# GAS SENSING PROPERTY OF Fe2O3 THIN FILMS USING E-BEAM EVAPARATION METHOD

# A dissertation submitted for the partial fulfillment of

# MASTERS OF SCIENCE (NANOSCIENCE AND TECHNOLOGY)

# BY

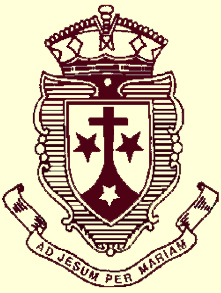
# PRABHAVATHI.C (M11NT05)

# SHALINI K.B. (M11NT08)

# Under the guidance of

# Dr. Mrs.UMA. V

# Mrs. CHAITRA. V



# MOUNT CARMEL COLLEGE

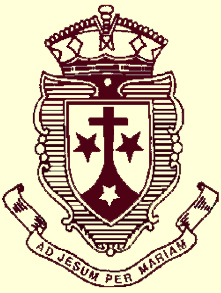
# (Autonomous)BANGALORE-560052

# APRIL-2013

**MOUNT CARMEL COLLEGE**

**(Autonomous)**

**BANGALORE-560052**



# CERTIFICATE

# This is to certify that the dissertation entitled GAS SENSING PROPERTIES OF FE2O3 THIN FILMS USING E-BEAM EVAPARATION METHOD Submitted by PRABHAVATHI.C and SHALINI K.B. is a bonafide record of work done by them under our guidance in the Department of Nnaoscience and Technology, Mount Carmel College (Autonomous), Bangalore, and this dissertation has not been previously submitted for any other degree.

# Signatureof the students (Dr.Mrs.UMA.V)

**(Mrs.CHAITRA.V)**

**DEPOSITION AND CHARACTERIZATION**

**OF Fe2O3 THIN FILM USING**

**e-BEAM EVAPORATION .**

**ACKNOWLEDGEMENT**

The endeavor that has been put in solely by us would not have given the expected result without the grace of **The Almighty.**

# We would like to take this opportunity to express our sincere gratitude to The Principal Sr. JUANITHA for providing us with all the required equipments and for her support.

# We owe our deepest gratitude to Dr.UMA. V, the Head of the Department of Nanoscience and Technology for help and encouragement through their cource of our project. We take a immense pleasure in thanking our project guides Dr.Mrs.UMA.V and Mrs.CHAITRA.V for their encouragement, their opitions, their valuable guidance and critical insights in helping us to well complete our final year project in field of Nanoscience and Technology.

# We would like to thank Mrs.Sudha Medappa, Dr.Prakash Rao, Mr.Pradeep.N, for their guidance.

# We are grateful to the teachers and our non teaching staffs of The department of Electronics, Department of Nanoscience and Technology, Department of Chemistry and The Research lab for their kind support in developing this project.

Finally, yet importantly, we would like to express our heartfelt thanks to our beloved parents for their blessings, our friends, our classmates for their help and wishes for the successful completion of this project.

# ABSTRACT

**INDEX**

**CHAPTER 1- Introduction**

* 1. What is nanotechnology?
  2. Top down and Bottom up Approaches
  3. Nanostructures and nano materials
  4. Applications of nanotechnology
  5. Sensors
  6. Metal oxide based Gas sensors
  7. Approach to the project

**CHAPTER 2- Theory**

2.1 Fe2O3 as a oxide material

2.2 Silicon properties and uses

2.3syntesys of Fe2O3nanoparticles

2.4 Déposition techniques

2.4.1 Spin coating

2.4.2 Spray pyrolysis

2.4.3 Dip coating

2.4.5 Chemical Vapour Depositionating

2.4..6 Physical Vapour Deposition –sputtering,e-Beam evaporation

2.5 Characterization techniques

2.5.1 surface charactérization - XRD,AFM

2.5.2 Optical Characterization – UV visible

Spectrophotometer

2.5.3 Electrical Characterization –

**CHAPTER 3- Experimental procedure**

* 1. Preparation of Fe2O3  nano particles
  2. Flow chart
  3. Deposition ofFe2O3 thin filmsusing e-Beam evaporation

