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New sensing method for H_2S gas based on LPDM

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Abstract
H_2S sensing method using photodiode, LED and copper acetate is reported. In order to directly convert the output signal of optical H_2S sensing system consisted of LED and photodiode module to electrical signal, a copper acetate film coated on glass is used. The experimental results show that the sensitivity and linearity of sensor are improved as the thickness of the copper film increases. The linearity of concentration-voltage characteristics is good in range from 1 to 50 ppm at 15 ℃. The sensitivity of this sensor is 0.2-0.4v/1 ppm and the voltage at LPDM (LED photodiode module) represents the concentration of H_2S. Four times repetition is also recorded, which all demonstrate the good stability and repeatability of this system.

Keywords: Optical H_2S gas sensor, copper acetate, LPDM, film

1. Introduction
H_2S is a toxic and flammable gas and the clinical effects of H_2S depend on its concentration and the duration of exposure [1]. Although H_2S has a distinctive rotten egg smell, above concentrations of 100 ppm it is practically undetectable by smell due to saturation of olfactory nerves. H_2S above concentrations of 100 ppm poses serious health risks, including shock, irritation of the eyes, skin and respiratory paralysis. Concentrations above 500 ppm are immediately fatal as it affects the uptake of oxygen in the blood. It is generated by the bacterial degradation of organic matter in the absence of oxygen, during volcanic eruptions, extraction of natural gas or oil, mining activity, refining of natural gas and crude oil, etc. [2].
Considering all these important aspects various research groups have been working to develop probes for efficient and convenient methods to track the trace amount of H_2S in solution or at cellular levels [3-6]. Most of the H_2S sensors are based on small molecules, metal complexes, polymers, quantum dots and nanoparticles. A vast variety of chemical sensing devices has emerged from both research and commercialization efforts. Earlier work by several authors has shown the potential of several novel solid state materials ranging from metal oxides to polymer composites as active sensing materials [7-9]. These sensors work under demanding operating conditions at different temperatures, atmospheres and concentrations which may result in various problems in terms of poor stability, low sensitivity and selectivity [10, 11]. Among different materials the sensors made from copper acetate have shown most promising results [12, 13]. Copper acetate reacts with hydrogen sulfide both in solution and in solid state [12]. In organic solutions it produces organosols [14], while in aqueous solutions it produces a black precipitate of copper sulfide [15]. Copper acetate films have also been reported to react directly with hydrogen sulfide to form copper sulfide [16].
The copper acetate films can be used as simple optical indicators, because of the characteristic colors of different copper compounds [8, 9]. It has been suggested that the change in color of the sensing material upon reaction with H_2S can also directly convert to electrical signal. After the mechanical contact and potentiometric sensors, optical sensors are probably the most popular for measuring position and displacement. Their main advantages are simplicity, the absence of a loading effect, and relatively long operating distances. They are insensitive to stray magnetic fields and electrostatic interferences, which makes them quite suitable for many sensitive applications.

An optical position sensor usually requires at least three essential components: a light source, a photo detector and light guidance devices, which may include lenses, mirrors, optical fibers, etc. In order to produce the optical H_2S sensor using the predominance copper acetate, it is necessary to convert the change in color of the sensing material upon reaction with H_2S to electrical signal.

In this work, we propose a novel optical H_2S sensor consisted of LPDM in order to directly convert the change in color of the sensing material to electrical signal, using copper acetate.

2. Sensor design and principle

The proposed optical H_2S sensor is shown in Fig. 1 schematically.

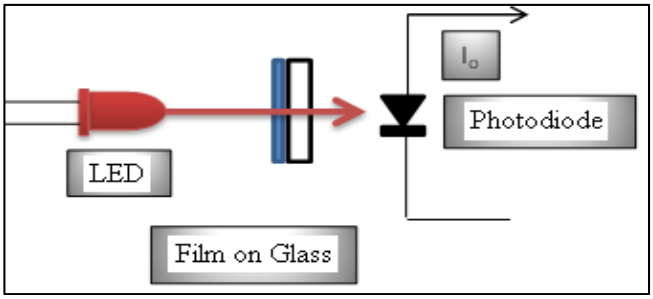


Fig 1: Schematic diagram for the operating mechanism of the proposed H_2S sensor.

