Chapter One

Introduction

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1.1 Introduction

Hydrogen is a highly flammable gas and will burn at concentrations as low as 4% in air. But has a larger window (4–75% v/v H_2) of flammability in comparison to natural gas, gasoline, propane, ethane, methane, propylene, etc. The flammability limit of hydrogen is seven times wider than methane table 1.1 [1]. It is, therefore, critical for a hydrogen sensor to have a wider measurement range (1–99% v/v H_2) for safety applications than most common fuels. Hydrogen is the lightest of elements and the smallest molecule. 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.

Table 1.1 Explosive Limits for Common Constituents in Process Industries

Fuel	LEL (%)	UEL (%)
Gasoline	1.4	7.6
Propane	2.1	10.1
Ethane	3	12.4
Hydrogen	4	75
Methane	5	15
Propylene	2.0	11.1

Several mechanisms for hydrogen sensing and detection have been studied since the early 1900s. Several traditional sensing mechanisms that are still widely used in the industry include the typical gas chromatography

(GC), mass spectrometry (MS), catalytic bead (CB), and thermal conductivity (TC). Semiconductor metal oxide and CB sensors are popular solid-state technologies, which employ heated catalysts to sense hydrogen. These sensors require heating to about 300°C (a potential source of ignition!) to enable surface reactions that promote hydrogen sensing. Electrochemical sensors are based on known electrolytic reactions of hydrogen. Sensors based on catalytic combustion are generally nonspecific yet. 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. The most promising solid-state technology is based on a hydrogen-specific material, palladium, which does not require oxygen for operation. Palladium-based sensors are gaining wide popularity in the industry due to their reliability and specificity to hydrogen. Any hydrogen sensor technology needs to satisfy the three basic requirements: sensitivity, selectivity, and specificity (i.e., the sensor should be functional over the range of measurement and be free of cross-interference from other gases present in the environment) [2].

1.2 Objective of the work

The main objective of this work is to design and implements of optical fiber sensor based on a hydrogen-especial material that is inert to variation environment, and rapid detection of Hydrogen leaks in hostile and harsh environments.

1.3 literature review

Hydrogen, the most basic element, has been used as a chemical for decades and is now emerging as a universal energy carrier with important environmental and energy security advantages. Hydrogen can be made from a variety of local resources and is used in many different applications, such as: producing electric power for the electric utility grid or micro-grids; fueling cars and buses; and providing power for materials handling equipment, and remote communications and security equipment. In addition, the military is evaluating hydrogen as a means to power the mobile equipment of military forces and unmanned vehicles because some hydrogen appliances, like fuel cells, are quiet, emit very little heat and can be lighter than loads of rechargeable batteries [3].

Hydrogen is potentially dangerous. It can be flammable and even explosive at concentrations above 40,000 ppm [4]. The famous explosion and burning of the Hindenburg in 1937 [5], illustrated just how dangerous this gas can be. The large airship, filled with hydrogen gas, caught fire and exploded and crashed to the ground, resulting in 36 fatalities. The fame of this accident caused airships to fall out of favor as transportation. A need for hydrogen sensors as a safety measure was seen as a result of this and other accidents involving fires and explosions.

A static hydrogen sensor is likely to be placed in a building or other enclosed space. This type of hydrogen sensor works in the same way as a hand-held unit but is linked to some type of monitoring system that may include alarms. In these applications, hydrogen sensors serve to detect a

buildup of the gas above normal atmospheric levels before it becomes dangerous.

Fiber-optic sensors have been made in a variety of configurations, most using one or more thin films at the end of the cable. Butler (1991, 1994) was suggested using a thin film of Pd as a mirror on the end of a fiber optic cable. Garcia (1996) and Mandelis (1998) describe a more sensitive (and much more complex) method of sensing hydrogen optically. Ito (1984) originally proposed using the palladium-catalyzed reaction of amorphous tungsten oxide with hydrogen in a fiber-optic hydrogen detector.

A different sensor design using a surface-plasmon resonance (SPR) configuration was also evaluated (Raether, 1988 and Chadwick, 1993). Chemochromic materials, such as tungsten oxide and certain Lanthanide hydrides (Griessen, 1997) were used in thin film stacks on a sensor head at the end of an optical fiber (Benson, 1998). Yet another variation of this sensor design uses a Pd coating on the sides of the fiber optic cable, after the jacketing material in a section of the fiber is removed (Tabib-Azar, 1999). In this configuration the Pd interacts with the evanescent field as the light beam propagates down the fiber via total internal reflection. When exposed to hydrogen, the complex index of refraction of the Pd film changes, affecting the transmission of light down the fiber. Detection of variations of light intensity at the end of the fiber signals changes in the Pd film due to the incorporation of hydrogen. This construction allows multiple sensors to be deposited along a single strand of fiber-optic cable. However, to allow this concept to identify the sensing station that has detected hydrogen, fiber optic Bragg gratings (FBGs) must be etched into the fiber at each station (Sutapun, 1999), and to improved upon the chemochromic materials used as

the sensing material under the Pd layer (Liu, 2002) [6]. A fiber-optic hydrogen gas sensor using catalyst-supported tungsten trioxide was characterized in 2003 by (S. Okazakia) [7]. In June 2004, (Hyeonsik) investigate hydrogen sensors based on Gasochromic Oxide Thin Films [8]. A Palladium coated PMMA (Poly Methyl Methacrylate) optical sensor to detect hydrogen in humid environment is presented by (M.A.Nabeerasool1) in 2010 [9]. The Improvement of ZnO and SnO2 hydrogen gas sensors in 2011 by Qahtan Ghatih Hial from University of Baghdad College of Science [10].

1.4 Thesis layout

This research work organized into five chapters. The first chapter is a general introduction, where the overview of the field of study and the scope of the work are carried outlined. The second chapter entitled briefly describes the background and working principle of the development of different types of sensors design and surveys the various methods used currently to improve the sensor characteristics. The fabrication and characterization of the sensing element as well as testing the sensors towards hydrogen reducing gas is discussed in the third chapter. Moreover, this chapter provides detailed information about the development of the gas sensor-testing chamber and the protocol of using the chamber.

Chapter four includes a detailed discussion of the experiments carried out and the results obtained from the tests applied to the development gas sensors. Finally, conclusions of the work and recommendations for further studies are summarized.

Chapter Two

Theoretical background

Chapter Two

Theoretical back ground

2.1 Hydrogen

Hydrogen is by far the most abundant element and may account for more than 90% of the atoms or about 75% of the mass of the universe. Helium atoms make up most of the remainder. All of the other elements together contribute only slightly to the total mass.

The chemical composition of the universe is undergoing continuous change. Hydrogen is being converted into helium, and helium is being changed into heavier elements. As time goes on, the ratio of heavier elements increases relative to hydrogen [11].

Hydrogen atom is the lightest element, with its most common isotope consisting of only one proton and one electron. Hydrogen atoms readily form H_2 molecules, which are smaller in size when compared to most other molecules. The molecular form, simply referred to as hydrogen is colorless, odorless, and tasteless and is about 14 times lighter than air, and diffuses faster than any other gas. [12].

Hydrogen was prepared many years before it was recognized as a distinct substance by Cavendish in 1766. It was named by Lavoisier. Hydrogen is the most abundant of all elements in the universe. It is thought that the heavier elements were, still being built from hydrogen and helium. In 1972, a Livermore (California) group reported a pressure-volume point centered at 2 Mbar. In 1973, a group of Russian experimenters have produced metallic

hydrogen at a pressure of 2.8 Mbar. It has been predicted that metallic hydrogen may be metastable; others have predicted it would be a superconductor at room temperature. On earth, hydrogen occurs chiefly in combination with oxygen in water, but also present in organic matter such as living plants, petroleum, coal, etc. Present as the free element in the atmosphere, but only to the extent of less than 1 ppm by volume. H_2 is the lightest of all gases, and combines with other elements, sometimes explosively, to form compounds.

Great quantities of hydrogen are required commercially for the fixation of nitrogen from the air in the Haber ammonia process and for the hydrogenation of fats and oils. H_2 is also used in large quantities in methanol production, in hydrodealkylation, hydrocracking, and hydrodesulfurization.

Hydrogen used as a rocket fuel, for welding, for production of hydrochloric acid, for the reduction of metallic ores, and for filling balloons.

Liquid hydrogen is important in cryogenics and in the study of superconductivity, as its melting point is only a 20°C above absolute zero. Hydrogen consists of three elements, most of which is ^{1}H. The ordinary isotope of hydrogen, H, is known as protium. In 1932, Urey announced the discovery of a stable isotope, deuterium (^{2}H or D) with an atomic weight of 2. Deuterium is present in natural hydrogen to the extent of 0.015%. Two years later an unstable isotope, tritium (^{3}H), with an atomic weight of 3 was discovered. Tritium has a half-life of about 12.32 years. Tritium atoms are also present in natural hydrogen but in much smaller proportion. Tritium is readily produced in nuclear reactors and is used in the production of the hydrogen bomb. Also is used as a radioactive agent in making luminous paints, and as a tracer. On August 27, 2001 Russian, French, and Japanese physicists working at the Joint Institute for Nuclear Research near Moscow

reported that had made “super-heavy hydrogen”, which had a nucleus with one proton and four neutrons. Using an accelerator, they used a beam of helium-6 nuclei to strike a hydrogen target, which is resulted in the occasional production of a hydrogen-5 nucleus plus a helium-2 nucleus.

Consideration is being given to an entire economy based on solar- and nuclear-generated hydrogen located in remote regions, power plants would electrolyze sea water; the hydrogen produced would travel to distant cities by pipelines. Pollution-free hydrogen could replace natural gas, gasoline, etc., and could serve as a reducing agent in metallurgy, chemical processing, refining, etc. It could also be used to convert trash into methane and ethylene. Hydrogen is being investigated as a substitute for deep-sea diving applications below 300 m. Hydrogen is readily available from air product suppliers [12].

2.1.1 Properties of Hydrogen

Table 2.1 Typical properties of Hydrogen [3].

Property	**Value**
Atomic Number	1
Atomic Mass	1.00794 g/mol
Group No	1
Group Name	Non-metals
State	gas at 298 K (the lightest gas known)
Color	Colorless
Classification	Non-metallic
Boiling Point	20.268K (-252.87°C)
Melting Point	14.01K (-259.14°C)
Critical temperature	33K (-240°C)
Density	0.08988g/cm^3

2.1.2 Hazards and Optical Properties

Hydrogen is a tasteless, colorless, odorless and extremely flammable gas. A refractive index as a (gas) is 1.000132, and refractive index as a (liquid) is 1.12.

2.1.3 Industrial syntheses

Hydrogen can be prepared in several different ways but the economically most important processes involve removal of hydrogen from hydrocarbons [13].

2.2 Palladium

Palladium discovered in 1803 by Wollaston. Palladium is found along with platinum and other metals of the platinum group in deposits of Russia, South Africa, Canada (Ontario), and elsewhere. Natural palladium contains six stable isotopes.

Twenty-nine other isotopes are recognized, all of which are radioactive. Separated from the platinum metals depends upon the type of ore. Palladium is attacked by nitric and sulfuric acid. In room temperatures the metal has the unusual property of absorbing up to 900 times its own volume of hydrogen, possibly forming Pd_2H. Hydrogen readily diffuses through heated palladium and this provides a means of purifying the gas. Palladium is a good catalyst that used for hydrogenation and dehydrogenation reactions. White gold is an alloy of gold decolorized by the addition of palladium, like gold. The metal is used in dentistry, watch making, and in making surgical instruments and electrical contacts. Palladium recently has been substituted for higher priced platinum in catalytic converters by some automobile companies. This has caused a large increase in the cost of palladium. Palladium, however, is less resistant to

poisoning by sulfur and lead, than platinum, but its proved usefulness in controlling emissions from diesel vehicles [12].

Palladium is a steel-white, ductile metallic element resembling and occurring with the other platinum group metals (PGMs) and nickel. It exists in three states: Pd^{0} (metallic), Pd^{2+} and Pd^{4+}. It can form organometallic compounds, only few of which have found industrial uses.

Palladium metal is stable in air and resistant to attack by most reagents except aqua regia and nitric acid. Currently, there are no published measurement methods that distinguish between different species of soluble or insoluble palladium in the environment [14].

2.2.1 Properties of Palladium

Table 2.2 Typical properties of Palladium [3].

