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Development of Hydrogen Zinc Oxide Film Sensor

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Abstract–Zinc Oxide-based hydrogensensors are gaining wide popularity in the industry due to their reliability and high specificity to hydrogen. Any hydrogen sensor technology needs to satisfy the sensitivity basic requirement. Number of sensors have been developed to detect hydrogen depending on the approach used.

In this research, Zinc Oxide thin films have been prepared on glass substrates and explored as a fast response sensor to hydrogen gas. Films were prepared by vaporization deposition technique with annealing temperature of about 600^0 C. The optical characteristics of the prepared films show that they are highly sensitive, but their properties vary considerably when the measurements are conducted in vacuum or in air. The response – recovery time of Zinc Oxide materials to hydrogen gas is characterized to be relatively extremely short.

The new sensor system success to detect hydrogen in a wide dynamic range especially in the range of 500-650nm,in quick and safe mode.

Keywords— *Catalytic Bead, Field Effect Transistors, Gaschromatography, Hydrogen Sensor, Mass Spectrometry, Metal Oxide Semiconductor, Metal Oxide Sensors, Solid-State, Surface Acoustic Wave, Thermal Conductivity.*

I. INTRODUCTION

Hydrogen, which occupies about more than 90% of the atmosphere is a highly flammable gas and will burn at concentrations as low as 4% in air. The lower explosive limit and upper explosive limit are the two most common terminologies used to indicate the flammable levels for many fuels including hydrogen. But it has a larger window (4–75% v/v H2) of flammability in comparison to natural gas, gasoline, propane, ethane, methane, propylene, etc. The flammability limit of hydrogen is seven times wider than methane. It is, therefore, critical for a hydrogen sensor to have a wider measurement range (1–99% v/v H2) for safety applications than most common fuels. Hydrogen is the lightest of elements and the smallest molecule; it, therefore, has the greatest tendency to leak. Thus, for a process safety application, a hydrogen leak can be more dangerous and its detection becomes more challenging than other gases [5].

II. MECHANISMS FOR HYDROGEN SENSING

Number of approaches have been used to sense and detect hydrogen. Numbers of which are used in industry including the typical Gas Chromatography (GC), Mass Spectrometry (MS), Catalytic Bead (CB), and thermal conductivity. Semiconducting metal oxide and CB sensors are popular in solid-state technologies, which employ heated catalysts to sense hydrogen. These sensors require heating to about 300°C to enable surface reactions that promote hydrogen sensing.

Electrochemical sensors based on known electrolytic reactions of hydrogen. Sensors based on catalytic combustion are generally nonspecific, electrochemical hydrogen sensors with liquid or solid type electrolytes having leakage issues. The hydrogen sensors based on thermal conductivity, CB, metal oxide, and electrochemical technologies require the presence of oxygen for sensor operation. Oxygen plays a crucial role in promoting the grain boundary formation in metal oxide sensors and electron transfer reactions in electrochemical sensors[7].

III. TRADITIONAL APPROACHES TO HYDROGEN SENSING

Number of traditional approaches that can be used to detect hydrogen, these may include:

a. Thermal Conductivity (TC): which is the most widely applied measuring principle for the determination of hydrogen. It'smeasuring principle based on the differences in thermal conductivity of the gases to be measured. A Thermal Conductivity Detector (TCD) measures the concentration of a gas in a binary gas mixture by measuring the thermal conductivity of the sample gas and comparing it to the thermal conductivity of a selected reference gas.
b. Gas Chromatography (GC): is also another widely applied measuring principle for hydrogen detection. The disadvantages of GC are long response times (minutes) due to the chromatography, time-intensive sample preparation, consumable (carrier and calibration gases), and labor-intensive handling procedures. An advantage, however, is the ability to measure other gases such as nitrogen, oxygen, and carbon dioxide in the presence of hydrogen. But, this adds time to the total analysis[5].