**CHAPTER 4- Results and discussion**

4.1 structural analysis

4.1.1 XRD

4.1.2 AFM

4.2 Optical analysis

4.2.1 UV visible Spectroscopy

4.3 Electrical Characterization

**CHAPTER 5**

5.1 conclusion

5.2 references

**FIGURES**

CHAPTER 1

INTRODUCTION

1.1 NANOTECHNOLOGY

Nanotechnology is the study of manipulating matter on and atomic and molecular

scale. Generally, nanotechnology deals with structures sized between 1 to 100 nanometer in at least one dimension, and involves developing materials or devices possessing at least one dimension within that size. The first use of the concepts found in ‘nanotechnology’ (but predating use of that name) was in “there’s Plenty of Room at the Bottom”, a talk given by physicist Richard Feynman at an American Physical Society meeting at California Institute of Technology (Caltech) on December 29, 1959. Feynman described a process by which the ability to manipulate individual atoms and molecules might be developed, using one set of precise tools to build and operate another proportionally smaller set, and so on down to the needed scale. Nanotechnology is the engineering of functional systems at the molecular scale. This covers both current work and concepts that are more advanced.

Researcher K.Eric Drexler was the first person to popularize this technology in the early 1980’s. Drexler was interested in building fully functioning robots, computers and motors that were smaller than a cell. He spent much of his 80’s defending his ideas against critics that thought this technology would never be possible. Today, the word nanotechnology means something a bit different. Instead of building microscopic motors and computers, researchers are interested in building superior machines atom by atom. Nanotech means that each atom of machine is a functioning structure on its own, but when combined with other structure, these atoms works together to fulfill a larger purpose.

Nanotechnology is the engineering of functional systems at the molecular scale. In its original sense, nanotechnology refers to the projected ability to construct items from the bottom up, using techniques and tools being developed today to make complete, high performance products. By convention, nanotechnology is taken as the scale range 1 to 100 nm following the definition used by the National Nanotechnology Initiative in the US.

Nanotechnology is very diverse, ranging from extensions of conventional device physics to completely new approaches based upon molecular self-assembly, from developing new materials with dimensions on the nanoscale to investigating whether we can scale. The basic reason that it makes sense to regulate nanotechnology as a separate category is that nanotechnology materials behave differently from conventional materials. The properties of nanotechnology materials are often not predictable from the laws of classical physics and chemistry. The laws of electricity that apply to bigger things may not hold for nanotechnology materials. A material that conducts electricity at normal size may be an electrical insulator at nanotechnology size, and vice versa.

Nanotechnology entails the application of fields of science as diverse as surface science, organic chemistry, molecular biology, semiconductor physics, microfabrication, etc. Nanotechnology may be able to create many new materials and devices with a vast range of applications, such as in medicine, electronics, biomaterials and energy production. On the other hand, nanotechnology raises many of the same issues as any new technology, including concerns about the toxicity and environmental impact of nanomaterials, and their potential effect on global economics, as well as speculation about various doomsday scenarios. These concerns have led to a debate among advocacy groups and government on whether special regulation of nanotechnology is warranted.

**1.2 TOP-DOWN AND BOTTOM-UP APPROACHES:**

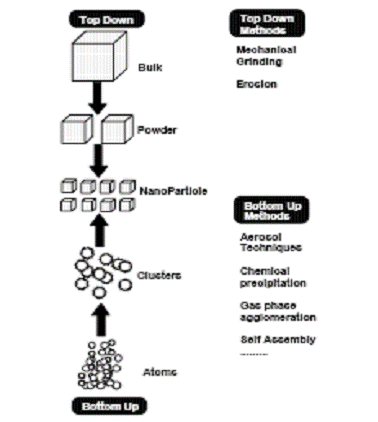
There are two approaches to the synthesis of nano materials and the fabrication of nanostructure: top down and bottom up. Attrition or milling is a typical top down method in making nanoparticles whereas the colloidal dispersion is a good examples for bottom up approach in the synthesis of nanoparticles.

