DESIGN OF MEMS BASED GAS SENOR WITH INPLANE INTEGRATED IDT AND MICRO-HEATER

A thesis submitted towards the partial fulfilment of the requirements
for the award of degree of

MASTER OF TECHNOLOGY
IN
VLSI DESIGN

Submitted by
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DECLARATION

I hereby declare that the thesis entitled "**Design of MEMS based gas senor with inplane integrated IDT and micro-heater**" is an authentic record of my own work carried out as the requirement for the award of degree of Master of technology in VLSI at Electronics and Communication Engineering Department of Thapar University, Patiala, under the guidance of **Dr. Anil Arora (Assistant Professor),** Electronics & Communication Engineering Department during the session 2014-2016.

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ABSTRACT

Gas sensors are employed to ensure that levels of various harmful (e.g. toxic, flammable, etc.) gases are within an acceptable range. These monitors are indispensable in industrial facilities that produce such gases (e.g. oil and natural gas refining), but are also critical for hazardous conditions in residential and other commercial settings. Hydrogen sensors have obtained increased interest with the widened application of hydrogen energy in recent years. In this work, MEMS (Micro-Electro-Mechanical Systems) sensor with interdigitated electrodes have been designed and simulated. It is observed that micro-heater design parameters imposed different impacts on the sensor performance. A gas chamber around the device has been designed and the effects of the hydrogen gas on the sensor device have been studied. In order to achieve small, robust, low cost and fast hydrogen sensor with high sensitivity, the designing and its optimization is performed. The effect of the applied voltage on the sensor is also studied.

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CHAPTER-1
INTRODUCTION

1.1 Micro-electro-mechanical-systems (MEMS)

Micro electro mechanical systems are the combination of both electrical as well as mechanical components. It is the amalgamation of both electrical as well as mechanical components. It includes the components of very small dimensions usually of micrometres or millimetres dimension. At microscopic level, various sensors like thermal sensors, gas sensors or pressure sensors can be designed. MEMS components are fabricated by micro machining processes. MEMS basically consists of four components as shown below [1].

Fig 1.1 Basic block diagram of MEMS

Input signal

It is the basic signal that we gave as input for processing.

Sensor

Next is the sensor, which senses the input signal and converts in to electrical form if it is in non-electrical form.

Processor

Processor does the processing by applying all the logical functions for the needed application.

Actuator

Actuator does the reverse operation of sensor by converting the non-electrical form of energy in to electrical form of energy.

For fabrication of MEMS, there are three fundamental steps- Deposition, Lithography and Etching [2]. In deposition, thin films of the suitable material are deposited on the substrate. There are two major methods which are used for deposition.

- Chemical reaction methods
- Physical reaction methods

Chemical reaction methods include chemical vapor deposition and thermal oxidation whereas physical reaction methods include physical vapor deposition (PVD) and casting.

1.2 MEMS Materials

MEMS consist of wide range of materials which can be used according to the application intended for. Some of them are discussed below [3]

Silicon

Silicon is the widely used material in industry to fabricate devices as it is abundant in nature, cost effective and also possesses suitable electrical and thermal properties which make it suitable for fabrication. It can survive in harsh environmental conditions and also has long life time. Not only silicon but silicon compounds like silicon dioxide are also very popular for MEMS applications [3].

Polymers

Polymers have high electrical resistivity, cost efficient and can be easily transformed in to desired structures but its poor conductivity limits it to certain applications.

Metals

Metals have high mechanical stability and are most reliable in nature but they do not possess the properties shown by silicon. Aluminium, copper, tungsten, nickel are widely used MEMS material.

Quartz

Quartz is a material which has high thermal dimensional stability. In some manner, they are better than silicon material as they have more flexible geometry and also are transparent to ultraviolet light which is used for species detection.

1.3 Advantages of MEMS

1) IC compatible
2) Batch fabrication
3) Ruggedness
4) Cost-effective
5) Low power

1.4 Applications of MEMS

MEMS can be used in numerous applications where miniaturisation is desired. Some of the applications for which it can be used are-

1) Automotive domain

MEMS devices are extensively used in automotive applications like designing airbag for cars, for automatic car locks considering security and safety purpose, anti-theft system and are also used in regulating temperature in vehicles [4].

2) Consumer oriented industry

   They are widely used in consumer oriented industry for various products like personal navigation devices, microphones, printers and in peripheral devices for computer.

3) Communication

   They are used in fabrication of communication devices like antennas, oscillators, and phase-shift circuits.

4) Integrated circuits

   IC devices mainly of silicon are extensively produced using MEMS technology as MEMS fabrication is compatible with IC devices

5) Biotechnology

   They are used for drug screening, for detection of hazardous chemicals, for DNA amplification and identification.

1.5 Gas sensors

Gas sensors detect the presence of the gas in the surrounding atmosphere. When the sensor interacts with the gas present around, the properties of the sensor changes which reflects the sensing mechanism. MEMS technology is very popular in gas sensors because of its minimal power consumption, ruggedness and very minute dimensions. Environmental conditions are deteriorating day by day. Among various gases, oxygen is vital and should be kept at adequate level while other hazardous gases should be controlled in order to maintain a healthy life. There are various types of sensors, among which metal oxide gas sensors are very popular as they have simple structure, long life time and robust nature [6].

Sensor device constitute two basic functions

1) To sense the gas in the surroundings

2) Signal translating mechanism that will translate the effects seen in the sensor to the corresponding measurable signals.

Gas sensing is done through the interaction of chemical species with the sensing surface. Chemical species which reacts with the sensing layer are absorbed on the layer and changes the properties of the sensing surface [6]. Metal oxide sensing layer that is used is sensitive to a particular gas. So, Sensitivity of the layer towards a particular gas is of a major concern.

1.6 Working principle of metal oxide gas sensor

When gases interact with the metal oxide sensing layer, some of the gas molecules are adsorbed on the surface which changes its properties. At some particular temperature, oxygen ions are adsorbed on the surface of oxide layer.

The sequence of adsorption process is as under [7]

O_2(Gas) $\Leftrightarrow$ O_2(absorbed) (1)

O_2 (Absorbed) +e− $\Leftrightarrow O^-$ (2)

O_2 $-$+ e− $\Leftrightarrow 2O^-$ (3)

O− +e− $\Leftrightarrow O_2$ $-$ (4)

1.7 Applications of Gas sensors

- Environmental monitoring
- Home safety
- Automobiles ventilation control
- Fire detection
- Leak detection
- Explosive gas detectors

Table 1.1 Types of Sensor and their detection principle [8]

Sensor	Detection Principle
Semiconductor gas sensor	Change of some electrical property
Catalytic gas senor	Heat or temperature
Optical sensor	Reflection or refractive index

1.8 Need of Micro-heater

In metal oxide sensor, oxidation or reduction process that takes place on the sensing layer occurs at a specific elevated temperature. Oxidation or reduction decides the number of free electrons on the layer which either increases or decreases the conductivity of the layer [9]. Micro-heater is required to achieve the adequate temperature for the reactions to take place.

Following are the issues that need to kept in mind while designing micro-heater

Uniform Heating

Heating should be uniformly distributed all over the surface. There should not be any hotspots and for uniform heating, non-uniform heater which has unequal strip gaps can be used. Also, there are many types of heater designs from which an appropriate heater should be chosen according to the application.

Low power consumption

In miniaturisation of devices, minimising power consumption has always been an issue as it should be as low as possible. It can be reduced by using cavity beneath the heater.

Mechanical stability

It should be rigid in nature and able to withstand high temperatures.

1.9 Structure of metal oxide based sensor

Basic layers of the device are given below

1) Substrate
2) Insulating platform
3) Micro-heater
4) Interdigitated electrodes
5) Sensing layer

Substrate

Substrate is the bottom layer which is basically provides base to the heater. Silicon substrates are popular because of its uniform mechanical properties.

Insulating platform

Insulating platform is a layer between the substrate and the micro-heater to avoid direct damage to the substrate and to reduce the heat losses.

Micro-heater

Micro-heater is essential in metal oxide gas sensors to detect the desired gas efficiently. The heating of micro-heater is based on the concept of joule of heating. Micro-heaters are used in humidity sensors, pressure sensors as well as gas sensors.

Interdigitated electrodes

Interdigitated electrodes are placed beneath the sensing layer which detect variations in resistance of metal oxide sensing surface when it respond to gases. The term interdigitated,means a digit like pattern of parallel electrodes which are used to measure the resistance when the gas is absorbed on the layer [10]. When the sensor is reacted with the atmospheric chemicals, conductivity of the material is changed. Interdigitated chemical sensors are cost efficient and so are very popular.

Metal oxide sensing layer

Metal oxide sensing layers are used in gas sensors as they have excellent sensitivity towards the hazardous gases. It can be made from many materials depending upon the gas to be sensed. The conductivity of sensing layer varies with respect to the gas concentration present around. The temperature limit of some popular metal oxide sensing layer are shown in Table 1.2 [11]

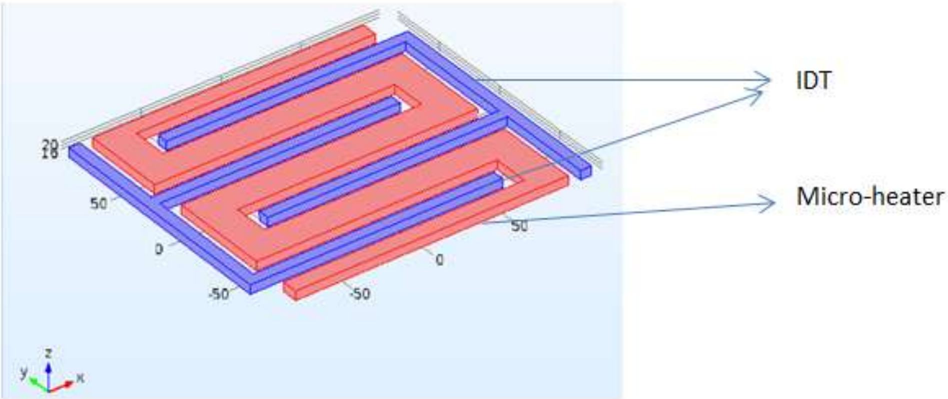


Fig1.2 Interdigitated electrodes placed coplanar to micro-heater

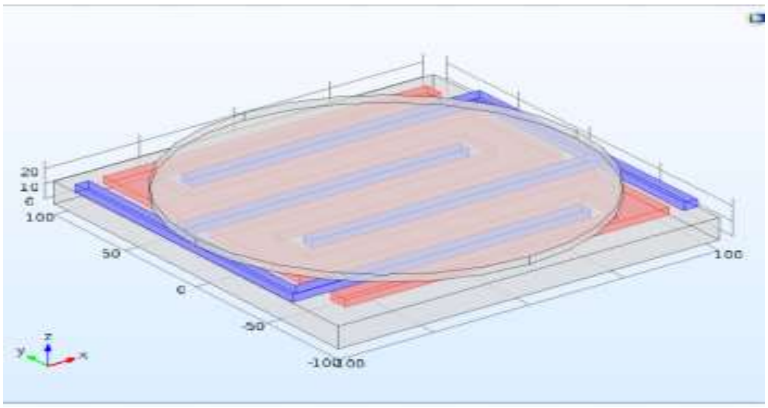


Fig 1.3 3D view of Micro-heater with IDT and sensing layer

Table 1.2 Temperature limit of some selected metal oxide sensing surfaces used in gas sensors [11]

Sr. No	Metal oxides sensing surface	Temperature range
1	Tin oxide (SnO_2)	300° C or above
2	Zinc oxide (ZnO)	300° C
3	Tungsten trioxide (WO_3)	Up to 500° C
4	Indium tin oxide (ITO)	300° C
5	Titanium oxide (TiO_2)	250° C
6	Cerium oxide (CeO_2)	400° C

Table 1.3 Typically used metal oxide sensing surface and their aimed gases [12, 13]

Sr.No	Metal Oxides sensing surfaces	Aimed gas
1	Tungsten trioxide (WO3), Zinc oxide (ZnO)	Nitrogen dioxide (NO2)
2	Tungsten trioxide (WO3)	Nitric oxide (NO)
3	Tin oxide (SnO2), Zinc oxide (ZnO)	Nitrous oxide (N2O)
5	Zinc oxide (ZnO), Titanium oxide (TiO2)	Carbon monoxide (CO)

1.10 Overview of hazardous gases

Hazardous gases are those gases that are considered dangerous to human health when present in excess proportion. They are broadly divided in to two categories: Toxic gases: Toxic gases are those which are potentially harmful to humans when are inhaled. This includes gases like ammonia, chlorine, sulphur etc. Flammable gases: Flammable gases are those which have the potential to burn in certain concentrations. They will burn when oxygen is present around; which usually is; as without oxygen there is a problem for workers around. In addition to the various hazardous gases, there are also sensors which are used to detect the low levels of oxygen which is an essential gas. A summary of various properties of the hazardous gases are shown in Table 1. TLV is the threshold limit of a particular that is allowed for repeated exposure without causing any health issue. [13]. Nitrogen dioxide which has the TLV of about 3 ppm is very harmful. Hydrogen sulfide (H2S) is one of the most dangerous gases, as it can disrupt the oxygen supply. It can be detected easily at lower concentrations from its rotten egg like smell but at higher concentrations, it affects the senses badly, and one is not able to sense it. Carbon monoxide is a colourless and odourless gas and is undetectable without a sensor. Ammonia (NH3) is a colourless gas with pungent odour. It is harmful to human health when its concentration reaches more than 25 ppm. Health effects due to excessive exposure of SO2 include respiratory problems. Persons with asthma are sensitive to it. Methane can explode in air when present below 5%. Nitrous oxide creates global warming to a large extent which causes disruption in ozone layer. Carbon dioxide (CO2) is a colourless and odourless gas. It is emitted by human activities. Environmental monitoring of these gases is must to ensure a good environmental health. As per Reference [14], one-third of Carbon dioxide is emitted during transportation. The use of fossil based fuels emits greenhouse gas and also degrades air quality. According to the pollution index rating 2016, out of top ten polluted cities in the world; three are Indian cities, which is definitely an issue of major concern. Demand of energy is increasing and the condition of fossil fuel supplies is at gloom. Hydrogen energy is renewable and pollution-free. Water vapour and heat are the by-products of hydrogen energy, not any other air pollutant. Because of these advantages, hydrogen fuelled products becoming popular. Safety is the most important issue that needs to be taken care while its development. The explosive range of hydrogen is from 4% to 74%. Therefore, hydrogen sensors should carefully monitor the hydrogen leak for safe use of hydrogen energy.