It consists of the light source, the copper acetate film coated on glass, and photodiode. When the light is launched into the copper acetate film, a portion of this light is transmitted and the rest is absorbed at the film and transmitted light through the film enters into the photodiode. The photodiode user is generally most interested in the internal current (*Io*) that is generated for each received watt (*Pr*) of incident light power. This is termed the responsivity (*r*) of the photodiode, with units of ampere per watt (A/W):

$$r = \frac{I_0}{P_r} \quad (1)$$

When the color of the sensing material upon reaction with H_2S changed, the absorption factor of the incident light at film change, therefore intensity the transmitted light through the film change, consequently changed the photo current.

Therefore, when concentration of the H_2S gas changed, deviation ΔI of photo current at the photodiode is related with the thickness of the film d and responsivity (*r*) of the photodiode. With increasing the thickness of film, sensitivity of the sensor is improved but the response time of it becomes long. In general, the response time of the proposed sensor become longer because the diffusion time of the H_2S gas is increased when the thickness of the copper acetate film increases.

3. Experimental

3.1 Device fabrication

The copper acetate based sensing material were prepared by dissolving the copper acetate salt in a mixture of water, ethylene glycol (EG) and 2-propanol (IPA) or in a mixture of water and 2-butoxyethanol (BE). Soda-lime glass with a thickness of 2mm was first thoroughly cleaned employing ultrasonic and chemical means and copper acetate film layer of 100, 200, and 300μm thick have been deposited on glass by spin coating as shown at Table 1.

Table 1: Spin coating process parameters

N_o	Thickness, Mm	Fist spin Speed, rpm	Fist spin Time, s	Second spin Speed, rpm	Second spin Time, s
1	100	300	20	1000	5
2	200	250	15	1000	5
3	300	200	10	1000	5

In Table.2 shows property of soda-lime glass used in this experiment.

Table 2: Property of soda-lime glasses

Wavelength(nm)	230	365	405	436
Permeability(%)	-	>80	>85	>90

Photodiode, which used in the experiment, is BPW21R and parameter of BPW21R is listed in Table 3.

Table 3: Basic Characteristics of Silicon PN Photodiode BPW21R ($T_{amb} = 25$ ℃)

Parameters	Test condition	Symbol	Min	Typ	Max	Unit
Forward Voltage	$I_F = 50mA$	V_F		1.0	1.3	V
Breakdown Voltage	$I_R = 20\mu A, E = 0$	$V_{(BR)}$	10			V
Reverse Dark Current	$V_R = 5V, E = 0$	I_{ro}		2	30	nA
Diode Capacitance	$V_R = 0V, f = 1MHz, E = 0$	C_D		1.2		nF
	$V_R = 5V, f = 1MHz, E = 0$	C_D		400		nF
Dark Resistance	$V_R = 10mV$	R_D		38		GΩ
Open Circuit Voltage	$E_A = 1klx$	V_o	280	450		mV
Temp. Coefficient of V_o	$E_A = 1klx$	TK_{V_o}		-2		mV/K
Short Circuit Current	$E_A = 1klx$	I_K	4.5	9		μA
Temp. Coefficient of I_k	$E_A = 1klx$	TK_{I_K}		-0.05		%K
Reverse Light Current	$E_A = 1klx, V_R = 5V$	I_{ra}	4.5	9		μA
Sensitivity	$V_R = 5V, E_A = 10^{-2} \cdots 10^5 lx$	S		9		nA/lx

In Fig.2 it is shown the current-voltage characteristic of photodiode BPW21R.