The “top-down” approach involves the breaking down of large pieces of material to generate the required nanostructures from them. This method is particularly suitable for making interconnected and integrated structures such as in electronic circuitry. The biggest problem with the top down approach is the imperfection of the surface structure. It is well know that the conventional top-down technique such as lithography can cause significant crystallographic damage to the processed patterns, and additional defects may be introduced even during the etching steps.

Bottom-up approach involves the building of structure, atoms by atom or molecule by molecule. This is a very powerful method of creating identical structures with atomic precision, although to date, the man-made materials generated in this way are still much simpler than nature’s complex structures. The wide variety of approaches towards achieving this goal can be split into three categories: chemical synthesis, self assembly and positional assembly. Positional assembly is a the only technique in which single atoms or molecules can be placed dileberatly one by one. More typically large number of atoms, molecules or particles are used or created by chemical synthesis and then arranged through naturally occuring process into a desired structure.

Figure 1

**Schematic representation of the ‘bottom-up’ and ‘top-down’ synthesis processes of nanomaterials with the popular techniques that are used.**



**1.3 NANOSTRUCTURES AND NANOMATERIALS**

With the emergence of nanotechnology and nanoscience the investigation and application of nanostructured materials is growing rapidly. By defination, nanostructured materials have atleast one dimension that is less than 500 nanometers. The different nanostructures includes:

* Zero-Dimensional Nanostructures: Nanoparticles.
* One-Dimensional Nanostructures: Nanowires and Nanorods.
* Two-Dimensional Nanostructures: Thin Films.

Nanostructure materials are materials with a microstructure the characteristics length scale of which is on the order of a few (typical 1-100) nanometers. The microstructure refers to the chemical compition, the arrangement of the atoms (the atomic structure), and the size of the solid in one, two or three dimensions. Nanostructured materials may be grouped under nanoparticles (the building blocks), nano-intermediates and nanocomposites. They may be in or far away from thermodynamics equilibrium.

**1.3.1 ZERO-DIMENSIONAL NANOSTRUCTURES: NANOPARTICLES**

Nanoparticles are defined as particulate dispersions or solid particles with a size in the range of 10-100nm. Nanparticles are currently made out of a very wide variety of materials, the most common of the new generation of nanoparticles being ceramics, which are the best split into metal oxide ceramics, such as titanium, zinc, aluminium and iron oxides, to name a promient few and silicate nanoparticles (silicates, or silicon dioxides, are alos ceramics), generally in form of nanoscale flakes of clay. Nanoparticles have been prepared most frequency by three methods: (1) dispersion of preformed polymers; (2) polymerization of monomers; and (3) ionic gelation or coacervation of hydrophilic polymers.

Particle size and size distribution are the most important characteristics of nanoparticles systems. They determine the in vivo distribution, biological fate, toxicity and the targeting ability of nanoparticle systems. In addition, they can also influence the drug loading, drug release and stability of nanoparticles. Generally nanoparticles have relatively higher intracellular uptake compared to microparticles and available to a wider range of biological targets due to their small size and relative mobility.

There is a wide variety of technique for producing nanoparticles. These essentially fall into three categories: condensation from the vapor, chemical synthesis and solid-state processes such as milling. Grinding or milling can be used to create nanoparticles. The milling material, milling time and atmospheric medium affect resultant nanoparticles properties. As the market for nanoparticles in high tech areas, such as computer an pharamaceutical industry, continues to expand, the demand for nanoparticles with a well defined size and/or shape in high olumes and allow cost continues to increase. This trend is responsible for a continuous refinement of existing manufacturing technologies and for the development of novel production techniques.

**1.3.2 ONE-DIMENSIONAL NANOSTRUCTURES: NANORODS AND NANOWIRES**

Nanorods and nanowires in nanotechnology are one morphology of nanoscale objects.