Property	**Value**
Atomic Number	46
Atomic Mass	106.42 (1) g/mol
Group No	10
Group Name	Precious metal or Platinum group metal
State	solid at 298 K
Classification	Metallic
Color	silvery white metallic
Boiling Point	3236K (2963°C)
Melting Point	1828.05K (1554.9°C)
Density	12.023g/cm3

2.2.2 Hazards

Fine powder may cause fire or explosion in air. It may cause skin, eye or respiratory tract irritation [13].

2.3 Zinc oxide

Recently, Zinc Oxide (ZnO) has attracted much attention within the scientific community as a ‘future material’. This is however, somewhat of a misnomer, as ZnO has been widely studied since 1935 [15]. ZnO is a versatile material which has been used in a variety of thin film devices, including surface acoustic wave devices, transparent conducting electrodes in solar cell, and thin film gas sensors [16]. Zinc Oxide has a wide range of industrial applications. An environmentally friendly, wide-band gap semiconductor [17]. This wide range of applications is a result of the fact that ZnO is both a piezoelectric and an electro-optic material, and a semiconductor which possesses a wide optical band gap (3.3 eV). In recent years, a great deal of effort has been devoted to the investigation of hydrogen in semiconductors as well as hydride forming metals. Research has been driven not only by technological requirements of state of the art applications, but also by the lack of a fundamental understanding on the role of hydrogen in those materials. Among any configurations, several notable results have been reported from observations on hydrogen in a compound semiconductor ZnO. Zinc Oxide thin film that used by surface acoustic device was required preferred orientation of c-axis, high resistivity and uniform surface morphology [16].

2.3.1 Properties of Zinc oxide

Table 2.3 Typical properties of Zinc Oxide [18].

Property	**Value**
Lattice constants	T=300K
a_o	0.32469 nm
c_o	0.52069 nm

Density	5.606 g/cm^3
Melting point	2248 K
Relative dielectric constant	8.66

2.4 Thin Film Deposition Techniques

2.4.1 Introduction

The most suitable methods for the preparation of such films are the physical vapor deposition methods (PVD). PVD are vacuum deposition methods, where the thin film material will be transferred from solid, liquid or gas phase precursors into a vapor phase by different vaporization methods and condensed after the transport as a molecular beam in vacuum or by diffusion through a diluted background gas on a suitable substrate, the temperature of which must be below the melting point of the deposit.) Chemical compounds are usually decomposed during their vaporization. Their deposition therefore requires a chemical reaction of the growing film surface with a suitable reactive gas.

The properties of the vaporized species can be strongly influenced with respect to the particle energy and distribution by the vaporization process. The methods can be classifying regarding the vaporization of the PVD as thermal evaporation and sputtering methods. The properties of the deposited films are determined essentially by the growth conditions during their deposition. There are many factors, which can influence the growth processes and modify the structure of the films [19].

2.4.2 Thin Film Deposition Methods

2.4.2.1 Thermal Evaporation

The thermal evaporation of a solid precursor of the thin film material takes place by heating the source above the melting point, where the vaporization of the melt increases with increasing. The most simple evaporation sources are resistance heated sources [20]. The basic components of a deposition system with a thermal evaporation source are illustrated in Figure (2.1).

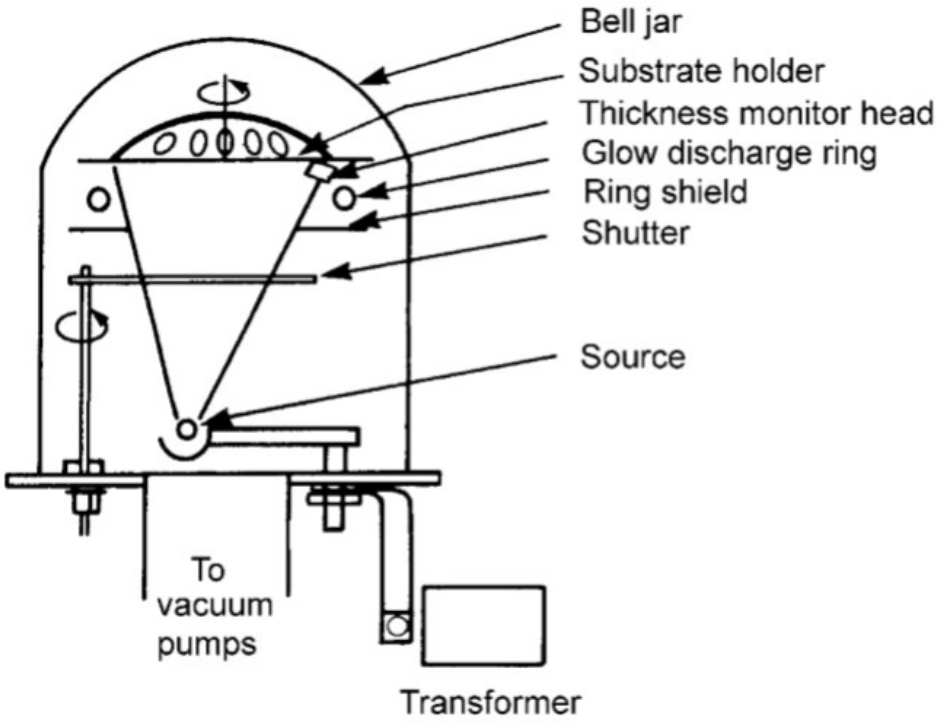


Figure (2.1) Vacuum system for deposition from resistance heated sources.

Because the distribution of the vapor flux has approximately a cos Θ characteristic, the condensation on planar substrates provides films of inhomogeneous thickness. Therefore a large source-substrate distance is required or the substrates must be moved in a suitable manner. Resistance heated sources are restricted to materials of low or moderate melting points because of undesired reactions between the melt and the resistance carrier and his temperature limitations. These disadvantages can be avoided by the

electron beam evaporation [21]. The main disadvantage of thermal evaporators is the different evaporation speed for different materials at the same temperature leading to deviations of the film composition compared with the source material, when it consists of alloys or compounds.

2.4.2.2 Sputtering Methods

In contrast to the evaporation methods the sputtering methods are characterized by a non-thermal mechanism for the transfer of the solid target material into the vapor phase, which is based on the impulse transfer from accelerated energetic particles to the surface and near surface atoms of the target [22]. The impact of energetic particles on the surface of a solid state results in a number of secondary processes.

Besides implantation, trapping, chemical effects, mixing and lattice destruction important essentially with respect to the growth of thin films under ion bombardment several types of particles can be emitted by single and multistep impulse transfer and a part of the incoming ions are simply reflected. The sputtered particles consist mainly of single neutral atoms and a small part of molecules [23].

2.4.3 Thin Film Growth

The properties of thin films are determined essentially by the growth conditions during their deposition. There are many factors, which can influence the growth processes and modify the real structure of the film. So that the optimization of the deposition parameters is dominated mainly by empirical points of view and experience. The reason is the depressingly large array of factors responsible for the growth processes and the developing of film structure and the difficulty to measure and control them in situ during

the film deposition. In general three steps are essential in the thin film deposition process: nucleation and coalescence, followed by different growth processes as special cases of crystal growth (columnar, polycrystalline, epitaxial) [19].

2.4.4 A classification of thin film configurations

As a guide for the development and application of concepts that are useful for describing mechanical behavior of solid thin film and multilayer materials, it is convenient to classify structures in terms of their geometrical configurations and the nature of constraint on their deformation imposed by their surroundings. The configurations are classified in terms of the relative extent of the solid bodies in three orthogonal directions, with the orientation of the reference coordinate system being dictated by the configuration. The degree of constraint is determined by the interaction of the thin film structure with other deformable solids to which it may be attached, or otherwise be in contact. The former situation requires compatibility of deformation, while the latter requires some restriction on its motion. The categories of configuration are termed film (or layer), line (or wire), and island (or dot); the categories of constraint are termed unconfined, partially confined and fully confined. These classes are illustrated in Figure (2.2), but its adoption can facilitate understanding of the ranges of applicability of the various ideas in the field. Reference to Figure (2.2), a structure of an extent that is small in one direction compared to its extent in the other two directions is termed a thin film; in structural mechanics, such configurations are identified as plates or shells. The qualifier `small' as used here means that the largest dimensions are at least twenty times greater than the small

dimension, and more commonly are hundreds of times greater than the small dimension. A structure that has small extent in two directions compared to its extent in the third direction is termed a line or wire; such configurations are usually identified as rods or bars in structural mechanics. Lastly, a structure that has small extent in all three directions, compared to the dimensions of its surroundings in this case, is termed an island or a dot. Concerning the degree of constraint on deformation, a small structure is said to be unconfined if the boundaries associated with its thin dimensions are free to displace without restriction. On the other hand, it is said to be fully confined if all boundaries associated with, its thin dimensions are constrained against deformation. In virtually all cases, the constraint at a boundary is due to another material which shares that boundary as a common interface.

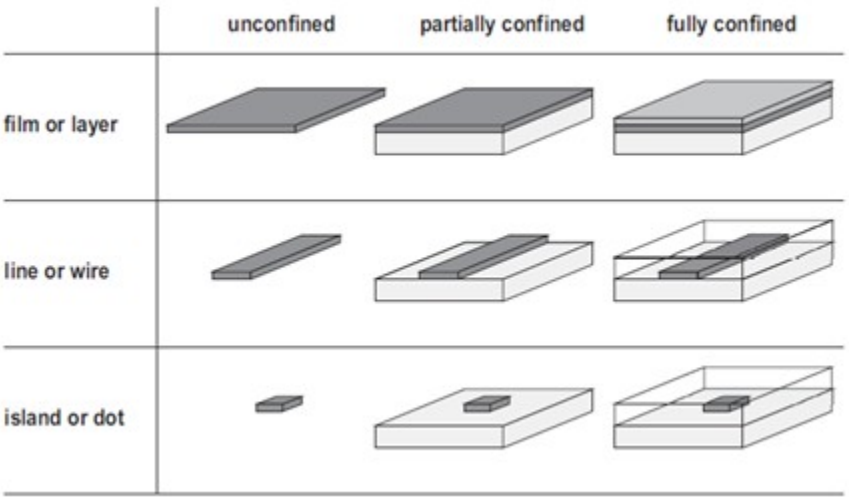


Figure (2.2): A categorization of small volume structures in terms of their general shapes and levels of constraints.

The structure is said to be partially confined if displacement of its boundaries associated with some, but not all, directions of thinness are

unconstrained. The classification matrix in Figure (2.2) includes some illustrations of varying degrees of confinement and thinness. As a specific example, consider a layer of a Si Ge alloy 1 μm in thickness, which is deposited on a 1cm by 1 cm area of a Si substrate that is 0.5mm thick. This configuration results in a partially confined thin film structure. A stripe of copper with a square cross-section that is 0.5 μm on a side and a length of 5mm deposited on a relatively thick Si substrate is a partially confined line. If the surfaces of the Si substrate and the wire are then completely covered over with a 1 μm thick coating of SiO2 to electronically isolate the wire, the structure becomes a fully confined line. An In As quantum dot being formed by stress driven surface diffusion is a partially confined island. If this configuration is then covered over by a blanket deposit of Al As, the final structure is a fully confined island or quantum dot configuration [24].

2.5 Interaction of Hydrogen with palladium

The interaction of atoms and molecules with surfaces is of great technological relevance. The rate of chemical reactions can be tremendously increased by the presence of a catalytic surface. Semiconductor devices are built by deposition and growth processes in molecular beam epitaxy. On the other hand, the performance of catalysts can be poisoned by the presence of adsorbents, and corrosion decreases the durability of mechanical systems. Again, these harmful processes can be avoided by coating the surfaces with some suitable material.

In particular, the interaction of hydrogen with palladium surfaces has been studied in great detail, both experimentally as well as theoretically. The bulk Pd can absorb huge amounts of hydrogen thus making it a possible

candidate for a hydrogen storage device in the context of the fuel cell technology [25].

Thin film palladium has been the leading material investigated for sensing hydrogen due to its unique material response to hydrogen. The sensing of hydrogen using palladium is a two-step process Figure (2.3). In the first step, molecular hydrogen dissociates on the surface of palladium and is converted into elemental hydrogen, which is the rate-limiting step for the detection. The elemental hydrogen then diffuses into the palladium lattice causing an expansion in the lattice and a phase transition. The main principle of hydrogen sensing is the conversion of the α phase (conductive metallic phase) of palladium, to the β phase (less conductive phase).

