Fundamentals of Sensors, Materials and Methods: A Review[1]

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ABSTRACT

The number of materials, methods and monitors are available for monitoring hazardous, toxic and inflammable gases. Bulk and nanoscaled material powders were utilized for fabricating the gas monitors, out of which, the nanoscaled materials show the challenging response to such gases. Thick film sensors exhibit the crucial response to such polluting gases which can be attributed with the porous nature of the film. This review paper shows the detailed study about the materials and monitors already available in the field.

Keywords: *Nanoscaled Material, Thick Films, Gas Monitor, etc.*

INTRODUCTION

The advancement and development of Science and Technology has been explosively enhanced all over the world, which has made available the easy and comfortable survival of human beings. However, the environment has not been preserved properly by man. During the competition of qualitative and quantitative production under development, many toxic and hazardous gases are released in the environment. Increasing industrialization results in unsustainable heights of pollution [Fig. 1]. Nearly, half of the population of the world is exposed to increasing air pollution as a result of which, 4.2 million deaths have been estimated annually due to particulate matters $PM_{2.5}$, declared WHO in 2016 [1-7]. In China alone, 1.2 million premature deaths occurred during 2010-2015, due to pollution [8]. The pollutants, so sadly affected all the components of environment such as air, water, soil, noise, etc. that the entire environment has become polluted and affected the living beings [9-11].

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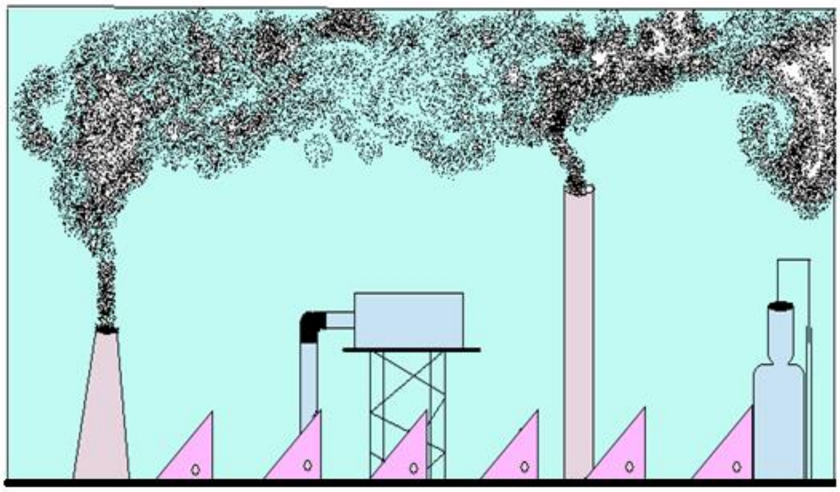


Fig. 1: Pollution Explosion by industry

Pollution intensifies global warming by which the whole living beings are affecting tremendously day by day from the destructive natural events, viz. Tsunamis, Cyclones, High tides as well as from the harmful diseases, viz. High fever, Typhoid, Malaria, Chikun Gunia, Bird flu, Swine flu, Dengue, etc. Industries, vehicles, few natural events and entire manmade events are the major sources of polluting gases. Carbon monoxide, carbon dioxide, sulphur dioxide, sulphur trioxide, nitric oxide, nitrogen dioxide, chlorine, ethanol, ammonia, etc. are toxic, hazardous and polluting gases, the leakages of which can reach to dangerous level up to 100 ppm and up to 150 ppm or above can cause death. The exposure of such gases can cause heart and respiratory track diseases, lung cancer, reduction in haemoglobin, impairment of nervous system, mental retardation, disorders of digestive system, disorder of reproductive system, blindness, forgetfulness, headaches, hypertension and what not? Explosive emission of CO_2 by vehicular and industrial exhausts caused decay in ozone layer and therefore, the intensity of the solar radiations reaching the earth is increasing day by day and exposure of those intense UV radiations on human skin has increased the chances of causing sunburns and skin cancers. So, it needs to monitor such gases at ppm, ppb or even sub ppb level, to make awareness among the Laymen and to control the liberation of such harmful gases.