Table 1.4 Properties of various hazardous gases [13]

Gas	Physical property	Source of emission	Environmental/health impacts	TLV
No2	Reddish-brown in colour, pungent odour	Mostly from automobile and industrial sector	Respiratory problems, corrode metals	3 ppm
NO	Non-flammable	Produced during combustion in air	Irritation in eyes	25 ppm
N2O	Colourless with a sweet odor	from nitrogen fertilizers, and from oceans	Causes greenhouse effect	50 ppm
H2S	Colourless, toxic and flammable in nature rotten egg smell	through volcanic gases and hot springs	Effects breathing	10 ppm
CO	Colourless, odourless, non-irritating gas .	Through incomplete carbon products like wood, coal.	Prevents absorption of oxygen in blood thus damaging vital organs	50 ppm
NH3	Colourless, pungent odour	During the decomposition of animal manures	Irritation in eyes	25 ppm
CH4	Odourless and Combustible gas	By an-aerobic digestion of organic materials	Explosive	1000 ppm
SO2	Lighter than air, Invisible gas with nasty smell	Produced from industrial emission	Irritation in nose, throat, and causes Shortness of breath, and heaviness in chest.	5 ppm
CO2	Colourless and odourless gas	Vehicle emissions	Creates suffocation and oxygen deficiency	5000 ppm
H2	Light, Non-toxic, odorless, highly flammable, easily reacts	Cylinders,lpgtanks, pipelines	High concentration causes oxygen deficient environment which leads to breathing shortness, explosive mixtures easily formed	Flammability limit 4%-75%

CHAPTER-2
LITERATURE REVIEW

2.1 Literature Review

TesturoSeiyam et al. in 1962 presented a new detector to sense various gaseous components by using borosilicate glass tube and zinc oxide (ZnO) as sensing layer. Electronic recorder can observe the changes in the sensing layer. Results are analysed for Toluene, Benzene, Propane and Carbon dioxide. Time response recorded by the electronic recorder is observed and the resulted output peaks represented change in thermal conductivity of zincoxide [15].

H. Meixner et al. in 1996 have analysed some metal oxides like, ZnO, SnO2, and TiO2. Metal oxides were less in application due to lack of knowledge at that time. They have also discussed fundamental requirements of metal oxides in next generation. They should have low SNR, high reducibility, long term stability and minimum breaking time [16].

Marius Dumitrescu et al. in 1998 simulated poly-Silicon based micro-hotplate structure of 110 µm x 110 µm dimensions having silicon as substrate material. “COMSOL” software is used for simulation of four “poly” suspended bridges structure and a central supporting pillar structure with the separation of 1 µm air gap to substrate. They achieved the temperature of about 673 K by applying 100mW power [17].

S. Semancik et al. in 2001 introduced micro-hotplate platform made of poly-Silicon material on the substrate made up of Silicon dioxide. They used four electrodes for analysing the change in sensing layer properties. Micro-hotplate designed is CMOS compatible and can be easily integrated on IC circuits [18].

Isolde Simon et al. in 2001 presented closed membrane metal oxide gas sensor and suspended membrane type gas sensor. They discussed sensing layer deposition techniques like, thick film deposition method and thin film deposition method. They concluded that both the sensing films differ in their thickness and structure which leads to different transducer functions [19].

M. Afridi et al. in 2002 presented monolithic MEMS gas sensor made up of poly-Silicon micro-heater. Gas sensor virtual components that are analog circuits and sensor itself should enclose in digital cell so one can make digital interface. They concluded that sensor and its complexity directly affect the response time. Heater efficiency, temperature sensor response and sensing film response with respect to gas concentration are analysed [20].

M. Baroncini et al. in 2004 proposed a double spiral micro-heater configuration with four probes for MEMS based gas sensor. They have used silicon as substrate and Silicon nitride as membrane of 1 mm X 1mm surface area. They also implemented double spiral geometry with two probes for voltage tab and two probes for micro-heater ends. Also sensing layer has two probes for measuring change in electrical properties. Surface temperature vs heating power characteristic is analysed [21].

A.z.sadek et.al in 2005 presents hydrogen and nitrogen dioxide gas sensors based on ZnOnano-belts. Response of hydrogen and nitrogen dioxide gases at different operating temperatures is observed. At 300 °C, highest magnitude response is recorded. Sensor with zinc oxide nano belt has lower optimum temperature in comparison to conductometric sensors of bare zinc oxide crystal layers [36]

Dimeo, Frank dio me at.al in 2006 presented a novel hydrogen gas sensor. Changes in resistance of >110% to 0.25 % hydrogen concentrations are measured, with response times 200ppm to > 1%. Low cross sensitivity is observed [34].

Ching-Liang Dai et al. in 2007 have implemented a chip nanowire WO3 (sensing surface material) based sensor with a poly-Silicon heater and an inverting amplifier using CMOS process. Humidity with respect to output voltage characteristic is observed [22].

J.F. Creemer et al. in 2007 have introduced micro-heater and micro-hotplate made up of TiN (Titanium nitride). They have compared spiral geometry structure for platinum and TiN micro-heater. Results are compared and analysed and they came to the conclusion that TiN have high melting point so it can survive very high temperature in comparison to platinum micro-heater [23].

Si-dong-kim et.al in 2007 presented co-planar-type gas sensors with high sensitivity and fast response are fabricated. Low power consumption is achieved by modifying the geometry. Maximum thermal efficiency is seen at 60um width. The fabricated gas sensor is expected to be useful for applications in detecting gases of automobiles because of the advantages like simple fabrication and low fabrication cost [35].

H.-Y. Lee et al. in 2008 came out with Wheatstone bridge (circular ring) platinum based micro-heater structure where four resistances are equal in normal condition and change can be measured with analog circuitry. This structure has multi-rings which spread heat so more uniformity can be achieved. They used SiO2 – Si3N4 – SiO2 (O/N/O) structure with platinum as micro-heater. Also they have proved that by using this structure high uniform temperature of 400° C can be achieved at central area of circular ring micro-heater [24].

Velmathi G. et al. in 2010 has introduced six different geometries of micro-heater. They have analysed plane plate structure with central square hole, double spiral, meander, fan type honey comb and s-type geometries. Their 2D surface temperature is observed, and was able to gain 400° C temperature. Also resistive heating vs applied voltage characteristic is analysed [25].

Jae-Cheol Shim et al. in 2010 have fabricated Nitric oxide sensor using 3C-SiC (cubic unit cell, zinc blende) material based micro-heater with Zinc oxide (ZnO) sensing film. They have used AlN/SiC membrane. They also analysed that platinum added ZnO has high sensitivity in comparison to bare ZnO sensing film. It was proven that SiC micro-heater can withstand till power value of 1.1 W that is quite higher than platinum micro-heater [26].

M. Gayake et al. in 2011 compared three different geometries of Polyimide based micro-heater. They changed track width and gap width of all the three geometries and came out with the conclusion that circular geometry has better response due to lesser edge losses. Also for micro-heater, heating response is observed using 5.2 V and 6.0 V supply voltage and noticed that 6.0 V supply gave good heating response [27].

Vineet Bansal et al. in 2011 simulated platinum spiral geometry. They have investigated four different cases [28]

1) Simple spiral micro-heater geometry that gave temperature up to 761.73 K.
2) Spiral geometry with Si cavity at centre which gave temperature response up to 1036.8K.
3) A suspended spiral micro-heater on four bridges that provides maximum temperature up to 919.1 K.
4) Spiral micro-heater with zinc oxide sensing layer.

Susmita Sinha et al. in 2011 simulated various shapes of micro-heater. They used DilverP1 (which is made of alloy of Ni, Co, Fe) material for micro-heater. Transient temperature response and relation between maximum temperature and power consumption is analysed and it is proved that that power consumption of DilverP1 is less in comparison to poly-Silicon and platinum [29].

BijoyKantha et al. in 2012 have made 3D analysis of MEMS based Dilver P1 micro-heater with Coventor ware design and simulator software. Structure has insulating layer of Si3N4, interdigitated electrodes of Aluminium and sensing layer made up of ZnO. Thickness and power consumption relation is observed; also voltage and power consumption relation for different thickness of micro-heater is analysed [30].

L. Sujatha et al. in 2012 have done simulations for poly-Silicon based micro-heater using finite element method of COMSOL Multiphysics 4.2 simulation tool. Four different geometries named, single meander, double meander, fan type and square type are simulated and 2-D simulation response is analysed. Maximum temperature value and temperature uniformity are analysed and they have concluded that square type geometry has better temperature uniformity among others. Maximum temperature and applied voltage relation is plotted [31].

Jianhai Sun et al. in 2013 made flow sensor having silicon as substrate and SiN as insulating layer. They used two heating/sensing elements to sense resistive variation in diagonal position which works as Wheatstone bridge connection. Variation in resistance can be observed as amplifier output voltage [32].

Monika et al.in 2013 have simulated curved spiral micro-heater geometry using Dilver P1 as micro-heater material. Micro-heater thickness is varied and power consumption is analysed. Power consumption of same curved spiral micro-heater made of different materials are investigated it is proved thatDilver P1 based micro-heater gives result with minimum power consumption [33].

S.Punnose at.al in 2015 presented MEMS based alcohol gas sensor simulated in Comsol Multiphysics. They presented alcohol sensor to detect the presence of alcohol by the theory of adsorption. Adsorption coefficients related to each material used is defined and according to that simulation of adsorption molecules is done.

2.2OBJECTIVES

The main objectives of presented work are as follows:

1. To simulate different micro-heater geometries using COMSOL Multiphysics and to analyse which geometry gives better temperature uniformity.
2. To create a virtual environment around the sensor device by creating a gas chamber
3. To analyse the effects of the gas on the sensing layer
4. To find an optimum temperature for the sensor.
5. To optimise the sensing layer in order to achieve better sensitivity.

CHAPTER-3
INTRODUCTION TO COMSOL MULTIPHYSICS

3.1 COMSOL MUTIPHYSICS

COSMOL Multiphysics is simulation software founded in Sweden and is used worldwide for various applications. It includes packages for engineering and medical applications. The purpose of the tool is to create a virtual environment for a particular task to get the similar results as of the real environment. It has various modules like AC/DC module, electrical, chemical, plasma, general physics etc.

It includes various interfaces and properties of different materials. User can propose their own variables and equations. COMSOL main window is shown in Fig 3.1.

Main Menu **Toolbar** **Graphic window**

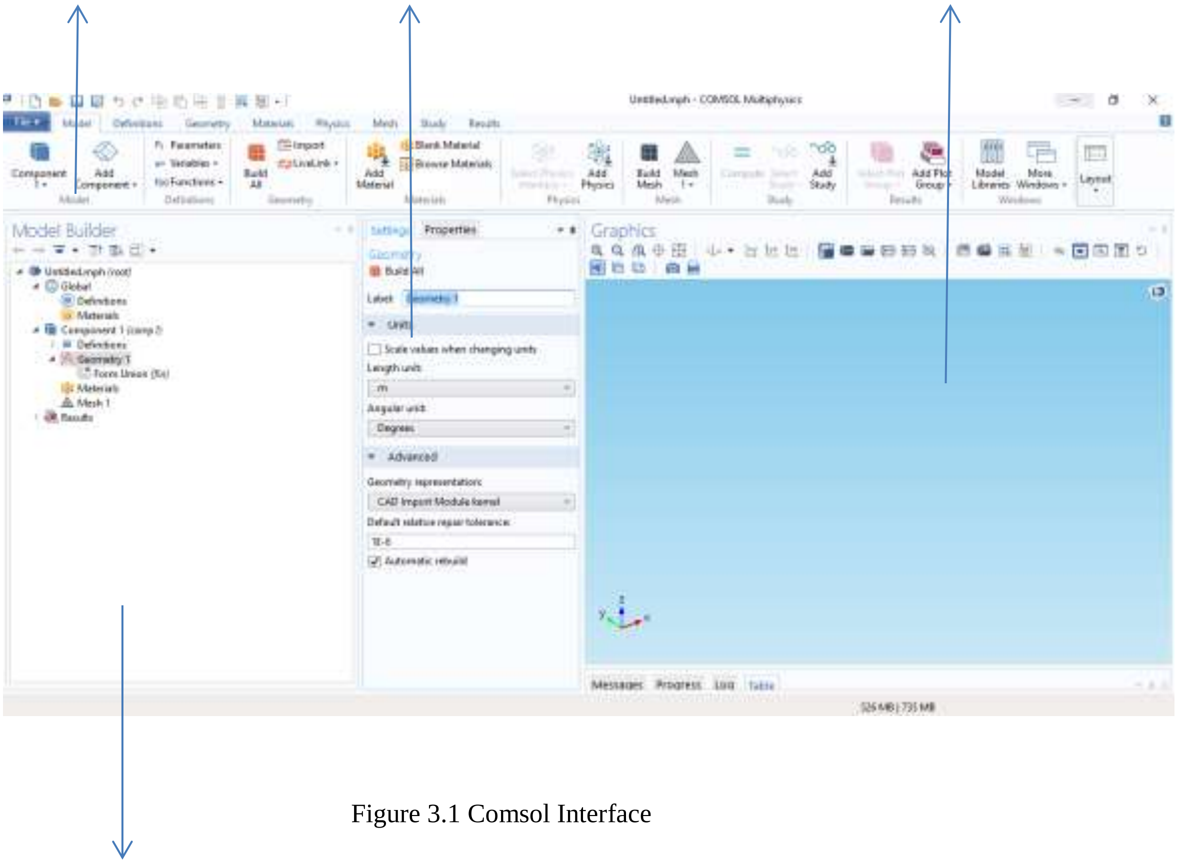


Figure 3.1 Comsol Interface

Model builder

3.2 Steps to model

Step 1

At opening of the COMSOL (5.2), three options are asked by the tool

1) Model Wizard
2) Blank Model
3) Application Wizard

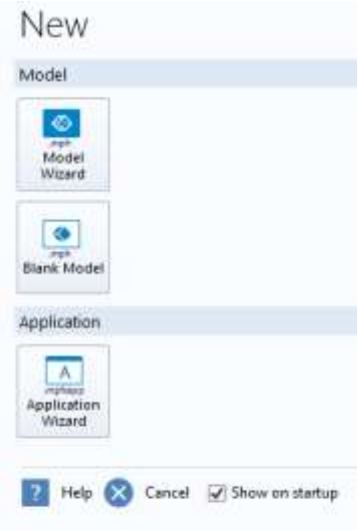


Figure 3.2 New Model

(1) Working in model wizard

The model wizard will ask for dimensions to work as shown in figure.