Fig. 2 shows the complete schematic current/voltage characteristic of a photodiode under three levels of illumination. In the third quadrant only a very small current flows (I_o). As the level of incident light is increased, the curves shift bodily downward the negative current direction. This shift is linear in incident power. Considering all these important aspects we selected the third quadrant to operating range of BPW21R. In Fig.3 shown light/voltage converter of photodiode

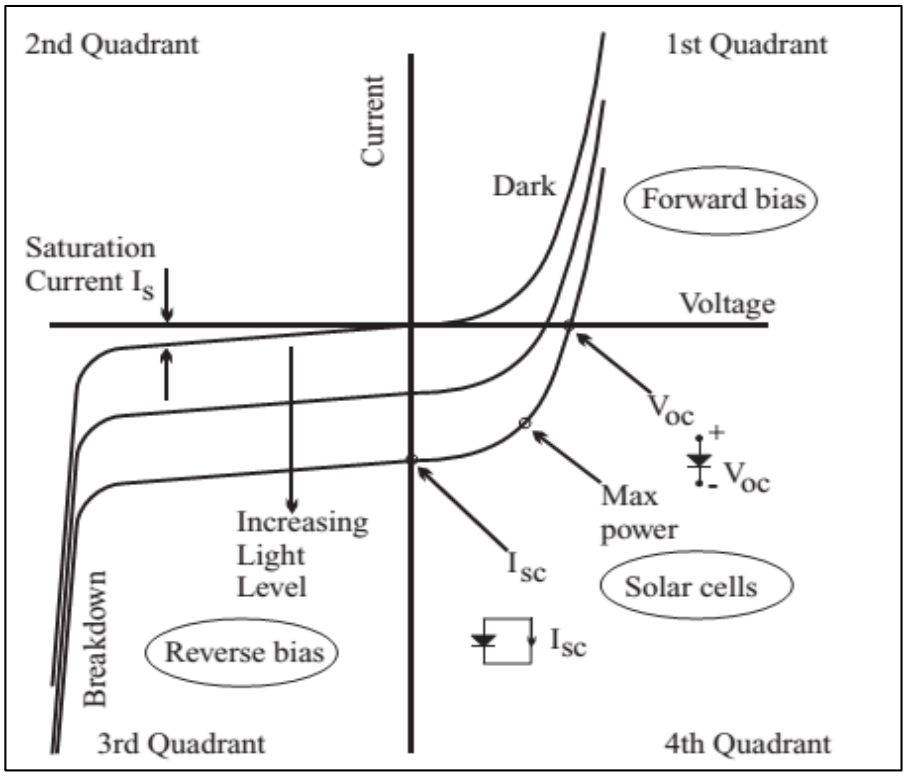


Fig 2: Four-quadrant current-voltage characteristic.

Without illumination this is similar to a conventional diode. Increasing illumination shifts the characteristic in the negative current direction. Detection is possible in quadrants 1, 3, and 4.

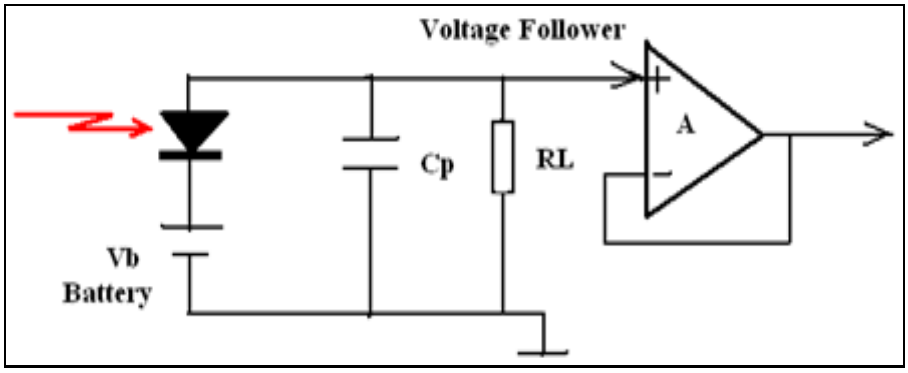
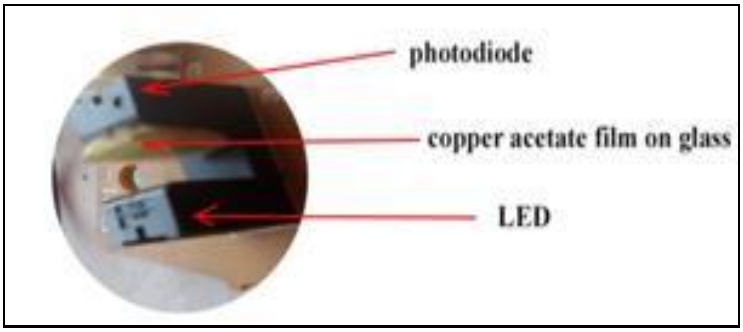