POLLUTANTS AND HEALTH HAZARDS

Beyond certain limit of leakage or exposure, polluting gases can cause undesirable and disastrous effects on living beings. Such gases are called as polluting gases or pollutants. Organic and inorganic gases, and particulate matters are the major air pollutants (Table 1). There are major two types of pollutants, primary and secondary pollutants. The polluting gases which are directly released from sources, e.g. CO_2, SO_2, NH_3, H_2S, NO, VOCs, vapours of petrol, diesel, kerosene, LPG, LNG, dust particles, etc. are called as primary pollutants. However, chemical interactions occurred among primary pollutants in the atmosphere would cause the production of secondary pollutants, e.g. NO_2, SO_3, O_3, Ketones, H_2SO_4, etc.

Table 1: Hazardous effects of few polluting gases

Sr. No.	Gases / PM	Hazardous effects
1	$PM_{2.5}$	Particulate Matters of size less than 2.5 µm in diameter (outdoor air pollution) causes 4.2 million deaths annually, WHO estimated [7].
2	CO	Carbon Monoxide is a colourless, odourless and tasteless gas, whose exposure leads causes of unintentional and suicidal poisonings, and it causes a large number of deaths annually both in Europe and in the United States [12-14]. It affects respiration, headaches, dizziness and nausea. Prolonged exposure to concentrations above 5000 ppm can increase the incidence of illness. CO combines with haemoglobin and myoglobin to stop the supply of oxygen to tissues affecting brain, myocardium and muscle tissues. On inhalation, it reacts with haemoglobin to form

		carboxy-haemoglobin, which can cause severe health effects at various percentage levels in blood: ✓ Loss of vigilance ability @ 3% - 5% ✓ Loss of hand to eye co-ordination @ 6% - 10% Higher concentrations may cause serious health hazards like coma and eventual death.
3	SO_2	Sulphur Dioxide is toxic, results in acid rain or snow or forms sulphate aerosol particles in the atmosphere [15].
4	H_2S	Hydrogen Sulphide is a major pollutant, hazardous and toxic in nature and colourless gas with foul odour [16-20].
5	NO_x	NO and NO_2 referred together as NO_x which are highly reactive and carcinogenic gases formed when O_2 and N_2 react at high temperatures during lightning. Nitric acid contributes to acid deposition and forms aerosol causing cough, breathlessness, irritation of upper airways, bronco spasms, nauseas, vomiting [21].
6	CO_2	Carbon Dioxide is an odourless gas vital to living and non living beings which exist in the atmosphere as a trace gas at a concentration of about 0.04 percent (400 ppm) by volume. It is the major responsible (50% alone) gas for green house effect [16]. Majorly, it is produced by animals and plants during respiration [22-24].
7	VOCs	Volatile Organic Compounds are highly inflammable and toxic which include hydrocarbons (CxHy). Benzene is the best example of an anthropogenic VOC which is one of the carcinogens. The importance of VOCs as precursors depends on their chemical structure and lifespan in the environment. VOCs on oxidation produce non volatile chemicals that condense to form aerosols. To avoid some cruellest events, it is necessary to detect the VOCs from the open environment [25].
8	LPG	Liquefied Petroleum Gas is a vital source of energy in the world as a clean and portable fuel. LPG is explosively utilized in industrial and domestic fields as fuels. LPG is potentially hazardous as explosion accidents might be caused when they leaks by any means [26-27].
9	LNG	Liquefied Natural Gas is a cryogenic liquid derived from natural gas. It consists primarily of Methane (67 – 97 volume %) and Ethane (3 – 29 volume %). It can cause frostbite on contact with eyes and skin [28]. Upon inhalation, it can depress the activities of central nervous system, nausea, headache, dizziness, vomiting, loss of consciousness, serious injuries or even death.
10	Cl_2	Chlorine is a disinfectant and toxic gas, having pungent smell and is yellowish green in colour. At higher concentrations, it can be detrimental for humans [29]. It results skin infection, liver damage, psychological disorders, etc. On contact with aqueous medium it forms dioxins. Dioxins are having bioaccumulation property; hence they easily become part of the food chain [16].
11	NH_3	Ammonia is a toxic in nature [30]. Its exposure causes chronic lung diseases, irritating and even burning the respiratory track, etc.
12	C_2H_5OH	Ethanol is used for beverages, fuels, scientific and industrial purposes which is a hypnotic (sleep producer) gas [31]. It depresses activity in the upper brain even though it gives the illusion of being a stimulant. Ethanol is also toxic like methanol. Abuse of ethanol is a major drug problem in most countries.

