer (EPP - 2000; Stellarnet, Inc., USA) having a spectral range of 200- 1200 nm and the transmitting fiber was coupled to the tungsten-halogen light source (SL1; Stellarnet, Inc., USA) having an emission wavelength range of 350- 2200 nm. The other end of the transmitting fiber connected to the photo detector (New port- 818) having a spectral response range of 400- 1100nm[8,9]. Initially, different concentrations of ammonia, ethanol, methanol, and acetone gases (100, 200, 300, 400 & 500 ppm) were prepared and mixed with the carrier gas (air - flow rate of 1 l/min) using air pump. This gas combination was injected into the gas chamber through the inlet and light output was recorded. The experiment was carried out at room temperature (about 25°C) with the relative humidity of about 71%[9].

III. RESULTS AND DISCUSSION

A. XRD and SEM Analysis

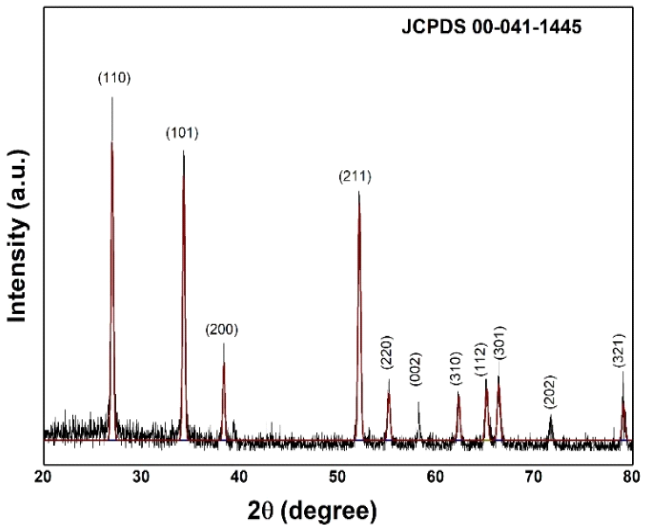


Fig. 2: XRD pattern of the SnO_2

The sample $SnO_2$was purchased from Sigma- Aldrich and used without any further purification. Fig.2 shows X-ray powder diffraction pattern of the SnO_2material using Rigaku X-ray diffractometer (Ultima III, Japan) with CuK_α radiation. A beam voltage of 40 kV and beam current of 32 mA be used. The data were collected at 2θ range (20-80°) with a continuous scan speed of 0.02 deg./min. A tetragonal phase of aluminum oxide was observed from the XRD. Scherer's formula,

$$D=0.9/\beta Cos\theta \qquad (1)$$

Where, λ (1.5406 Å) is the CuK_α wavelength, β is the full width half maximum (FWHM), and θ is the diffraction angle were used for predicting the crystallite size which was found to be about 35nm.

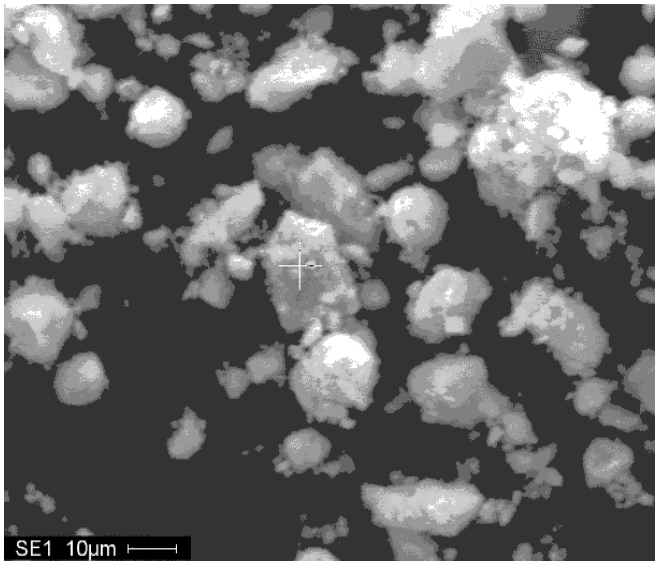


Fig. 3: SEM micrograph powder of SnO_2

In fig. 3 shows that the surface morphology of the as-prepared SnO_2 powder using scanning electron microscope (SEM) and the particles are spherical shape with highly agglomerated.

B. Spectral Response of the Sensor

Fig. 4 (a-d) shows the output spectral response of radiation detection mode for ammonia, ethanol, methanol and acetone gases. The peak wavelength of 652nm was observed for all the gases and it remains same.