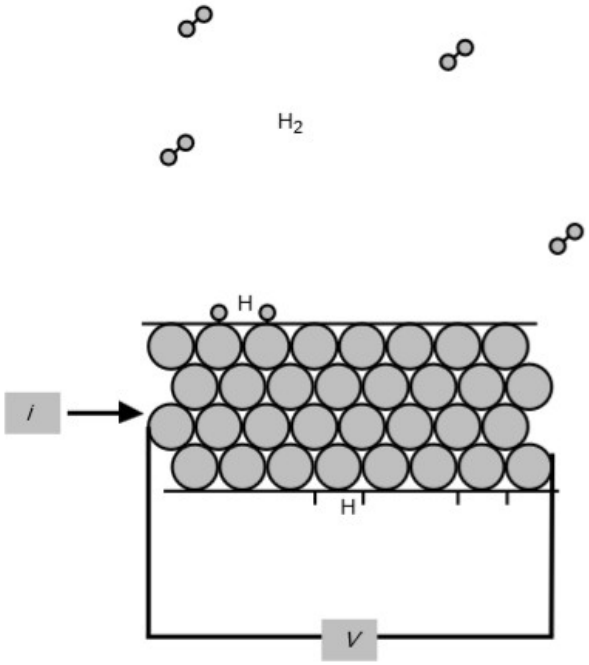


Figure 2.3: The mechanism of hydrogen sensing with palladium thin films.

In the presence of the Pd coating, the evanescent fields are altered. As the Pd film absorbs the hydrogen, it expands; because of the formation of the

palladium- hydride, the real and imaginary part of its refractive index change. The expansion of the Pd thin film induces a strain in the fiber and through the elasto-optic effects; it influences the phase of the guided light. As the Pd film adsorbs the hydrogen, it expands; because of the formation of the palladium- hydride, the real and imaginary part of its refractive index change. The change in the imaginary part of the refractive index results in a change in the adsorption of the light and, hence, it can be detected by monitoring the light intensity. The change in the real part of the palladium's refractive index results in effective phase changes in the light that can be detected [2].

2.6 Interaction of Hydrogen with Zinc Oxide

Hydrogen is known to be a good electron donor in ZnO [26]. It is experimentally demonstrated that, when the ZnO surface is covered with H, substantial electron donation from H to the ZnO substrate occurs and the surface exhibits metallic properties [27]. Considering the large band-gap energy of ZnO, such a H-induced change in the band structure is unexpectedly drastic. Thus, understanding the metallization mechanism is important from scientific and technological viewpoints [28].

Typical columnar structure and circular shaped grains appeared none hydrogen, but increasingly collapsed columnar structures with elongated textured were obtained above 4 vol% hydrogen. The resistivity of ZnO films decreased sharply in the range of 0~24 vol% hydrogen and exhibited a minimum for 24 vol % hydrogen [29].

2.7 Adsorption Mechanisms

Physical adsorption (physisorption) is defined as an adsorption event where no geometric change occurs to the adsorbed molecule and van der Waals forces are involved in the bonding between the surface and adsorbate [30].Chemical adsorption (chemisorption) is the formation of a chemical bond between the molecule and the surface during the adsorption process [31].

2.8 Traditional Approaches to Hydrogen Sensing

2.8.1 Thermal Conductivity

Thermal conductivity is the most widely applied measuring principle for the determination of hydrogen. The measuring principle is 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.

2.8.2 Gas Chromatography

Gas chromatography is the second most 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.

2.8.3 Solid-State Approaches to Hydrogen Sensing

A wide variety of solid-state sensors based on hydrogen-specific palladium, metal oxide semiconductor (MOS), electrochemical, and surface

acoustic wave (SAW) technology are used in the industry for several years. Micro electromechanical 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.

2.8.3.1 Hydrogen-Specific Palladium-Based Sensors

There are three major classes of palladium-based hydrogen sensors [32]. 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 the second class, where in 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.

2.8.3.2 Metal Oxide and Catalytic Bead Sensors

Metal oxide sensors are also known as semiconductor-based sensors since they use a semiconducting film as the sensing element [33]. 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. 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.

2.8.3.3 Electrochemical-Based Sensors

Electrochemical sensors represent the most commercially successful sensor type due to the simplicity of their operation, high sensitivity, and proven sensor mechanisms [34]. The sensor architecture mimics a battery containing an anode, cathode, and a supporting electrolyte. These sensors can operate well at room temperature in the presence of oxygen. Hydrogen is detected using a specific electrochemical reaction between the anode and the cathode. The resulting signal is either a current or a voltage change in the external electrical circuit.

2.8.3.4 Nanotechnology Approaches for Hydrogen Sensing

Nanotechnology involves the manipulation of materials in the molecular level to provide significant material property enhancements. Carbon nanotubes, nanoparticles, nanowires, nanowhiskers, metallic nanotubes, metal oxide nanostructures, and nanoclusters are some of the nanomaterials investigated for hydrogen sensing. Most of these materials are nanoscale components of bulk materials, which have been investigated for hydrogen sensing for several years. Nanomaterials have high surface to bulk ratio resulting in higher sensitivity, faster response time, and high selectivity in comparison with their bulk counterparts.

2.8.4 Types of Palladium Hydrogen Sensors

Several types of palladium-based hydrogen sensors have been reported in the literature. The most notable ones are based on Pd thin-film resistors, FETs, Pd nanowires, Pd nanoparticle networks, Pd nanoclusters, and Pd nanotubes

2.8.4.1 Palladium Field-Effect Sensors

Hydrogen sensors based on the “field effect” of palladium have been investigated extensively in the literature [35]. The field effect results due to the rapid dissolution of hydrogen in the palladium surface arranged in a Pd–SiO2–Si configuration. The sensor relies on an electric field resulting from the charge transfer between palladium and hydrogen on its surface. The FETs [36] and metal–insulator–semiconductor (MIS) [37] are the two major types of device structures that have been studied for palladium-based hydrogen sensing. Palladium is catalytically active, permeable to hydrogen, and can be readily used in FET and MIS devices.

2.8.4.2 Palladium-Based Resistors

Thick- and thin-film palladium-based resistors have been reported for hydrogen sensing [38]. The thick-film device uses printed palladium paste on a ceramic substrate in a four- resistor network (Wheatstone bridge). Two opposed resistors are covered to isolate them from the ambient atmosphere. The exposure of the uncovered resistors to hydrogen results in a change in resistivity of the thick-film material and a shift in the balance point of the bridge, which can be scaled to the hydrogen concentration. The thin-film device is equivalent in design to the thick film; here, much thinner films (typically vacuum deposited) are used as the resistors. Thin-film palladium detectors have been prepared by depositing palladium through electron beam

evaporation [39], RF magnetron sputtering [40], micro contact printing [41], and wet electrochemistry. Most palladium resistors have fouling issues on the palladium surface due to impurities and pollutants in, or reaction with the air. The fouling on the palladium surface can be reduced by the addition of a second metal (alloy) to palladium.

2.8.4.3 Palladium-Coated Fiber Optic Sensors

A fiber optic hydrogen sensor consists of a palladium coating at the end of an optical fiber that senses the presence of hydrogen in air. When the coating reacts with the hydrogen, its optical properties are changed. Light from a central electro-optic control unit is projected down the optical fiber where it is either reflected from the sensor coating back to central optical detector or is transmitted to another fiber leading to the central optical detector. A change in the reflected or transmitted intensity indicates the presence of hydrogen. The fiber optic detector offers inherent safety by removing all electrical power from the test sites and reduces signal-processing problems by minimizing electromagnetic interference.

2.8.5 Metal Oxide and Catalytic Bead Hydrogen Sensors

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 SnO_2-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

2.8.6 Electrochemical Hydrogen Sensors

Electrochemical sensors have been used for several decades for monitoring several toxic and volatile organic compounds. There has been little research in the electrochemical hydrogen sensors in comparison to the palladium-based sensors. Electrochemical sensors operate similar to a battery with a liquid or solid electrolyte. The electrochemical sensors generally have a two or three electrode configuration with a membrane for gas transport and a solid housing to prevent electrolyte leakage [2].

2.9 Optical Fiber

Optical fibers are dielectric waveguide [42] structures that are used to confine and guide the light. An optical fiber is composed of three parts; the core, the cladding, and the coating or buffer. The core is a cylindrical rod of dielectric material and is generally made of glass. Light propagates mainly along the core of the fiber [43].

The cladding layer is made of a dielectric material with an index of refraction. The index of refraction of the cladding material is less than that of the core material. The cladding is generally made of glass or plastic. The cladding executes such functions as decreasing loss of light from core into the surrounding air, decreasing scattering loss at the surface of the core, protecting the fiber from absorbing the surface contaminants and adding mechanical strength. The coating or buffer is a layer of material used to protect an optical fiber from physical damage. The material used for a buffer is a type of plastic. The buffer is elastic in nature and prevents abrasions [43]. The light-guiding principle along the fiber is based on the “total internal reflection”. Optical fibers are divided into two groups called single mode and multimode. In classifying the index of refraction profile, we differentiate between step index and gradient index. Step index fibers have a

constant index profile over the whole cross section. Gradient index fibers have a nonlinear, rotationally symmetric index profile, which falls off from the center of the fiber outwards. Figure (2.4) shows the different types of fibers. [44].

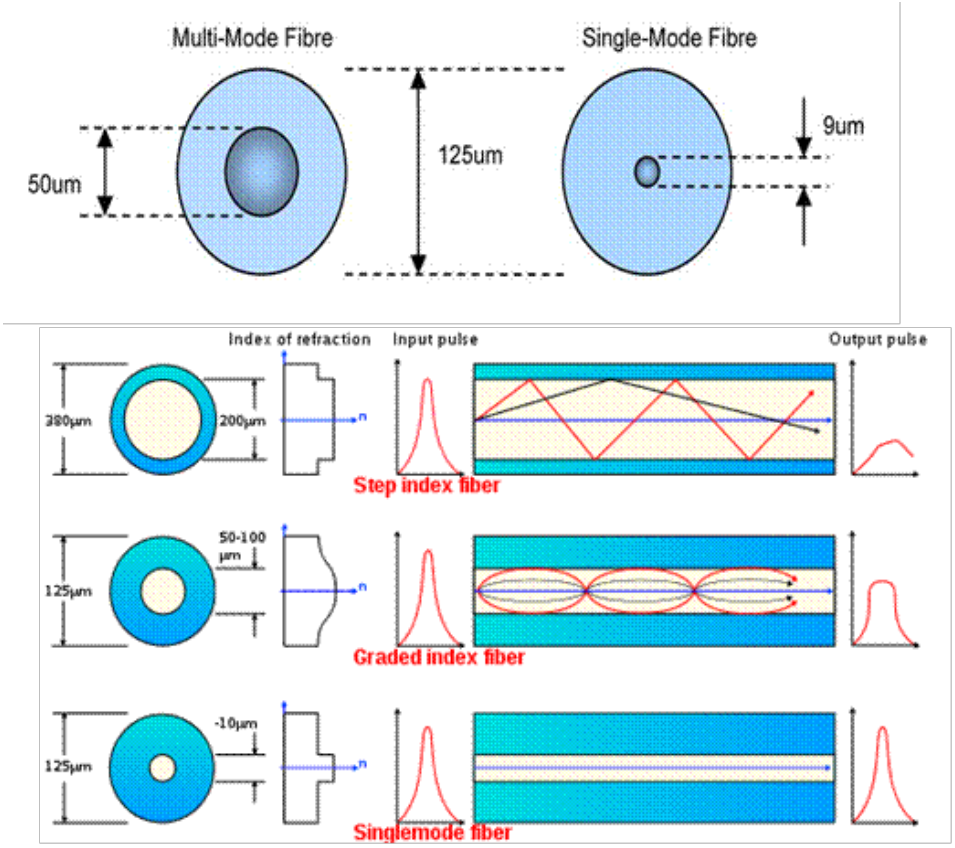


Figure (2.4): Different types of optical fiber

2.10 Pressure and Vacuum

The term pressure is used to describe either negative (below atmospheric) or positive (above atmospheric) pressure. Positive pressure is called gauge pressure. The term vacuum is used to describe the region of pressure below one atmosphere of pressure, also referred to as negative pressure. When speaking of vacuum, one must remember it as the opposite of pressure; high vacuum means low pressure.