SENSORS IN GENERAL

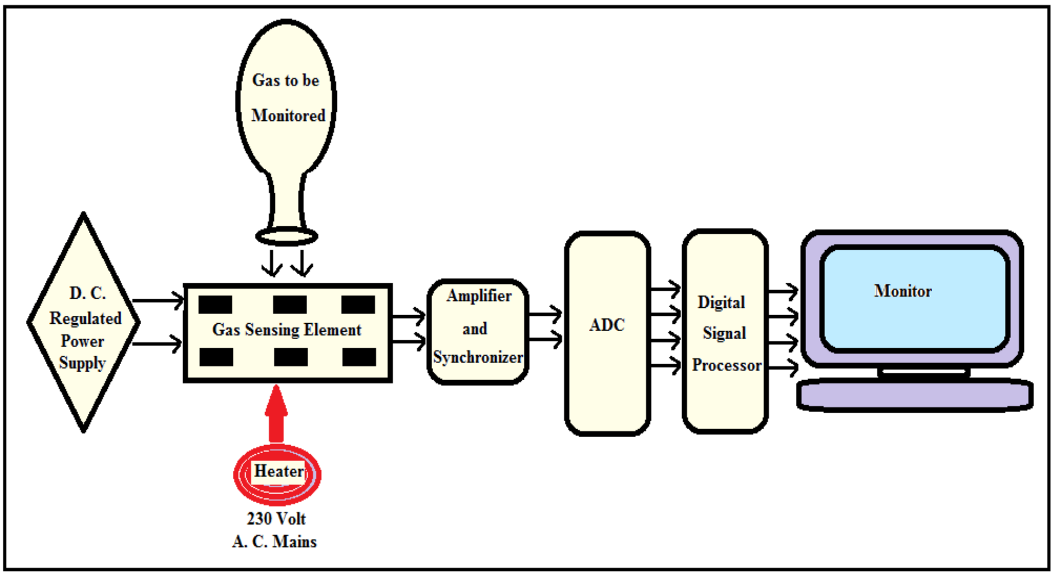
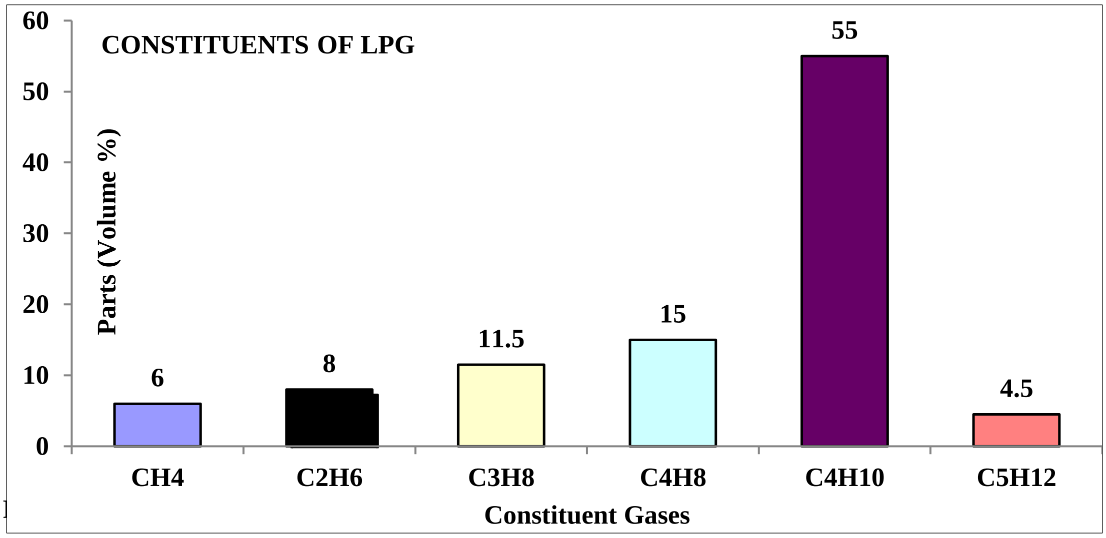


Fig. 2: Block diagram of gas monitoring system (in general)

Sensor is an intelligent organ of an instrument which is used to measure the traces of quantities which are not perceivable by human sensory organs. Sensors are the input ports of the electronic devices used to detect the disastrous levels of some quantities. The electronic device which responds to physical and chemical quantities, and generates the proportional output is known as a sensor. The output signal may be in the form of electric, magnetic or any other type of signal. A device that converts one type of signal into another is called as a transducer. Fig. 2 depicts the block diagram of gas monitoring system in general. The system consists mainly, d. c. regulated power supply (0-30V), current meter (pico-ammeter), sensor holder, heater mounted just below the sensor, gas inject unit, thermocouple, monitor, etc.