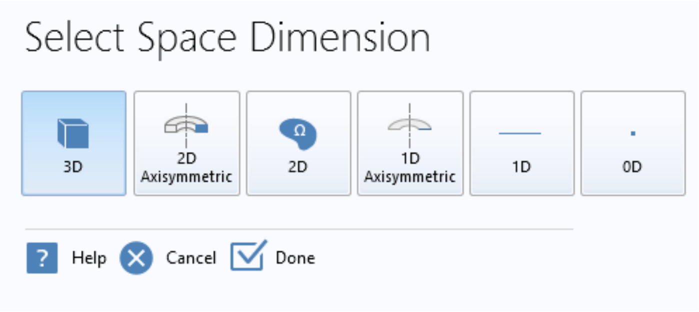
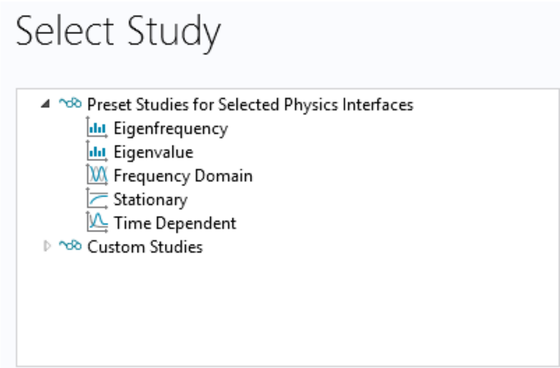


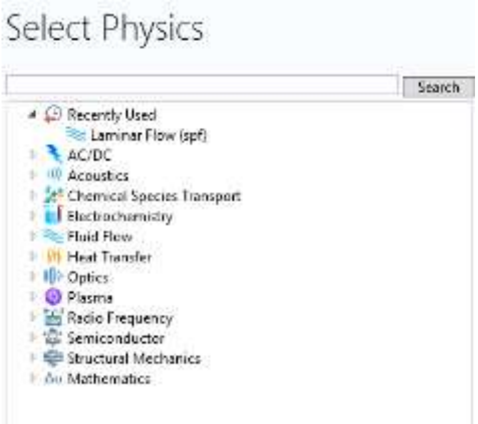
Figure 3.3 Select space dimensions



Select Study

- Preset Studies for Selected Physics Interfaces
  - Eigenfrequency
  - Eigenvalue
  - Frequency Domain
  - Stationary
  - Time Dependent
- Custom Studies

Figure 3.4a Selection of Study



Select Physics

Search

- Recently Used
  - Laminar Flow (spf)
- AC/DC
- Acoustics
- Chemical Species Transport
- Electrochemistry
- Fluid Flow
- Heat Transfer
- Optics
- Plasma
- Radio Frequency
- Semiconductor
- Structural Mechanics
- Mathematics

Fig 3.4b Selection of Physics

After selecting dimensions, tool will show the option for various studies like stationary and time dependent. User can select the desired one according to the work. Various Physics can be added in to one model.

(2) Working in blank model

The blank model does not ask for any study or component. User can add the component by clicking on model tree. It provides the various functionalities of the simulation. Various materials and studies can be added from this toolbar

Figure 3.5 Quick access toolbar

Application wizard

An application of the model simulated can be made by it by hiding all the back end data. Only the Input data has to be provided by the user and the application wizard will simulate the intended application.

3.3 Joule heating Multiphysics

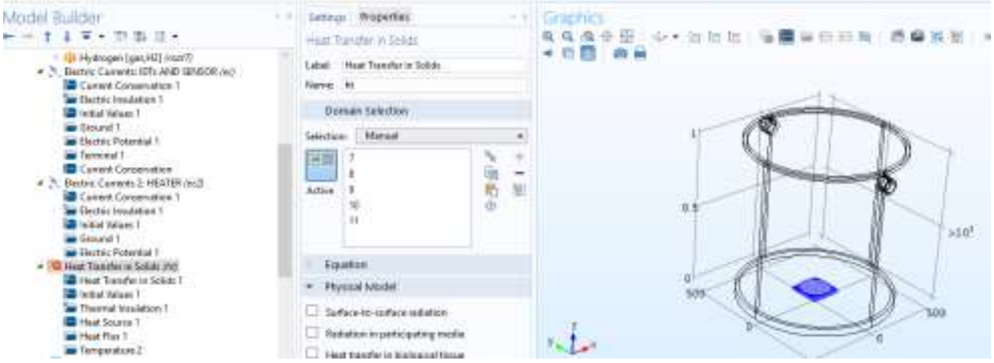
The Joule Heating multi-physics interface includes two physics that are Electric Currents and Heat Transfer in Solids. They will be combined together by Multiphysics.

The Heat Transfer in Solids and Electric Currents studies has the settings for heat conduction and current conduction, respectively. In Electric Currents study, the Current Conservation mode shows electric current at domain level and the Electric Insulation option represents the boundary condition for Electric Currents.

In Heat Transfer in Solids study, the domain level Heat Transfer in Solids mode shows the heat conservation and the Thermal Insulation mode presents the boundary condition for heat Transfer.

They are defined as

- Current conservation
- Electric insulation
- Initial values
- Ground
- Heat transfer in solids
- Thermal insulation
- Heat flux



Label: Heat Transfer in Solids

Name: ht

Selection: Manual

7
8
9
10
11

☐ Surface-to-surface radiation

☐ Radiation in participating media

☐ Heat transfer in biological tissue

Figure 3.6 Model Builder interface

3.4 Transport of diluted species

The Transport of Diluted Species gives a domain for evaluating the species carried by diffusion. It is assumed that all the species present are dilute means their concentration is small in comparison to the solvent.

This interface helps in the evaluation of the chemical species carried by convection or diffusion in different dimensions. It ca compute various models over time and also in stationary mode, when concerned parameters are provided by the user.

It considers the mass balance equation as given in

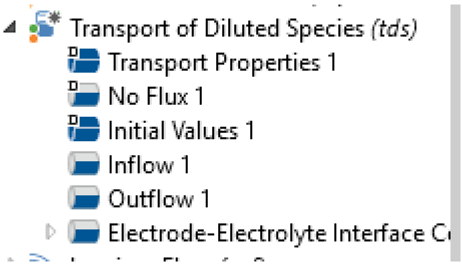
$$\frac{\partial c}{\partial t} + u.\tilde{\nabla}c = \tilde{\nabla}.\,(D\,\tilde{\nabla}c) + R$$

Where, c is concentration of the species (mol/m^3)

D is diffusion coefficient (m^2/s)

R is Reaction rate for the species (mol/ (m^3.s))

U is velocity vector



Transport of Diluted Species (tds)

Transport Properties 1

No Flux 1

Initial Values 1

Inflow 1

Outflow 1

Electrode-Electrolyte Interface C

Figure 3.7 Components of added physics

3.5 Laminar Flow

The Laminar Flow physics comes under the Single-Phase Flow node in model Wizard. It includes conditions for simulation of fluids.

Domain selection

By default it has all the possible domains in the model. Specific domains can be chosen form manual in the drop down list.

Physical model

It controls the various properties of the Laminar Flow.

Compressibility

Compressible flow <0.3 is used as default. It can also be changed manually.

Dependent variables

Following are the dependent variables—

Velocity field (u) (SI unit: m/s)

Pressure (p) (SI unit: Pa)

Variable names and component names can be edited. If the current field matches with the existing field of the same type, then degrees of freedom and variable names will be merged. So, a new variable name should not coincide with any earlier name defined by the user.

Advanced settings

Advanced settings can be chosen by clicking on the show button ( ). Advanced Physics Options can be selected from there. In Normal conditions, user does not need to change these settings.

Discretization

Discretization can again be selected from the show () button. It selects the order of velocity and pressure. It can be

P1+P1 (by default),

P2+P1 or P3+P2

P1+P1 (default) includes linear elements for velocity as well as pressure filed. Linear elements are cheaper in computation in comparison to higher-order elements.

P2+P1 represent second-order elements for velocity and pressure field.

P3+P2 represent third-order elements for velocity and pressure field. It adds additional accuracy but additional degrees of freedom are also included in comparison to P2+P1 elements.

Fluid Properties

Material properties can be added by a simple right click on it.

Domain selection

Domains can be chosen from the selection menu.

Model inputs

Variables that are given as input to the flow equations can be edited if needed. For fluid flow, model inputs are given when any material needing inputs has been applied.

Fluid properties

Density: By default it is shown as ρ (SI unit: kg/m3). User defined option can be selected to add another value.

Dynamic Viscosity: By default it is shown as μ (SI unit: Pa·s).

Initial Values: This requires an initial value for the velocity field and the pressure.

Domain Selection: It has list from which domains can be chosen to enter the initial values.

Inlet

The Inlet node gives a set of conditions which describes the fluid flow at the specified boundary.

CHAPTER-4
ANALYSIS OF MICRO-HEATER GEOMETRIES

4.1 Selection of micro-heater geometry and material

There are different types of micro-heater geometries and each fits to a specific interest. Among all, we have to choose one which fulfils our requirements. Following are the major factors that are to be taken care while choosing the material for the micro-heater [25].

- Thermal conductivity
- Coefficient of thermal expansion
- Electrical conductivity

Thermal Conductivity:

Thermal conductivity is the measure up to which any material can conduct. It determines the cooling time and heating time of the micro-heater. Higher the thermal conductivity of material, higher is the rise time.

Coefficient of thermal expansion

Coefficient of thermal expansion measures the degree up to which an object changes its dimension. If its value is higher than the material deforms at a faster rate. So, it’s desirable to have lower thermal coefficient of expansion while choosing micro-heater material.

Electrical conductivity

Electrical conductivity shows how the temperature increases due to joule heating effect. Dilver P1 possesses high yield stress and minimal thermal expansion but it is not a well-known material. So for present study, Poly silicon (Poly-Si) and Platinum (Pt) are preferred as materials for micro-heater design. Poly-Silicon (Poly-Si) material has high resistivity which makes it more suitable for micro-heater. Whereas Platinum (Pt) has very high temperature stability (as its melting point is high) so it can easily withstand high temperatures. Therefore, micro-heater geometries are compared and analysed using these materials.

4.2 Simulation of micro-heater geometries and their results

Different micro-heater geometries are simulated and analysed using simulation tool COMSOL Multiphysics 5.2 to achieve high temperature uniformity and low power consumption. Different geometries namely, single meander, double meander, S-shape are analysed for designing micro-heater in metal oxide gas sensors. In the present study, all micro-heater geometries are made up of poly-silicon with thickness 3 μm and 120 x 120 μm^2 area using SiO_2 substrate as thermal insulation is needed for better temperature throughout the geometry [37]. One end of micro-heater is provided 3 V and the second end is at 0 potential. Comparison is done for the following geometries-

- Single meander
- Double meander
- S-shape

Simulations are done to discover the suitable geometry which gives throughout uniformity of temperature. Every simulation result represents distribution of temperature by various colours.

Single meander geometry

Design of single meander geometry and its simulation is shown in fig. 4.1 and fig. 4.2, where fig 4.1 shows single mender geometry and fig. 4.2 represents simulation result of temperature distribution. Simulation result displays minimal uniformity in temperature.

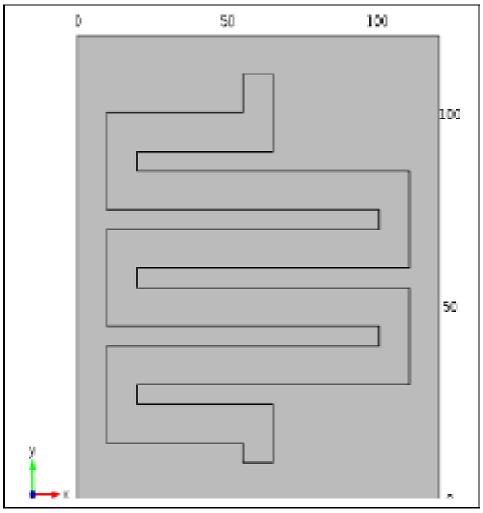


Fig 4.2 Temperature distribution of single meander geometry

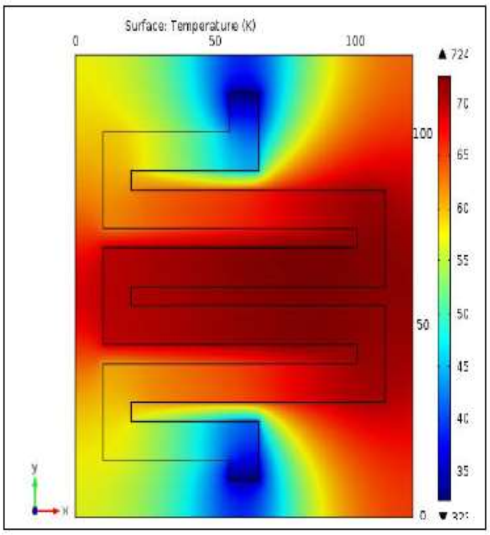
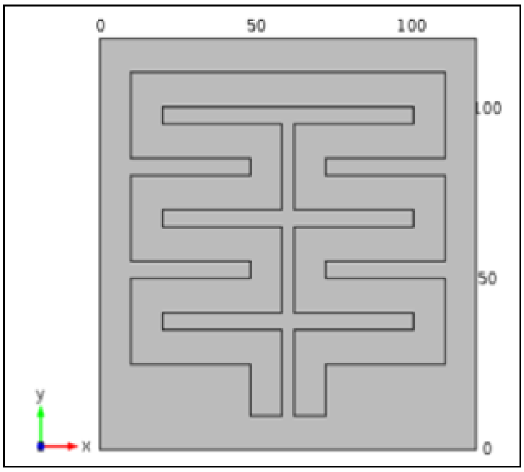


Fig 4.2Tempearture distribution of single Meander geometry

Double meander geometry

Fig 4.3 demonstrates the double mender geometry and Fig. 4.4 represents simulation result. It shows an improvement in temperature uniformity in comparison to the geometry simulated in fig 4.1 [37].