The term vacuum is generally used to denote a volume or region of space in which the pressure is significantly less than 760 torr. In the traditional measurement system, normal pressure is expressed in millimeters of a column of mercury, and 760 millimeters of mercury is equal to 1 standard atmosphere [40]. A vacuum (HgV) reading is similar to gauge pressure (pounds per square inch gauge PSIG), in that the gauge reading is referenced to the current atmospheric or barometric pressure (which changes over time and place to place). When the reading is referenced to the theoretical absolute zero for a unit of measure, the reading is called an absolute value (Pounds per square inch absolute PSIA, HgA).

Chapter Three

Sensor Component

Chapter Three

Sensor Component

3.1 Introduction

A wide variety of solid-state sensors based on hydrogen-specific palladium, Metal Oxide Semiconductor, electrochemical, and Surface Acoustic Wave technology used in the industry for several years. Micro electromechanical systems and nanotechnology-based devices for the measurement of hydrogen are the recent developments. In this research work a palladium and Zinc Oxide thin film based gas sensors have been developed. This sensor proved to be more sensitive depending on the optical characteristics of the sample used.

3.2 Samples

The developed sensor sensitivity depends on the sample sensor used. In this research six samples have been prepared, four of them prepared from palladium metal (Pd (1), Pd (2), Pd (3), and Pd (4)), and two other samples prepared from Zinc Oxide (ZnO (1), and ZnO (2)). Palladium and Zinc Oxide thin films have been prepared on glass substrates. Films are prepared by vaporization deposition technique as described in chapter two. Samples Pd (1), Pd (2), Pd (3), Pd (4), and ZnO (1) were annealed in temperature of 600 degrees centigrade, while the last sample ZnO (2), was annealed in 200 degree centigrade. Figure (3.1) shows Zinc Oxide as one of the sample used.

Figure: (3.1) Zinc Oxide sample, annealing at 200 C.

The vacuum unit system, supplied by Blazers Model [BL 510] used to prepare thermally evaporated Zinc Oxide and Palladium films. Thin film sensor coatings deposited by thermal evaporation. Zinc oxide powder (99.9%) evaporated from a resistively heated tungsten effusion source. Palladium evaporated from tungsten boats. The vacuum unit system is consisting of the power supply, rotary pump, diffusion pump and the molybdenum boats that located inside the chamber. The nature of the substrate is extremely important because it greatly influences the properties of the films deposited on explain. The effectiveness of cleaning of substrates has strong effect on the adhesion properties of the deposited films. The procedures for cleaning the substrate are as follows:

Step 1: using detergent with water to remove any dust that might attached to the surface of substrate and then placed under tap water.

Step 2: repeating step 1 by replacing the distilled water with pure alcohol solution, which reacts with contamination such as dusts and some oxides. The slides eventually dried by blowing air and wiped with soft tissue.

Step 3: Finally, Putting the powder of the Zinc Oxide or Palladium thin films inside the molybdenum boats and the location of the glass substrate in the upper of the holder. Discrete the distance between the boats and the glass

substrate that equal to 15 cm could change it by the users and close the chamber, began of the operate system, by operating the rotary pump after close the chamber to cleaned from the atmospheric reach to (10^{-2}-10^{-3} bar) and open the diffusion pump also reach to the value (10^{-5}-10^{-6} bar) after that causes the process.

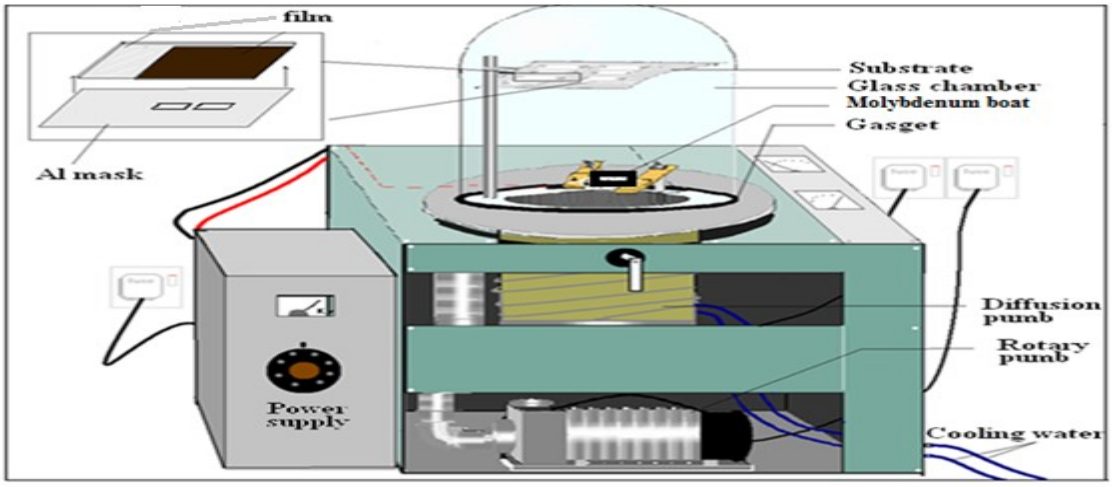


Figure (3.2): Typical experimental setup of thermal evaporation system

3.3 Samples evaluation

Before using the samples for hydrogen sensing, they were subject to three tests:

i. The X-Ray Diffraction (XRD) to check the crystalline structure of the samples,
ii. Atomic Force Microscope (AFM) to show the surface topography of the samples.
iii. The scanning electron microscope (SEM) to take images of surface topography of samples.

3.3.1 Crystalline structure of the samples

The crystalline structure was analyzed by SHIMADZU XRD-6000 x-ray diffractometer (λ = 0.154 nm) in 2Θ range from 20° to 60°. The interplaner distanced (h, k, L) for different planes were measured by using Bragg's law:

$2d\sin\Theta = n\lambda$ ……..……. (3.1)

Where,

n is integer, d is the interplanner distance, λ is the wavelength for incident radiation and Θ is Bragg angle.

The d-values are compared with the ASTM (American Society for Testing Materials) cared data file for Zinc Oxide and Palladium.

The grain size (D), in a polycrystalline film measured by Scherer's formula [41]:

$$D = \frac{0.9\lambda}{\Delta(2\theta).\cos(\theta)}$$ ..... (3-2)

Where Δ (2θ) is the full width at half maximum (in radians) of the peak, θ is the Bragg angle.

3.3.2 Samples surface flatness

The morphological surface analysis carried out that employing an atomic force microscope, AFM, (Scanning Probe Microscope SPM) used to scanning the sample surface.

The AFM can be operating in a number of modes, depending on the application. In general, possible imaging modes divided into static modes (also called contact) and a variety of dynamic modes (non-contact) where the cantilever vibrated and the tip. AFM used to determine the roughness and the average of grain size of Zinc Oxide and Palladium on the glass substrate

and give the statistical distribution of grain size. The most important part of an AFM is the tip with its nano-scale radius of curvature. The atomic force microscopy has three modes static (contact, non-contact and mapping topography) which used to investigate the morphology and intermittent tapping. The tip attached to a micro-scale cantilever, which reacts to the Van der Waals interaction and other forces between the tip and sample.

3.3.3 Scanning electron microscopy (SEM)

The first scanning electron microscope (SEM) debuted in 1938 (Von Ardenne) with the first commercial instruments around 1965[42]. Fortunately, many scientist and technologies quickly recognized the SEM ability to yield three-dimensional from the surface of the bulk specimen over a large range of length scales.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time and produces an image that is a good representation of the three-dimensional sample. SEM used in this work to take images of Zinc Oxide and Palladium samples that used in this work for gas sensing system.

3.4 The developed gas sensor system

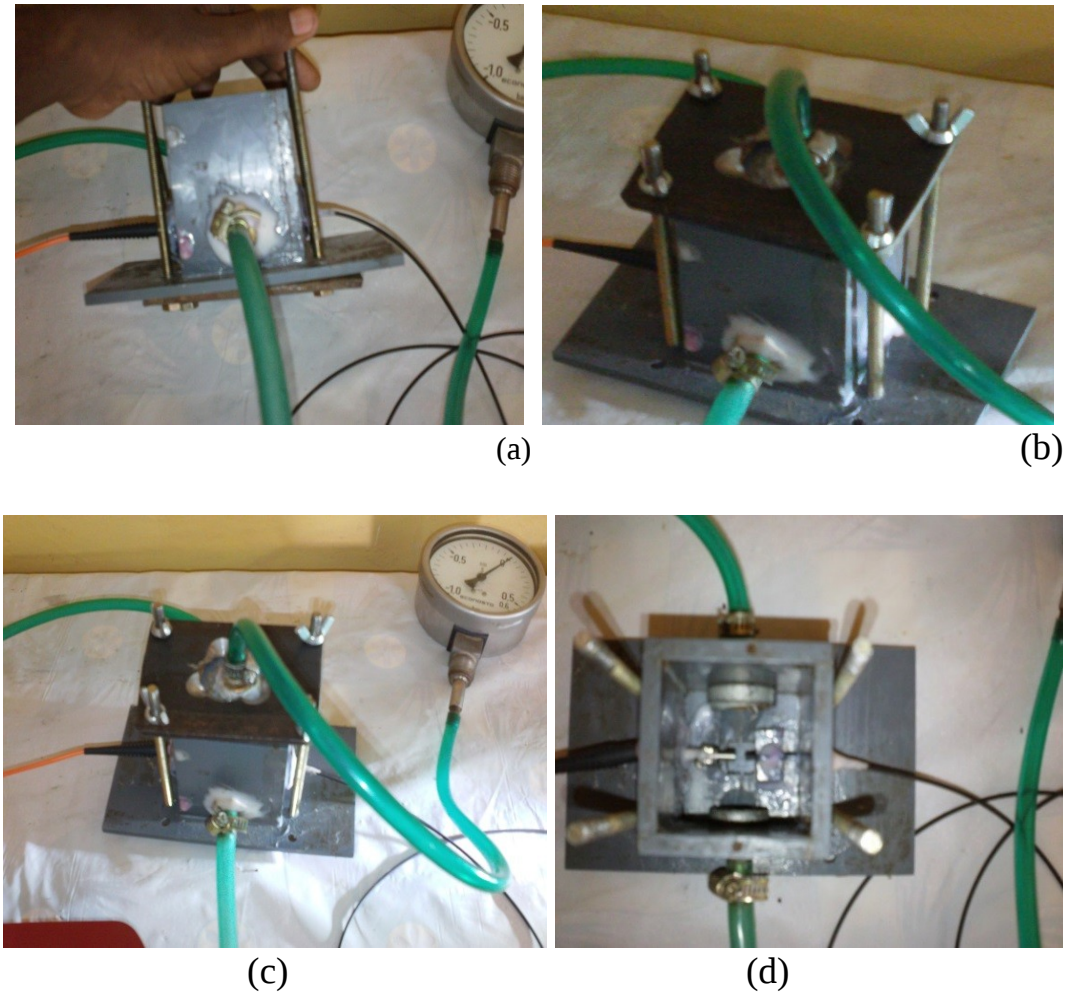
This system integrates seven components. These components include: samples, chamber, white LED lamp, spectrometer, vacuum unit, gauge pressure, cylinder of hydrogen gas, and computer.

i. **Sample:** The coated glass substrate, Palladium and Zinc Oxide thin films.
ii. **Chamber:** A cubical polymer test chamber of 50 mm square base and of 75 mm height and effective volume of 187500 mm^3; was developed

with removable 3mm iron cover and base. Four metal screws were used to close the chamber firmly. The cubical chamber was designed with five holes to serve the fowling:

a. Ejecting the gas inside the chamber,
b. Evacuating the gas from the chamber,
c. Inserting the fibre optic,
d. Taking the fibre optic outside the chamber and
e. Measuring the current pressure by the Gauge pressure.

The develop chamber contains two holders; one holder carry the fibre optic while the other holder carry the sample as shown in figure (3.6).



(a) (b)

(c) (d)

Figure (3.6): The chamber sensor.

(a) the iron base of the chamber, (b) the iron cover and tighten screws, (c) the two inside chamber holders and (d) the chamber during measurement .

iii. **White LED lamp:** 3W and 220V light source.

iv. **CCS Spectrometer:** The Spectrometer is an instrument that measures the amount of light absorbed by a sample. The Spectrometer technique is to measures light intensity as a function of wavelength. It does this by diffracting the light beam into a spectrum of wavelengths, detecting the intensities with a charge-coupled device and displaying the results as a graph on the detector and then the display device.
The specifications of this Spectrometer are listed in table (3.1) below.