LPG Sensor and its Need

The electronic device which detects the LPG at trace level, i. e. the concentration of LPG in the environment before noticeable to human nose or LEL, and producing the proportionate output signal, is called as LPG sensor. LPG, usually referred as cooking gas, is a vital source of energy around the world as a clean and portable fuel, which consists chiefly of butane (55 vol%), as depicted in Fig. 3 [16].



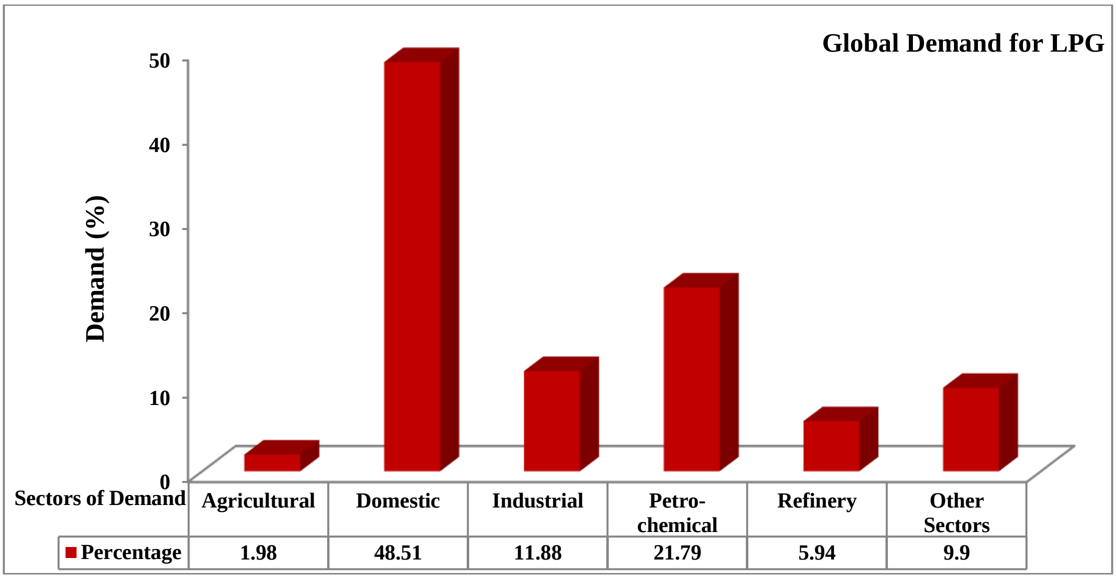


Fig. 4: Global Demand for LPG

As it is a colourless and odourless gas, it is usually mixed with compounds of sulphur (methyl mercaptan and ethyl mercaptan) of foul smell. Due to this mixing, the leakage of LPG can be easily noticed by human nose. LPG, LNG, VOCs, etc. are potentially hazardous because explosion accidents might be caused when they leak out by any means. Lower explosive limit (LEL) of LPG is 1.8 volume % in the atmosphere, which means that, if it is leaked in the environment below LEL, it does not explodes and it is not dangerous. But, in case, the leakage is continued and its concentration reaches more than LEL (1.8 volume %), then it is potentially dangerous and it explodes instantly. Recently, India became the second largest consumer of LPG in the world after Government's Ujjwala push [32]. Fig. 4 depicts how the global demand for LPG has been increased tremendously in domestic field, which may increase the risks of accidents in large extent, attributed due to the leakage of LPG [33]. If the leakage of LPG is noticeable by human nose, that concentration is much higher than the lower explosive limit (LEL) of LPG. It should be monitored before noticeable to human nose. So, there is a great need to detect it for the purpose of control and safety applications in domestic as well as in industrial fields. It leads to the research and development of a wide range of sensors using simple and low cost materials and technologies. So, the research on gas sensing is the need of society. For the good quality, sensor must have the specifications described in Table 2.