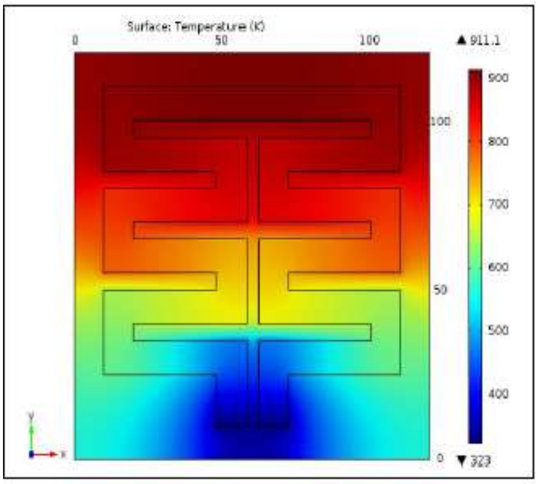


Fig 4.3 Design of double meander geometry

Fig 4.4 Tempearture distribution of double meander geometry

S-shaped geometry

S-shape geometry has more material deposition at centre. More temperature and higher uniformity is observed in this particular geometry. But these types of geometries have to handle high stress due to thermal expansion [37]. Fig. 4.6 shows the simulation result of S-shape geometry where larger temperature uniformity can be seen.

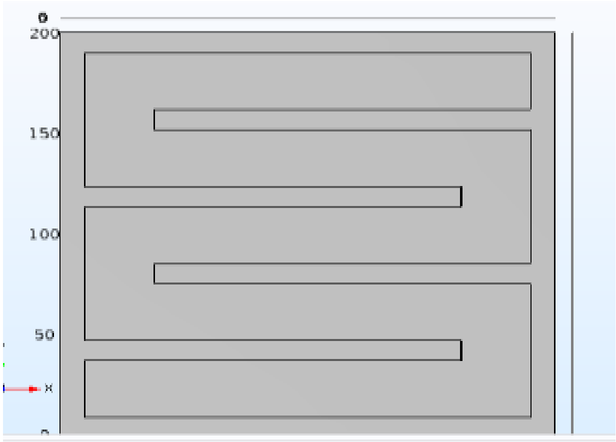


Fig 4.5 Design of S-shape geometry

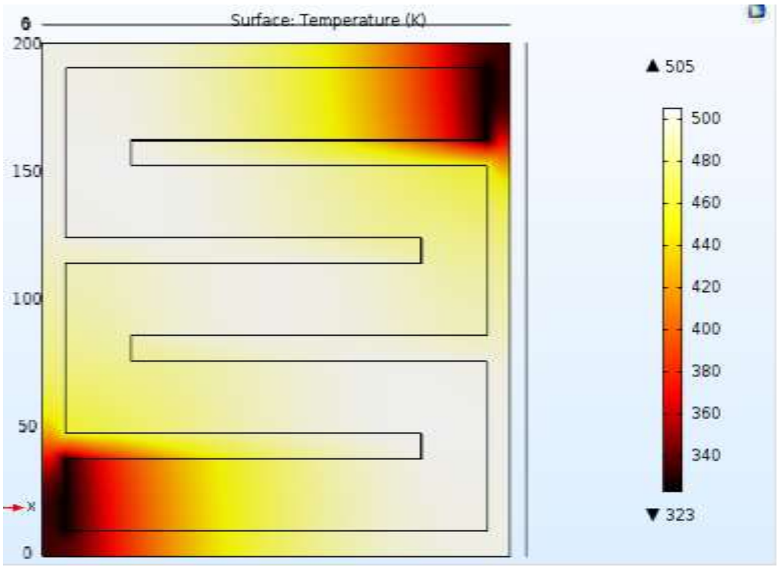


Fig 4.6 Temperature distribution across S-shape geometry

S-shape and double meander geometries gave more temperature uniformity. But for further use in our application, we will use S-shape geometry as we can easily apply IDTs in it.

4.3 Design of S-Shape geometry for simulation

Geometry which is given in Fig. 4.7 is designed on 200 x 200 μm^2 substrate made up of silicon dioxide which has 2 µm thickness. Properties of Silicon dioxide and Platinum are defined in **Appendix 1**. Geometry has equal stripe width of 20 µm. Place IDT in the same plane that as of micro-heater. The IDE’s are also made of Poly-Si and have same stripe width of 7 µm throughout.

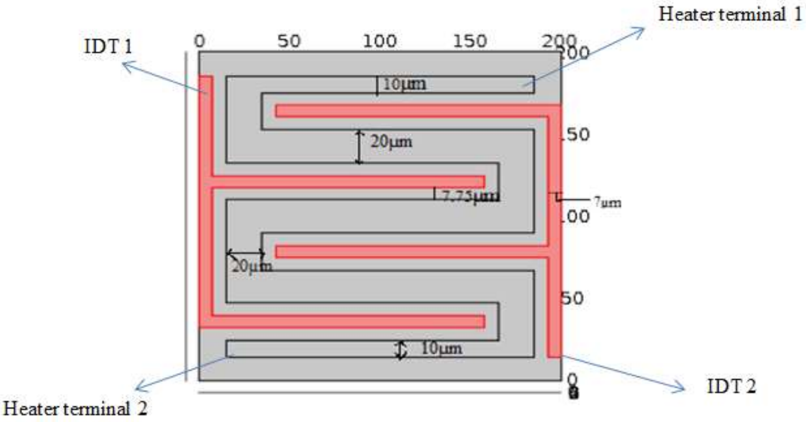


Fig 4.7 IDTs placed coplanar to the micro-heater

Tin oxide is used as a material for sensing layer. Whenever there is a gas present around it will react with the layer and heater will provide the temperature required for reaction. A circular layer is designed over the surface of micro-heater as shown in Fig. 4.8. Thickness of 2 µm is given to the sensing layer.

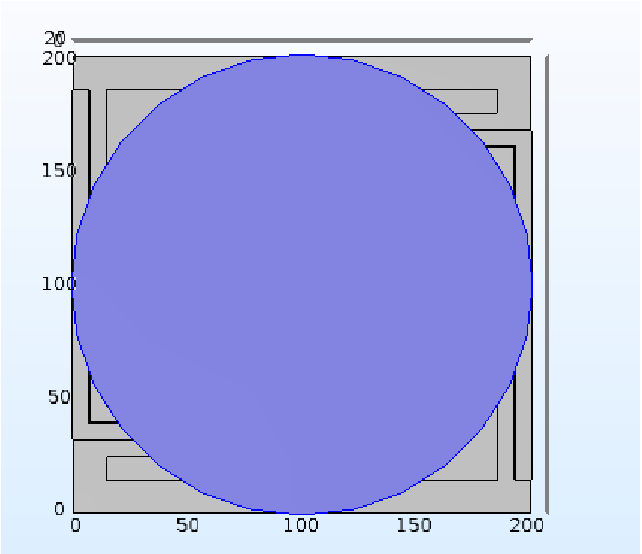


Fig 4.8 Sensing layer placed on the micro-heater

4.4 Simulation Results of uniform S-shape Micro-heater with IDT and Sensing Layer

Voltage of 3 V is applied across the terminals of heater to obtain the required temperature. Silicon dioxide of thickness 3 µm is providing the base to the device to protect it from any damage. Fig. 4.9 is showing the temperature distribution across the geometry designed.

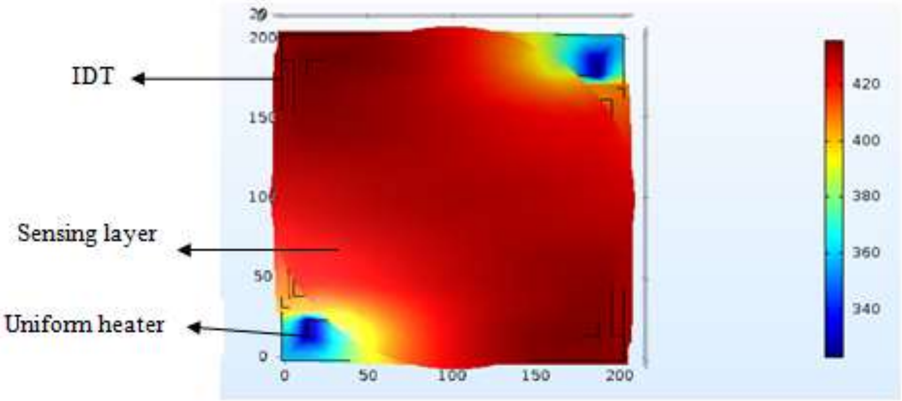


Fig 4.9 Simulation result of uniform S-shape micro-heater

4.5 Non-uniform micro-heater to achieve better temperature uniformity

Changing the width of micro-heater will help in providing more uniformity. Stripe width is 10 μm on the upper and lower edges and 20 μm width in remaining parts.Voltage of 3V is applied across the micro-heater to obtain the required temperature. Fig. 4.10 represents temperature distribution across the design.

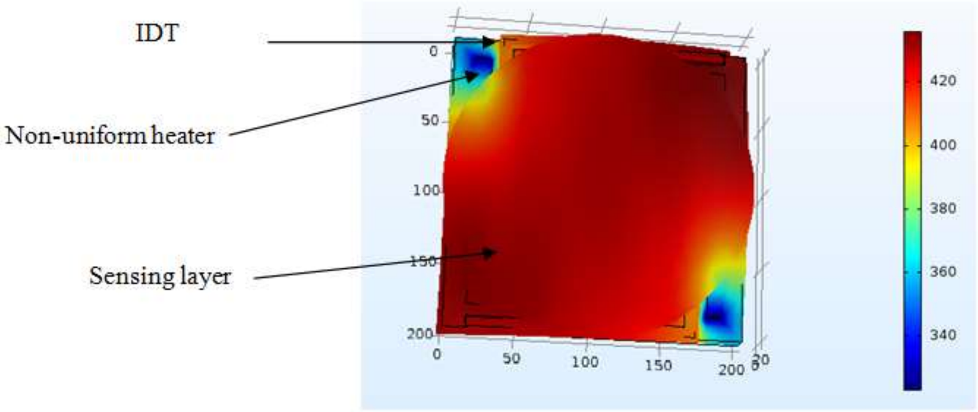


Fig 4.10 Simulation of varying width micro-heater

Comparing Fig. 4.9 and Fig. 4.10 it can be easily observed that the non-uniform micro-heater provides better temperature uniformity than the uniform width micro-heater. Graph of temperature comparison for both geometries is shown in Fig. 4.11. The red line represents the temperature variation for uniform width s-shaped micro-heater and blue line represents non-uniform width micro-heater. Fig.4.11 shows that more temperature uniformity is achieved in non-uniform micro-heater.

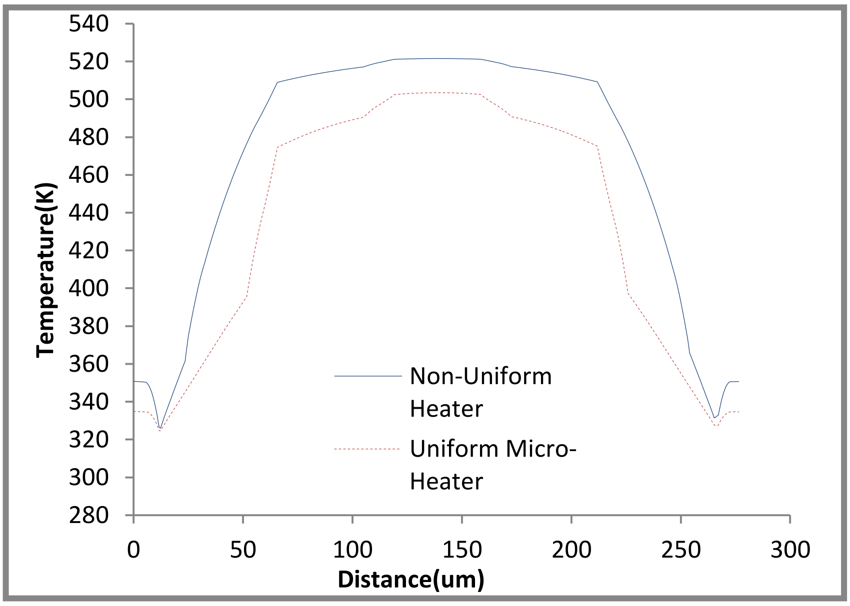


Fig 4.11 Comparison of non-uniform heater with uniform heater

4.6 Material for Gas sensing layer

Sensing layer can be made up of different materials. But each gas reacts with the layer differently as every sensing layer is sensitive to a particular gas or a particular type of group of gases only. So it is very important to study the sensitivity of layer material to the corresponding gas which is to be sensed

Tin oxide(SnO_2)

Tin oxide is one of the most researched materials that are used in sensor industry**.** Any material is chosen for an application on the basis of its intrinsic properties. The material properties are classified into bulk and surface properties. It must be noted that the exact status of physics and chemistry are not fully developed, in order to know the effects of surface properties on the sensitivity of the gas [38].

