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

# A dissertation submitted for the partial fulfillment

# BY

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 **DEPOSITION AND CHARACTERIZATION**

**OF Fe2O3 THIN FILM USING**

**e-BEAM EVAPORATION .**

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CHAPTER 1

INTRODUCTION

1.1 NANOTECHNOLOGY

The study of atomic and molecular scale manipulation of matter is known as nanotechnology.

Generally, nanotechnology deals with structures sized between 1nm to 100 nm 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 Engineering systems at the molecular level is known as nanotechnology. This covers both the most recent research and more complex ideas current work and concepts that are more advanced.

 The technology was first made popular in the early 1980s by researcher K.Eric Drexler. Drexler was interested in developing completely functional, cell-sized robots, processors, and motors. He devoted a significant portion of his 80s to defending his theories against those who held the opinion that this technology was unattainable. The definition of nanotechnology has changed somewhat over time. Researchers are interested in creating improved machines atom by atom rather than developing minuscule motors and processors. With nanotechnology, each machine atom functions as a separate structure on its own, but when joined with other machine atoms, these atoms cooperate to achieve a more comprehensive goal.

 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 encompasses a wide range of disciplines, from inventing new materials with nanoscale dimensions to examining if we can scale, from extending traditional device physics to whole new ways based upon molecule self-assembly.

 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:**

Top down and bottom up are the two methods used to synthesise nanomaterials and create nanostructures. A typical top-down way for creating nanoparticles is attrition or milling, whereas a good example of a bottom-up method .

 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 10nm to 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.

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**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:Nanotechnology has several applications. Applications of nanotechnology have directly led to the development of numerous daily products. The development of material with nanotechnology entails working with particles that are smaller than atoms. These many fields include:

**1)MEDICINE**: In to deliver medications, heat, light, or other things to particular types of cells, nanotechnology is used in medicine (such as cancer cells). It is possible to directly treat damaged cells by using particles that have been designed to attach to certain cells. This method enables earlier disease identification and lessens harm to the body’s healthy cells.

**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:** Food science is being impacted by nanotechnology in a number of ways, including how food is grown and processed. In lightweight bottles, cartons, and packaging sheets, clay nanocomposites are being employed to create an impermeable barrier to gases like oxygen or carbon dioxide.

**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:** Nano-sized fibres or particles can be used to create composite fabrics, which allow fabric qualities to be improved without significantly increasing weight, thickness, or stiffness. For instance, adding nano-whiskers to pants-making fabric creates a thin, water- and stain-resistant fabric.

**1.5 SENSORS:**

 A Sensor, often known as a detector, is a device that senses a physical quantity and then transforms it into a signal that an observer or an instrument can decipher. The sensitivity of a sensor describes how much the output of the sensor changes when the quantity being measured changes. A mercury-in-glass thermometer, for instance, turns the temperature being measured into the expansion and contraction of a liquid, which can then be read on a calibrated glass tube. Most sensors are calibrated against established standards for accuracy.

Sensors are employed in commonplace items like touch-sensitive lift buttons and lamps whose bases can be touched to dim or brighten the light. Many individuals are unaware of the countless applications for sensors that exist. Applications span the automotive, industrial, aerospace, medical, manufacturing, and robotics industries.

CHAPTER 2

THEORY

Iron(III) oxide is an inorganic substance with the formula Fe2O3. It is sometimes referred to as red iron oxide or hematite. All types of rocks naturally contain this chemical. As a solid, it appears reddish-brown. It has no smell. The pH level of it is 7.



In ultrahigh vacuum, ordered iron oxide ultrathin films were created on a single-crystal Mo(110) substrate by either depositing iron in air or oxidising films that had already been made of Fe(110).

By using a variety of surface analytical techniques, the surface structure and electrical structure of the iron oxide coatings were studied. The findings show that films made of metastable FeO(111) and O-terminated Fe2O3(0001) underwent surface structural changes to become Fe3O4(111), respectively. Whereas the latter heavily depends on the annealing temperature, the former is greatly influenced by oxygen pressure and substrate temperature. Understanding the mechanisms of surface structure change in iron oxides is aided by our experimental observations. For further research on chemical interactions, the model surfaces of Fe-oxide films, in particular O-terminated surfaces, can be used (e.g., in catalysis).

**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.