Table 2: Characteristics to select a good quality sensor

Sensor Parameters	**Expected Values**
Gas response	High
Selectivity	Highest
Response Time	Low (~sec)
Recovery Time	Low (~sec)
Sensitivity	High
Cross Sensitivity	Zero (Low)
Longer Life	Few years
Repeatability and Stability	Good, Linear
Resolution	ppm, ppb or even sub-ppb
Power consumption	Low (~mW)
Cost	Very low
Size	Portable (Pocket)
Temperature range	Room (or low) Temperature
Error	As low as possible

Available Sensors: An Overview

Figaro Engg. Inc, Sierra Monitors Inc, IST, CAPTEUR LTD, etc., are some of the world leading industries who have fabricated the sensor models already [6]. Few sensor models manufactured by these industries are listed in Table 3. Here, TGS (Taguchi Gas Sensors) are fabricated by Figaro Engg. Inc. and other sensors are fabricated by Sierra monitors.

Table 3: Sensors models with applications

Sensor Application	Target Gas	Sensor Model Numbers
Combustible Gas Monitoring	General hydrocarbons	TGS 813, TGS 816
	LPG, LNG	TGS 09, TGS 109 TGS 2610, TGS 2612
	Methane Natural Gas	TGS 815, TGS 842 201-00, 2001-00
	Hydrogen	TGS 821
Solvent Vapour Monitoring	Alcohol, Toluene, Organic Solvents	TGS 822, TGS 823 TGS 2600, TGS 2620
Toxic Gas Monitoring	Carbon Monoxide	TGS 203 206-00, 2006-00
	Ammonia	TGS 826
	Hydrogen Sulphide	TGS 825 203-00, 2003-00
	Sulfur compounds	TGS 550
Green House Gas Monitoring	Chlorofluorocarbons	TGS 830, TGS 831
	Refrigerant gas	TGS 832
Odour Monitoring	Ammonia Amines	TGS 826
Cooking Control	Volatile Gases, Fumes from Food (Alcohol, Odour, Humidity)	TGS 880, TGS 881 TGS 882, TGS 883
Air Quality Control	General Air Contaminants (Cigarette smoke, gasoline vapors, etc.)	TGS 100, TGS 800
Automobile Ventilation	Gasoline Exhaust	TGS 822

We should develop the smart gas sensors which may have the capability of detecting the gaseous species below TLV, low cost, low power consumption, portable size and shape, etc. for large applicability and affordable to Laymen.

LEL and UEL of Combustible Gases

Lower explosive limit (LEL) and upper explosive limit (UEL) are the key points for combustible gases to avoid their hazards. These terms should be displayed where combustible and explosive gases are utilized in large extent. The minimum gas concentration present in open environment below which it does not supports the fire and, at or above which it just supports fire, is called as LEL, whereas, the gas catches fire instantly in UEL. Explosive limits are measured either in volume percent or in ppm. Table 4 depicts the LEL and UEL for various gases [34-38]. The smart sensors are needed to detect the inflammable gas concentrations below LEL.

Table 4: LEL and UEL for combustible gases

Gas	LEL (Volume %)	UEL (Volume %)
LPG (Butane: 55%, Fig. 3)	1.8	8.4
LNG (Methane: 67 - 97% and Ethane: 3 – 29%)	5.0	17
Methane	5.0	17
Ethane	3.0	12.4

Propane	2.1	9.5
Butane	1.8	8.4
Pentane	1.4	7.8
Hexane	1.2	7.4
Heptane	1.1	6.7
Petrol vapours	1.2	8.0
Ethanol	3.3	19
Ammonia	15	28
Methyl Mercaptan	3.9	21.8
Turpentine	0.7	---

LITERATURE SURVEY

Table 5 depicts the detailed literature survey of the sensing materials, techniques employed for fabrication of the sensors, target gases with year of publications. The survey leads to conclude that, ZnO, SnO_2 and their nanocomposites with few metal oxides proves their candidature as the functional materials for combustible gases including LPG [39-63]. However, if ZnO and SnO_2 form the nanocomposites with other metal oxides, they respond to hazardous and toxic gases [64-135]. Very few reports are available on the Bi_2O_3 based LPG sensors. From the actual work performed in our laboratory, we observed that, Bi_2O_3 and its nanocomposites gives very less response to inflammable or combustible gases easily. So, the study on Bi_2O_3 based gas sensors is not included in detail.