The output spectral response increased for ammonia (968-1128) and methanol (1001-1093), whereas, it decreased for ethanol (955-939) and acetone (1053-1032) gases for increasing gas concentration.

C. Gas Sensitivity

The gas sensitivity is defined as the relative amount of changes in the sensor output to the change in the gas concentration (Fig. 5 (a-d)). In radiation mode, for increasing gas concentration, a highest gas sensitivity of $39x10^3$counts/kPa was obtained for methanol gas and lower gas sensitivities for ammonia (6×10^3counts/kPa), ethanol ($4x\ 10^3$counts/kPa) and acetone ($3\ x\ 10^3$counts/kPa) gases. Whereas, in transmitting modes the sensitivity is found to be 1.3 μW/kPa for methanol and <1μW/kPa observed for other gases.

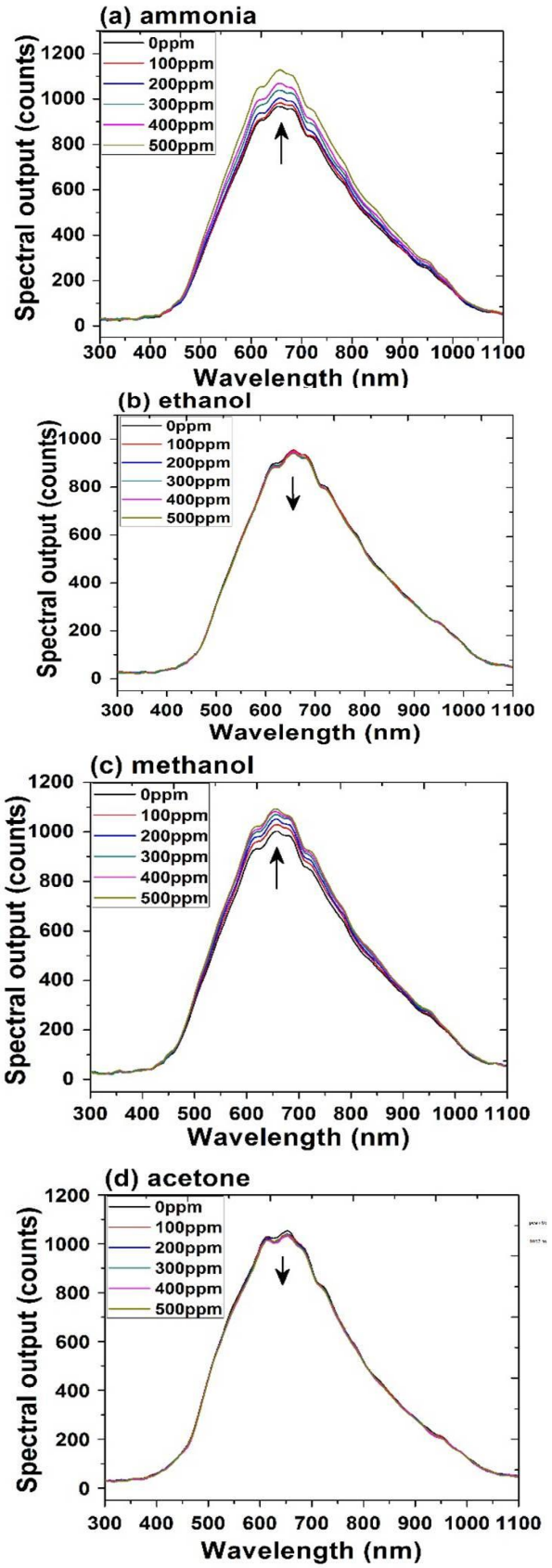


Fig. 4(a-d): Output spectral response for radiation detection mode for different gases with increasing concentration

It is seen that in radiation mode an increase response observed for ammonia and methanol gases and decrease response observed for ethanol and acetone gases, whereas, in transmitting mode all the gases decrease for the increasing gas concentrations. In room temperature the gases have a different vapour pressures, hence the ppm are converted into vapour pressures.