Table (3.1) CCS Spectrometer specifications

Item	Data
Wavelength Range	200 – 1000 nm
Spectral Resolution	< 2.0 nm FWHM at 633 nm
Slir (WxH)	20 µm x 2nm
Grating	600 Lines/mm, 800nm Blaze
Detector Range	200 - 1100nm
CCD Pixel Size	8 µm x 200 µm (8 µm pitch)
CCD Sensitivity	160 V / (lx · s)
CCD Dynamic Range	300
CCD Pixel number	3648
Resolution	4 px/nm
Trigger Frequency Max	100 Hz
Trigger Pulls Length Min	0.5 µs
Interface	Hi-Speed USB2.0 (480 Mbit/s)
Dimensions	(LxWxH)(122 x 80 x 30) mm
Weight	< 0.4 kg
Fiber Connector	SMA 905

v. **Vacuum unit:** to evacuate the test chamber.

vi. **Gauge pressure:** to measure the current pressure of the chamber, the gauge reading between (-1 to 0.6 bar).
vii. **Cylinder of hydrogen gas:** of a known concentration and have certificates bellow.
viii. **Computer:** to process the recorded signal.

Other additional parts were also used to assist in experimental work such as Lap Hood to evacuate the gas from vacuum unit outside the lap.

3.5 Sensor testing procedure

Hydrogen gas can be measured through applying the following procedure:

- Sample should be placed inside the developed polymer test chamber.
- White LED light source should be power on to enlighten the sample sensor.
- CCS Spectrometer used to measure transmitted light through the sample.
- Vacuum should be applied to evacuate the test chamber by vacuum unit.
- Chamber pressure should then measure using the gauge pressure.
- Hydrogen gas of a known concentration should then allow to flow from the gas cylinder to the test chamber during measurement.
- The measured transmitted light signals are then sent to computer to be processed.

3.6 Experimental setup

The components of the developed system are arranged and setup as demonstrated in figure (3.12). The next figure is a real image of the system figure (3.13).

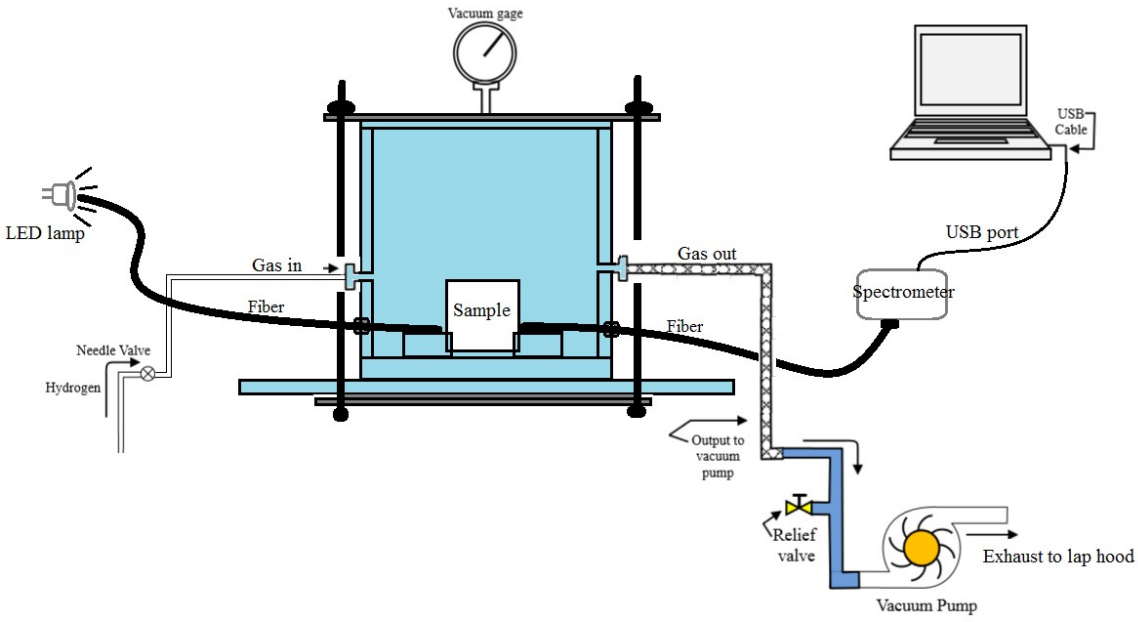


Figure (3.12): System setup.

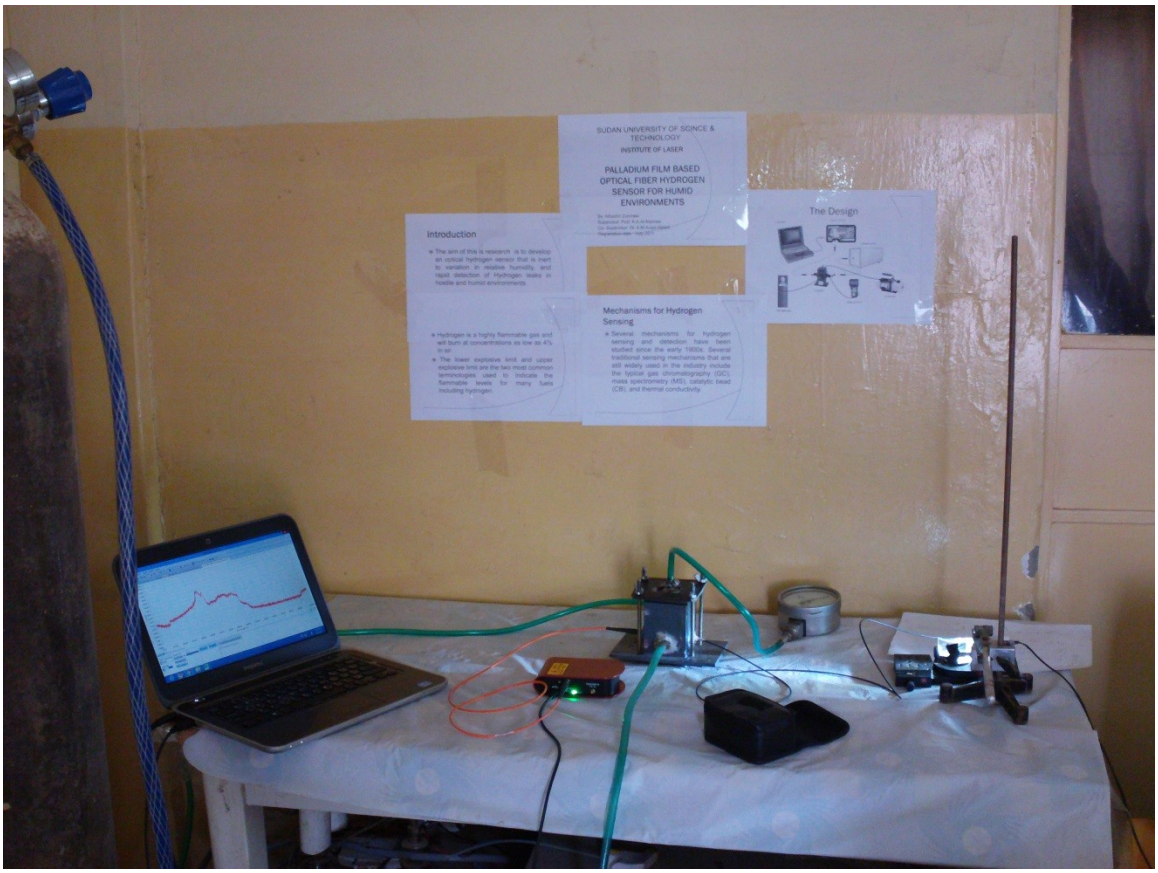


Figure (3.13): Real image of the system setup

Chapter Four
Measurement and Results

Chapter Four
Measurement and Results

4.1 Measurement

This research work is arranged to design an optical Hydrogen sensor based on a hydrogen-specific material used. The approach of the new develop sensor is based on passing the gas to the chamber to adsorb on the sample (Palladium, or Zinc Oxide), and transmitted the light through the sample. The spectrometer is used to measure and record the signal and processed by computer.

4.1.1Sample used

Two types of materials used in this work; Zinc Oxide, and four sample of Palladium. These samples were subjected to two tests before using in the measurement process. The first test is to check the crystallization of the sample, and the second test for surface flatness testing.

4.1.2 Crystalline structural of the samples

The structure and lattice parameters of samples (Palladium and Zinc Oxide thin film) were analyzed by x-ray diffractmeter (XRD) as illustrated in figure (4.1). The crystalline structure was analyzed by a SHIMADZU 6000 X-ray diffractometer using Cu Kα radiation (1.5406 Å) in reflection geometry. X-ray diffraction pattern for the deposited Zinc Oxide thin films were obtained and all the samples checked under identical conditions.

The main purpose of this step is to investigate the material structure of the samples and to ensure that the structure is crystalized. The result was showed many peaks in the XRD spectrum as illustrated it in Figure (4-1). The particle size of the deposited films uses the Debye-Scherrer formula. The XRD spectrum was applied for Zinc Oxide films as:

$$D = (0.9\lambda / \beta \cos \theta)$$

Where,

λ is the wavelength of the X-ray employed which in this case equal to 0.15418 nm for Cu-k α.

β is the FWHM (full-width at half maxima) and

θ is the Bragg's angle in degrees.

Where 'D'; the average particle of the prepared Zinc Oxide films was found to be about 28nm for 2θ equal to 38.600° and the Mullier induces is (101). The average particle for 2θ equal to 44.860° was about 26.1 nm, and the Mullier induces was (102).

The average particle for the structure Zinc Oxide films was found to be 27.1 nm, the Mullier induces was (202) and the structure is for Zinc Oxide films. The average particles for Palladium was equal to 28 nm for 2θ equal to 38.580° , and the Mullier induces was (111). The average particle for 2θ equal to 44.820° was about 28.4nm and the Mullier induces was (200) for Pd films. The average particle for the structure Palladium films was 28.2 nm and the structure for the Palladium is polycrystalline. Since the peaks were sharp it was evident that the films deposited are polycrystalline and are hexagonal structure for Zinc Oxide films. The peaks for Palladium films were sharp it is evident that the films deposited are cubic.

Tables (4.1): show the calculation of the grain particles.

2θ	**2θ/2**	**FWHM**	**Cos(θ)**	**(h,k,l)**	**D(nm)**
38.600°C(ZnO)	19.3	0.0053	0.9438	(101)	28
44.860°C(ZnO)	22.43	0.0057	0.929	(102)	28.4
38.580° C(Pd)	19.3	0.0053	0.9438	(111)	28
44.820° C(Pd)	22.41	0.0057	0.924	(200)	26.1

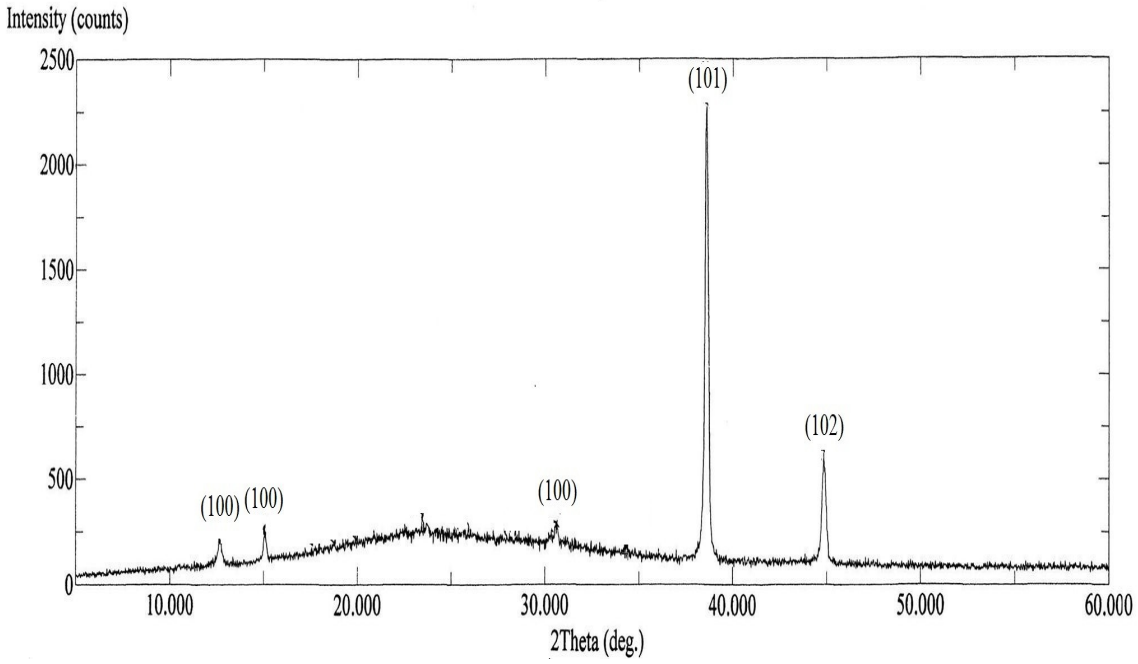


Figure (4-1): X-ray diffraction patterns of the Zinc Oxide films.