Table 5: Literature Survey of Gas Sensors

Materials	**Techniques**	**Gases**	**Year**	**Reference**
Ppy-Bi_2O_3-MOx	Ex-situ-approach	LPG	2018	39
PANI-ZnO-PEO	Thin Film by Electro-Spinning	LPG	2015	40
Pd Loaded Fe-SnO_2	Co-Precipitation	LPG	2014	41
Pd-Fe-SnO_2	Thick Film	LPG	2014	42
ZnO	Sol-Gel	LPG	2013	43
Bi_2O_3-Y_2O_3 PANI	Thin Film by Chemical route	LPG	2013	44
Graphene / Bi_2O_3	Quantum dots	LPG	2013	45
Cu - ZnO	Thick Film	LPG	2010	46
Cr_2O_3- ZnO	Thick Film	LPG	2009	47
ZnO	Thin film	CH_4	2009	48
SnO_2	Thin film	CH_4 and CO	2009	49
CdO	Thin film by spray pyrolysis	LPG	2009	50
ZnO	Hydrazine method	LPG	2008	51
Al- ZnO	Thin film	LPG	2008	52
CdO	Thin film by SILAR	LPG	2008	53
CdO	Thin film by Chemical route	LPG	2008	54
Sr - SnO_2	Thick Film	LPG	2007	55
RuO_2 - SnO_2	Thick Film	LPG	2007	56
ZnO	Thin Film by Spray Pyrolysis	LPG	2007	57
ZnO	Chemical Synthesis	LPG	2007	58
ZnO	Thick Film	LPG	2006	59
PdO: ZnO	Thin film by Chemical deposition	LPG	2004	60
ZnO	Thin film by Spray Pyrolysis	CH_4	2001	61
ZnO	Thin film	H_2, SF_6, C_4H_{10}	2000	62
ZnO	Thin film by CBD	LPG	1998	63

ZnO / SnO_2	Surface metallization	H_2	2015	64
Ga_2O_3-Core / ZnO-Shell nanowires	DC sputtering	NO_2	2015	65
$BaSnO_3$	Thin film	SO_2	2015	66
Ga - ZnO	Thin film	CO	2014	67
Al doped ZnO	Sol – Gel	CO	2014	68
ZnO - NiO	Nano-wire	Polluting Gases	2014	69
ZnO	Thin Film by CVD	Ethanol and Acetone	2014	70
ZnO	Sol – Gel	NH_3	2014	71
MOx	Thin Film	H_2	2014	72
Zeolite -ZnO	Thick Film	Polluting Gases	2014	73
ZnO - PANI	Thick Film	NH_3	2014	74
ZnO (Ga)	Wet Chemical Method	NO_2	2013	75
ZnO	Thick film	NH_3	2013	76
ZnO	Thin Film	Ethanol	2012	77
ZnO	Thin Film	Ethanol	2012	78
Bi_2O_3-Core / In_2O_3-shell	Thin Film	NO_2	2012	79
Ga - ZnO	Thin film	CO	2012	80
Ni, Ce-SnO_2	Thick Film	Acetone	2012	81
SnO_2–ZnO	Thin film	Ethanol	2011	82
SnO_2–ZnO-Noble metal	NWs Array	NO_2 and H_2S	2011	83
SnO_2–Bi_2O_3	Thick Film	Immittance	2011	84
Bi_2O_3	Electrode	DNA biosensors	2011	85
Bi_2O_3	HRP film	H_2O_2	2011	86
In / Ga / ZnO	Thin film	NH_3 and Acetone	2011	87
ZnO	Thin film	CO and H_2	2010	88
ZnO	Thick Film	O_3, NO_2 and CO	2009	89
ZnO	Thin Film	Solar Cell	2009	90
MOx	Thin Film	Gas Sensing	2009	91
Ga - ZnO	Thick film	Formaldehyde	2009	92
SnO_2	Thin film	CH_4 and CO	2009	93
SnO_2	Thick film	H_2S	2009	94
Sn - ZnO	Thermal evaporation	LPG	2009	95
Ce - SnO_2	Thin film	H_2S	2009	96
SnO_2	Thin Film by Atomic Layer Deposition	CO	2008	97
ZnO	Thin Film (FET)	Polluting Gases	2008	98
M-TiO_2 / SnO_2	Thick Film	Dichloromethane	2008	99
ZnO / YX $LiNbO_3$	Thin Film by RF Sputtering	H_2	2007	100
ZnO	Thin Film by Spray Deposition	NO_2	2007	101
ZnO	Thin Film	Ethanol	2007	102
Pd- ZnO- SnO_2	Thick Film	LPG	2007	103
SnO_2-Bi_2O_3, SnO_2-Sb_2O_3	Thin film by Chemical Method	O_2	2007	104
SnO_2, SnO_2-MoO_3, SnO_2-Fe_2O_3	Wet chemical synthesis	NH_3	2007	105
SnO_2	Thin film	Hydrogen and Ethene	2007	106
Al_2O_3- ZnO	Thick film	Ethanol	2007	107