Structural Properties of Tin oxide (SnO_2)

Tin (Sn) is a naturally found element which appears in group 14 (4A) of the periodic table. Several organic and inorganic compounds can be formed with tin. Tin gives an outlook of a silver-white metal which is malleable and ductile to some extent. Tin oxide is a semiconductor which has tetragonal structure. The unit cell has six atoms, two Sn, and four O2 [38]. Every Sn atom is at the centre of six O_2 atoms which are at the corners of an octahedron, and three Sn atoms are at the corners of an equilateral triangle surround every O2 atom; which results in 6:3 coordination as shown in figure 4.12 [39].

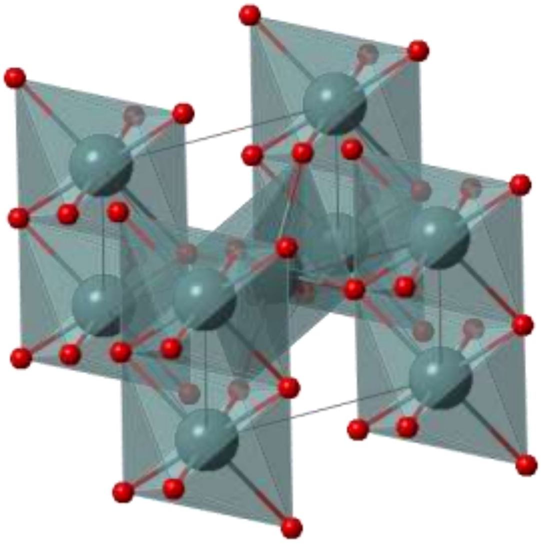


Fig 4.12 Structure of tin oxide (Source: Wikipedia)

SnO_2 is anefficient gas sensor because of the combined effects of bulk as well as surface properties. Reactivity of any gas with the sensing layer depends on the chemical species that are adsorbed on the surface layer of the material. Generally, most of the gases are detected through the influence that they have on the O_2 stoichiometry of the surface. H_2 can reduce the SnO_2 surface, through chemisorbed O_2 which results in increasing surface conductance.

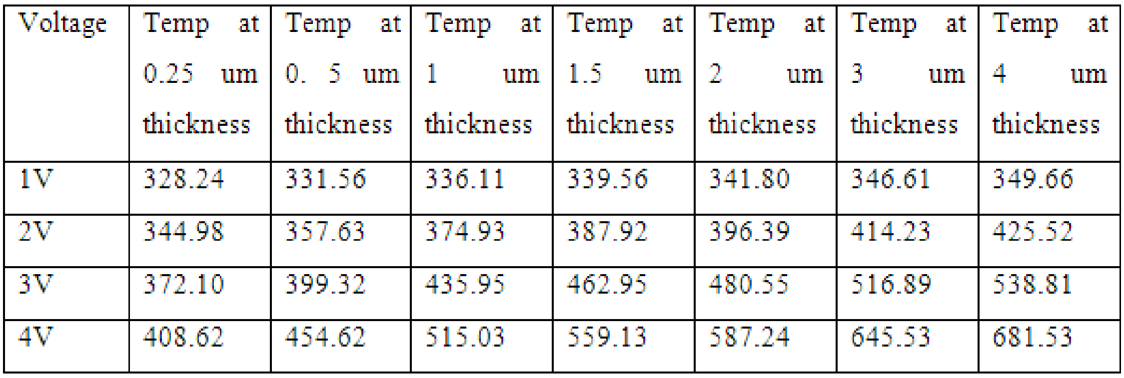
Chapter-5
RESULTS AND DISCUSSIONS

5.1 Analysis of MEMS Micro-heater

As previously discussed that micro-heater can be made up from different materials. But considering the properties of different materials, platinum and poly-silicon are widely used. Simulations are done using both materials and their respective results are compared and analysed

Analysis of Poly-silicon Micro-heater at different voltages w.r.t varying thickness of Micro- heater is given below-

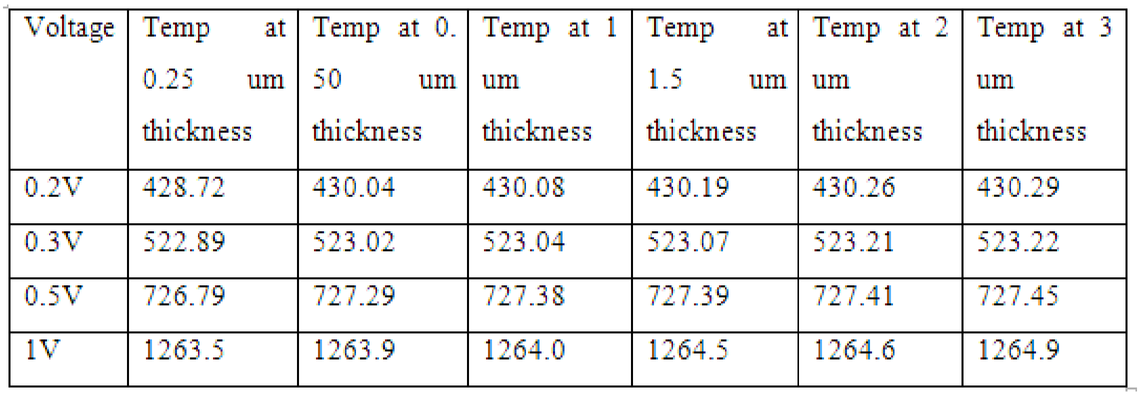
Table 5.1 Analysis chart of poly-silicon micro-heater



Voltage	Temp at 0.25 um thickness	Temp at 0. 5 um thickness	Temp at 1 um thickness	Temp at 1.5 um thickness	Temp at 2 um thickness	Temp at 3 um thickness	Temp at 4 um thickness
1V	328.24	331.56	336.11	339.56	341.80	346.61	349.66
2V	344.98	357.63	374.93	387.92	396.39	414.23	425.52
3V	372.10	399.32	435.95	462.95	480.55	516.89	538.81
4V	408.62	454.62	515.03	559.13	587.24	645.53	681.53

Analysis of Platinum Micro-heater at different voltages w.r.t varying thickness of micro-heater is given below-

Table 5.2 Analysis chart of Platinum micro-heater



Voltage	Temp at 0.25 um thickness	Temp at 0. 50 um thickness	Temp at 1 um thickness	Temp at 1.5 um thickness	Temp at 2 um thickness	Temp at 3 um thickness
0.2V	428.72	430.04	430.08	430.19	430.26	430.29
0.3V	522.89	523.02	523.04	523.07	523.21	523.22
0.5V	726.79	727.29	727.38	727.39	727.41	727.45
1V	1263.5	1263.9	1264.0	1264.5	1264.6	1264.9

Platinum has excellent thermal conductivity so it reaches the same temperature at less voltage in comparison to poly-Si but it is more expensive and has less resistivity.

5.2 Design of gas chamber

A virtual environment is created for the reactions to take place with the sensing layer. For that, a gas chamber has been designed and how the physiochemical properties of sensing layer changes w.r.t the target gas in the chamber has been analysed.

A gas chamber of 1mm x 1mm has been designed with an inlet of radius 0.05mm and sensor device is placed inside the chamber at the bottom.

Inlet-Inlet is designed for the entering of the gas.

Outlet-Outlet is designed if the gas has to be flushed out.

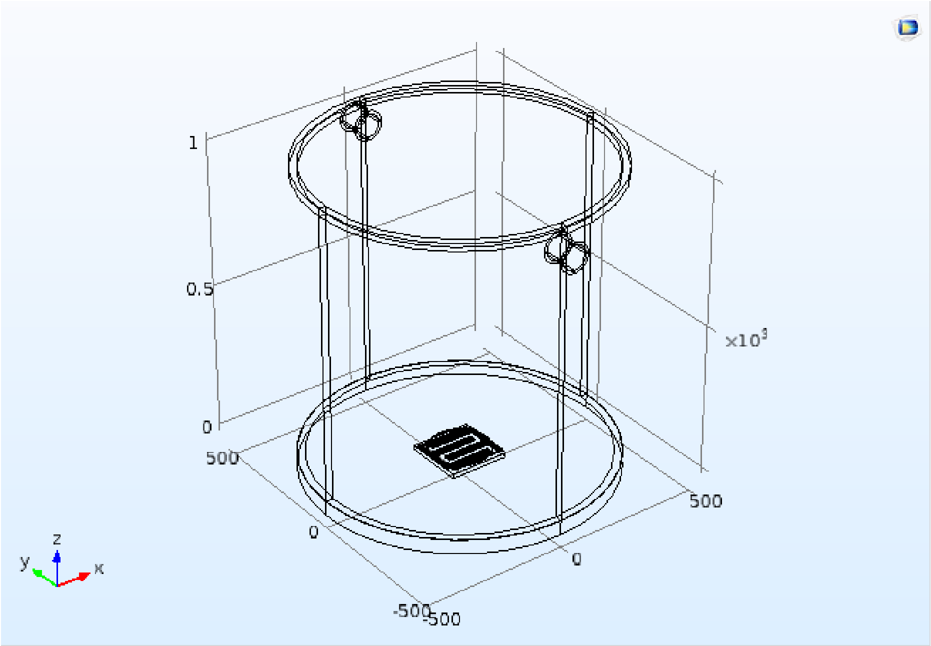


Fig 5.1 Design of gas chamber

5.3 Study of average temperature of the sensor device w.r.t time

In figure temperature of the sensor is increasing with time. At 30 ms, maximum temperature at 2V is about 370k and at 3V it is about 500K which implies that by increasing the voltage applied to the heater, response time of the sensor heating is getting better.

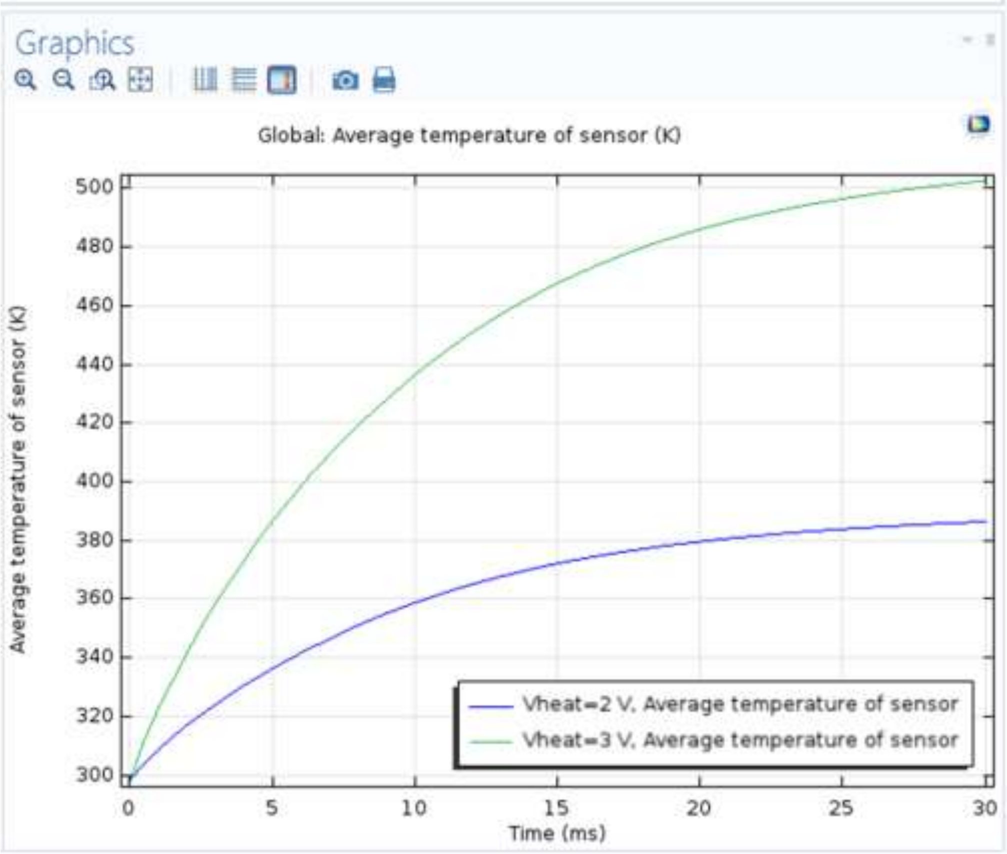


Fig 5.2 Comparison of average temperature of sensor at different voltages

5.4 Study of average concentration of the gas species at the sensingsurface

The behaviour of the gas introduced in the chamber and the average gas concentration of the gas on the sensor device is shown. The gas which is introduced in the gas chamber through the inlet has not been fully absorbed on the layer. The gas which is introduced and the proportion which has reached on the sensing surface are compared.

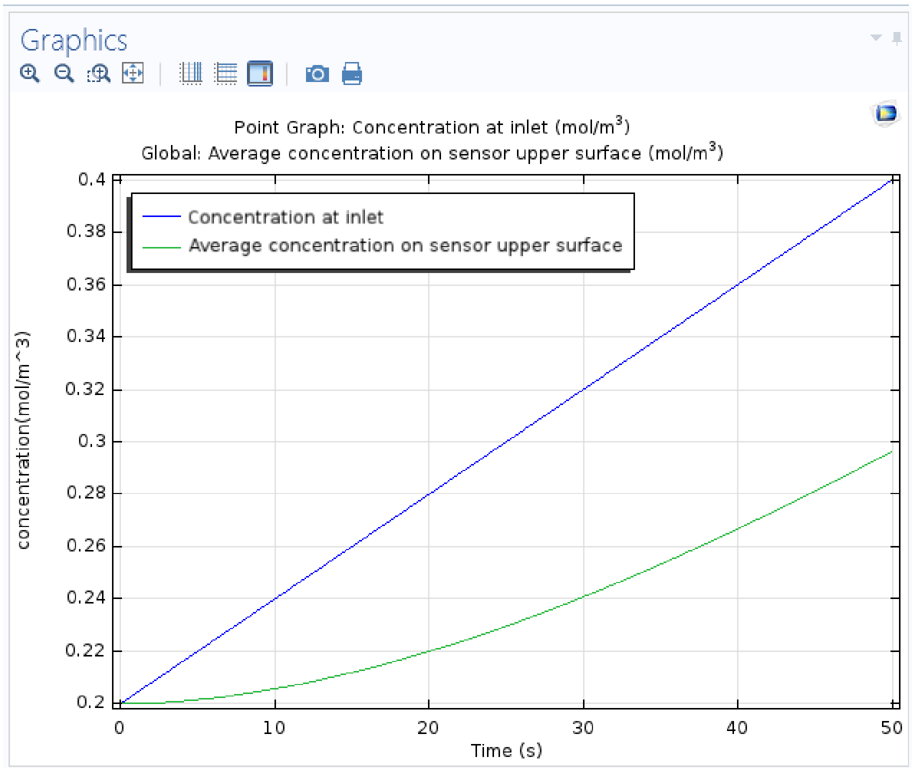


Fig 5.3 Comparison of the average concentration of the gas in chamber and at the sensing layer

5.5 Study of effect on the resistance of sensing layer with varying concentration

The resistance of the gas with respect to the increasing concentration of the gas is observed when the gas is varied from 1 mol/m^3 to 50 mol/m^3.The voltage is kept constant at 3V.