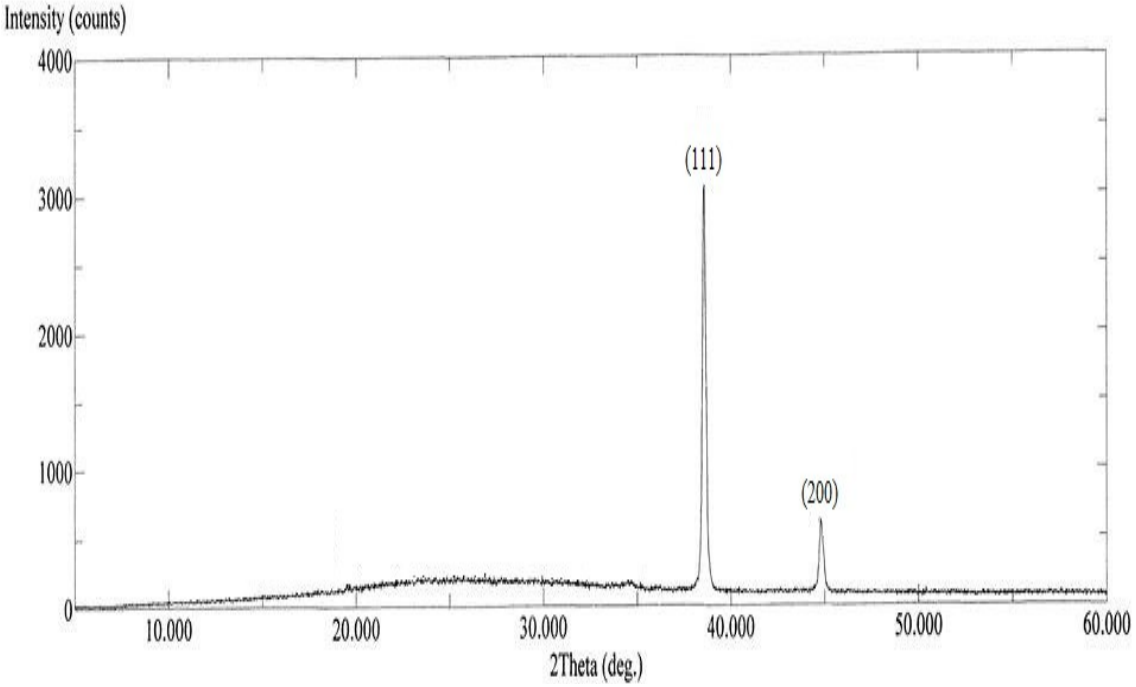
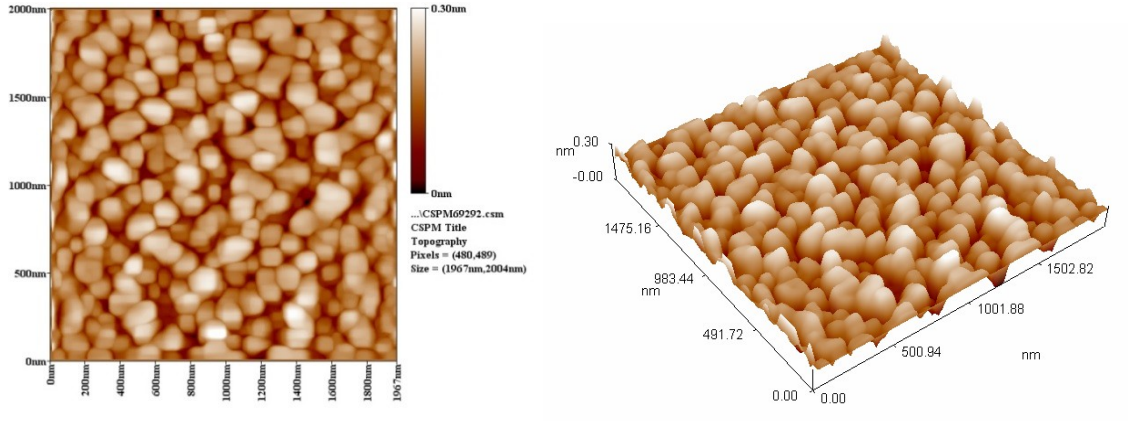


Figure (4-2): X-ray diffraction patterns of the Palladium films**.**

4.1.3 Surface flatness

SPM scanning probe Microscope (AFM micrograph) was used to study the surface flatness of each sample used. This test is provided granularity distribution of the sample. Figures (4.3) and (4.4) bellow illustrates Scanning Probe Microscope images of each samples and its accumulation distribution chart of two models of the sample. The first for Palladium sample while the second for Zinc Oxide sample.

The surface morphology of the films studied from the AFM images; the average grain size gets to increase with increasing of Zinc Oxide content and while the roughness is decreases. Also for Palladium, the thickness of Palladium and Zinc Oxide increases the roughness decreases while the average grain size increases. From AFM images, observed that the grain size become larger and the crystalline was improved. The root mean square (rms) of the film surface roughness for Zinc Oxide thin film is very smooth. This surface characteristic is important for a gas sensor.



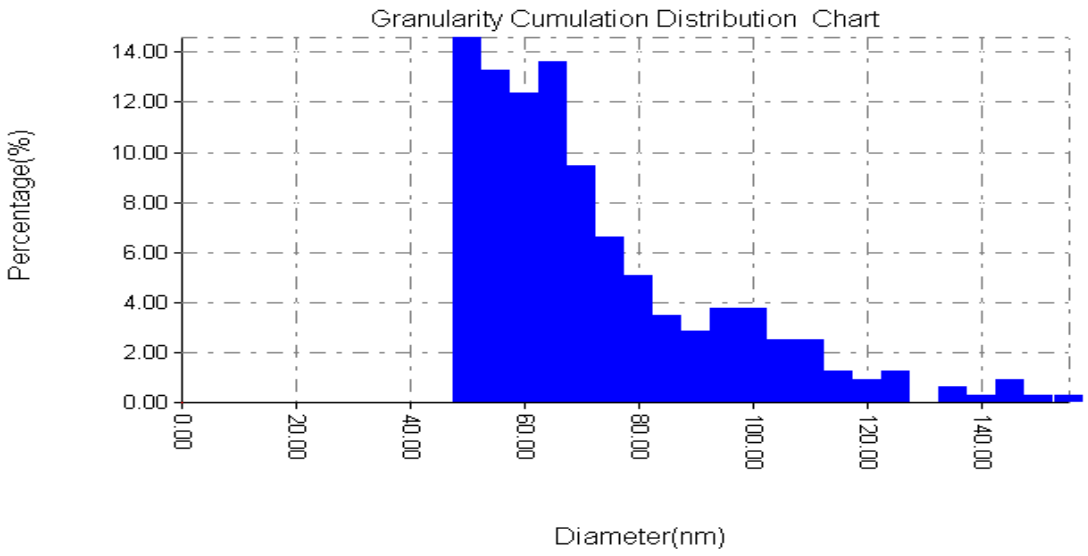
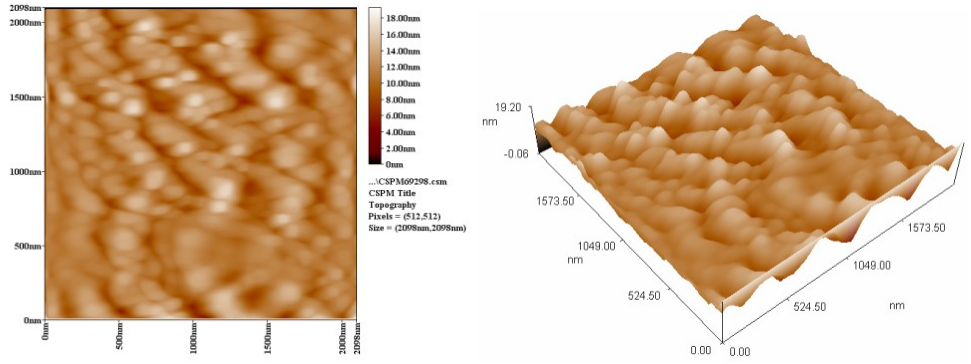


Figure (4.3) Scanning Probe Microscope images of Palladium thin film sample and its Accumulation Distribution.



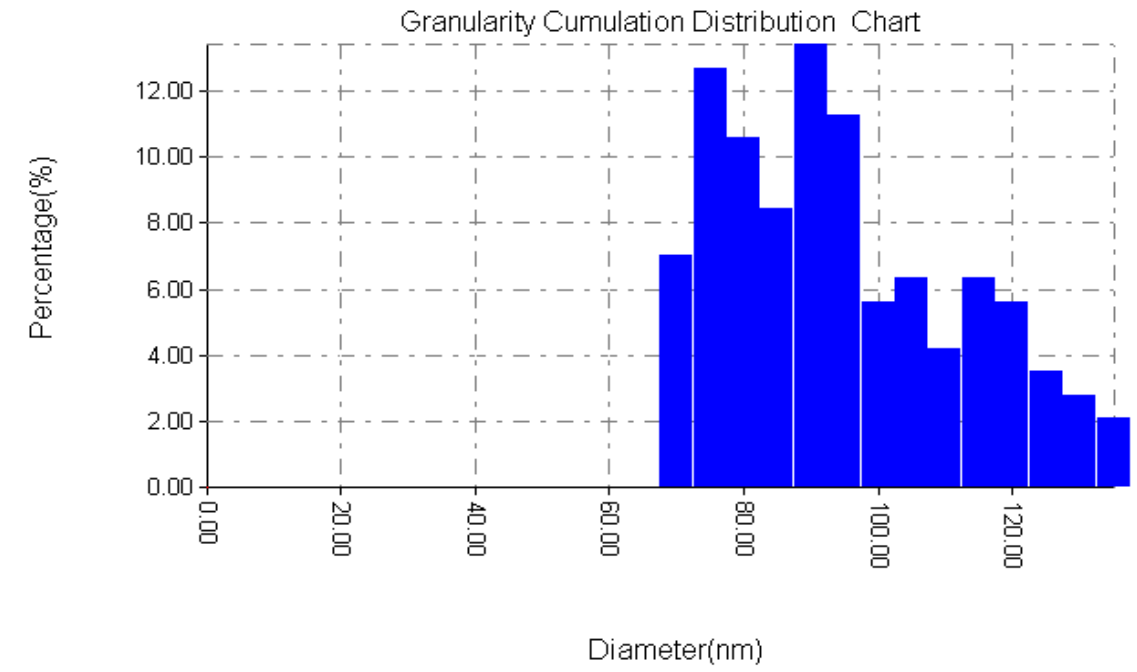
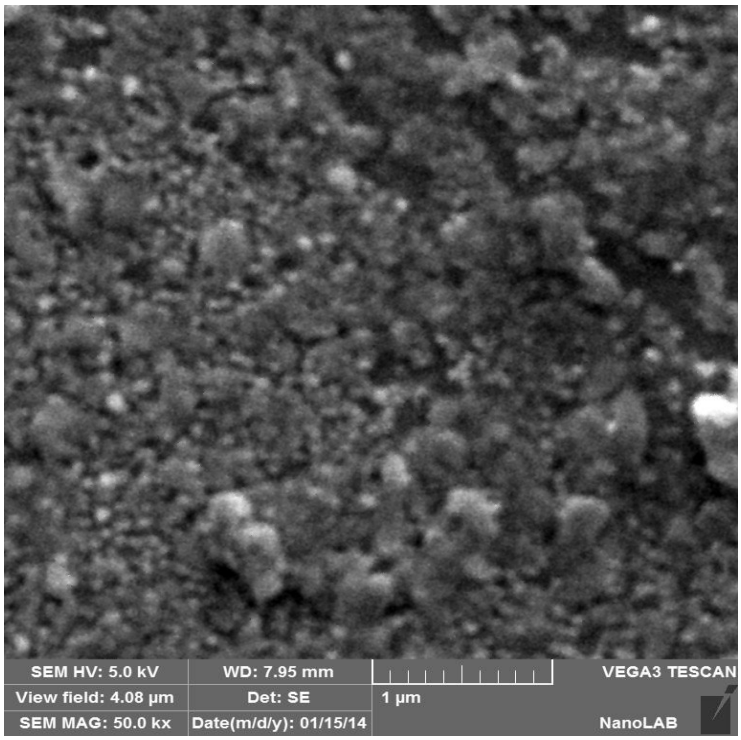