* NANORODS: Each of their dimension ranges from 1-100nm. They may be synthesized from metal or semiconducting materials. Standard aspect ratio (length divided by width are 3-5.
* NANOWIRES: It exhibits aspect ratios (length-to-width ratio) of 1000 or more. As such they are reffered to as one-dimensional materials. Nanowires have many interesting properties that are not seen in bulk or 3-D materials. This is becauseelectrons in nanowires are quantum confined laterally and thus occupy energy levels that are different from the traditional continum of energy levels or bands found in bulk materials.

The common characteristics of these structures is that have a nanometer size in one of the dimensions, which produces quantum confinement in the material and changes its properties.

Many techniques have been developed to synthesize these structure and can be grouped into four categories:

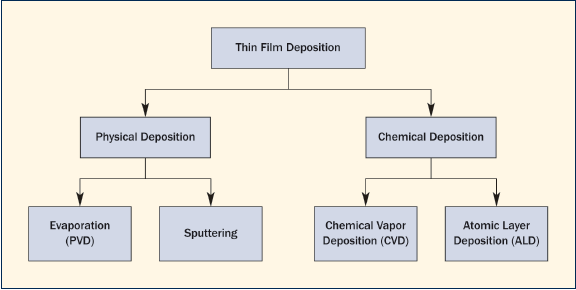
1. Spontaneous growth.
2. Template synthesis.
3. Electrospinning.
4. Lithography.

Spontaneous growth results in the formation of nanowires or nanorods due to a preferential crystal growth direction depending on the crystal structure and surface properties of the nanowires materials. His method commonly conducts to single crystal nanowires. On the other and template synthesis produces polycrystalline structures or even amorphous nanowires.

**1.3.3.TWO-DIMENSIONAL NANOSTRUCTURES: THIN FILM**

A thin film is a layer of material ranging from fractions of a nanometer (monolayer) to several micrometers in thichness. Most deposition techniques control layer thickness within a few tens of nanometers. Molecular beam epitaxy allows a single layer of atoms to be deposited at a time. Electronic semiconductor devices and optical coating are the main applications benefiting from thin film construction.

The act of applying thin film to a surface is thin film deposition- any technique for depositing a thin film of material onto a substrate or onto previously deposited layers. The preparation of thin films of the size of a nanometer is important bacause os their potential applications in the various fileds of science and technology, including the diverse fileds of electronics, optics, space science, aircraft science, defence and other industries.

****

**Figure 2: Deposition techniques**

**1.4 APPLICATIONS**

There are numerous application of nanotechnology. Many everyday products are the direct result of nanotechnology applications. Nanotechnology involves the creation of material derived from the manipulation of particles as smaller than atoms. Such various fields are:

**1)MEDICINE**: Nanotechnology in medicine involves employing nanoparticles to deliver drugs, heat, light or other substances to specific types of cells (such as cancer cells). Particles are engineered so that they are attracted to diseased cells, which allows direct treatment of those cells. This technique reduces damage to healthy cells in the body and allows for earlier detection of disease.

**2) ELECTRONICS:** Nanotechnology in electronics involoves building transistors from carbon nanotubes to enable minimum transistor dimensions of a few nanometers and developing techniques to manufacture integrated circuits built with nanotube transistors.

**3)FOOD:** Nanotechnology is having an impact on several aspects of food science, from how food is grown to how it is packaged. Clay nanocomposites are being used to provide an impermeable barrier to gases such as oxygen or carbon dioxide in lightweight bottles, cartons and packaging films.

**4)SPACE:** Nanotechnology may hold the key to making space-flight more practical. Advancements in nanomaterials make lightweight spacecraft and a cable for the space elevator possible. By significantly reducing the amount of rocket fuel required, these advances could lower the cost of reaching orbit and travelling in space.

**5)FABRICS:** Making composite fabric with nano-sized particles or fibers allows improvement of fabric properties without a significant increase in weight, thickness, or stiffness. For example incorporating nano-whiskers into fabric used to make pants produces a lightweight water and stain repellent material.