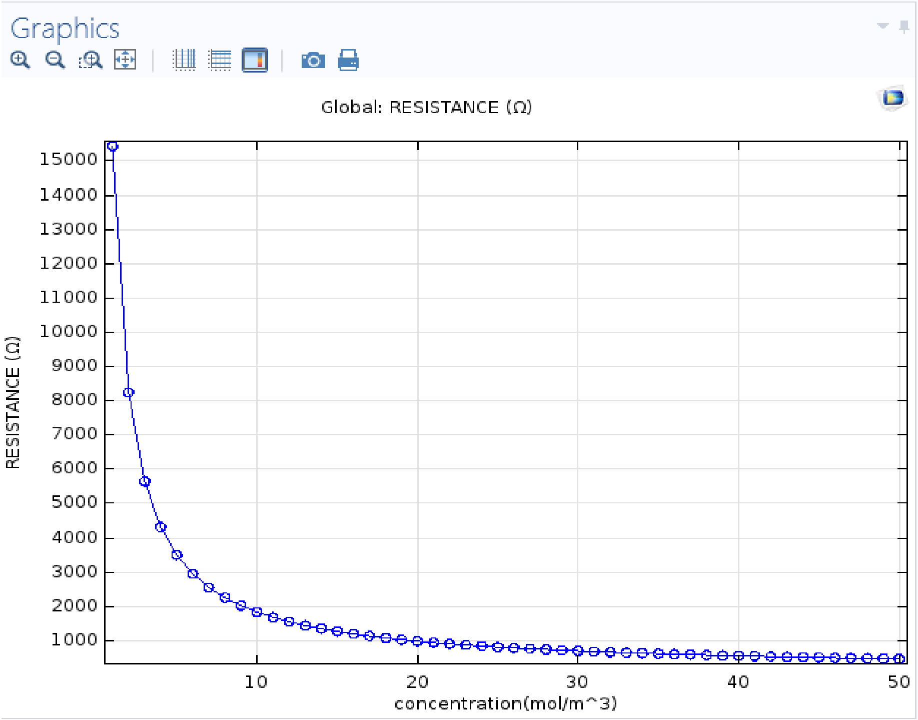


Fig 5.4 Variation in resistance of the sensing layer w.r.t concentration

5.6 To ensure the gas sensing mechanism by introducing a input pattern of gases

Input pulses of the concentration are given to the chamber with respect to time to ensure the gas sensing mechanism. A time slot of 1600s is taken and concentration is varied up to 15 mol/m^3.

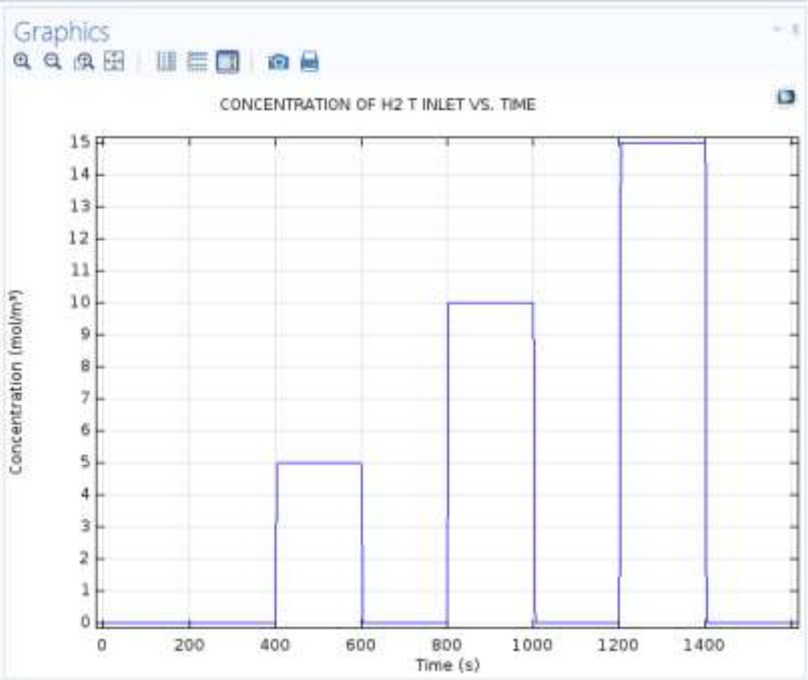


Fig 5.5 Input pulses of concentration given to the chamber

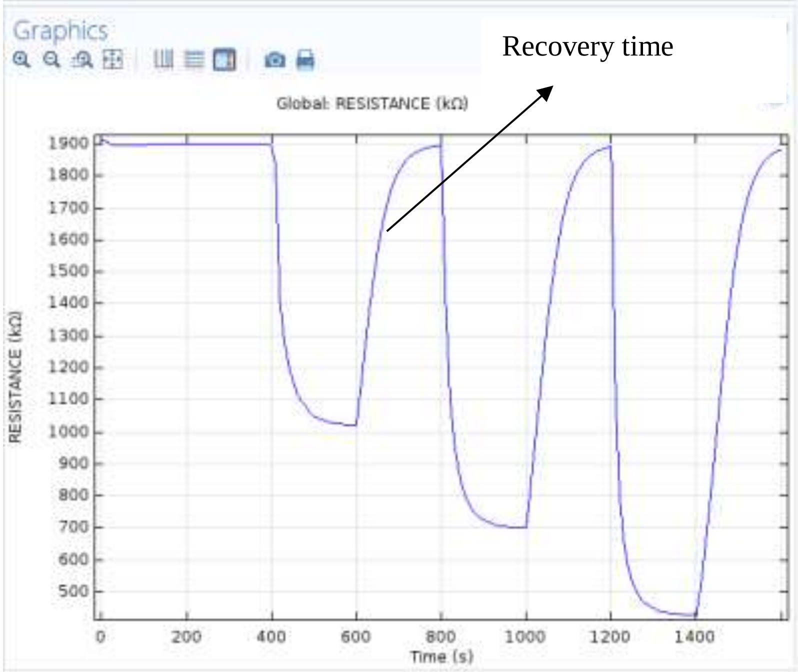


Fig 5.6 Dynamic response of sensor to input concentration pulses of figure 5.5

Resistance change w.r.t the input concentration given is observed and it can be seen that when the concentration is constant up to a 400 sec, resistance is also constant up to that time and when concentration starts increasing at 400 sec, resistance shows a corresponding decrease, and at the purge resistance is again increasing ensuring the gas sensing mechanism w.r.t the reducing behaviour of the hydrogen gas.

5.7 Study of resistance of sensing layer at various operating voltages

As the voltage is increased, the average temperature over the sensor is showing an increase which will correspondingly fasten the reaction rate and hence resistance of the layer will decrease.

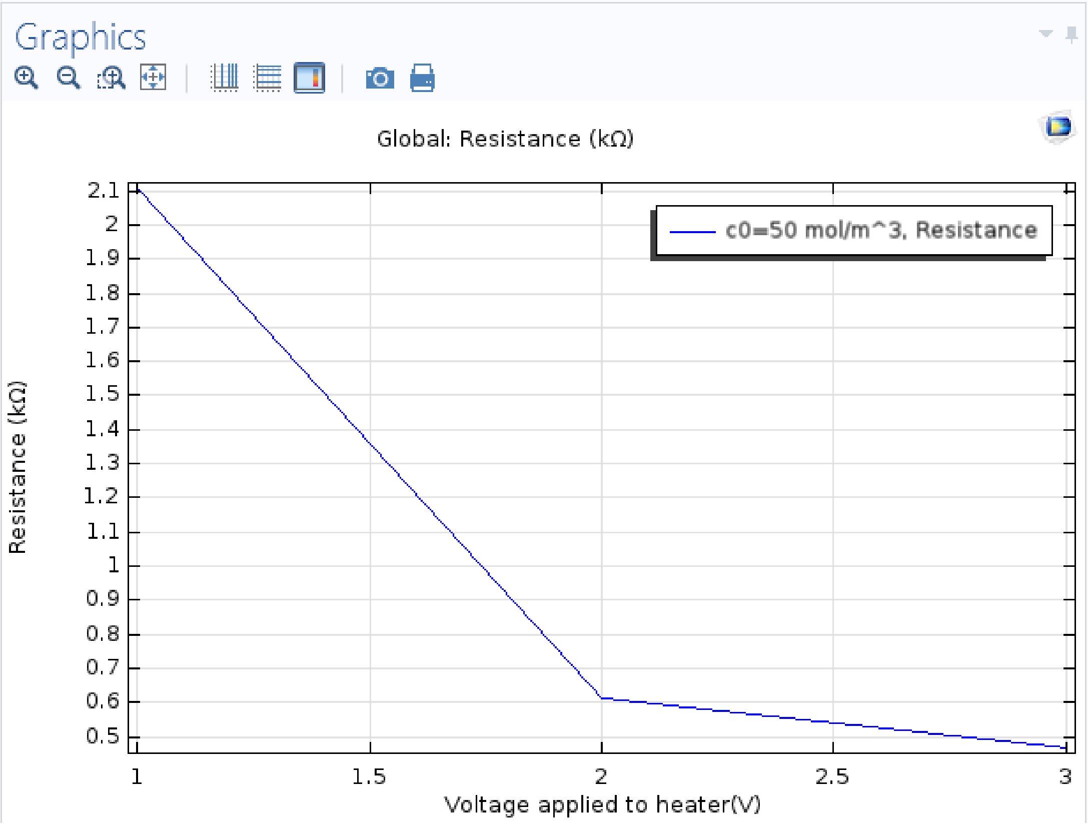


Fig 5.7 Resistance change of the sensor w.r.t varying voltage

5.8 Study of response time of the sensor

Response time of the sensor device is showing a decrease when the voltage applied is increased, as with the increased voltage, an average temp of the sensor will increase which will fasten the rate of reactions on the layer and the response time is showing an evident decrease. Blue, green and red lines are showing the response time at heater voltage of 1V, 2V, 3V respectively.

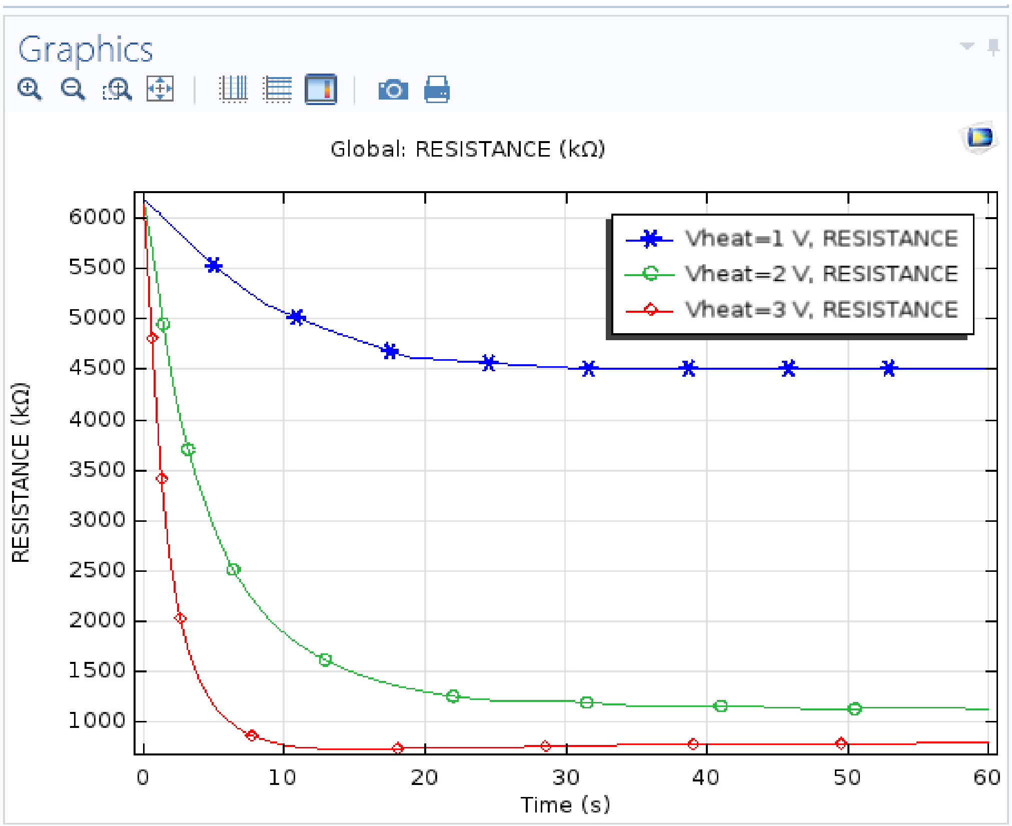


Fig 5.8 Response time of sensor w.r.t applied voltage to heater

5.9 Study of an optimised value of temperature for the sensor device

The sensor is simulated at temperatures between 300 K to 600 K as shown in figure 5.9. Adsorption and desorption of gases are temperature initiated process. Hydrogen gas pulses of 10 mol/m^3,50 mol/m^{3}and 100 mol/m^3 at different operating temperatures are given as input to the device. It is found that sensor shows maximum response at about 450 K.