Fig 3: The light-voltage converter of photodiode

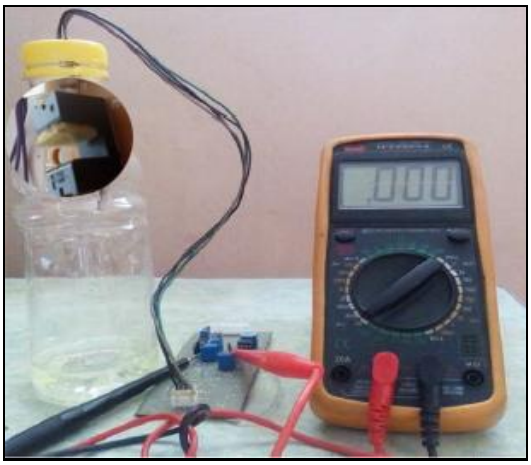
The output voltage at DC is as before just the photocurrent I_o flowing through the load resistor R_L:

$V_o = I_o R_L$ (2)

Output voltage of Light-voltage converter V_o be in proportion to photo current, finally it decrease with an increase of H_2S concentration. The wavelength and intensity of light source are 650 nm and 30mW, respectively. In our experiment, the light source, the copper acetate film deposited on glass and photodiode are assembled in black plastic carcass (LPDM) as shown in Fig.4 a), b is the photograph of experimental setup.



a)



b)

Fig 4: Gas-sensing setup a) Light source, the copper acetate film and photodiode assembled in black plastic carcass b) The gas-sensing setup

3.2 Experimental setup

The gas-sensing setup is shown in Fig.4.b. A gas measurement chamber consisted of a resin box with the diameter of 10 cm and length 12 cm, a H_2S sensor (Alpha Sense, with operation range of 0-200 ppm and response time < 35 s), a humidity and a temperature sensor (Sensirion SHT75, with a response time of 8 s)

4. Results and Discussion

4.1 Sensor sensitivity

Sensitivity is the ability to detect small concentrations of analyte that is how much the sensors output (voltage in our case) changes when the measured quantity (an H_2S concentration) changes. Typically sensitivity is measured as the slope of the response curve. A set of H_2S concentrations ranging from 1 ppm to 50 ppm were selected to study the response of the sensor. A 45% relative humidity was recorded during the measurements. Figure 5 shows several typical measurement of output voltage versus of H_2S concentrations with different thickness of films.

4.2 Response speed

Figure 6 shows the output voltage measured by sensor versus time when changing the H_2S gas concentration rapidly from 1 ppm to 50 ppm (absorption) for different thickness of films.