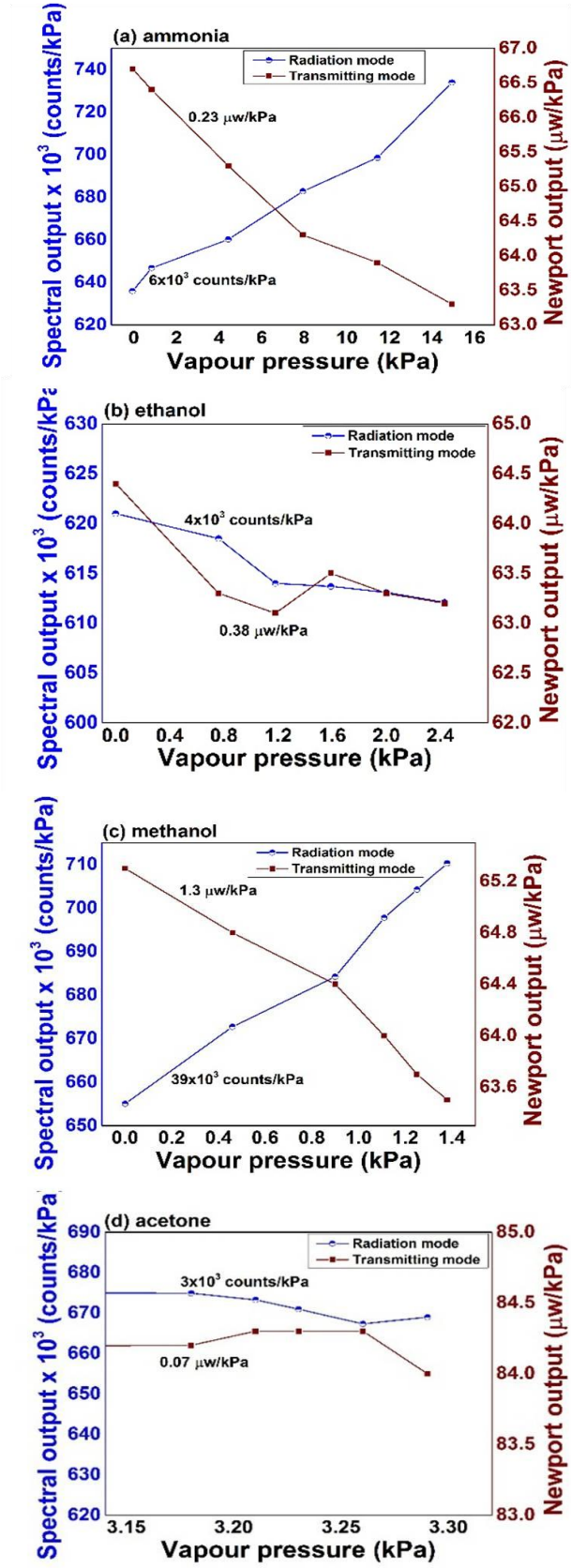
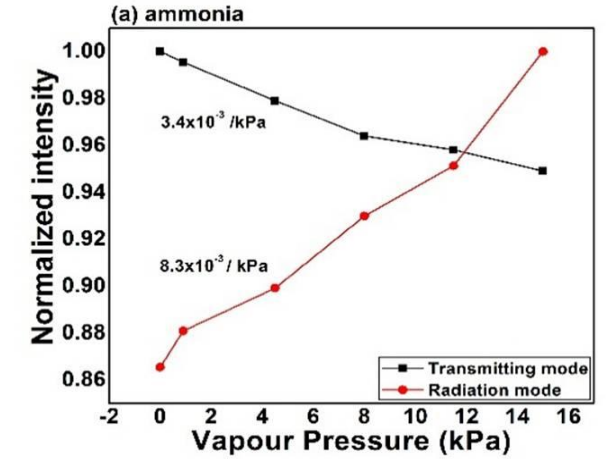
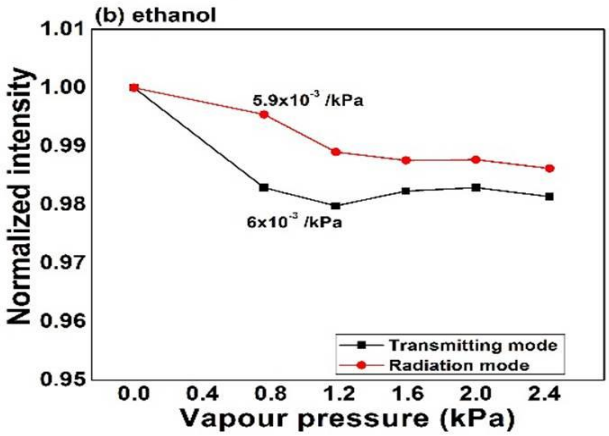