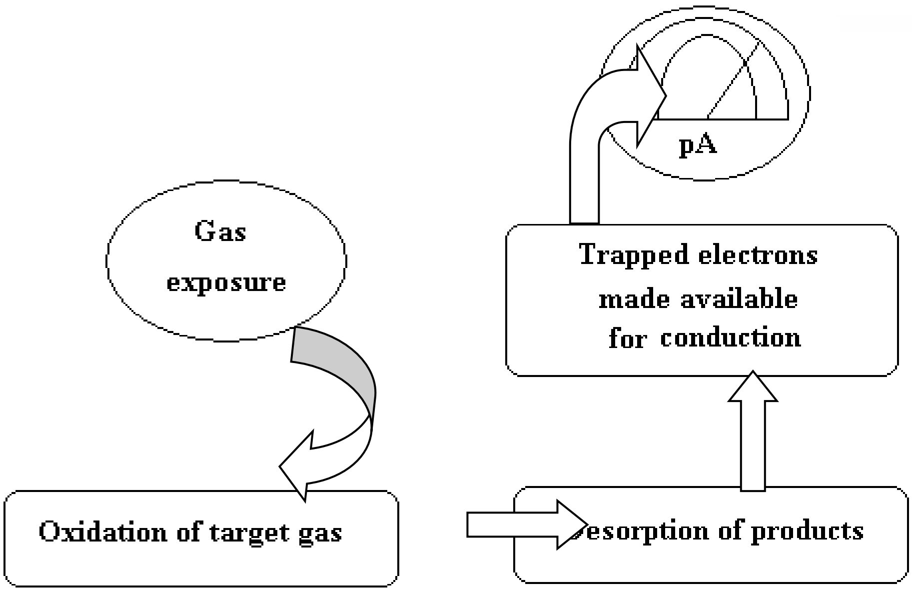
Cr_2O_3- ZnO	Thick film	NH_3	2007	108
ZnO	Thick film	NH_3	2006	109
Fe_2O_3–ZnO	Thick film	NH_3	2006	110
ZnO	Thick Film	NH_3	2006	111
ZnO	Thin film	Ethanol	2006	112
ZnO	Thin film	Polluting Gases	2006	113
SnO_2–ZnO	Thin Film	NO_2	2005	114
ZnO	Thin film	Polluting Gases	2005	115
ZnO	Thin Film	Acetone	2005	116
ZnO- Bi_2O_3	Thin / Thick film	H_2	2005	117
Cupricated SnO_2-ZnO	Thick Film	H_2S	2004	118
ZnO	Thin film by Sol-Gel	Alcohol	2004	119
ZnO	Thin film by RF Sputtering	O_2	2004	120
ZnO	Thin film by Sputtering	O_2	2003	121
SMO	Thick Film	Combustible gases	2003	122
ZnO	Thin Film	CH_4 / H_2 / H_2O	2003	123
ZnO	Thin Film by Sol-Gel	CO	2003	124
SnO_2	Thick film	O_2	2003	125
CuO-doped SnO_2-ZnO	Pellets	CO and H_2	2002	126
ZnO-CuO	Thin Film	CO	2000	127
SnO_2	Screen printing	NO and CO_2	2000	128
ZnO	Thin Film	CH_4 and H_2S	1999	129
ZnO	Thin film	Ethanol	1999	130
ZnO-Al_2O_3	Thick film	H_2	1997	131
ZnO	Thin Film	H_2	1995	132
SnO_2 and ZnO	Thin film	Hydrocarbon, CO, H_2	1992	133
SnO_2 (Bi_2O_3)	Thin film	H_2	1991	134
ZnO	Thin film (Electrode)	H_2	1984	135