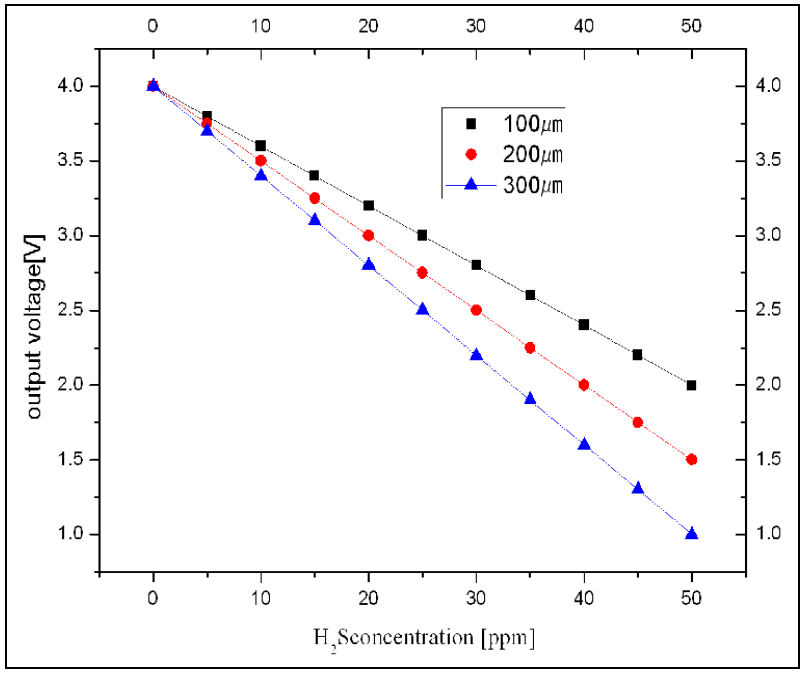


Fig 5: Output voltage versus of H_2S concentrations with different thickness of films.

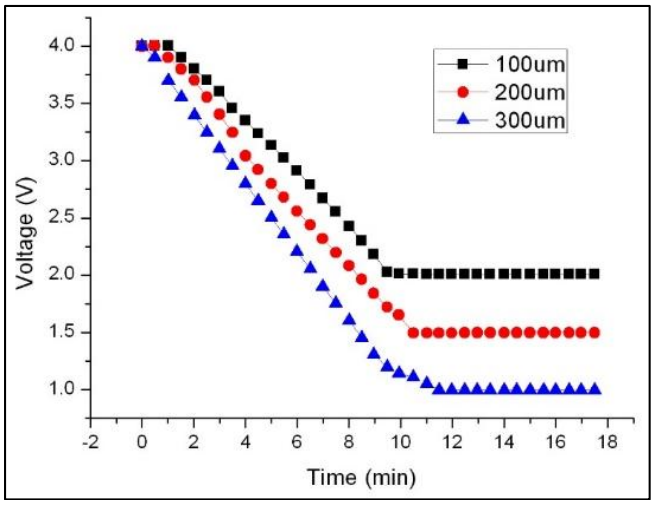


Fig 6: Output voltage measured by sensor versus time

4.3 Stability

The sensor is aged in a humidity chamber at 60%RH, 26 °C for 7 days. But no systematic drift is observable.

4.4 Effect of humidity

The influence of humidity on the performance of the sensor is very important since the sensor sometimes is performed at high humidity atmospheres (>80%). The sensor was exposed to different humidity atmospheres ranging from 14% RH to 80% RH. No humidity background optical response was obtained from the sensor when exposed up to 80% RH for four hours.

4.5 Selectivity and cross sensitivity

A hydrogen sulfide gas sensor is often cross sensitive to other sulfur containing compounds. Considering sensor applications in food packaging, the most common interfering gases can be CO_2, as well as amine-based, and sulfur containing compounds [10]. The cross sensitivity of sensor was tested towards SO_2, CO_2, and CH_3 NH_2 (methyl amine). The sensor exhibited no electrical or optical response towards either CO_2 or SO_2 during two hours of exposure (data not shown). It clearly shows that unlike gags no change color of the film similarity humidity.

5. Conclusion

In this work, the optic H_2S gas sensor system has been designed and evaluated to detect and measure H_2S gas. Experiments have verified a very satisfactory performance of the H_2S gas sensor in terms of its sensitivity, selectivity, and response to the H_2S gas and long-term stability. The sensor is fabricated and tested. Owing to the advantages of high-sensitivity and compactness, this proposed optic H_2S gas sensor will likely be developed for the practical applications. We already manufactured into one chip assembled with a LPDM (LED photodiode module), and a copper acetate film coated on glass. And it has been applied in several branches of industry.

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