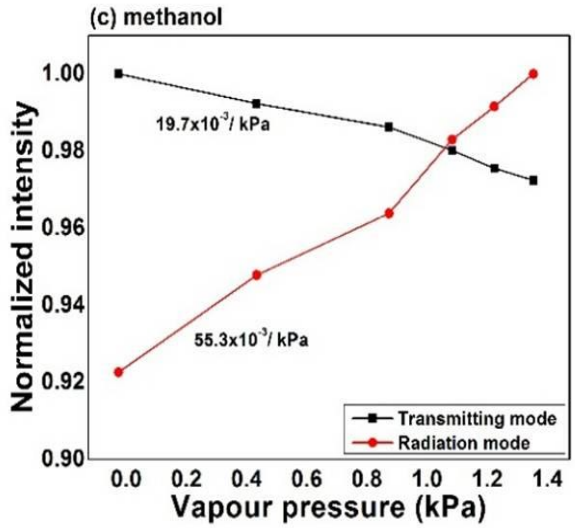
Fig. 5(a-d): Gas sensitivity of radiation mode and transmitting mode for different gases with increasing gas concentrations

Table. 1: Gas sensitivity of radiation and transmitting modes.

Gases	Sensor Sensitivity		Normalized Sensitivity (10^{-3}/kPa)	
	RD 10^3x(counts/kPa)	TM (µW/kPa)	RD	TM
Ammonia	6	0.23	8.3	3.4
Ethanol	4	0.38	5.9	6
Methanol	39	1.3	55.3	19.7
Acetone	3	0.07	3.9	0.79







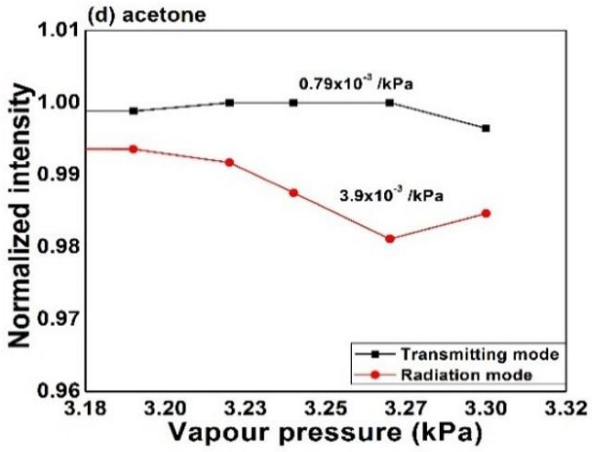


Fig. 6(a-d). Normalized sensitivity of radiation mode and transmitting mode for different gases with increasing gas concentrations.

Fig. 6(a-d) show the comparison plots for the normalized sensitivity of the radiation mode (proposed method) with the transmitting mode. It is seen that for methanol gas the sensor output changed largely for the radiation mode (0.92 - 1.0) compared to the transmitting mode (0.97- 1.0) (Fig. 5(c)).

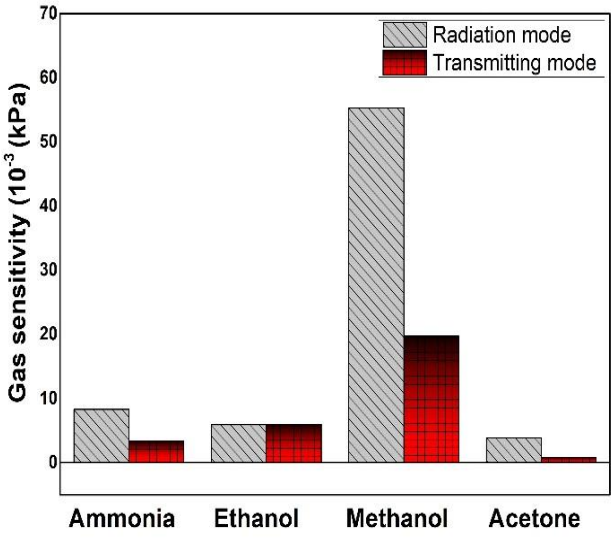


Fig. 7: The comparison chart of radiation and transmitting modes for different gases

The highest gas sensitivity of about 55.3 x 10^{-3}/kPa is observed for methanol in the radiation mode and about 19.7 x 10^{-3}/kPa in the transmission mode. The radiation mode exhibits higher gas sensitivity (about 2.8 times) for methanol compared to the transmission mode (Table. 1).