IV. SOLID-STATE APPROACHES TO HYDROGEN SENSING

A wide variety of solid-state sensors based on hydrogen-specific palladium, "Metal Oxide Semiconductor (MOS), CB, electrochemical, and Surface Acoustic Wave (SAW) technology are used in the industry for several years. Microelectromechanical systems (MEMS) and nanotechnology-based devices for the measurement of hydrogen are the recent developments. These developments are mainly driven by the demands of the fuel cell industry. Solid-state approaches are gaining rapid popularity within the industry due to their low cost, low maintenance, replacements, and flexibility of multiple installations with minimal labor.

Hydrogen-Specific Palladium-Based Sensors are of three major classes of palladium-based hydrogen sensors[2]. The most popular class of palladium-based sensors is based on palladium resistors. A thin film of palladium deposited between two metal contacts shows a change in conductivity on exposure to hydrogen due to the phase transition in palladium. The palladium Field- Effect Transistors (FETs) or capacitors constitute is the second class, wherein the sensor architecture is in a transistor mode or capacitor configuration. The third class of palladium sensors includes optical sensors consisting of a layer of palladium coated on an optically active material that transforms the hydrogen concentration to an optical signal.

V. METAL OXIDE AND CATALYTIC BEAD SENSORS

Metal oxide sensors(MOS) which known also as semiconductor-based sensors, since they use a semiconducting film as the sensing element. The MOS have to be heated up to high temperatures and require the presence of oxygen. At high temperatures (300–500°C), there is a grain boundary formation in the MOS that enable detection of a number of gases. The MOS consume high power and have high false alarm issues [7]. CB sensors are a variation of this type and have an active sensing element with a coated catalyst and a passivated reference element for ambient temperature and pressure compensation.

Metal oxides are semiconductor-type materials that constitute a major category of gas sensors. The most widely researched metal oxide used for sensing applications is tin oxide (SnO_2). Typical processes for manufacturing SnO2-based sensors involve a thick semiconducting film made of micron size particles. Electron transport between particles is limited by the huge energy barriers at their grain boundaries

VI. THE DEVELOPED HYDROGEN ZINC OXIDE FILM SENSOR

This system consists basically on seven components including chamber, white photo diode, spectrometer, vacuum unit, gauge pressure, cylinder of hydrogen gas, and computer as shown schematically in figure: 1 below.

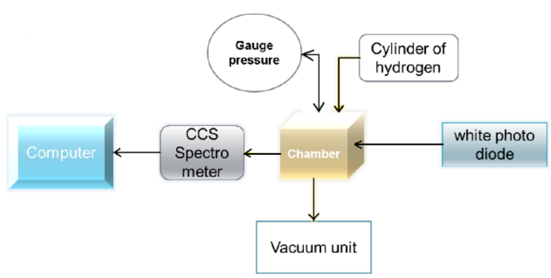

Figure 1: Sensor system components.

This system consists basically of seven components including; chamber, white photo diode, spectrometer, vacuum unit, gauge pressure, cylinder of hydrogen gas, and computer.

A sample of Zinc Oxide coated glass slide sensor should be prepared first to detect hydrogen. Also an X-ray sample testy is necessary to cheek its crystallization.After set up of the developed sensor system, each component should be responsible of the following:

- The coated glass substrate (Zinc Oxide thin films) should be placed inside the developed polymer square test chamber of 50 mm square base and of 75 mm height with the top removable cover. The effective volume of the chamber is 187500 mm^3.It has an inlet to allow the test gas to flow in.
- White photo diode is the sensor light source.
- CCS Spectrometer used to measure transmitted light.
- Vacuum unit used to evacuate the test chamber.
- Gauge pressure is to measure the current pressure of the chamber.
- Cylinder of hydrogen gas of a known concentration used to flow through the test chamber during measurement.
- Computer attached to process's recorded signals.