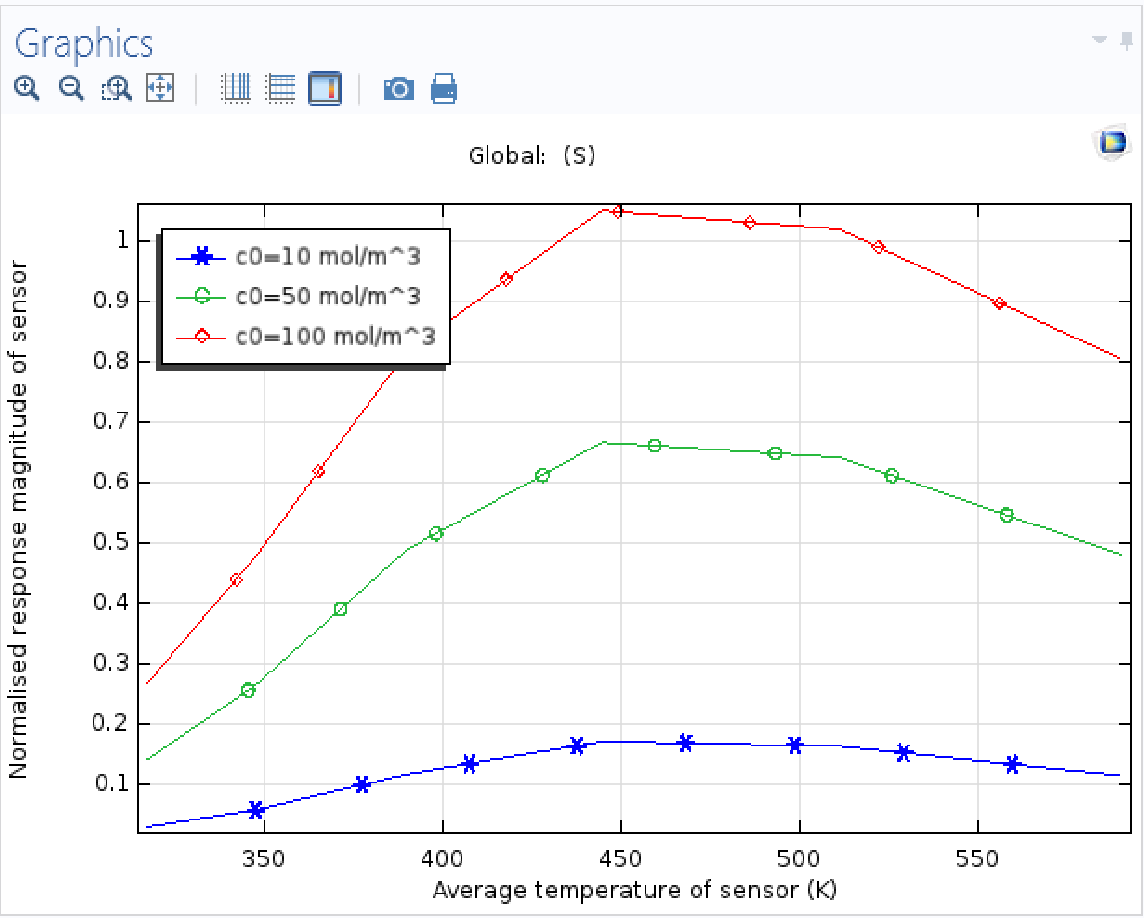


Fig 5.9 Comparison of normalised response magnitude of sensor for different gas concentrations vs operational temperature

5.10 Sensitivity of layer w.r.t temperature at different thickness of sensing layer

Thick films consists loosely connected crystallites that features several cracks that helps in the inflow of gas molecules in to the layer and thus improves sensitivity. Sensitivity decreases when the sensing layer is thickened. Here blue, green, and red lines represent the sensing layer thickness at 20, 10,5um respectively to study the effect of thickness of layer on the sensitivity of the sensor device.

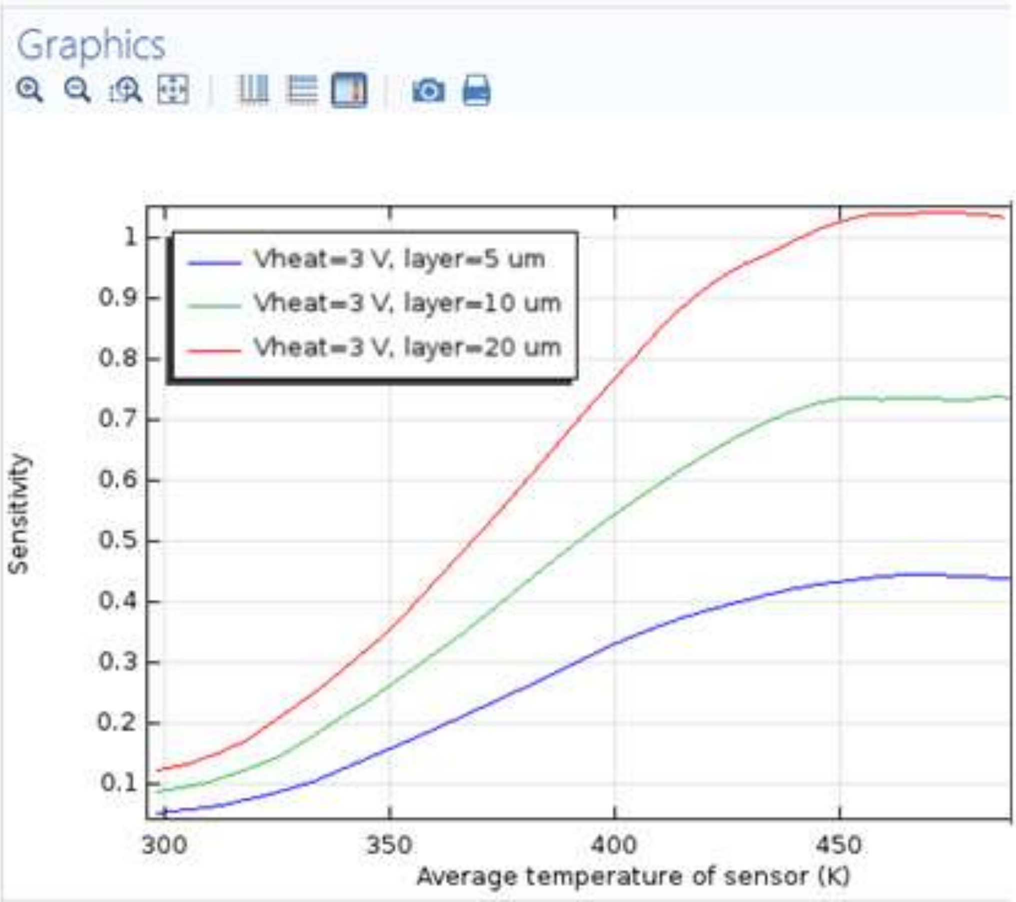


Fig 5.10 Sensitivity of layer at various thicknesses' of sensing layer

CHAPTER-6
FABRICATION STEPS

6.1 PROPOSED FABRICATION OF MEMS BASES GAS SENSOR

Design and simulation of MEMS based gas sensor has been done. Following is the proposed methodology for the fabrication process.

WAFER CLEANING

Silicon wafers which are to be used in fabrication should be clean before use. One such method is piranha cleaning. Piranha mixture is made by mixing sulfuric acid and hydrogen peroxide. Being an oxidising agent it cleans all the organic matter. Solution can be made by mixing the both agents in different ratios(x: y where x refers to sulfuric acid and y refers to hydrogen peroxide) like 3:1, 4:1 or 7:1. Piranha solution cleans the surface by dissolving surface impurities. Solution should be properly mixed as it is very corrosive in nature. Hydrogen peroxide should be added to sulfuric acid very slowly and never vice versa. Explosion can occur if peroxide solution concentration exceeds 50%.cleaning is normally done in 10 to 40 minutes, after which the substrate is removed from the existing solution. Hydrogen peroxide self decomposes itself therefore the solution prepared should be used freshly and not to be stored.

OXIDATION

There are various methods of oxidation like wet oxidation, plasma oxidation and diffusion oxidation.

Wet oxidation method is mostly preferred in fabrication of gas sensor. It is explained below-

At first, the thermal wet oxidation furnace is turned on to grow 100 nm oxide layers and temperature has to be raised to 500℃. Then nitrogen atmosphere has to be created in the furnace. Next, wafers are to be loaded in the furnace. Raise the temperature from 500℃ to 1000℃. After it, flow of oxygen gas in to the furnace for dry oxidation. Direct supply of oxygen is given for a period of 10 minutes. Now, pass oxygen gas through water bubbler for 3 hrs. Disrupt the oxygen supply after 3 hrs and flow the nitrogen gas in to the furnace (0.5 litre/min).Unload the wafers out of furnace but only after decreasing the furnace temperature up to 37℃. This oxide is treated as hard mask during the etching process.

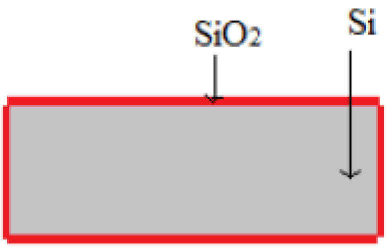


Fig 6.1 Structure after wet oxidation

Lithography

Oxidized wafer is to be coated properly with S1813 photoresist at 4000 rpm for 40 sec where temperature is kept at 125 ℃ for 1 minute. Ultraviolet bulb is turned on for 10 minutes. Mask can be loaded according to the procedure mentioned by the software. Mask is transferred by the coated photoresist by UV exposure.MF26A solution is used for developing pattern followed by rinsing with DI water and nitrogen drying.

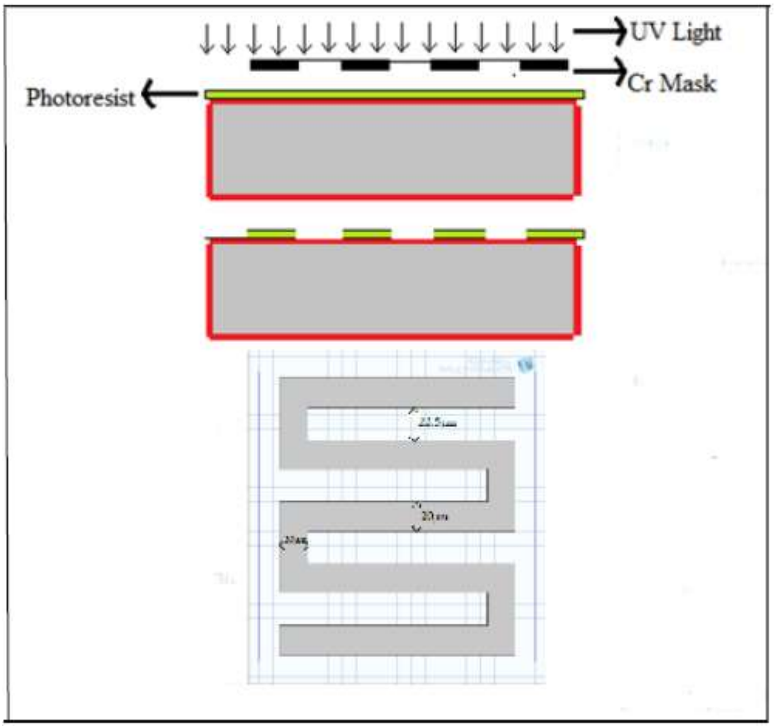


Fig 6.2 Top view of mask layout designed of micro-heater with dimension

Platinum deposition

Deposition of 200 nm platinum is done after lithography process by sputtering method.

Parameters which are used for sputtering are
Voltage given to plate 1.5 KV, incident power and reflected power are 60w and 25w.Current given to plate is 60mA. Pre-sputtering time and deposition time are 2 min and 8 min respectively.

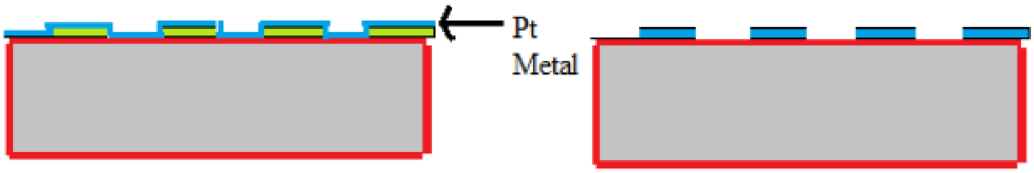


Fig 6.3 Structure after platinum deposition

Silicon nitride deposition

Silicon nitride of thickness 80nm is deposited as a mask for silicon etching.
Parameters which are used for sputtering are as-
Voltage given to plate is 1.5 KV, Incident power and reflected powers are 60 W and 20 W respectively. Current given to plate is 60mA. Pre-sputtering time and deposition time are 15 minutes each. After that annealing is done at 700 °C temperature for about 45 minutes.

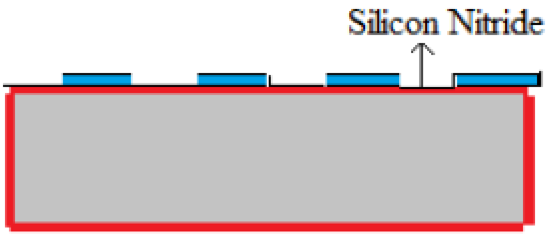


Fig 6.4 Structure after Silicon Nitride deposition

Backside membrane patterning

Backside of wafer is coated with S1813 photoresist at about 4000 rpm. Time slot given is 40 secs and prebaked temp is 125°C. Membrane pattern is aligned with respect to top layer. Ultraviolet bulb is turned on for 10 minutes for stabilisation. Masked pattern is transferred to the coated photoresist by UV exposure. MF26A developer solution is used for developing pattern for 1 min and nitrogen drying is done afterwards.