D. Gas Selectivity

Fig. 7shows the bar diagram of gas sensitivities for different gases in the radiation (RD) and transmission (TM) modes. It is seen that in radiation mode the gas sensitivity is higher for methanol, which is about 6.7 times higher than the ammonia gas. Whereas, in transmitting mode methanol gas obtains more sensitivity (3-times) than ethanol gas.

Therefore, a clad modified (SnO_2) fiber optic gas sensor is more sensitive and selective to methanol gas among other gases in the radiation mode.

E. Mechanism

The gas sensing mechanism is related to the adsorption and desorption of atmospheric oxygen (appears as O^-, $O^{2-}$and O_2^-) on the surface of the metal oxides (SnO_2)[10]. In the proposed fiber optic sensor, a layer of adsorbed oxygen may be present on surface of the SnO_2 and the above phenomena may change the optical property (refractive index) of the layer when the sensing gases present (Fig.8).

The working mechanism of the proposed fiber optic gas sensor works in leaky mode condition as the refractive index of the modified clad (n_1= 2.006)[11]is higher than the core refractive index (n_{core}= 1.4589). The leaked light undergoes reflection and refraction at the interface of SnO_2 region – adsorbed oxygen layer (A). The amount of light reflection and refraction at this interface may change in the presence of a gas leading to the change in the sensor output[12].

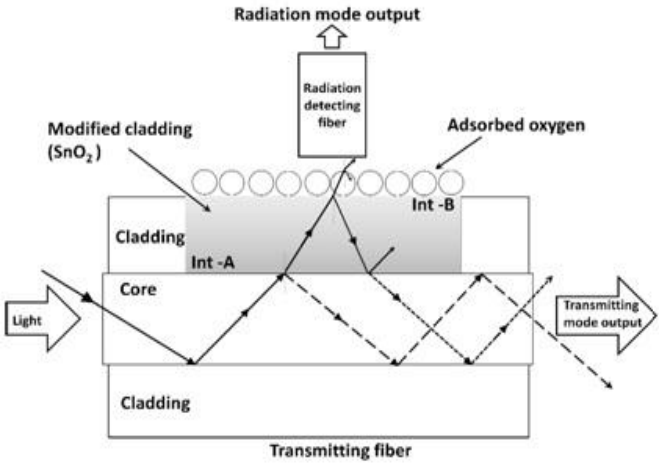


Fig. 8: Sensing mechanism of the proposed sensor

F. Time Response of the Sensor

Fig. 9. shows time response characteristics of the sensor in the radiation mode for methanol gas at 500 ppm. The response time was determined by passing the gas instantaneously to the gas chamber and recovery time by discharging the gas immediately from the chamber. The response time was calculated by observing the time period of the sensor signal (at 652 nm) took for raising from 10% to 90% of the maximum and for recovery time, the intensity falling from 10% to 90% of the maximum. The response time and recovery time of methanol were found to be about 5.3 min and 3.5 min, respectively.

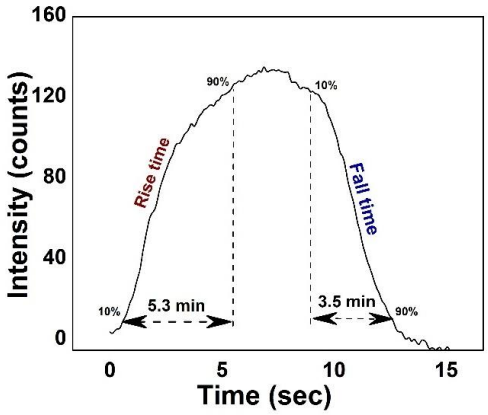


Fig. 9: Time response for methanol at 500ppm

IV. CONCLUSION

Gas sensing characteristics of SnO2coated (side polished) fiber optic sensor was studied for ammonia, ethanol, methanol and acetone gases at room temperature. In radiation mode the output intensity of the sensor was found to be increased for ammonia and methanol and decreased for ethanol and acetone with increasing gas concentration (100 to 500 ppm). The sensor showed highest response for methanol (39 x 10^3 counts/kPa) and lower responses observed for ammonia, ethanol and acetone gases of about 6 x 10^3 counts/kPa, 4 x 10^3 counts/kPa and 3 x 10^3 counts/kPa, respectively.

In comparing radiation and transmission modes the normalized gas sensitivity of methanol was found to be about 55 x 10^{-3}/kPa and 19.7 x 10^{-3}/kPa, respectively, which is about 2.8 times better in sensitivity in the radiation mode compared to the transmitting mode. The response and recovery times of the sensor were found to be about 5.3 min. and 3.5 min., respectively.

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