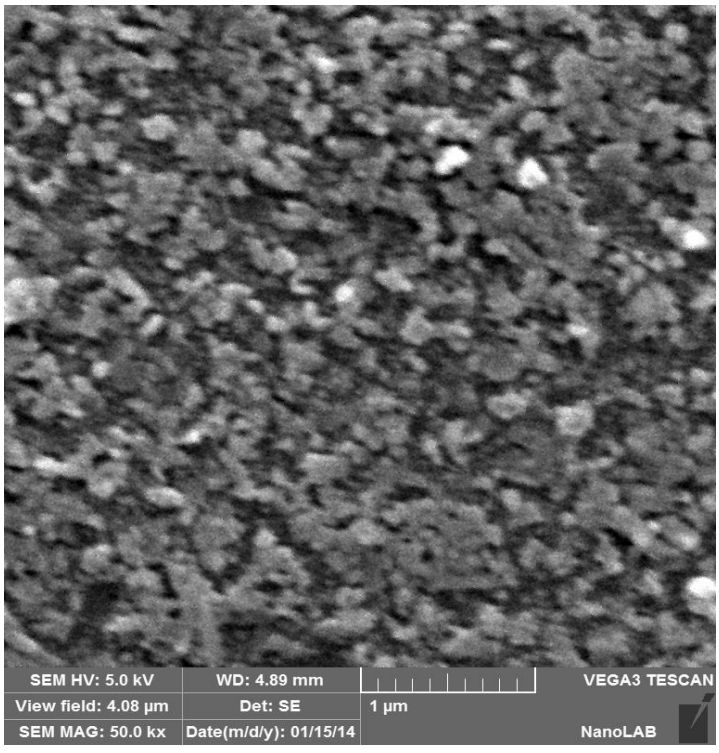
Figure (4.4): Scanning Probe Microscope images of Zinc Oxide thin film sample and its Accumulation Distribution.

4.1.4 Surface topography

Scanning electron microscope (SEM) used to take images of surface topography for Zinc Oxide and Palladium samples show in figures (4.5) and (4.6).



Figures (4.5): SEM images for Zinc Oxide.



Figures (4.6): SEM images for Palladium.

4.2 Results

The experiments' components setup as shown in figure (4.7).

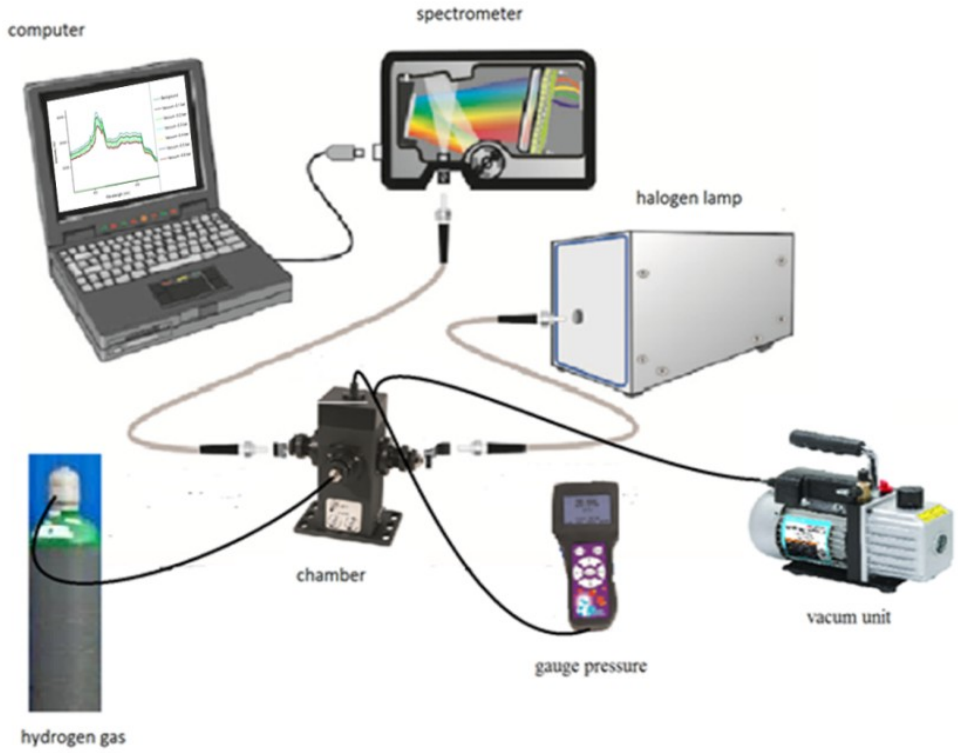


Figure (4.7) the experimental setup.

Measurements were taken relative to the steps mentioned that includ:

- Each sample was placed inside the design polymer test chamber.
- White LED light source was power on.
- CCS Spectrometer was used to measure transmitted light through each sample.
- The test chamber was evacuated using vacuum unit.
- Chamber pressure was then measure using the gauge pressure.

- Hydrogen gas of the flowing characteristics was allowed to flow from the gas cylinder to the test chamber during measurement.

Table (4.2) characteristics of Hydrogen gas

Purity	99.999%
Moisture	Less 0.5 ppm
Oxygen	Less 0.1 ppm
Nitrogen	Less 0.5 ppm
Carbon Dioxide	Less 0.1 ppm

- The light signals were detected, then sent to CCS Spectrometer and processed by computer.

In this work, six readings were measured for each sample under pressures of -0.6, -0.5, -0.4, -0.3, -0.2 and -0.1bar, frequency against transmitted light intensity were then plotted for each sample.

Four tests were carried out for palladium samples as shown in figures (4.7), (4.8), (4.9) and (4.10). The figures investigation can be noted that the intensities of transmitted light are always subject to Hydrogen concentrations, which implies the design sensor is working probably. Slights difference between results from the graphs can be due to uneven distribution of coating process.