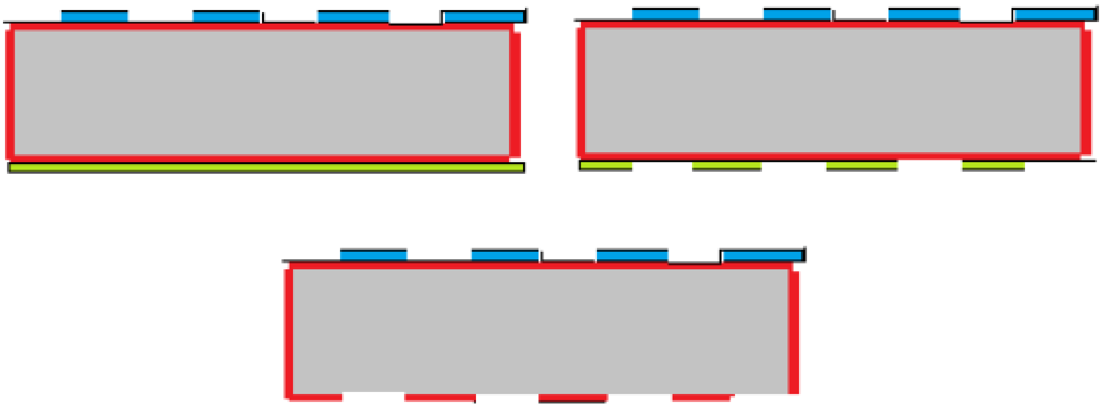


Fig 6.5 Structure after membrane patterning

Silicon etching using KOH + IPA

Preparation of 30% KOH mixture

Take 85% pure KOH weighing 105g in to 250ml beaker and add 190ml DI water and stir it properly to dissolve the KOH. Place the solution on hotplate and raise the temperature up to 75℃. Now add 50 ml of IPH in to it. Silicon etching is done by putting the wafer in to the solution for about 2hr and 30min.Next is to clean the wafer with DI water properly and dry with nitrogen.

Preparation of 5% TMAH solution

40ml of TMAH solution (25%) is added to 200ml of DI water in a 250 ml furnace. After then solution is placed on hotplate after setting the temperature at 75℃. Time is to be set at 15 minutes. Load etched wafer in to heated TMAH solution for 3 hours. Take out the sample from the solution and rinse it properly with DI water properly followed by methanol.

Fig 6.6 Silicon etching using KOH + IPA

Silicon Nitride Removal

Silicon nitride is to be etched using 1:3 BFH solution. After etching wafer is properly rinsed with DI water and dried up with nitrogen.

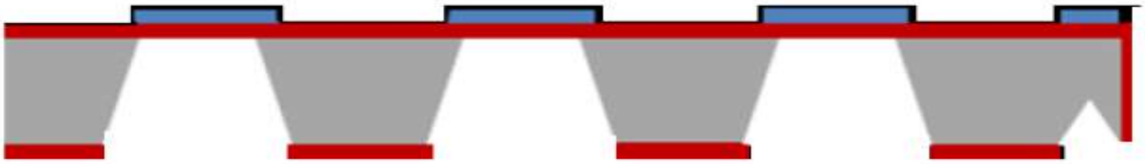


Fig 6.7 Structure after Silicon Nitride removal

IDT deposition

For IDT deposition, same procedure is followed which was used for micro-heater deposition. Idt is used to detect the resistance change in sensing surface.

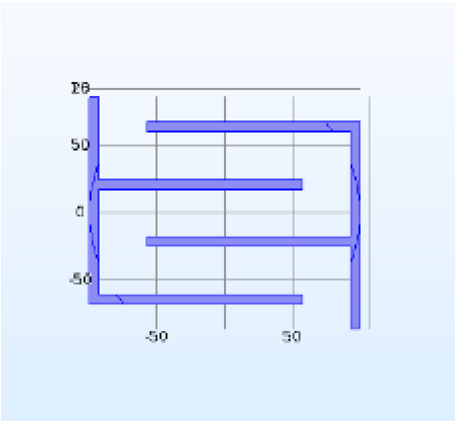


Fig 6.8 IDT structure

Deposition of sensing layer

Uppermost layer is the sensing layer where reactions take place. Tin oxide and Zinc oxide are the most popular materials used as sensing surface

CHAPTER-7
CONCLUDING REMARKS AND FUTURE SCOPE

CONCLUSION

In this work, MEMS based gas sensor has been designed and simulated. Various micro-heater geometries are studied and the one giving more uniform heating is selected for the further use. Poly-Silicon and platinum materials are studied for the micro-heater. A virtual environment has been created by designing a gas chamber around the device and the behaviour of the sensor is studied in the presence of gas. COMSOL MULTIPHYSICS 5.2 is used for the simulations. Out of various micro-heater geometries, S-shaped and meander shaped geometries were found to be having more temperature uniformity, but for our intended purpose. S-shaped geometry is used as it is difficult to apply IDTs across the double meander geometry. Gas is put in the chamber and the behaviour of the sensor device is studied when the concentration of the gas is increased. Hydrogen is a reducing gas, thus resistance is found to be decreasing w.r.t to the increasing concentration. Transient analysis of the device in the presence of the hydrogen gas is done. Various heater voltages were given to the device and it was observed that more is the applied voltage to the heater, more is the reaction rate, thus lesser is the response time of the sensor device. Optimised value of temperature for different concentrations of gas at a constant voltage of 3 V was noted and it was found to be near 450 K.

FUTURE SCOPE

In the present study tin oxide material is used as a sensing layer to study the response of the sensor. But there are some limitations to it when it comes to the selectivity of the sensor among various gases. For future applications, various doping can be done to enhance the selectivity of the sensor.

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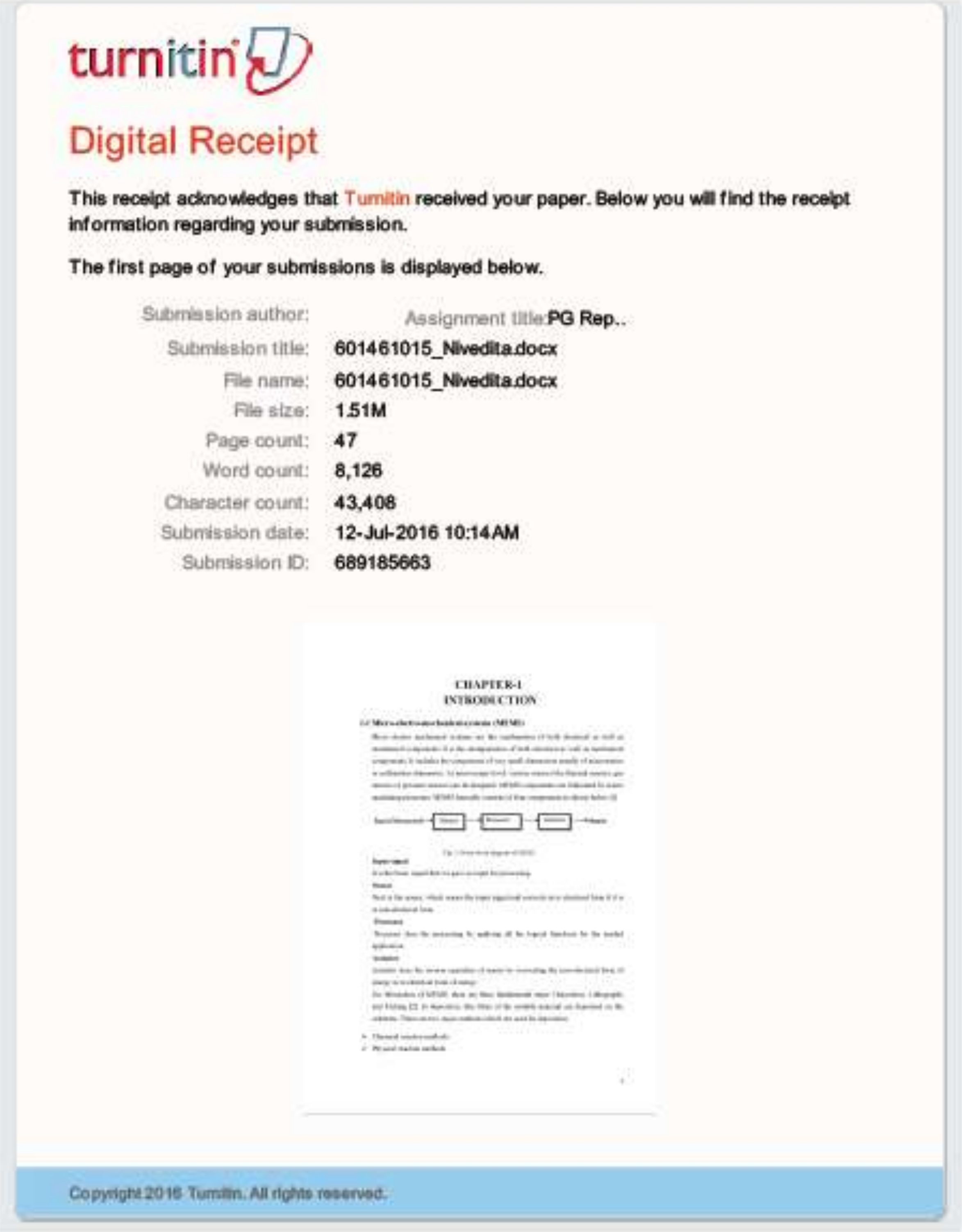
APPENDIX 1

Material properties used in simulation

Sr.No	**Properties**	**SiO_2**	**Platinum**	**Poly-Silicon**
1	Co efficient of thermal expansion in 1/K	0.5e-6	8.80e6	2.6e-6
2	Heat capacity at constant pressure in J/kg-K	730	133	678[J/(kg*K)]
3	Relative permittivity	4.2	7.0	4.5
4	Density in kg/m^3	2200	21450	2320
5	Thermal Conductivity in W/m-K	1.4	71.6	34
6	Young’s modulus in Pa	70e9	168e9	160e9
7	Poisson’s ratio	0.17	0.38	0.22
8	Electrical Resistivity in Ω-m	10e18	10.6e-8	2e5

LIST OF PUBLICATIONS

Nivedita, Anil Arora, "Design of MEMS Microheater Based H2 Gas Sensor." *International Journal of Advanced Research in Computer Engineering & Technology (IJARCET)* Volume 5, Issue 5, May 2016.



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CHAPTER-1
INTRODUCTION

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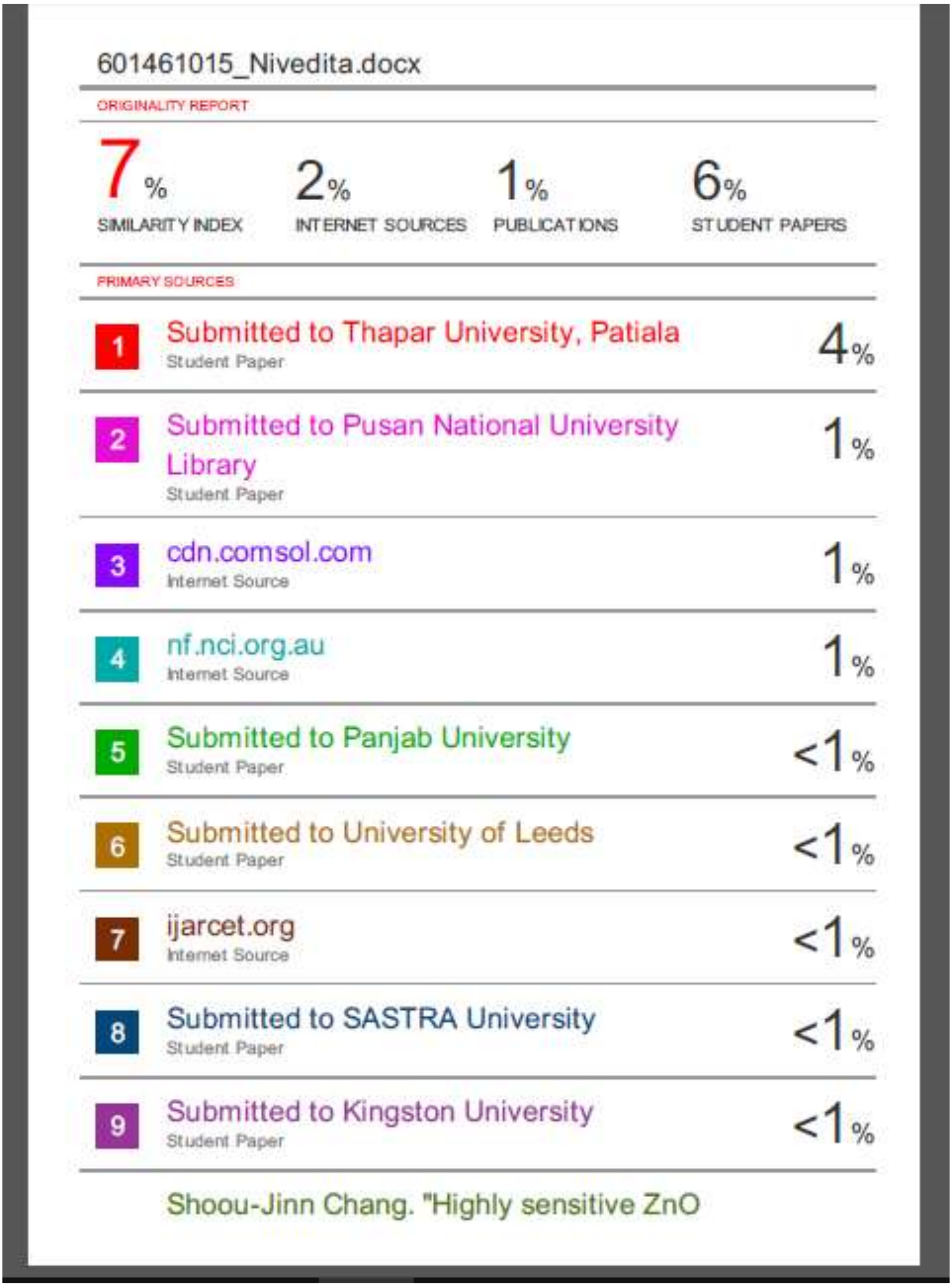

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