THICK FILM TECHNOLOGY FOR FABRICATION OF GAS SENSORS

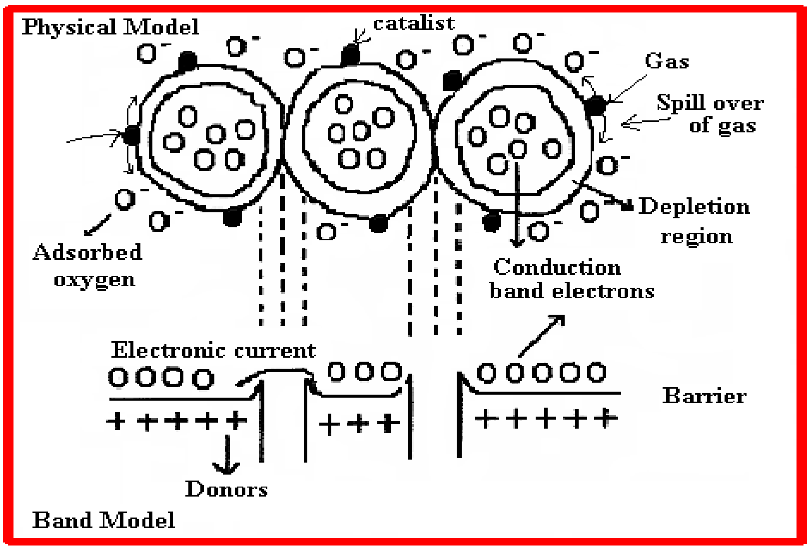
Thick film technology involves the screen printing of thixotropic pastes of functional materials on the substrates in a definite pattern followed by firing process. For thick film fabrication, paste of the functional material is prepared with the use of some temporary organic binder [136-146]. The paste is screen-printed on an insulating substrate (e.g. alumina), which can withstand at higher temperatures, to get definite pattern. The printed film is dried under an IR lamp to remove the temporary organic binder and then it is fired at a particular higher temperature with a definite time-temperature profile to obtain stability and better adhesion of the film to the substrate. Thick film technology offers the following advantages for gas sensing, over thin films or pellet.

- ✓ Low cost technique and easier mass production,
- ✓ A good control over the thickness and microstructure is possible,
- ✓ Easier modification of surface and even bulk of materials,
- ✓ Easier fabrication, etc.

BASIC MECHANISM OF GAS MONITORING



(a)



(b)

Fig. 5: Gas sensing mechanism (a) Gas sensing process and (b) Physical and band model of semiconductors with catalysts dispersed on the grain surface.

The gas sensing mechanism involves oxygen adsorption / desorption type of mechanism on the surface of the film. The surface of the sensor should be clear and large so that, the gas molecules can easily adsorb on it. The exposed target gas molecules are oxidized by capturing the adsorbed, atomic or molecular oxygen which holds the electrons on the surface, trapping behind the electrons constituting the increase in surface current [16, 147]. Fig. 5 describes the oxygen adsorption – desorption type of mechanism and band model of the sensor.

RESEARCH GAP

The entire world is facing the problems of explosion of cooking gas (LPG) in kitchens, CNG in vehicles and in industries. Also, we are facing the hazardous and toxic effects of gases and polluting materials liberated by the industries in the open environment. So, being as a student in this advanced age, we should turn our attention to resolve these problems, even though, at small scale. Literature survey leads to conclude that, there is a big gap between the research work done and practical applicability of the sensor as a device. Lot of research work has been done in the gas sensing field using bulk as well as nanotructured materials. However, some limitations persist till today, viz. lack of selectivity and high sensitivity at lower concentration of gas (ppm or even ppb), repeatability, long term stability, etc. The numerous metal oxides such as ZnO, SnO_2, Fe_2O_3, TiO_2, WO_3, CuO, ZrO_2, Cr_2O_3, etc. and their composites have also found the applications in the field of gas sensors. While, since last two decades, nanostructured metal oxides have attracted great attention due to their potential properties and applications, such as nanoelectronics, optoelectronics, catalysis and gas sensors. Size reduction in nanostructured metal oxides leads to significantly enhanced gas sensing performance due to enhanced surface to volume ratio, nanoscale microstructure and quantum-size effect, their properties and structural ability are very different from their bulk counterparts.

As per literature survey, ZnO and SnO_2 have been extensively used in wide range of applications in the field. ZnO and SnO_2 are important metal oxide semiconductors exhibit excellent physical properties such as wide band gap, high conductivity, etc. Considering few unique characteristics, nanostructured ZnO and SnO_2 thick films are expected to exhibit crucial performance in LPG sensing studies.

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