**1.5 SENSORS:**

A **sensor** (also called **detector**) is devices that senses a physical quantity and then converts it into a signal which can be read by an observer or by an instrument. A sensor’s sensitivity indicates how much the sensor’s output changes when the measured quantity changes. For example, a mercury-in-glass thermometer converts the measured temperature into expansion and contraction of a liquid which can be read on a calibrated glass tube. For accuracy, most sensors are caliberated against known standards.

Sensors are used in everyday objects such as touch-sensitive elevator buttons and lamps which dim or brighten by touching the base. There are also innumerable applications for sensors of which most people are never aware. Applications include cars, machines, aerospace, medicine, manufacturing and robotics.

CHAPTER 2

THEORY

**CHAPTER 3**

**EXPERIMENTAL PROCEDURE**

**3.1 PREPERATION OF Fe2O3 NANOPARTICLES:**

**Material used:**

* + - * Ferric nitrate (Fe(NO3)3.9H2O)
      * Citric acid (C6H11O7.H2O)
      * Deionized water
      * Ammonium hydroxide (NH4OH)

**Method used:**

* + - * Sol gel method
      * Combustion method

**Preparation of Fe2O3 nanoparticles using sol gel method:**

11.54g of Ferric nitrate (Fe(NO3)3.9H2O) and 6g of citric acid (C6H11O7.H2O) were dissolved in 17.4ml of deionized water and solution pH value was adjested to 7 using ammonium hydroxide (NH4OH). Solution was heated to 600C and continuously stirred using magnetic bit for 2hrs the solution became homogeneous brown gel, then the gel was dried at 1200C in hot oven for 10hrs and became a brown dry gel. Dry gel was combusted under muffle furnace for 3hrs at 6000C. loose, brown and very fine Fe2O3 powder was formed

**3.2 FLOW CHART**

|  |
| --- |
| **Ferris nitrate + citric acid+ Deionized water= solgel** |

pH is adjusted to 7 using

ammonium hydroxide

|  |
| --- |
| **Stirred using magnetic bit** |

|  |
| --- |
| **Brown dry gel is formed and kept it under hot air oven for dring** |

2 hours at 600

|  |
| --- |
| **Dry gel is obtained** |

8 hours at 1200C

|  |
| --- |
| **Kept under muffle furnace for combustion** |

|  |
| --- |
| **Fine** **Fe2O3** **powder is obtained** |

3 hours at 6500C

**3.3Deposition of Fe2O3 thin films using e-Beam evaporation**

**Pellet making:**

**Substrate cleaning:**

The glass substrate and silicon substrate is first cleaned with water then it is dried and immerresed in a beaker containing acetone and kept in an ultrasonicator for 15 minutes and then used it for deposition.

**Deposition process:**

System main switch and water tank is in on.

Then the rotary vacuum switch is switched ON ,then indicator lamp rp is ON.In this stage all valves must be in closed condition (0.01mbar vacuum)

Combination valve is turned ON to backning position and wait for 2 minutes.

On the first start the rotary pump vacuum must be checked by means of guage head -1 in pirani guage.

The cooling water supply is then allowed to flow to diffusion pump.

The diffusion pump is switched ON and wait for 30 minutes to reach the operating temperature 2000C.

The pellet of Fe2O3  is taken and glass , Silicon substrate is fixed on substrate holder.

When the substrate is closed ,close the air admittance valve.

The combination valve is in backing position is turn to roughing position.

For Gauged head-2 pirani gauge is selected when guage head reaches 0.05mbar then the combination valve is turned to backing position .

The high vacuum valve is slowly opened so that high vacuum is created then the penny guage is switched ON to read high vacuum.

LT operation:

The system is allowed to pump the chamber after high vacuum switch ON.

HTCB knob and green button is switched ON.

By using e-Beam remote control increase the current by maintaining 5-6KV voltage .

When there is glow in e-beam reduce the current using remote control and switch OFF LTCB , HTCB and pirani guage.

Finaly work holder is unloaded after some time.