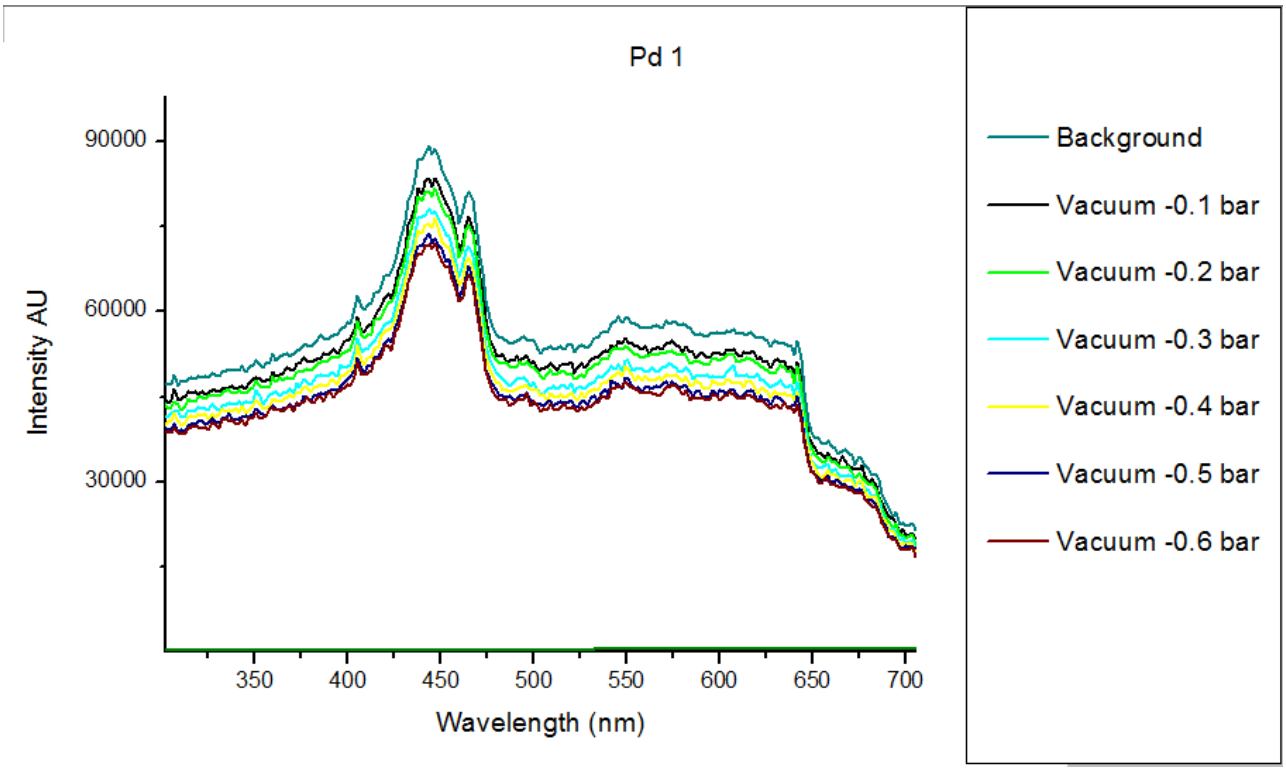


Figure (4.7) Palladium sample one

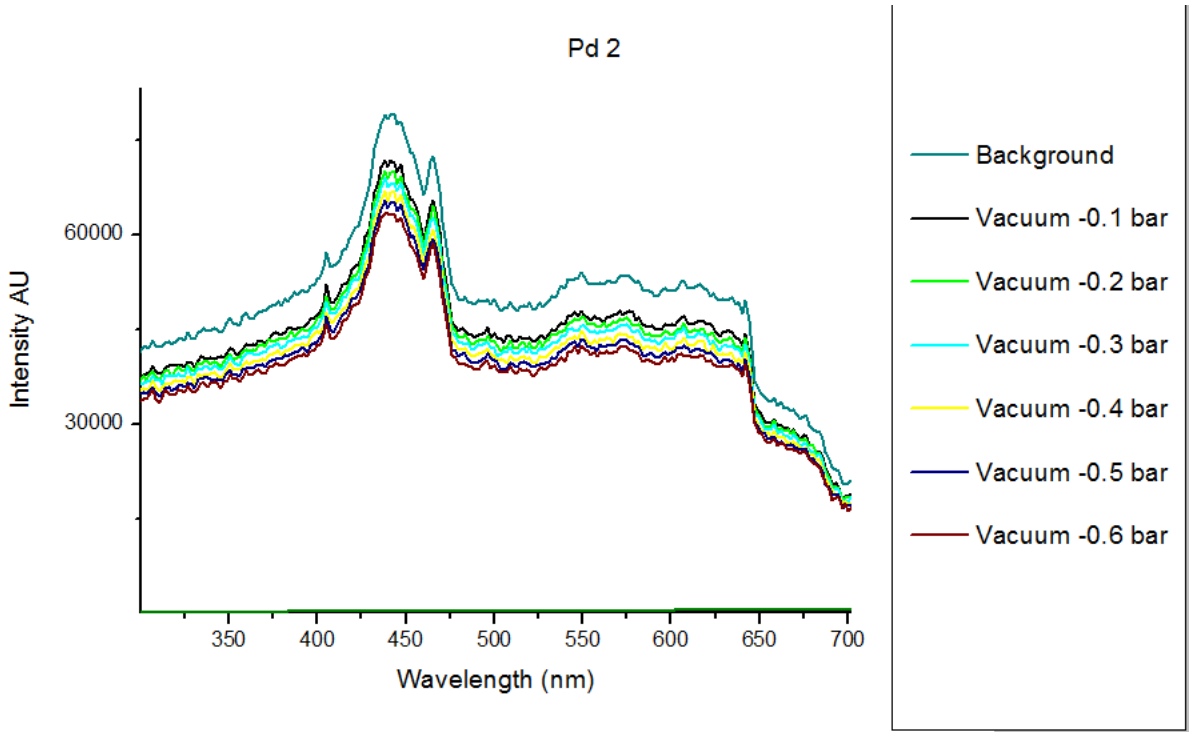


Figure (4.8) Palladium sample two

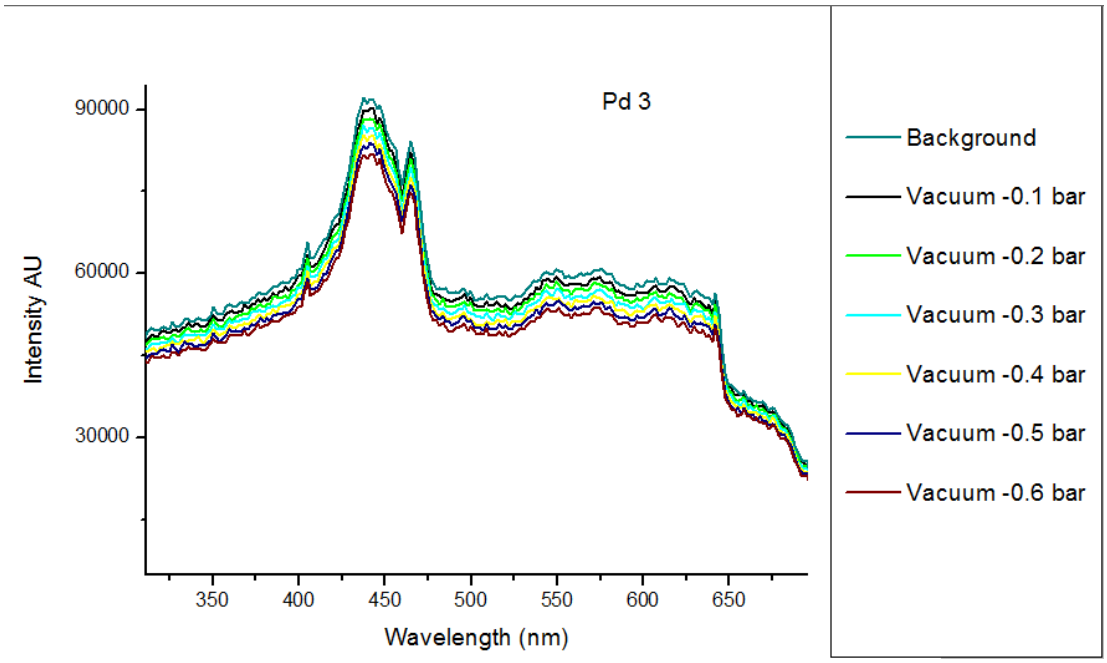


Figure (4.9) Palladium sample three

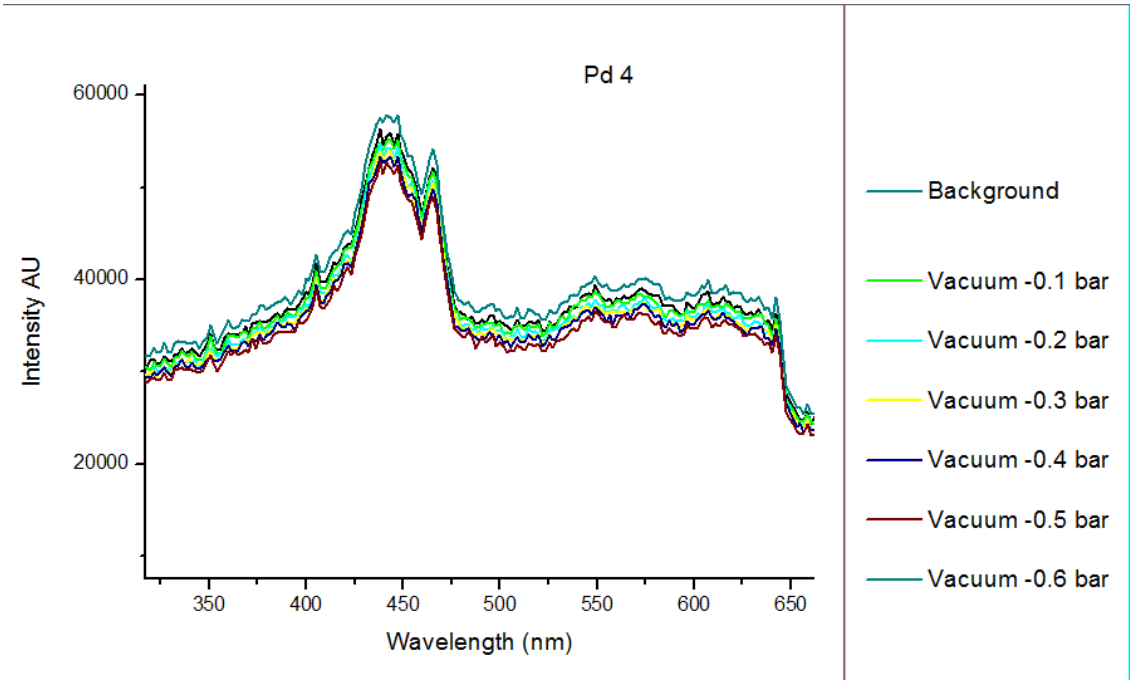


Figure (4.10) Palladium sample four

The two samples of Zinc Oxide were examined through applying the previous procedures. Results of these two tests were illustrated in figures (4.11) and (4.12).

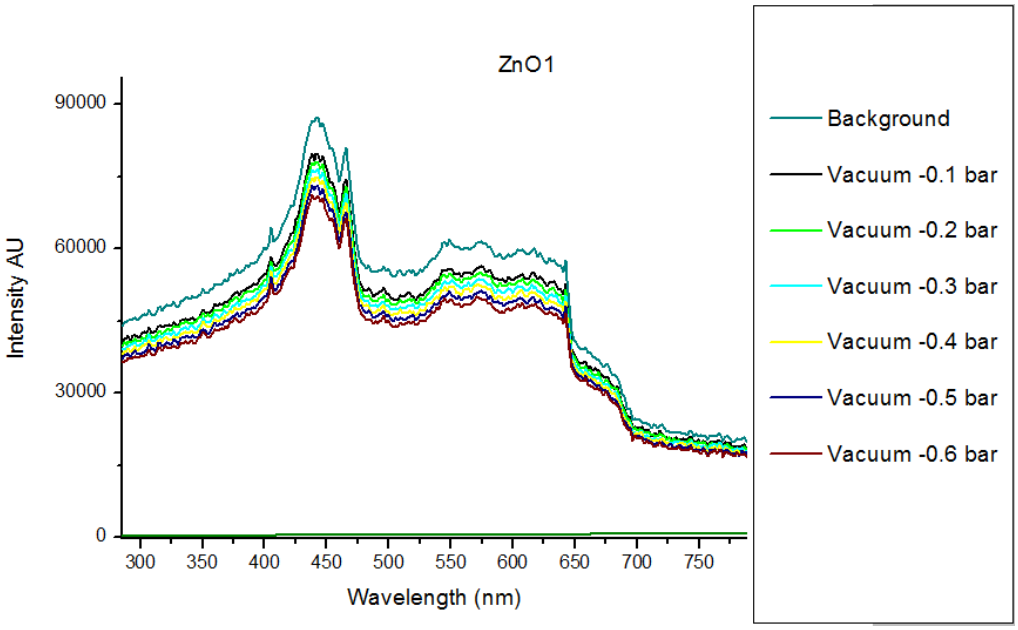


Figure (4.11) Zinc Oxide sample one

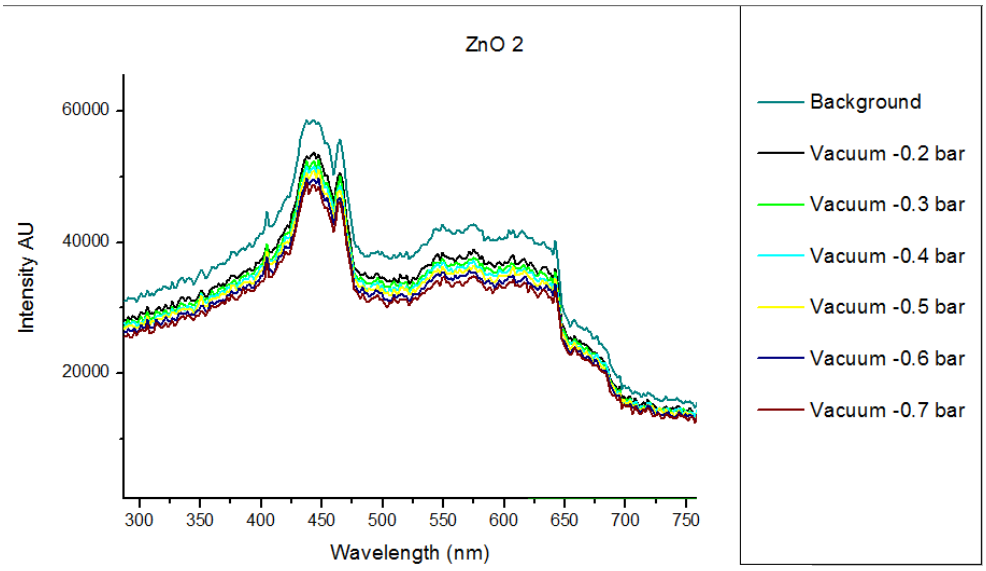


Figure (4.12) Zinc Oxide sample two

4.3 Discussions

The experiment work was started by allowing light to transmit through the sample. Then chamber evacuated from the air. This can be noted by referring to the resultant curve of the background (the higher curve in blue colour) that shows the maximum transmitted light intensity.

After inserting hydrogen to the chamber the transmitted light intensity decreased to the lowest level at pressure of -0.6 bar. Again this can be obviously demonstrated by examining the resultant curve below (in brown colour), where the hydrogen under vacuum. Insert more hydrogen through the chamber lead to increase the transmitted light intensity through the samples. Again, this could be noted by referring to the other curve.

On the other hand, by the increase the hydrogen, the pressure decrease in the chamber until to -0.1 bar. The second curve from the higher drown in black color illustrates this phenomenon.

Also, from the figures of the results, it can be noted that for some samples, the curves of the measurement appeared to be close to the curve of the background, especially for the sample of palladium (sample Pd)3(, Pd) 4() figure (4.9 and 4.10), this can be due to better quality of the coating of the sample (number of atoms). This can be extracted from the sample test (AFM, SEM) figures, in addition to the granularity accumulated distribution chart. In the better coated samples the background-measuring curve was far from the other curve because transmittance depends on the sample of material coating, that interact with hydrogen in the chamber. However, when the sample is not fully coated transmitted light will be increased.

Referring to the figures derived from the carried out tests, it can be noted that by increasing of concentration of hydrogen gas in the test chamber a transmitted light is also increase, and sensitivity increase linearly

as the gas concentration increases. These leads to judge that the palladium and zinc oxide thin film sample is successfully detect hydrogen gas. This is again proving the success of the design sensor which is working probably.

The design optical fiber Hydrogen gas sensor based on a palladium or zinc oxide material can be design of Hydrogen sensor, moreover it can provide number of advantages over the traditional sensors.

Chapter five

Conclusion and Recommendation

Chapter Five

Conclusion and Recommendation

5.1 Conclusion

In this research, a new approach of hydrogen sensor was designed, manufactured and tested. Palladium and Zinc Oxide were successfully tested. The conclusion of these measurements and results obtained, proved that the new designed sensor is working probably and can be used successfully to detect hydrogen. Moreover, the new design provides number of advantages that include:

- Rapid response to low hydrogen concentration,
- Wide dynamic range but more sensitive in 500-650 nm range.
- Free from electromagnetic interference.
- Multiplexing possibilities.
- Fully functional in radioactive environments
- Light weight and resistance to high temperature

5.2 Recommendations

This research work design and developed a new approach of an optical Hydrogen sensor using (Pd and ZnO) materials. The work does not cover a wide range of materials and other options. Therefore, recommendations for further studies can include:

- Examining additional material for optical Hydrogen sensing,
- Applying the same experimental setup for other gases,
- Limiting the source of light in the visible range,
- Assuming other approach for Hydrogen sensing,
- Study the effect of changing chamber dimensions.

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