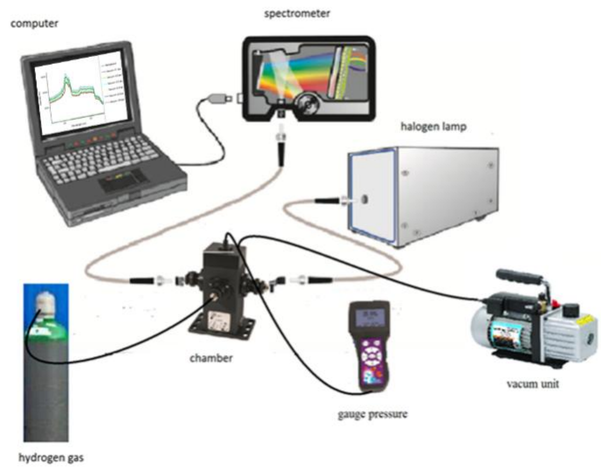

Figure 2: Sensor component setup.

VII. MEASUREMENT AND RESULTS

Tow testes were carried out to examine the developed Zinc Oxide thin film sensor. In both tests, results were obtained through adopting the following steps:

- The test chamber opening to place the Zinc Oxide thin film sensor on the sensor holder and close it.
- The necessary light source then directed by optical fiber and allowed to pass through the sample to the spectrometer.
- The rotary pump switched on to evacuate the test chamber to about -0.7 bar.
- Next, the hydrogen gas of a known concentration allowed to pass from the cylinder through the special inlet to the test chamber by opening the cylinder valve.
- Test chamber pressure measured by observing the gauge pressure.
- Spectrometer detected and analyzed transmitted signals and sent the data to the computer that process and record signals.

Numbers of measurements were carried out in different pressures. Six readings were observed for each sample test including pressures at -0.6, -0.5, -0.4, -0.3, -0.2 and -0.1bar. Frequency against transmitted light intensity graphs were produced for each reading. The six graphs were then combined together in one diagram with a background graph representing -0.7 pressure without Hydrogen.

Figure: 3 and figure: 4 below represent the resultant combined graphs for both sample tests.

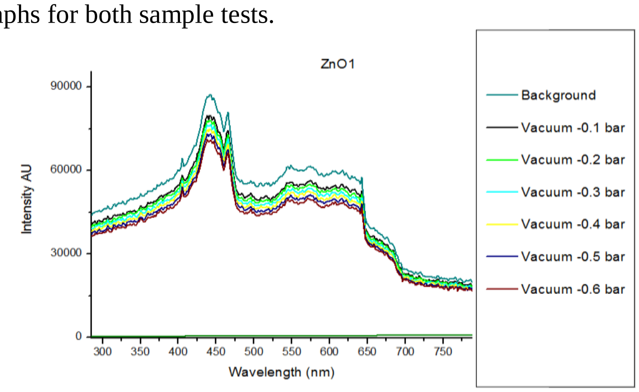

Fig: 3Combined graphs of the first sample

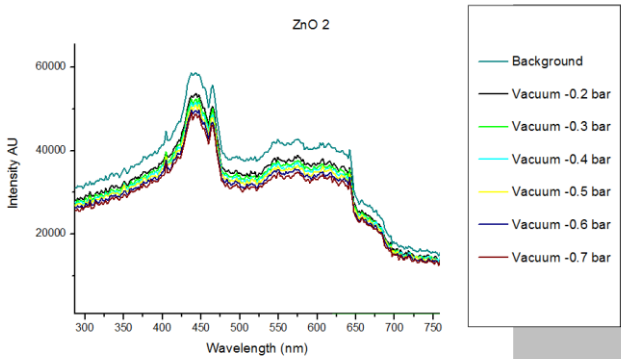

Figure 4: Combined graphs of the second sample

From the figures obtained above it can be obviously noted that by increase of concentration of hydrogen gas in the test chamber a transmitted light is also increase which means that the Zinc Oxide thin film sample successfully detect hydrogen gas.

Also it can note that the suitable wavelength that can be used to detect hydrogen gas is a visible band that located in the range of 500-650 nm.

CONCLUSION

This work is directed to develop a new sensor design that depends on the Zinc Oxide coated glass thin film which able to detect hydrogen gas. From measurements carried out and results obtained it can be concluded with that the new proposed sensor detects hydrogen successfully. Moreover, the new design provides number of advantages which includes:

- Rapid response to low hydrogen concentration,
- Wide dynamic range but more sensitive in 500-650 nm range.
- Electromagnetic Interference free,
- Safe to be use.

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